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(19) **United States**(12) **Patent Application Publication****Rouse et al.**(10) **Pub. No.: US 2020/0040529 A1**(43) **Pub. Date: Feb. 6, 2020**(54) **FIBROUS STRUCTURES COMPRISING
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The disclosure provides fibrous structures and tissue products comprising a volatile agave compound selected from the group consisting of limonene, hexanaldehyde and 6-Methyl-5-hepten-2-one. The volatile agave compounds may be provided by forming the structures at least in-part from agave fiber, such as *Agave tequilana* fiber. In other embodiments the volatile agave compounds may be incorporated in the fibrous structures by adding the compounds during formation of the fibrous structure, such as by adding the compounds to the furnish, or by topically applying the compounds to the fibrous structure after formation, in order to provide a lemon-like scent to the fibrous structure.

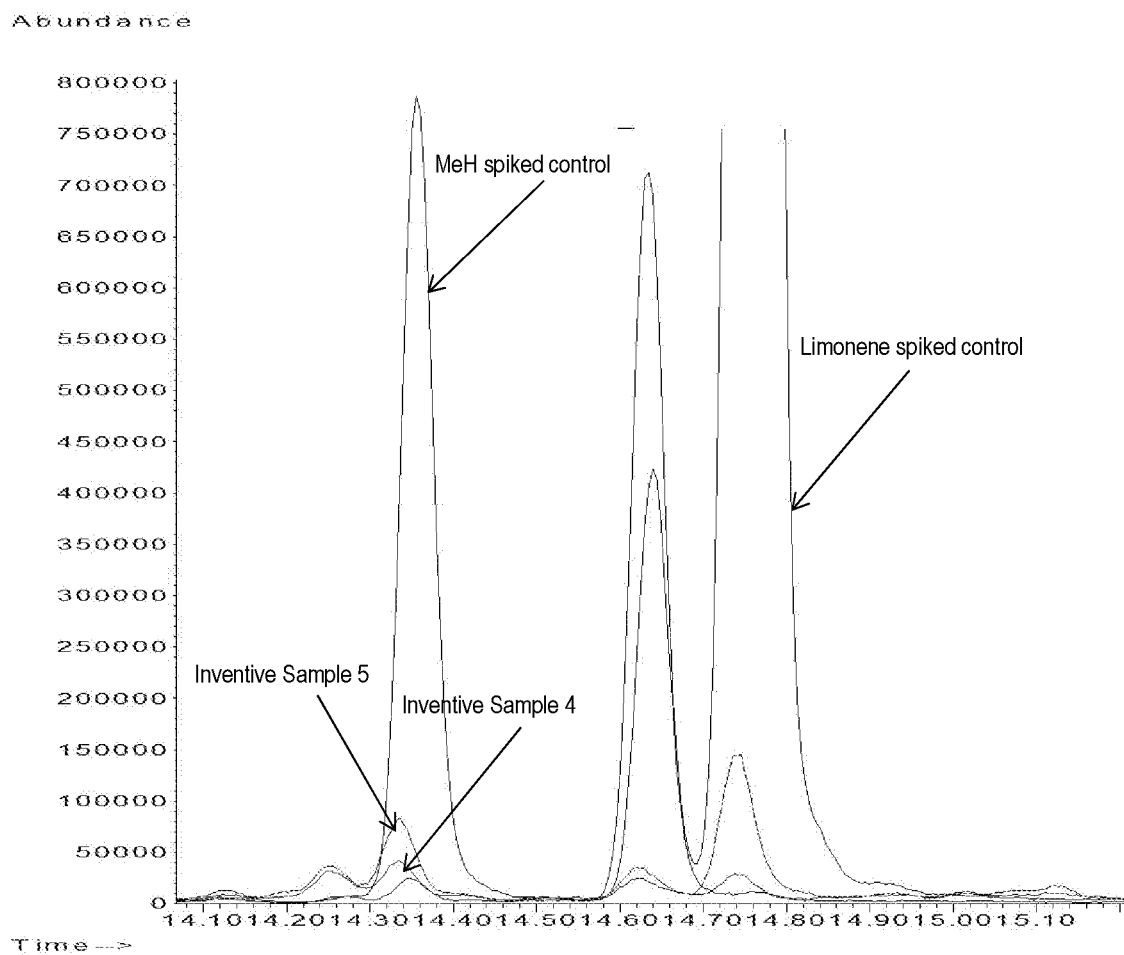


FIG. 1

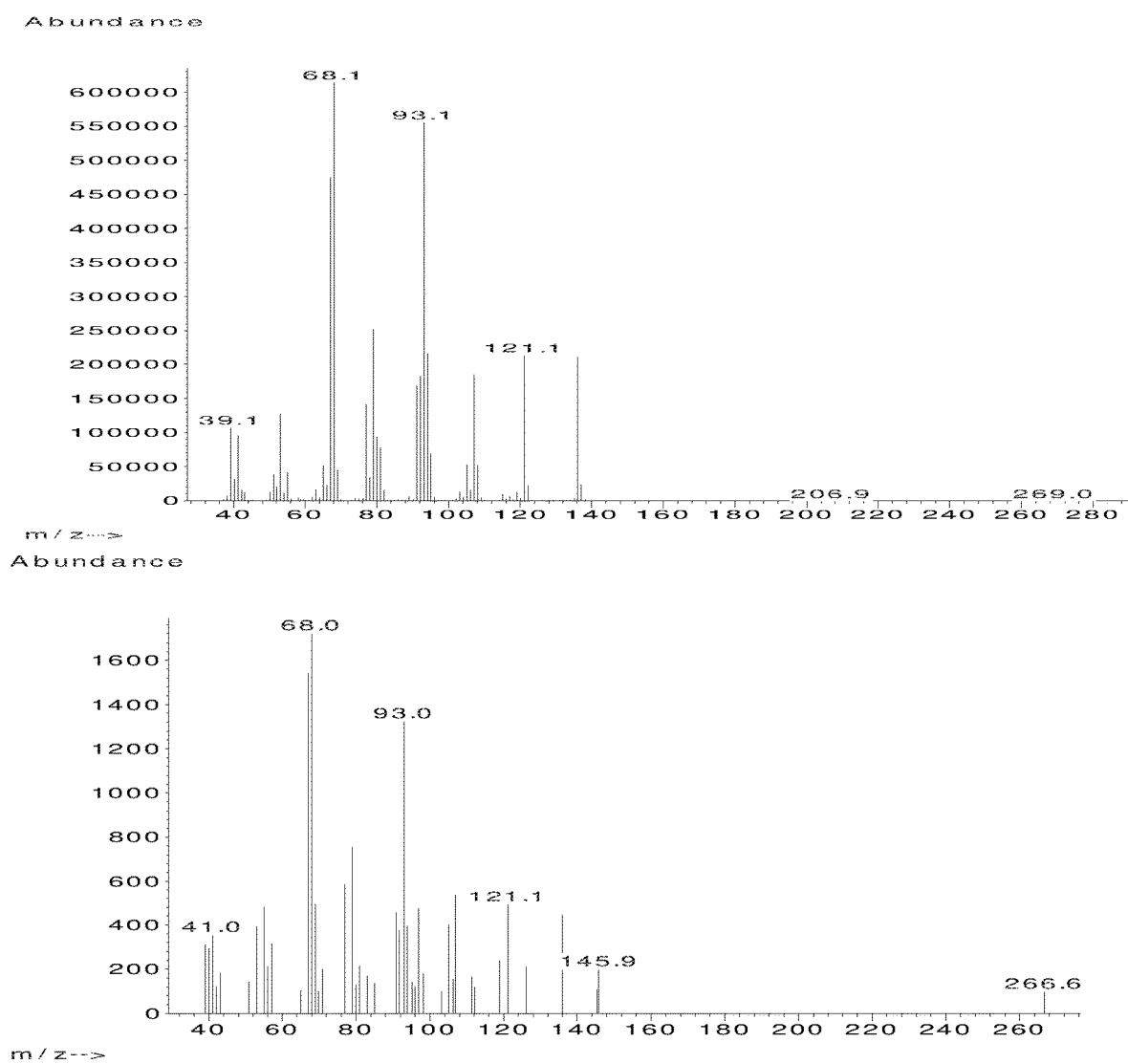
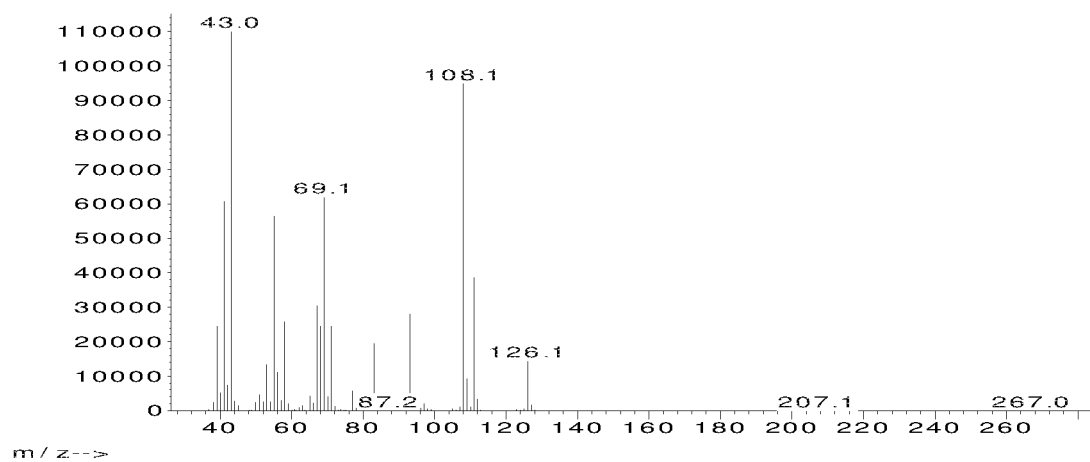


