



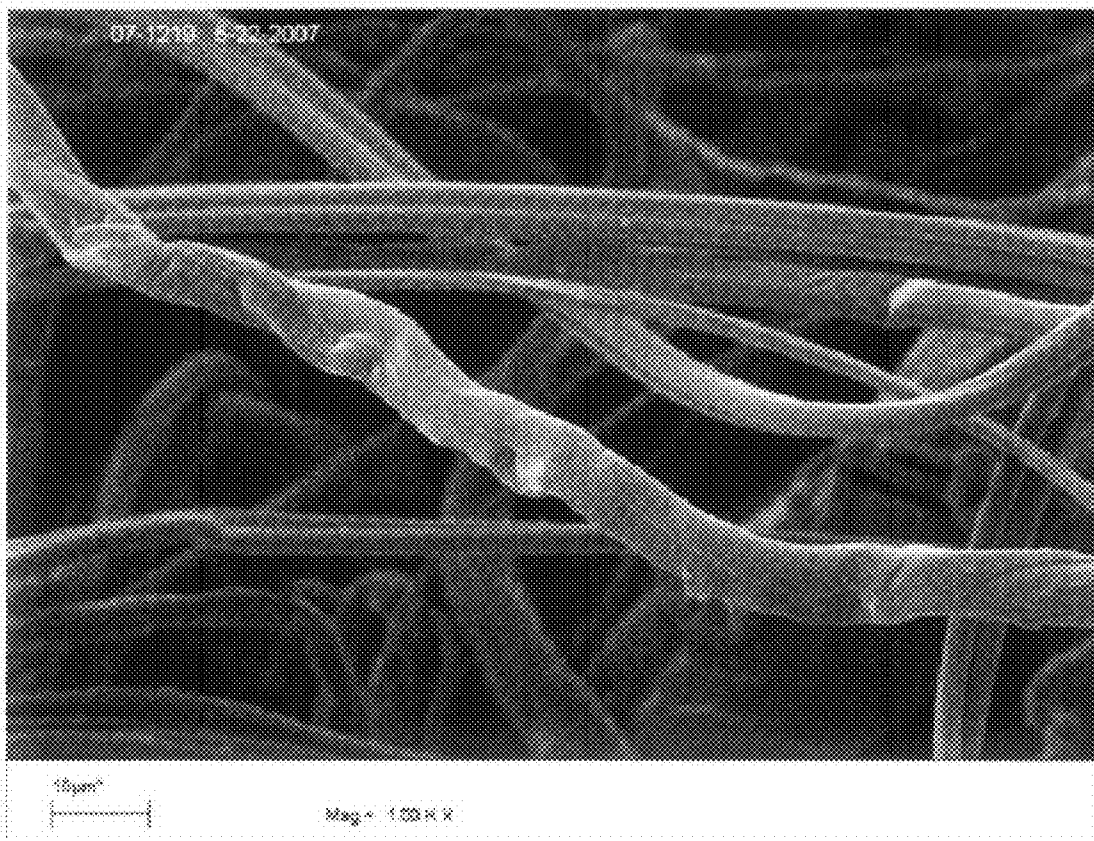
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(19) **United States**(12) **Patent Application Publication**
Luo(10) **Pub. No.: US 2009/0004474 A1**(43) **Pub. Date: Jan. 1, 2009**(54) **LYOCELL FIBERS WITH ANTI-MICROBIAL
ACTIVITY**(75) Inventor: **Mengkui Luo**, Auburn, WA (US)

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WEYERHAEUSER COMPANY**INTELLECTUAL PROPERTY DEPT., CH 1J27****P.O. BOX 9777****FEDERAL WAY, WA 98063 (US)**(73) Assignee: **Weyerhaeuser Co.**, Federal Way,
WA (US)(21) Appl. No.: **11/771,875**(22) Filed: **Jun. 29, 2007****Publication Classification**(51) **Int. Cl.**
D02G 3/00 (2006.01)(52) **U.S. Cl.** **428/397**(57) **ABSTRACT**

Meltblown lyocell fibers are disclosed which show a high degree of antimicrobial activity against *E. coli* and *C. albicans*. The fibers are prepared by adding inorganic and organic compounds to the NMNO dope prior to spinning. The additives are uniformly distributed throughout the fiber cross section and longitudinal sections show relatively smooth or grainy, rough surfaces depending on the additive used.