FIG. 2

Abundance



Abundance

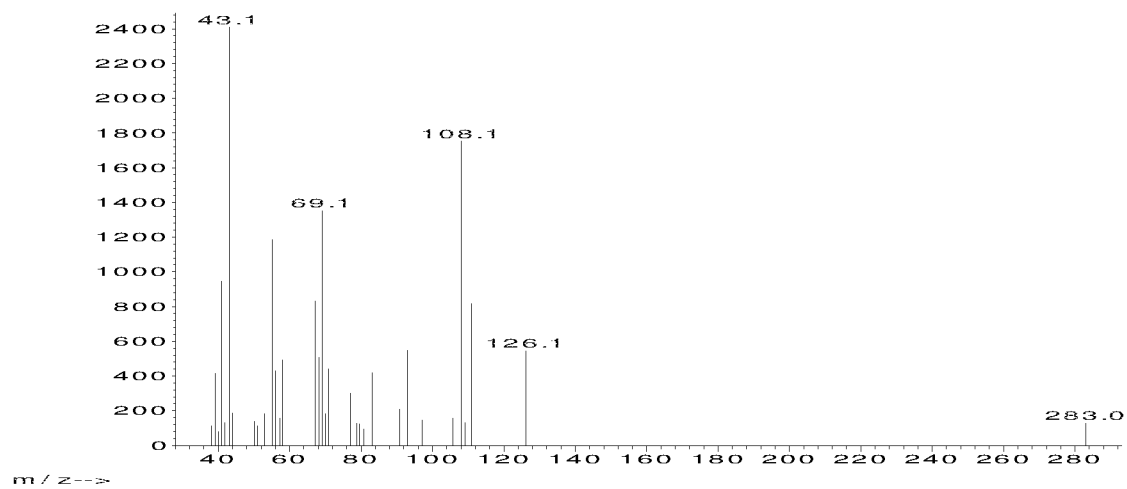


FIG. 3

FIBROUS STRUCTURES COMPRISING VOLATILE AGAVE COMPOUNDS

BACKGROUND

[0001] Manufacturers of fibrous structures have long sought to introduce compositions having a scent, such as a perfume, fragrance-emitting substance, and the like to their products to enhance their appeal to consumers and to provide a more favorable consumer experience. The manufacturers of tissue, and rolled tissue products, such as bath tissue and paper towels, in particular, have concerned themselves with adding scents to the core or to the dispensing mechanism. For example, U.S. Pat. Nos. 3,017,117, 5,857,621, 6,425,530 and 6,688,551 are directed to various roll holder and dispensing systems for rolled bath tissue that deliver a scent in-use. In each instance the user must purchase and install some additional component to provide scent to a roll of bath tissue. Requiring users to purchase and install additional components adds complexity and cost to delivering a scented tissue product. Moreover, the foregoing systems are generally limited to the storage and dispensing of rolled tissue products and not readily adapted to storing and dispensing folded tissue products, such as facial tissue.

[0002] In other instances a scent may be applied to the core during manufacture of the rolled tissue product such as disclosed in US Patent Publication No. 2014/0230950. Adding a scent directly to the core eliminates the need for a user to purchase a separate scented dispenser, but adds complexity to the manufacturing process and may not deliver a long-lasting scent. Further, the scent may not be perceived in-use by the consumer as it is not incorporated in the tissue sheet itself.

[0003] Therefore there remains a need in the art for a tissue product comprising a scent, where the scent is incorporated in the tissue sheet itself, is long lasting and provides the consumer with a sensory cue.

SUMMARY

[0004] The present inventors have now discovered that agave fiber is suitable for forming fibrous structures, such as rolled bath tissue and towels, having favorable physical characteristics such as low stiffness and good bulk. In addition to having favorable physical properties, the tissue products have a pleasant odor, which consumers describe as citrusy, grassy and fatty. These scents, in-turn, may act as a sensory cue to convey to a consumer product benefits and attributes such as natural, freshness and cleanliness. Thus, the present invention provides fibrous structures comprising volatile agave compounds, particularly compounds selected from the group consisting of limonene, hexanaldehyde and 6-Methyl-5-heptene-2-one.

[0005] Accordingly, in one embodiment the present invention provides a fibrous structure comprising agave fiber and at least one volatile agave compound selected from the group consisting of limonene, hexanaldehyde and 6-Methyl-5-heptene-2-one.

[0006] In another embodiment, the present invention provides a tissue product comprising agave fiber and a volatile agave compound selected from the group consisting of limonene, hexanaldehyde and 6-Methyl-5-heptene-2-one and combinations thereof.

[0007] In other embodiments, the present invention provides a tissue product comprising at least about 1.0 percent,

by weight of the tissue product, agave fiber and a volatile agave compound selected from the group consisting of limonene, hexanaldehyde and 6-Methyl-5-heptene-2-one and combinations thereof, wherein the tissue product contains at least 10 ppb of the volatile agave compound.

[0008] In still other embodiments, the present invention provides manufacturing a tissue web having a lemon-like odor note comprising the steps of forming a fiber slurry comprising agave fiber and wood pulp fiber, forming a tissue web from the fiber slurry, the web having a first and a second side; depositing on at least a first side of the web a formulation comprising a sensorily effective amount of a volatile agave compound selected from the group consisting of limonene, hexanaldehyde and 6-Methyl-5-heptene-2-one and combinations thereof and a carrier.

DESCRIPTION OF THE DRAWINGS

[0009] FIG. 1 depicts the total ion chromatogram (TIC) obtained by SPME GC/MS of two tissue products comprising agave fiber and two volatile agave compound standards;

[0010] FIG. 2 depicts mass spectra obtained from limonene standard (top) and a tissue product comprising 40 weight percent agave fiber (bottom); and

[0011] FIG. 3 depicts mass spectra obtained from 6-methyl 5-heptene-2-one standard (top) and a tissue product comprising 40 weight percent agave fiber (bottom).

DETAILED DESCRIPTION

[0012] The present invention provides fibrous structures adapted for use in tissue products such as facial tissues, bathroom tissues, toilet tissues and the like comprising a volatile agave compound selected from the group consisting of limonene, hexanaldehyde and 6-Methyl-5-heptene-2-one. In certain embodiments fibrous structures may be provided with volatile agave compounds by forming the structures at least in-part from agave fiber. In other embodiments the volatile agave compounds may be incorporated in the fibrous structures of the present invention by adding the compounds during formation of the fibrous structure, such as by adding the compounds to the furnish, or by topically applying the compounds to the fibrous structure after formation. For example, the volatile agave compounds may be incorporated into a formulation and topically applied to a fibrous structure to provide the fibrous structure with a sensory cue in-use. Alternatively, one or more volatile agave compounds may be formulated in a lotion, which may in-turn be applied to the external surface of the fibrous structure or otherwise, as desired.

[0013] Volatile agave compounds useful in the present invention are selected from the group consisting of limonene, hexanaldehyde and 6-Methyl-5-heptene-2-one. The foregoing compounds have been found in agave fiber, which may be useful in forming fibrous structures according to the present invention. The compounds provide the fibrous structures with a pleasant odor, which may act as a sensory cue to users and reinforce properties of the fibrous structures. In other instances the sensory cue may reinforce the user's perception of cleanliness and freshness.

[0014] In certain embodiments the invention provides a tissue product comprising limonene (1-methyl-4-(1-methylethenyl)-cyclohexene) having the formula $C_{10}H_{16}$, which is preferably provided as d-limonene and which may impart the tissue product with a citrus, lemon or piney odor. In other

embodiments the invention provides a tissue product comprising hexanaldehyde, also known as hexanal, and having the chemical formula $C_6H_{12}O$, which may impart the tissue product with a grassy odor. In still other embodiments the invention provides a tissue product comprising 6-Methyl-5-hepten-2-one having the chemical formula $C_8H_{14}O$, which may impart the tissue product with a fatty, green and citrus odor.

[0015] Fibrous structures may comprise a single volatile agave compound, or may comprise a combination of two or more volatile agave compounds. Further, the amount of volatile agave compound may vary depending on the desired physical properties of the fibrous structure. As such, the tissue products may comprise at least one volatile agave compound selected from the group consisting of limonene, hexanaldehyde and 6-Methyl-5-hepten-2-one, the volatile agave compound having a concentration of at least about 5 parts per billion (ppb), more preferably at least about 10 ppb and still more preferably at least about 15 ppb, such as from about 5 to about 500 ppb and more preferably from about 10 to about 300 ppb.

[0016] In certain embodiments the volatile agave compound is provided in an amount that exceeds its odor thresholds. Generally, when a compound is provided in excess of its odor threshold, the compound is said to be present in a "sensorily effective amount." For example, the fibrous structure may comprise at least about 10 ppb limonene, such as from about 10 to about 500 ppb. In other embodiments the fibrous structure may comprise at least about 2,500 ppb hexanaldehyde, such as from about 2,500 to about 5,000 ppb. In still other embodiments the fibrous structure may comprise at least about 50 ppb 6-Methyl-5-hepten-2-one such as from about 50 to about 500 ppb.

[0017] The volatile agave compounds may be incorporated in a wide range of fibrous structures. Suitable fibrous structures include, for example, a web, such as a wet laid tissue web or air laid web, gauze, swab, pad or the like. Particularly preferred fibrous structures include fibrous webs, including flushable and non-flushable cellulosic webs and nonwoven webs of synthetic fibrous material. Useful webs may be wet laid, air laid, meltblown, or spunbonded. Suitable synthetic fibrous material includes meltblown polyethylene, polypropylene, copolymers of polyethylene and polypropylene, bicomponent fibers including polyethylene or polypropylene, and the like. Useful nonwoven webs may be meltblown, coform, spunbond, airlaid, hydroentangled nonwovens, spunlace, bonded carded webs.

[0018] In one particularly preferred embodiment the volatile agave compounds are incorporated in tissue webs and products. Tissue webs useful in the present invention may be manufactured using any number of different techniques for manufacturing tissue webs, including but not limited to known wet-laid papermaking processes and air-laid papermaking processes. Such processes typically include steps of preparing a fiber composition in the form of a suspension in a medium, either wet, more specifically aqueous medium, or dry, more specifically gaseous, i.e. with air as medium. The aqueous medium used for wet-laid processes is oftentimes referred to as a fiber slurry. The fibrous slurry is then used to deposit a plurality of fibers onto a forming wire or belt such that an embryonic tissue product is formed, after which drying and/or bonding the fibers together results in a tissue product.

[0019] In particularly preferred embodiments the tissue webs and products of the present invention are formed by through-air drying and may be either creped or uncreped. For example, in one embodiment, at least one web of the tissue product is formed by an uncreped through-air drying process, such as the process described, for example, in U.S. Pat. Nos. 5,656,132 and 6,017,417, both of which are hereby incorporated by reference herein in a manner consistent with the present disclosure. Alternatively, at least one web of the tissue product is formed by the creped through-air dried process.

[0020] Further processing of the tissue product may be carried out such that a finished tissue product is formed. For example, in typical papermaking processes, the finished tissue product is the tissue product that is wound on the reel at the end of papermaking, and may subsequently be converted into a finished product, such as a rolled tissue product or a folded and stacked tissue product. Rolled tissue products may comprise a plurality of individual sheets separated from one another by perforations, which connect the sheets but allow individual sheets to be dispensed for use.

[0021] The tissue products may be single-ply or may be multi-ply. In those embodiments where the tissue product comprises more than one ply, each ply may be made of different materials or can have been manufactured in different ways. As used herein, the term "single-ply tissue product" means that it is comprised of one ply of tissue; the ply can be substantially homogeneous in nature or it can be a multi-layered tissue paper web. As used herein, the term "multi-ply tissue product" means that it is comprised of more than one ply of tissue. The plies of a multi-ply tissue product can be substantially homogeneous in nature or they can be multi-layered tissues. Further, the plies can be of the same construction and content or include different materials or processing steps prior to their combination.