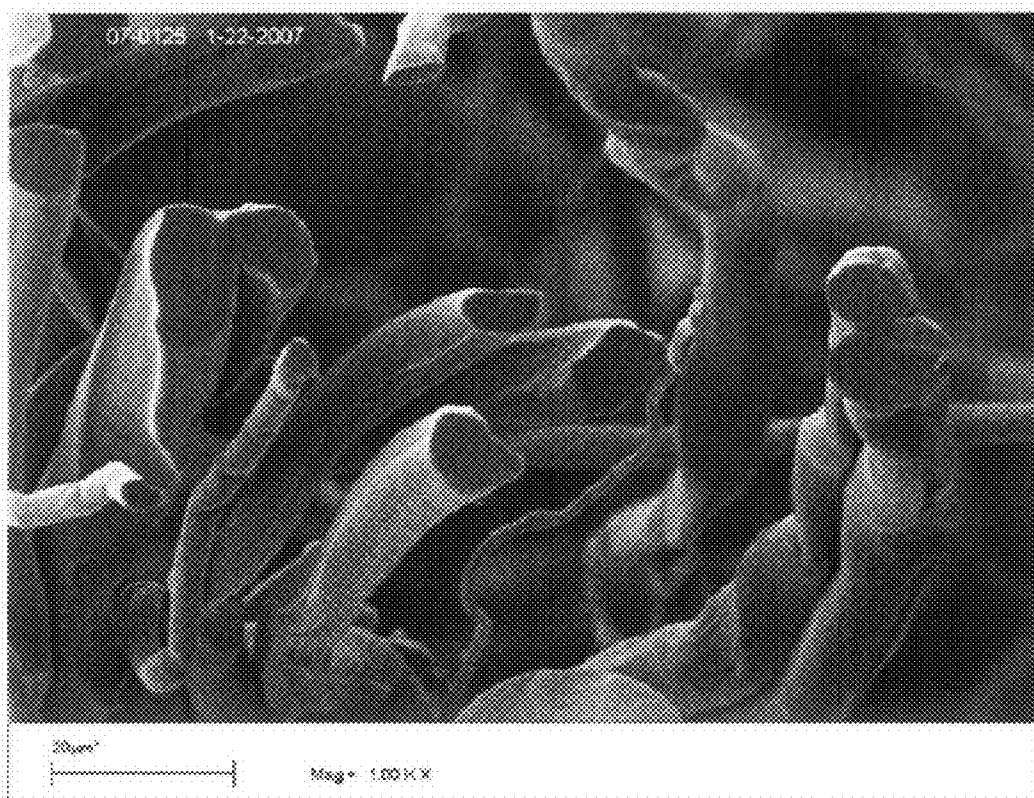


Fig 1

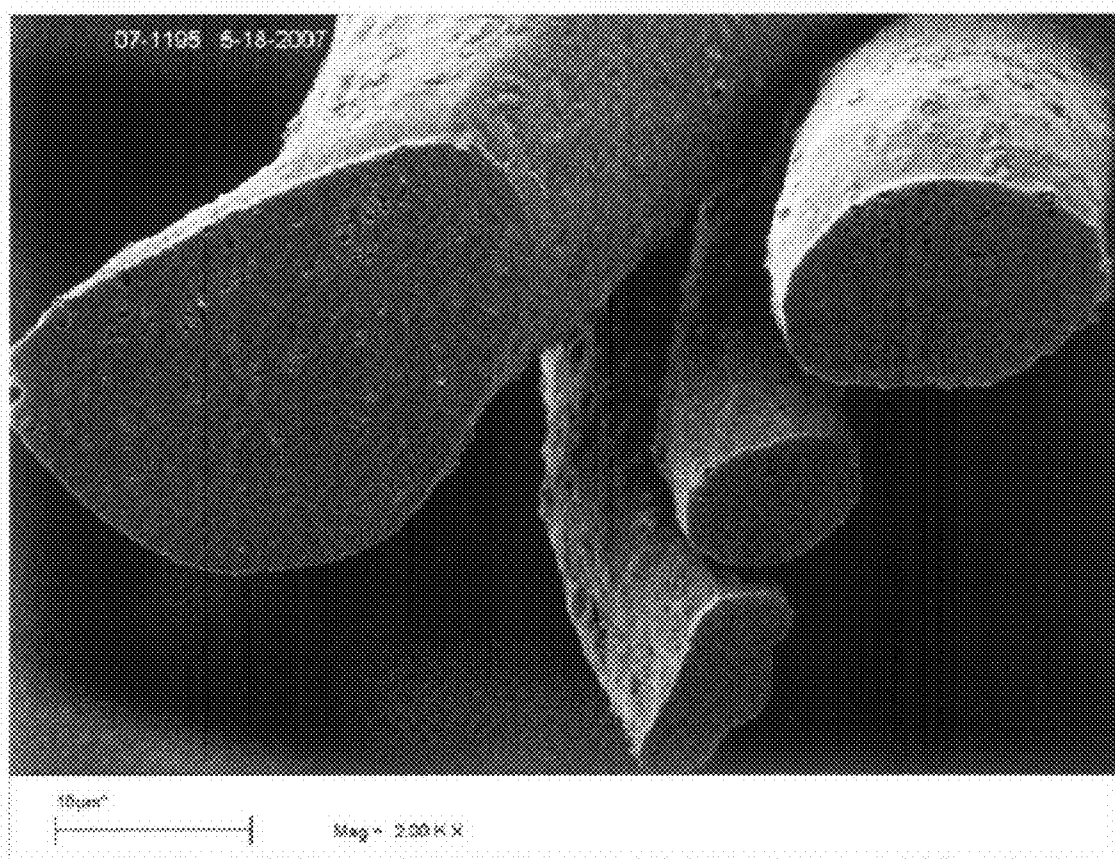


Fig 2

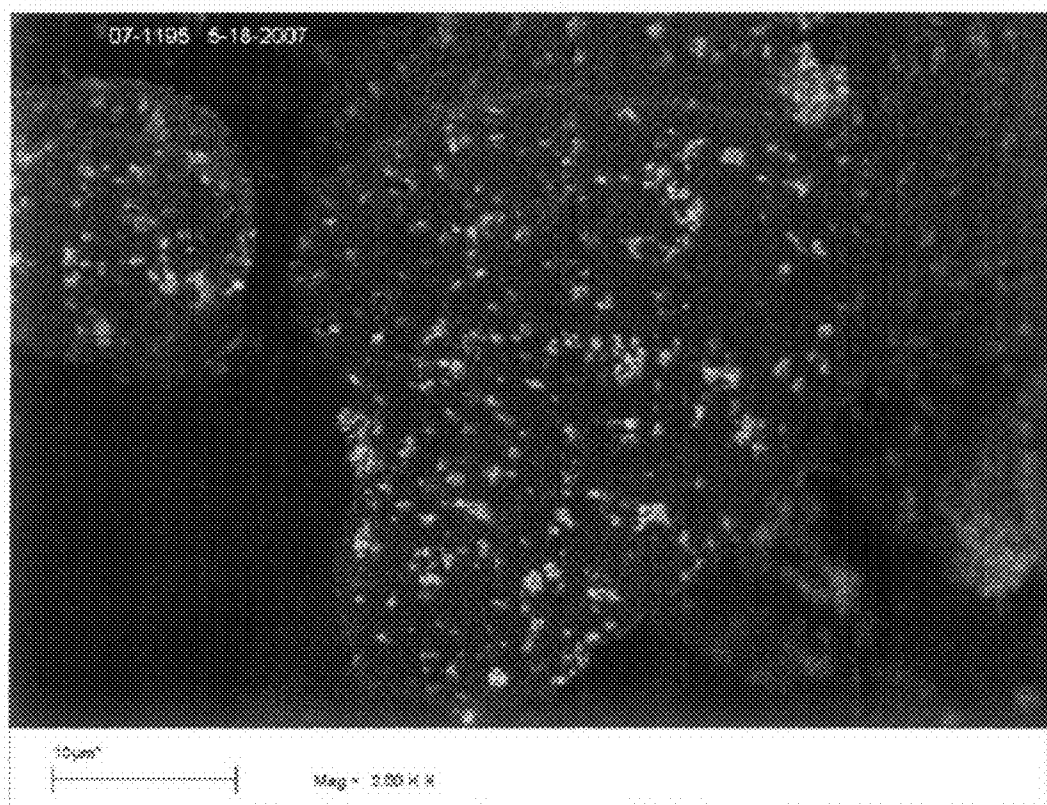


Fig. 3

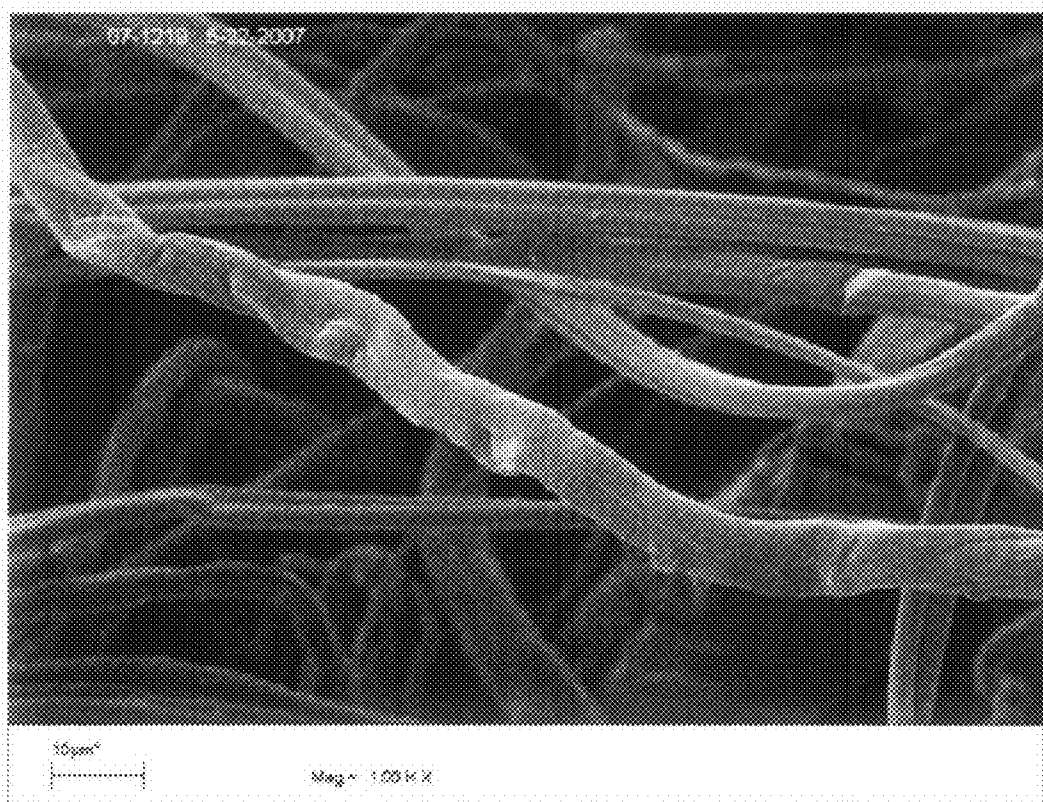


Fig. 4

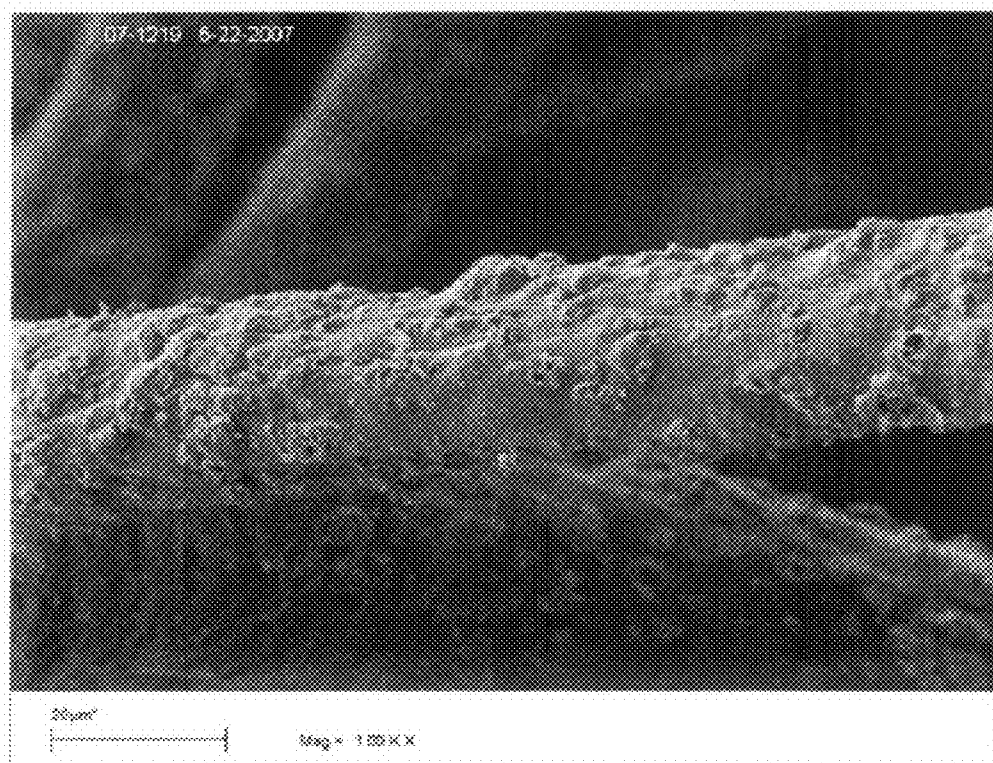


Fig 5

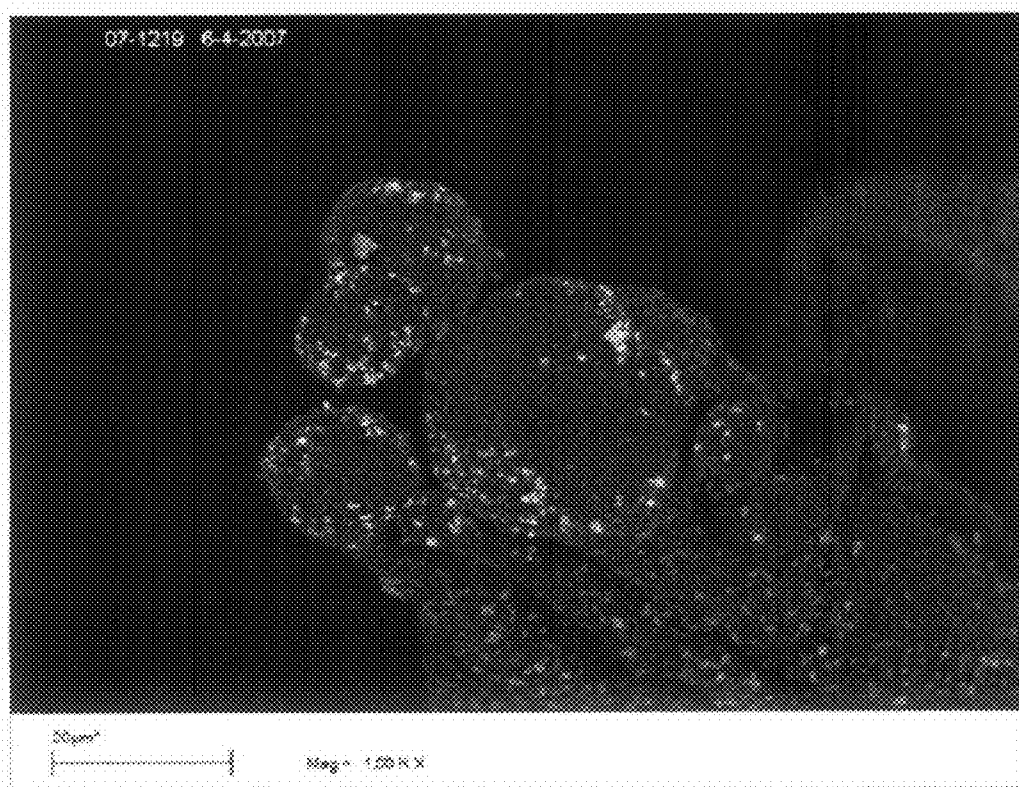


Fig 6

LYOCELL FIBERS WITH ANTI-MICROBIAL ACTIVITY

FIELD

[0001] The present application relates to lyocell fibers with antimicrobial activity.

BRIEF DESCRIPTION OF THE DRAWINGS

[0002] FIG. 1 is a scanning electron photomicrograph at 1000× of the longitudinal section of control Sample A.

[0003] FIG. 2 is a scanning electron photomicrograph at 1000× of the cross section of Sample 6.

[0004] FIG. 3 is a backscattering electron photomicrograph at 2000× of the cross section of Sample 7.

[0005] FIG. 4 is a scanning electron photomicrograph at 1000× of the longitudinal section of Sample 11.

[0006] FIG. 5 is a scanning electron photomicrograph at 1000× of the longitudinal section of Sample 15.