[0022] The tissue product of the present invention may have a basis weight from about 10 to about 120 gsm, more preferably from about 10 to about 80 gsm and still more preferably from about 10 to about 60 gsm. Basis weight, as used herein, generally refers to the bone dry weight per unit area of a tissue and is generally expressed as grams per square meter (gsm). Basis weight is measured using TAPPI test method T-220. In particularly preferred embodiments the tissue products comprise one or more plies and have a basis weight from about 10 to about 80 gsm and still more preferably from about 15 to about 60 gsm.

[0023] The tissue product of the present invention may have a geometric mean tensile (GMT) greater than about 400 g/3". GMT refers to the square root of the product of the machine direction tensile strength and the cross-machine direction tensile strength of the tissue product, measured as described in the test methods section below and having units of grams per three inches (g/3"). In particularly preferred embodiments the tissue products may have a GMT from about 400 to about 3,000 g/3" and more preferably from about 600 to about 2,500 g/3". In certain preferred embodiments the tissue product may have a basis weight from about 30 to about 60 gsm and a GMT from about 700 to about 1,200 g/3". In another embodiment the tissue product may have a basis weight from about 45 to about 70 gsm and a GMT from about 1,200 to about 2,500 g/3".

[0024] The tissue product of the present invention may have a sheet bulk greater than about 5.0 cc/g. Sheet bulk refers to the quotient of the caliper (μm) divided by the bone

dry basis weight (gsm). The resulting sheet bulk is expressed in cubic centimeters per gram (cc/g). Tissue products prepared according to the present invention generally have a sheet bulk greater than about 5.0 cc/g, more preferably greater than about 8.0 cc/g and still more preferably greater than about 10.0 cc/g, such as from about 5.0 to about 20.0 cc/g and more preferably from about 8.0 to about 15.0 cc/g.

[0025] In a particularly preferred embodiment the present invention provides a through-air dried tissue product comprising agave fiber, and more preferably high yield agave fiber, and at least one volatile agave compound selected from the group consisting of limonene, hexanaldehyde and 6-Methyl-5-hepten-2-one, the product having a GMT greater than about 400 g/3" and a sheet bulk greater than about 8.0 cc/g.

[0026] In certain embodiments the addition of volatile agave compounds to the tissue products may be achieved by forming the tissue product at least in-part from fiber derived from non-wood plants of the genus *Agave*, of the family Asparagaceae, such as *Agave tequilana*, *Agave sisalana* and *Agave fourcroydes*. Where the compounds are provided by forming the tissue product in-part from agave fiber, it is generally preferred that the tissue product comprise at least about 0.5 percent, by weight of the product, agave fiber and more preferably at least about 1.0 percent, and still more preferably at least about 5.0 percent, such as from about 0.5 to about 50 percent agave fiber and more preferably from about 1.0 to about 20 percent and still more preferably from about 5 to about 15 percent. In particularly preferred embodiments the agave fibers are incorporated into the instant tissue products as a replacement for a portion of the long fiber fraction of the papermaking furnish, such as Northern Softwood Kraft (NSWK) or Southern Softwood Kraft (SSWK).

[0027] In a particularly preferred embodiment the present invention provides a three layered web where the agave fiber is selectively disposed in the middle layer and the two outer layers consist essentially of hardwood kraft fibers, such as *Eucalyptus* Hardwood Kraft (EHWK) and are substantially free of agave fiber. In addition to agave fiber, the middle layer also comprises wood pulp fibers having an average fiber length greater than about 2.0 mm. As used herein, the term "average fiber length" generally refers to the length weighted average length of fibers determined utilizing a fiber analyzer such as those commercially available from Kajaani Oy Electronics (Kajaani, Finland). The agave fiber comprises at least about 1.0 percent by weight of the middle layer, and more preferably at least about 5.0 percent and still more preferably at least about 10 percent, such as from about 1.0 to about 75 percent by weight of the middle layer. In the foregoing example, the agave fiber may comprise from about 0.5 to about 30 percent, by weight of the tissue product, and the product may comprise from about 1.0 to about 30 percent, by weight of the product, less wood pulp fibers having an average fiber length greater than about 2.0 mm, such as NSWK or SSWK.

[0028] In still other embodiments the agave fiber may replace substantially all of the long fiber fraction of the papermaking furnish, such that the tissue product is substantially free from wood pulp fibers having an average fiber length greater than about 2.0 mm. In such embodiments the agave fiber may be selectively incorporated into the middle layer of a three layered tissue web such that the middle layer

consists essentially of agave fiber and the two outer layers comprise wood pulp fibers having an average fiber length less than about 2.0 mm.

[0029] Further, in certain embodiments it may be desirable to provide a layered tissue web where the agave fiber is selectively disposed in a layer of the tissue which is not brought into contact with a dryer, such as a Yankee dryer during manufacture. Without being bound by any theory, selectively disposing the agave fiber in a layer that is not contacted by a dryer may preserve the volatile agave compound and ensure that the resulting tissue web and products have a pleasant odor.

[0030] The tissue webs may be incorporated into tissue products that may be either single- or multi-ply, where one or more of the plies may be formed by a multi-layered tissue web having agave fibers selectively incorporated in one of its layers. In one embodiment the tissue product is constructed such that the agave fibers are not brought into contact with the user's skin in-use. For example, the tissue product may comprise two multi-layered through-air dried webs wherein each web comprises a first fibrous layer substantially free from agave fibers and a second fibrous layer comprising agave fibers. The webs are plied together such that the outer surface of the tissue product is formed from the first fibrous layer of each web and the second fibrous layer comprising the agave fibers is not brought into contact with the user's skin in-use.

[0031] Generally agave fibers useful in the present invention are derived from non-woody plants in the genus *Agave* of the family Asparagaceae. Suitable species within the genus *Agave* include, for example, *Agave tequilana*, *Agave sisalana* and *Agave fourcroydes*.

[0032] In certain embodiments the agave fibers are processed by a high yield pulping process, such as mechanically treating the fibers. High yield pulping processes include, for example, mechanical pulp (MP), refiner mechanical pulp (RMP), pressurized refiner mechanical pulp (PRMP), thermomechanical pulp (TMP), high temperature TMP (HT-TMP), RTS-TMP, thermopulp, groundwood pulp (GW), stone groundwood pulp (SGW), pressure groundwood pulp (PGW), super pressure groundwood pulp (PGW-S), thermo groundwood pulp (TGW), thermo stone groundwood pulp (TSGW) or any modifications and combinations thereof. Processing of agave fibers using a high yield pulping process generally results in a pulp having a yield of at least about 50 percent, more preferably at least about 65 percent and still more preferably at least about 85 percent, such as from about 50 about 95 percent and more preferably from about 65 to about 90 percent.

[0033] The high yield pulping process may comprise heating the agave fiber above ambient, such as from about 70 to about 200° C., and more preferably from about 90 to about 150° C. while subjecting the fiber to mechanical forces. Caustic or an oxidizing agent may be introduced to the process to facilitate fiber separation by the mechanical forces. For example, in one embodiment, a solution of 3 to about 8 percent NaOH and a solution of 3 to about 8 percent peroxide may be added to the fiber during mechanical treatment to facilitate fiber separation.