[0007] FIG. 6 is backscattering electron photomicrograph at 1000× of the cross section of Sample 13.

DESCRIPTION

[0008] The present application is directed to lyocell fibers with antimicrobial activity. In particular it is directed to lyocell fibers comprising antimicrobial agents in which the fibers are extruded by the meltblown process.

[0009] The need exists for low cost disposable consumer products such as nonwoven wipes and towels with attributes that make them suitable for specific end use applications. Meltblown lyocell fibers with antimicrobial activity are particularly suitable for use in nonwoven applications because of their characteristic soft feel, water absorption, microdiameter size, biodegradability and the ability of these fibers to be combined in the spinning process to form either selfbonded or spunlaced webs. Fibers made from pulp with a high hemicellulose content are particularly suited for this application because of the added interfiber bonding attributed to hemicellulose.

[0010] Currently available lyocell fibers are produced from high quality wood pulps that have been extensively processed to remove non-cellulose components, especially hemicellulose. These highly processed pulps are referred to as dissolving grade or high α (high α) pulps, where the term α refers to the percentage of cellulose remaining after, extraction with 17.5% caustic. Alpha cellulose can be determined by TAPPI 203. Thus, a high α pulp contains a high percentage of cellulose, and a correspondingly low percentage of other components, especially hemicellulose. The processing required to generate a high α pulp significantly adds to the cost of lyocell fibers and products manufactured therefrom. Typically, the cellulose for these high α pulps comes from both hardwoods and softwoods; softwoods generally have longer fibers than hardwoods.

[0011] Since conventional Kraft processes stabilize residual hemicelluloses against further alkaline attack, it is not possible to obtain acceptable quality dissolving pulps, i.e., high α pulps, through subsequent treatment of Kraft pulp in the bleaching stages. A relatively low copper number, reflective of the relative carbonyl content of the cellulose, is a desirable property of a pulp that is to be used to make lyocell fibers because it is generally believed that a high copper number causes cellulose and solvent degradation, before, during, and/or after dissolution in an amine oxide solvent. The

degraded solvent can either be disposed of or regenerated, however, due to its cost it is generally undesirable to dispose of the solvent.

[0012] A low transition metal content is a desirable property of a pulp that is to be used to make lyocell fibers because, for example, transition metals accelerate the undesirable degradation of cellulose and NMMO (N-methyl morpholine N-oxide) in the lyocell process.

[0013] In view of the expense of producing commercial dissolving grade pulps, it is desirable to have alternatives to conventional high α dissolving grade pulps as a lyocell raw material.

[0014] Low α (e.g., high yield) pulps can be used to make lyocell fibers. Preferably, the desired low α pulps will have a low copper number, a low lignin content and a desirably low transition metal content but broad molecular weight distribution.

[0015] Pulps which meet these requirements have been made and are described in U.S. Pat. No. 6,797,113, U.S. Pat. No. 6,686,093 and U.S. Pat. No. 6,706,876, the assignee of the present application. While high purity pulps are also suitable for use in the present application, low cost pulps such as Peach®, Grand Prairie Softwood and C-Pine, all available from Weyerhaeuser are suitable. These pulps provide the benefit of lower cost and better bonding for nonwoven textile applications because of their high hemicellulose content. Selected pulp properties are given in Table 1.

TABLE 1

Pulp	Pulp Properties				
	R ₁₀	R ₁₈	% Xylan	% Mannan	α -cellulose
Peach	85	88	7.05	6.10	86
Grand Prairie		19*	7.59	6.2	
Softwood					
C-Pine	87.4	88.0	7.50	5.86	

*18% solubility by TAPPI T235

[0016] As used in this application the degraded shorter molecular weight components in the pulp are measured by the R₁₈ and R₁₀ content as described in TAPPI 235. R₁₀ represents the residual undissolved material that is left extraction of the pulp with 10 percent by weight caustic and R₁₈ represents the residual amount of undissolved material left after extraction of the pulp with an 18% caustic solution. Generally, in a 10% caustic solution, hemicellulose and chemically degraded short chain cellulose are dissolved and removed in solution. In contrast, generally only hemicellulose is dissolved and removed in an 18% caustic solution. Thus, the difference between the R₁₀ value and the R₁₈ value, ($\Delta R = R_{18} - R_{10}$), represents the amount of chemically degraded short chained cellulose that is present in the pulp sample. In one embodiment the pulp has a ΔR from about 2 to a ΔR of about 10. In another embodiment the ΔR is from about 4 to a ΔR of about 6.

[0017] The term hemicellulose refers to a heterogeneous group of low molecular weight carbohydrate polymers that are associated with cellulose in wood. Hemicelluloses are amorphous, branched polymers, in contrast to cellulose which is a linear polymer. The principal, simple sugars that combine to form hemicelluloses are: D-glucose, D-xylose, D-mannose, L-arabinose, D-galactose, D-glucuronic acid and D-galacturonic acid.

[0018] Hemicellulose was measured in the pulp and in the fiber by the method described below for sugar analysis and represents the sum of the xylan and mannan content of the pulp or fiber.

[0019] Lyocell fibers prepared with the antimicrobial agents can be spun from cellulose dissolved in NMMO by various processes. In one embodiment the fibers are spun by the meltblown process. Where the term meltblown is used it will be understood that it refers to a process that is similar or analogous to the process used for the production of thermoplastic fibers, even though the cellulose is in solution and the spinning temperature is only moderately elevated. In another embodiment the fibers are spun by the centrifugal spinning process, in another embodiment the fibers are spun by the dry-jet-wet process and in yet another the fibers are spun by the spun bonding process. Fibers formed by the meltblown process can be continuous or discontinuous depending on air velocity, air pressure, air temperature, viscosity of the solution, D.P. of the cellulose and combinations thereof; in the continuous process the fibers are taken up by a reel and optionally stretched. In one embodiment for making a nonwoven web the fibers are contacted with a non solvent such as water by spraying, subsequently taken up on a moving foraminous support, washed and dried. The fibers formed by this method can be in a bonded nonwoven web depending on the extent of coagulation or if it is spunlaced. Spunlacing involves impingement with a water jet. A somewhat similar process is called "spunbonding" where the fiber is extruded into a tube and stretched by an air flow through the tube caused by a vacuum at the distal end. In general, spunbonded fibers are longer than meltblown fibers which usually come in discrete shorter lengths. Another process, termed "centrifugal spinning", differs in that the polymer is expelled from apertures in the sidewalls of a rapidly spinning drum. The fibers are stretched somewhat by air resistance as the drum rotates. However, there is not usually a strong air stream present as in meltblowing. The other technique is dry jet/wet. In this process the filaments exiting the spinneret orifices pass through an air gap before being submerged and coagulated in a liquid bath. All four processes may be used to make nonwoven fabrics.

[0020] In one embodiment the fibers are made from a pulp with greater than 3% percent by weight hemicellulose. In another embodiment the fibers are made from a pulp with greater than 8% by weight hemicellulose. In yet another embodiment the fibers are made from a pulp with greater than 12% by weight hemicellulose.

[0021] In one embodiment the fibers contain from about 4 to 18% by weight hemicellulose. In another embodiment the fibers contains from 7 to 14% by weight hemicellulose and in yet another embodiment the fibers contain from 9% to 12 percent by weight hemicellulose.