[0034] In other embodiments the high yield pulping process may comprise treating agave leaves with an alkaline pulping solution such as that disclosed in U.S. Pat. No. 6,302,997, the contents of which are incorporated herein in a manner consistent with the present disclosure. Alkaline

treatment may be carried out at a pressure from about atmospheric pressure to about 30 psig and at a temperature ranging from about ambient temperature to about 150° C. The alkaline hydroxide may be added, based upon the oven dried mass of the agave leaves, from about 10 to about 30 percent. Suitable alkaline pulping solutions include, for example, sodium hydroxide, potassium hydroxide, ammonium hydroxide, calcium hydroxide and combinations thereof. After alkaline treatment, the agave is mechanically worked and then treated with an acid solution to reduce the pH to an acid pH.

[0035] In other embodiments the high yield pulping process may comprise impregnating agave leaves with a solution of nitric acid and optionally ammonium hydroxide at ambient temperatures under atmospheric pressure, such as described in U.S. Pat. No. 7,396,434, the contents of which are incorporated herein in a manner consistent with the present invention. The impregnated leaves are then heated to evaporate the nitric acid followed by treatment with an alkaline solution before being cooled.

[0036] Although a caustic, such as NaOH, or oxidizing agent, such as nitric acid or peroxide, may be added during processing, it is generally preferred that the agave fiber is not pretreated with a sodium sulfite or the like prior to processing. For example, high yield agave pulps are generally prepared without pretreatment of the fiber with an aqueous solution of sodium sulfite or the like, which is commonly employed in the manufacture of chemi-mechanical wood pulps.

[0037] The use of agave fiber, and in a particularly preferred embodiment high yield agave pulp fibers, results in tissue webs and products having one or more volatile agave compounds selected from the group consisting of limonene, hexanaldehyde and 6-Methyl-5-hepten-2-one. The volatile agave compounds may provide the tissue products with an odor, which consumers may find desirable and provide the consumer with a sensory cue. Thus, the present invention may provide a sensory cue that can remain with the consumer for a period of time, does not rely on the consumer viewing or reading packaging content, may be more impactful than a visual cue and may provide a more lasting impression.

[0038] While in certain embodiments the volatile agave compounds may be provided by forming the fibrous structure at least-in part from agave fiber, in other embodiments the volatile agave compounds may be formulated and added to a fibrous structure to enhance the sensory cue and reinforce to a user the presence and benefits of agave fiber in the fibrous structure. In such embodiments the fibrous structure may comprise less than about 10 percent, by weight, agave fiber, such as less than about 5.0 percent, and in certain instances less than about 2.0 percent, such as from about 0.1 to about 10 percent and more preferably from about 1.0 to about 5.0 percent and the web may further comprise a formulation comprising a volatile agave compound and a carrier disposed thereon.

[0039] Suitable formulations may comprise a volatile agave compound selected from the group consisting of limonene, hexanaldehyde and 6-Methyl-5-hepten-2-one and combinations thereof and a carrier. Amounts of the volatile agave compounds in the foregoing formulation may range from 0.000001 to 2 percent, by weight of the formulation, more preferably from 0.00001 to 1 percent, and still more preferably from 0.0001 to 0.5 percent, and optimally from

0.001 to 0.1 percent. Amounts of the carrier may range from 1 to 99.9 percent, by weight of the formulation, preferably from 70 to 95 percent, optimally from 80 to 90 percent. Among the useful carriers are water, emollients, fatty acids, fatty alcohols, humectants, thickeners and combinations thereof. The carrier may be aqueous, anhydrous or an emulsion. Preferably the formulations are aqueous, especially water and oil emulsions. Water when present may be in amounts ranging from 5.0 to 90 percent, by weight of the formulation, preferably from 20 to 70 percent, optimally from 35 to 60 percent. Alternatively, emollient materials may serve as carriers. These may be in the form of silicone oils, synthetic esters and hydrocarbons. Amounts of the emollients may range anywhere from 0.1 to 95 percent, by weight of the formulation, preferably between 1 and 50 percent.

[0040] In other embodiments the volatile agave compounds may be incorporated in a lotion and applied to a tissue web or product. In certain embodiments, particularly those in which the composition is applied to a web, it may be desirable that the composition provide certain physical attributes, such as having a smooth, lubricious, non-greasy feel; the ability to at least partially transfer from the web to the user's skin; the capability to be retained on the web at about room temperature; or the ability to be compatible with the web manufacturing process. In certain embodiments it is preferred that at least a portion of the composition is transferred from the tissue to the user's skin in-use.

[0041] Incorporating volatile agave compounds into lotions and applying the same to a tissue product provides the added advantage of transferring the volatile agave compounds to a user's skin in the region where the effect is mostly intended. For example, the tissue product may be in the form of a facial tissue product intended for nose blowing and wiping, and in-use the lotion is transferred to the user's skin near the nose enabling highly effective transport of the compounds to receptor sites of the mucous skin in the nasal cavity. According to the present invention the lotion can be the carrier for the volatile agave compounds.

[0042] As used herein, the term "lotion" generally refers to a composition added to the tissue in order to improve its softness or handfeel of the tissue and which may be transferred to the user's skin in-use. The lotion may comprise tissue softening and/or debonding agents, emollients, immobilizing agents and mixtures thereof. Suitable softening and/or debonding agents include quaternary ammonium compounds, polysiloxanes, and mixtures thereof. Suitable emollients include propylene glycol, glycerin, triethylene glycol, spermaceti or other waxes, petrolatum, fatty acids, fatty alcohols and fatty alcohol ethers or esters having from 12 to 28 carbon atoms in their fatty acid chain, mineral oil and mixtures thereof. Suitable immobilizing agents include waxes, fatty alcohols, fatty acids, polyhydroxy fatty acid esters, polyhydroxy fatty acid amides, and mixtures thereof. In most cases, the lotions contain at least one immobilizing agent and an emollient. Lotions can be emulsions or dispersions.

[0043] Thus, in certain embodiments a lotion useful in the present invention may consist essentially of a volatile agave compound selected from the group consisting of limonene, hexanaldehyde and 6-Methyl-5-hepten-2-one, a cationic softening compound, such as a quaternary ammonium compound, a polyhydroxy compound having a molecular weight at least about 1,000 and optionally a silicone or glycerin, or

mixtures thereof. For example, the softening composition may consist essentially of a quaternary ammonium compound, a polyhydroxy compound having a molecular weight at least about 1,000, a silicone and glycerin.

[0044] Other chemicals commonly used in papermaking can be added to the softening composition described herein, or to the papermaking furnish so long as they do not significantly and adversely affect important tissue product properties, such as strength or absorbency of the tissue product, or negatively effect the softening provided by the softening compositions of the present invention. For example, dry strength additives such as starch or carboxymethyl cellulose may be added to the furnish to improve the tensile strength of the tissue products. In other embodiments wet strength resins, such as polyamide-epichlorohydrin resins may be added to the furnish to improve the tensile strength of the tissue product when wet. In still other embodiments a temporary wet strength agent may be added to the furnish, such as modified starch and more particularly cationic starches.

[0045] Other additives may include humectants and skin protectants. Suitable humectants include lactic acid and its salts, sugars, ethoxylated glycerin, ethoxylated lanolin, corn syrup, hydrolyzed starch hydrolysate, urea, and sorbitol. Suitable skin protectants include allantoin, kaolin, zinc oxide, aloe vera, vitamin E, petrolatum and lanolin. Again, the foregoing additives are generally complementary to the volatile agave compound containing lotion and generally do not significantly and adversely affect important tissue product properties, such as strength or absorbency of the tissue product, or negatively effect the sensory cue provided by the volatile agave compounds.

[0046] The formulations and lotions comprising volatile agave compounds described above, may be added to the tissue web at any point after the web has been formed and at least partially dewatered. In a particularly preferred embodiment a formulation or lotion comprising volatile agave compound is applied to the web after it has been dried to final dryness, such as a moisture content less than about 10 percent (by weight of the tissue web) and more preferably less than about 5 percent. For example, a lotion may be applied after the drying section of the tissue machine where the tissue sheet has a consistency of from about 90 to about 100 percent. Formulations and lotions may also be applied via a secondary post treatment process where the tissue sheet has a consistency of from about 90 to about 100 percent.

[0047] The method by which the formulation or lotion is applied to the tissue sheet may be accomplished by any method known in the art. For example, in one embodiment a formulation or lotion may be applied by contact printing methods such as gravure, offset gravure, flexographic printing and the like. In other embodiments, non-contact printing methods such as ink jet printing, digital printing of any kind, and the like may be used.

[0048] In other embodiments the formulation or lotion may be sprayed onto the tissue sheet. For example, spray nozzles may be mounted over a moving tissue sheet to apply a desired dose of a solution to the tissue sheet. Nebulizers may also be used to apply a light mist to a surface of a tissue sheet. In other embodiments the lotion may be applied to a moving belt or fabric by spray or other means and the belt or fabric may in-turn contact the tissue sheet to apply the lotion to the tissue sheet.

[0049] In still other embodiments the lotion may also be applied by coating onto the tissue sheet by slot coating, blade coating, air knife coating, short dwell coating, cast coating, and the like. Preferred methods of application include gravure printing, flexographic printing, WEKO and spraying. A particularly preferred method of application is rotogravure printing such as described in U.S. Pat. No. 5,665,426, the contents of which are incorporated by reference in a manner consistent with the present disclosure.

[0050] In one embodiment the lotion may be applied by an indirect application process where the lotion is applied to the web via a transfer/applicator roll. For example, the web to be treated may be threaded from an unwind roll through a nip between the transfer/applicator roll and a backing roll. The lotion is added to a second nip created between a Mayer rod and the transfer/applicator roll. Mayer rods are well known in the art and are provided in a number of different configurations that allow different volumes of fluid to be put onto the transfer/applicator roll. The lotion applied to the transfer/applicator roll by the Mayer rod is subsequently disposed on the web.

[0051] The foregoing application method generally only applies the lotion to one side of the web. However, there is also an option to coat both sides of the web with a machine configuration change where the backing roll is replaced with a transfer/applicator roll.

[0052] The lotion may be applied to only a single surface of the tissue or may be applied to both the upper and opposed lower surfaces. The add-on amount of the lotion can be from about 0.5 to about 6 dry weight percent based on the weight of the tissue, more specifically from about 1 to about 5 dry weight percent, and still more specifically from about 2 to about 4.5 dry weight percent. The higher add-on amounts are more likely to leave behind a detectable residue on the skin, whereas the lower add-on amounts are less likely to do so. Surprisingly, the instant lotion may be added at relatively low levels, such as less than about 10 dry weight percent, and more preferably less than about 4 dry weight percent, such as from about 0.5 to about 6 dry weight percent, and still provide a significant sensory cue to the user.

[0053] When applied to the tissue web, the lotion can cover the entire surface area of the web or a portion of the web. For example, the composition can be applied so as to cover from about 20 to about 80 percent of the surface area of the web, and particularly from about 30 to about 60 percent of the surface area of the web. In certain embodiments, depending on the composition of the lotion, leaving untreated areas on the web, the web remains easily wettable, which can be a concern when applying hydrophobic additives.

[0054] Thus, in one embodiment, the present invention provides a method of making a soft, single- or multi-ply tissue product wherein one or more of the plies of the tissue product comprises a lotion disposed on its outer surface, the composition comprising a cationic softener, such as a quaternary ammonium compound, a polyhydroxy compound, a volatile agave compound selected from the group consisting of limonene, hexanaldehyde and 6-Methyl-5-hepten-2-one and combinations thereof and optionally a silicone or glycerin. Methods of forming the foregoing tissue web generally comprise (a) forming a fiber slurry comprising agave fiber and wood pulp fiber; (b) depositing the fiber slurry onto a forming fabric to form a wet tissue sheet; and, (c) dewatering the wet tissue sheet to form a dewatered tissue sheet; (d)

drying the dewatered tissue sheet to form a dried tissue sheet; and, (e) topically applying a lotion on the dried tissue sheet, the composition comprising a cationic softener, such as a quaternary ammonium compound, a polyhydroxy compound, a volatile agave compound selected from the group consisting of limonene, hexanaldehyde and 6-Methyl-5-hepten-2-one and combinations thereof, and optionally a silicone or glycerin.

Test Methods

Gas Chromatography/Mass Spectrometry

[0055] Samples of tissue products were prepared by cutting and weighting 2.5 g of each tissue product and placing into a 60 mL vial. The tissue samples were then sampled by poking a small hole through the vial cap septa using a needle. Through the hole, a SPME fiber was inserted and the fiber exposed for one hour at 85° C. to collect the volatiles for analysis. The SPME fiber used was:

[0056] 85 μ m Carboxen/polydimethylsilicone (Supelco catalog No. 57334-U, light blue) recommended for gases and low molecular weight compounds (MW 30-225).

[0057] 57330-U manual fiber holder.

Control samples were spiked with standards and analyzed similarly to the samples. Area counts generated from the standards were used to calculate amounts present in the sample. Three replicates were run for each sample.

[0058] Analysis was carried out using a series 5973N quadrupole mass spectrometer (Agilent Technologies Inc., Santa Clara, Calif.). The gas chromatograph was equipped with an Agilent J&W DB-624MS column (30 m, 0.25 mm ID, 1.8 μ film). Helium was used as the carrier gas. The helium flow rate was 1.5 mL/minute. The gas chromatograph temperature was programmed as follows:

Level	Rate (° C./min)	Final Temp (° C.)	Final Time (min)
Initial	—	0	1
1	7.5	100	0
2	10	240	—

The temperature of the GC inlet was 240° C. and the split ratio was 5:1. Mass spectra and reconstructed chromatograms (total ion current “TIC”) were obtained by automatic scanning in the mass range from 35 to 350 Daltons with a threshold of 200 Daltons. GC/MS data were processed with Agilent ChemStation software and the Wiley NIST11 mass spectra library.

Sheet Bulk

[0059] Sheet Bulk is calculated as the quotient of the dry sheet caliper (μ m) divided by the basis weight (gsm). Dry sheet caliper is the measurement of the thickness of a single tissue sheet measured in accordance with TAPPI test methods T402 and T411 om-89. The micrometer used for carrying out T411 om-89 is an Emveco 200-A Tissue Caliper Tester (Emveco, Inc., Newberg, Oreg.). The micrometer has a load of 2 kilo-Pascals, a pressure foot area of 2500 square millimeters, a pressure foot diameter of 56.42 millimeters, a dwell time of 3 seconds and a lowering rate of 0.8 millimeters per second.