[0022] In one embodiment the D.P. of the fibers is from about 200 to about 2000. In another embodiment the D.P. is from about 350 to about 900 and in yet another 4 embodiment the D. P. is from about 400 to about 800. As defined herein, degree of polymerization (abbreviated D.P.) refers to the number of alhydro-D-glucose units in the cellulose chain. D. P. was determined by ASTM Test 1795-96.

[0023] Antimicrobial fibers may be used in a wide variety of fibrous products, among them textiles and garments (including athletic wear, incontinence and medical garments, etc.), air and water filters, wound and burn care dressings, medical wipes and gowns, shoe components, and institutional

and home furnishings including bed sheets, pillow cases, mattress pads, blankets, towels, drapes, bedspreads, pillow shams, carpets, walk-off mats, napkins, linens, wall coverings, upholstered furniture, liners, mattress ticking, mattress filling, pillow filling, carpet pads, upholstery fabric and the like.

[0024] Antimicrobial agents can be inorganic or organic compounds. Inorganic compounds include but are not limited to compounds that contain tin, copper, silver and zinc and may be in the form of the oxide or carried in such compounds as zirconium phosphate, a zeolite or similar carriers. Other inorganic compounds containing metals such as potassium, magnesium and calcium can also be used as antimicrobial agents. Inorganic antimicrobial agents include silver zeolite complexes sold by Milliken Chemical as ALPHASAN, and AGION by Agion Technologies. In the present application, zeolite of the formula $\text{Ag}_{84}\text{Na}_2(\text{AlO}_2)_{86}(\text{SiO}_2)_{106}\times\text{H}_2\text{O}$ was obtained from Aldrich as a silver exchanged zeolite in granular form and was ground to pass through a screen of <20 micron. Zinc oxide, grade AZO 66USP was obtained from US Zinc; >99.9 percent of the particles passed through a 325 mesh screen. Calcium carbonate was obtained from Aldrich (CAS 471-34-1) and had a particle size of less than 10 microns.

[0025] In one embodiment the inorganic antimicrobial agent is added at a level of from about 1 percent by weight on pulp to about 40 percent by weight on cellulose. In another embodiment the additive is added at a level of from 10 percent by weight on cellulose to about 25 percent by weight on cellulose. In yet another embodiment it is added at a level of from about 15 percent by weight on pulp to about 20 percent by weight on cellulose.

[0026] In one embodiment the inorganic antimicrobial agent is at a level of from about 1 percent by weight to about 40 percent by weight in the fiber. In another embodiment the, additive is at a level of from 10 percent by weight to about 25 percent by weight in the fiber. In yet another embodiment it is at a level of from about 15 percent by weight to about 20 percent by weight in the fiber.

[0027] Organic compounds useful as antimicrobial agents include but are not limited to Triclosan, quaternary ammonium compounds, diammonium ring compounds, chitosans, N-halamine siloxanes and chlorine. Organic compounds depend on the antimicrobial agent to leach or migrate from inside the fiber to the surface.

[0028] Processing and fiber properties of meltblown fibers containing antimicrobial agents are shown in Table 2 and Table 4; Table 3 and Table 5 show the results of antimicrobial testing of the meltblown fibers.

[0029] In the present application, SILVIO®, manufactured by InikTec, Korea, an organic silver complex compound at a concentration of 5% in water was used. The antimicrobial agent is highly efficacious and can be used at extremely low levels to produce a significant reduction in colony forming units. In one embodiment the additive is added at a level of from about 0.01 to about 5 percent by weight of a 5% by weight solution of the organic silver complex compound on cellulose. In another embodiment the additive is added at a level of from about 0.5 to about 3% by weight solution of the organic silver complex compound on cellulose. In one embodiment the fibers contain from about 5 to about 1000 ppm silver. Lyocell fibers with silver compounds have lower brightness than a control sample but combining silver based antimicrobial agents with zinc oxide or calcium carbonate

improves fiber brightness (sample 11, Table 4, v sample 10 Table 2 and sample 11 v sample 17 Table 4, respectively).

[0030] Meltblown lyocell fibers containing inorganic zinc compounds reduced *E. coli* colony forming units at twenty four hours by at least 98 percent compared to a control at the same time. *C. albicans* count was reduced at least ninety five percent at four and twenty four hours compared to a control at the same times. Both the inorganic additive calcium carbonate and the organic additive SILVIO containing an organic silver complex reduced the four and twenty four hour colony forming units of *E. coli* by more than ninety five percent compared to a control at the same times. Zinc oxide reduced the four hour colony forming units of *C. albicans* by at least ninety five percent and the twenty four hour colony forming units of both *E. coli* and *C. albicans* by at least ninety five Percent.