Tensile

[0060] Tensile testing was done in accordance with TAPPI test method T-576 “Tensile properties of towel and tissue products (using constant rate of elongation)” wherein the testing is conducted on a tensile testing machine maintaining a constant rate of elongation and the width of each specimen tested is 3 inches. More specifically, samples for dry tensile strength testing were prepared by cutting a 3 $\pm$ 0.05 inch (76.2 $\pm$ 1.3 mm) wide strip in either the machine direction (MD) or cross-machine direction (CD) orientation using a JDC Precision Sample Cutter (Thwing-Albert Instrument Company, Philadelphia, Pa., Model No. JDC 3-10, Serial No. 37333) or equivalent. The instrument used for measuring tensile strengths was an MTS Systems Sintech 11S, Serial No. 6233. The data acquisition software was an MTS TestWorks® for Windows Ver. 3.10 (MTS Systems Corp., Research Triangle Park, N.C.). The load cell was selected from either a 50 Newton or 100 Newton maximum, depending on the strength of the sample being tested, such that the majority of peak load values fall between 10 to 90 percent of the load cell’s full scale value. The gauge length between jaws was 4 $\pm$ 0.04 inches (101.6 $\pm$ 1 mm) for facial tissue and towels and 2 $\pm$ 0.02 inches (50.8 $\pm$ 0.5 mm) for bath tissue. The crosshead speed was 10 $\pm$ 0.4 inches/min (254 $\pm$ 1 mm/min), and the break sensitivity was set at 65 percent. The sample was placed in the jaws of the instrument, centered both vertically and horizontally. The test was then started and ended when the specimen broke. The peak load was recorded as either the “MD tensile strength” or the “CD tensile strength” of the specimen depending on direction of the sample being tested. Ten representative specimens were tested for each product or sheet and the arithmetic average of all individual specimen tests was recorded as the appropriate MD or CD tensile strength of the product or sheet in units of grams of force per 3 inches of sample. The geometric mean tensile (GMT) strength was calculated and is expressed as grams-force per 3 inches of sample width. Tensile energy absorbed (TEA) and slope are also calculated by the tensile tester. TEA is reported in units of gm $\cdot$ cm/cm². Slope is recorded in units of kg. Both TEA and Slope are directional dependent and thus MD and CD directions are measured independently. Geometric mean TEA and geometric mean slope are defined as the square root of the product of the representative MD and CD values for the given property.

[0061] Multi-ply products were tested as multi-ply products and results represent the tensile strength of the total product. For example, a 2-ply product was tested as a 2-ply product and recorded as such. A basesheet intended to be used for a two ply product was tested as two plies and the tensile recorded as such. Alternatively, a single ply may be tested and the result multiplied by the number of plies in the final product to get the tensile strength.

Example

[0062] Single-ply uncreped through-air dried (UCTAD) tissue webs were made generally in accordance with U.S. Pat. No. 5,607,551. The tissue webs and resulting tissue products were formed from various fiber furnishes including, EHWK pulp, NSWK pulp, and high yield agave (HYA) pulp.

[0063] HYA was produced by processing *Agave tequilana* leaves using a three stage non-wood pulping process com-

mercially available from Taizen America (Macon, Ga.). The resulting HYA pulp had an average fiber length of about 1.1 mm and a fiber coarseness of about 12.74 mg/100 m.

[0064] The EHWK furnish was prepared by dispersing about 120 pounds (oven dry basis) EHWK pulp in a pulper for 30 minutes at a consistency of about three percent. The fiber was then transferred to a machine chest and diluted to a consistency of 1 percent. The NSWK furnish was prepared by dispersing about 50 pounds (oven dry basis) of NSWK pulp in a pulper for 30 minutes at a consistency of about three percent. The fiber was then transferred to a machine chest and diluted to a consistency of 1 percent. The HYA was prepared by dispersing about 50 pounds (oven dry basis) HYA pulp in a pulper for 30 minutes at a consistency of about 3 percent. The fiber was then transferred to a machine chest and diluted to a consistency of 1 percent.

[0065] Stock solutions were diluted to 0.75 percent consistency and pumped to a 3-layer headbox to form a three layered web. The relative weight percentage and composition of the layers is summarized in Table 1 below. In those instances where starch was added, it was added on as a kilogram per ton of furnish basis as indicated in Table 1 and added to all layers.

TABLE 1

Sample	Furnish Layering (wt %)	Redibond 2038 A (kg/ton)	Middle Layer Furnish Refining (min)
Control 1	30 EHWK/40 NSWK/30 EHWK	0	0
Control 2	30 EHWK/40 NSWK/30 EHWK	3	0
Control 3	30 EHWK/40 NSWK/30 EHWK	6	0
Inventive 1	30 EHWK/30 NSWK 10 HYA/ 30 EHWK	0	2
Inventive 2	30 EHWK/30 NSWK 10 HYA/ 30 EHWK	2	2
Inventive 3	30 EHWK/30 NSWK 10 HYA/ 30 EHWK	4	2

[0066] The formed web was non-compressively dewatered and rush transferred to a transfer fabric traveling at a speed about 28 percent slower than the forming fabric. The web was then transferred to a T-1205-2 TAD fabric (commercially available from Voith Fabrics, Appleton, Wis. and previously disclosed in U.S. Pat. No. 8,500,955, the contents of which are incorporated herein in a manner consistent with the present disclosure). The web was then dried and wound into a parent roll. The effect of agave fibers on various tissue properties, including tensile, durability and softness, is summarized in the tables below.

TABLE 2

Sample	Basis Weight (gsm)	Sheet Bulk (cc/g)	GMT (g/3")	GM Slope (kg)	CD Tensile (g/3")	CD TEA (g · cm/ cm ²)	CD Stretch (%)
Control 1	36.1	14.5	587	4.96	392	3.35	8.7
Control 2	35.8	13.4	856	6.51	607	5.40	9.7
Control 3	35.9	14.5	983	6.84	709	6.46	10.2
Inventive 1	36.9	13.7	735	6.34	514	4.14	9.0
Inventive 2	35.9	13.0	829	6.11	589	5.31	10.9
Inventive 3	37.3	13.9	1024	7.00	742	6.74	11.3

TABLE 3

Sample	Stiffness Index	Dry Burst (gf)	Burst Index	GM Tear (gf)	CD Tear (gf)	CD Durability Index
Control 1	8.46	521	8.89	8.30	9.9	19.72
Control 2	7.60	727	8.50	13.90	16.5	21.30
Control 3	6.96	832	8.46	15.25	18.5	21.93
Inventive 1	8.63	623	8.48	10.61	12.0	19.35
Inventive 2	7.38	687	8.28	10.59	12.5	22.03
Inventive 3	6.84	919	8.97	14.29	16.2	22.52

[0067] To further assess the quantity of volatile agave compounds present in the inventive products, additional tissue products comprising 20 and 40 percent, by weight of the tissue product, HYA pulp were produced as described above. The tissue products were analyzed by solid phase micro extraction (SPME) and high resolution gas chromatography coupled to mass spectrometry (GC-MS) to quantify odor compounds limonene and 6-methyl-heptene-2-one (MeH), as described in the Test Methods section above. FIG. 1 illustrates the SPME analysis of limonene and MeH for tissue products comprising 20 and 40 percent, by weight of the tissue product, agave fiber, compared to limonene and MeH standards. FIG. 2 illustrates the profile of volatile hydrocarbons obtained by the separations shown in FIG. 1. The amount of limonene and MeH in the various tissue product samples is summarized in the table below.

TABLE 4

Sample	HYA (wt %)	MeH (ppb)	Limonene (ppb)
Inventive 4	40	29.58	13.07
Inventive 5	20	22.89	9.92

[0068] While tissue webs, and tissue products comprising the same, have been described in detail with respect to the specific embodiments thereof, it will be appreciated that those skilled in the art, upon attaining an understanding of the foregoing, may readily conceive of alterations to, variations of, and equivalents to these embodiments. Accordingly, the scope of the present invention should be assessed as that of the appended claims and any equivalents thereto and the foregoing embodiments:

[0069] In a first embodiment the present invention provides a fibrous structure comprising at least about 0.5 percent, by weight of the fibrous structure, agave fiber and a volatile agave compound selected from the group consisting of limonene, hexanaldehyde and 6-Methyl-5-hepten-2-one and combinations thereof.

[0070] In a second embodiment the present invention provides the fibrous structure of the first embodiment comprising at least about 10 ppb limonene.

[0071] In a third embodiment the present invention provides the fibrous structure of the first or the second embodiments comprising from about 10 to about 200 ppb limonene and from about 10 to about 500 ppb 6-Methyl-5-hepten-2-one.

[0072] In a fourth embodiment the present invention provides the fibrous structure of any one of the first through the third embodiments having a geometric mean tensile (GMT) greater than about 400 g/3", such as from about 400 to about 2,500 g/3".

[0073] In a fifth embodiment the present invention provides the fibrous structure of any one of the first through the fourth embodiments having a basis weight greater than about 10 grams per square meter (gsm) and a sheet bulk greater than about 5 cc/g.

[0074] In a sixth embodiment the present invention provides the fibrous structure of any one of the first through the fifth embodiments comprising from about 1.0 to about 40 percent, by weight, agave fiber and from about 5.0 to about 30 percent, by weight of the product, wood pulp fibers having an average fiber length greater than about 2.0 mm.

[0075] In a seventh embodiment the present invention provides the fibrous structure of any one of the first through the sixth embodiments wherein the fibrous structure comprises high yield agave pulp fibers.

[0076] In an eighth embodiment the present invention provides the fibrous structure of any one of the first through the seventh embodiments comprising from about 1.0 to about 40 percent, by weight of the fibrous structure, agave fibers and substantially free from wood pulp fibers having an average fiber length greater than about 2.0 mm.

[0077] In a ninth embodiment the present invention provides the tissue product of any one of the first through the eighth embodiments wherein the agave fibers are high yield agave pulp fibers having a lignin content from about 10 to about 15 weight percent.

[0078] In a tenth embodiment the present invention provides the fibrous structure of any one of the first through the ninth embodiments wherein the fibrous structure comprises at least one multi-layered tissue web having a middle layer and two outer layers wherein the agave fiber is selectively disposed in the middle layer and the two outer layers are substantially free of agave fiber.

[0079] In an eleventh embodiment the present invention provides the fibrous structure of any one of the first through the tenth embodiments wherein the fibrous structure comprises a through-air dried fibrous structure. In particularly preferred embodiments the fibrous structure is an uncreped through-air dried fibrous structure and in other embodiments it is a creped through-air dried fibrous structure.

[0080] In a twelfth embodiment the present invention provides the fibrous structure of any one of the first through the eleventh embodiments wherein the fibrous structure comprises a single-ply uncreped through-air dried fibrous structure.

What is claimed is:

1. A fibrous structure comprising at least about 0.5 percent, by weight of the fibrous structure, agave fiber and a volatile agave compound selected from the group consisting of limonene, hexanaldehyde and 6-Methyl-5-hepten-2-one and combinations thereof.

2. The fibrous structure of claim 1 wherein the fibrous structure comprises at least about 10 ppb limonene.

3. The fibrous structure of claim 1 wherein the fibrous structure comprises from about 10 to about 200 ppb limonene and from about 50 to about 500 ppb 6-Methyl-5-hepten-2-one.

4. The fibrous structure of claim 1 wherein the fibrous structure comprises from about 1.0 to about 40 percent, by weight of the fibrous structure, agave fiber and from 60 to about 99 percent, by weight of the fibrous structure, wood pulp fibers.

5. The fibrous structure of claim 4 wherein the agave fibers are high yield agave pulp fibers.

6. The fibrous structure of claim 1 wherein the fibrous structure comprises a throughdried fibrous structure.

7. The fibrous structure of claim 1 wherein the fibrous structure comprises an uncreped through-air dried fibrous structure.

8. The fibrous structure of claim 1 having a first and a second side and a formulation comprising a volatile agave compound selected from the group consisting of limonene, hexanaldehyde and 6-Methyl-5-hepten-2-one and combinations thereof and a carrier disposed on the first or second side.

9. A tissue product comprising at least one tissue web, the tissue web comprising at least about 0.5 percent, by weight of the fibrous structure, agave fiber and a volatile agave compound selected from the group consisting of limonene, hexanaldehyde and 6-Methyl-5-hepten-2-one and combinations thereof, the product having a basis weight from about 10 to about 60 gsm, a geometric mean tensile (GMT) greater than about 400 g/3" and a sheet bulk greater than about 5.0 cc/g.

10. The tissue product of claim 9 wherein the fibrous structure comprises at least about 10 ppb limonene.

11. The tissue product of claim 9 wherein the fibrous structure comprises from about 10 to about 200 ppb limonene and from about 50 to about 500 ppb 6-Methyl-5-hepten-2-one.

12. The tissue product of claim 9 wherein the fibrous structure comprises from about 1.0 to about 40 percent, by weight of the fibrous structure, agave fiber and from 60 to about 99 percent, by weight of the fibrous structure, wood pulp fibers.

13. The tissue product of claim 9 wherein the tissue web is a creped tissue web.

14. A method of manufacturing a tissue web having a lemon-like odor comprising the steps of:

- a. Forming a fiber slurry comprising agave fiber and wood pulp fiber;
- b. Forming a tissue web from the fiber slurry, the web having a first and a second side; and
- c. Depositing on at least a first side of the web a formulation comprising a volatile agave compound selected from the group consisting of limonene, hexanaldehyde and 6-Methyl-5-hepten-2-one and combinations thereof and a carrier.

15. The method of claim 14 wherein the tissue web comprises at least about 10 ppb limonene.

16. The method of claim 14 wherein the tissue web comprises from about 10 to about 200 ppb limonene and from about 50 to about 500 ppb 6-Methyl-5-hepten-2-one.

17. The method of claim 14 wherein the volatile agave compounds comprise from about 0.0001 to 0.5 percent, by weight of the formulation.

18. The method of claim 14 wherein the carrier is selected from group consisting of water, emollients, fatty acids, fatty alcohols, humectants, thickeners and combinations thereof.

19. The method of claim 14 wherein the carrier is a water and oil emulsion and comprises from about 35 to about 60 percent, by weight of the formulation.

20. The method of claim 14 wherein the tissue web comprises from about 1.0 to about 30 percent, by weight of the tissue web, agave fiber.

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