[0031] Meltblown lyocell fibers containing various antimicrobial agents are shown in FIGS. 2-6. FIG. 1 is a scanning electron photomicrograph (SEM) of a control sample showing a longitudinal section and cross section of the fibers at 1000 $\times$. The fibers are relatively smooth with oblong to circular cross sections. FIG. 2 is a SEM at 2000 $\times$ of the cross section of Sample 6 showing uniformly distributed zinc oxide particles in the fiber and a grainy or granular surface. FIG. 3 is a backscattering electron photomicrograph (BSE) at 2000 $\times$ of the cross section of fibers of Sample 7 showing the uniform distribution of zinc in the fibers. FIG. 4 is a SEM at 1000 $\times$ of the longitudinal section meltblown fibers containing about 0.1 percent by weight of SILVIO. Fibers containing this additive have a relatively smooth surface. FIG. 5 is a SEM at 1000 $\times$ of the meltblown lyocell fibers of Sample 15 containing 22.39 percent by weight calcium carbonate in the fiber. The fibers are characterized by a rough and granular surface. FIG. 6 shows a BSE at 1000 $\times$ of the cross section of Sample 13 containing 1.9 percent by weight zeolite in the fiber and the distribution of zinc in the fiber and on the fiber surface.

[0032] Depending on a number of factors such as air velocity, air pressure, air temperature, viscosity of the solution, D.P. of the cellulose and combinations thereof, a wide range of fiber properties can be obtained by the meltblowing process. In one embodiment the fibers have a fiber diameter of from about 5 μ to about 50 μ . In another embodiment the fibers have a fiber diameter of from about 10 μ to about 30 μ and in yet another embodiment the fibers have a fiber diameter of from about 15 to about 20 μ . Fiber diameter measurements

represent the average diameter of 100 randomly selected fibers and measurement with a light microscope.

[0033] Birefringence of the antimicrobial fibers indicates a high degree of molecular orientation of the cellulose fibers which is virtually unchanged from the control. Control values ranged from 0.026 to 0.034 and samples with zinc oxide were unchanged at 0.026. This suggests that in spite of the zinc oxide additive, the Molecular orientation is not adversely affected. In one embodiment the birefringence is at least 0.02. In another embodiment the birefringence is at least 0.025. Birefringence was determined by the method described below. A typical birefringence value for lyocell is 0.045, for viscose staple, 0.022, Modal, 0.038, for cotton, 0.047, for ramie, 0.074 and NB416, a commercially available market pulp available from Weyerhaeuser, 0.026.

[0034] Fiber brightness was determined by TAPPI T452.

EXAMPLE

[0035] In a representative example, Peach®, a bleached kraft southern pine pulp, available from Weyerhaeuser, Federal Way, Wash., was acid hydrolyzed and treated with sodium borohydride to yield a pulp having an average degree of polymerization of about 420, a hemicellulose content of 12.0% by weight hemicellulose in pulp (6.5% and 5.5% by weight xylan and mannan, respectively) and an R₁₀ and R₁₈, of about 77 and 87, respectively. The pulp was dissolved in NMMO (N-methyl morpholine N-oxide) as follows. A 250 mL three necked flask was charged with, for example, 66.4 g of 97% NMMO, 24.7 g of 50% NMMO, 10.4 g pulp, 0.1 g of propyl gallate, and 1.2 g of zinc oxide. The flask was immersed in an oil bath at 120 C, a stirrer inserted and stirring continued for about 1 hr. A readily flowable dope resulted that was suitable for spinning. The cellulose concentration in the dope was about 12% by weight. The dope was extruded from a melt blowing die that had 3 nozzles having an orifice diameter of 457 microns at a rate of 1.0 gram/hole/minute. The orifices had a length/diameter ratio of 5. The nozzle was maintained at a temperature of 95 C. The dope was extruded into an air gap 30 cm long before coagulation in water and collected on a screen as either continuous or discontinuous filaments depending on dope rheology and meltblown conditions. Air, at a temperature of 95 C and a pressure of about 10 psi, was supplied to the head. Air pressures of from 8 to 30 psi were used to achieve varying fibers diameters shown in Table 2 and Table 4.

TABLE 2

Processing and Fiber Properties Of Meltblown Fibers Containing Antimicrobial Agents										
	Control				Sample No.					
	A	B	C	D	1	2	3	4	5	6
97% NMMO g	66.4	66.4	66.4	66.4	66.4	66.4	66.4	66.4	66.4	66.4
50% NMMO g	25.4	25.4	25.4	25.4	24.7	24.7	24.7	24.7	24.7	24.7
Propyl gallate g	0.1	0.1	0.1	0.1	0.1	0.1	0.1	0.1	0.1	0.1
Pulp g	10.4	10.4	10.4	10.4	10.4	10.4	10.4	10.4	10.4	10.4
SILVIO ® (5%) (g)										
Cellulose %	10.18	10.18	10.18	10.18	10.25	10.25	10.25	10.25	10.25	10.25
Additive	no	no	no	no	ZnO	ZnO	ZnO	ZnO	ZnO	ZnO
Additive g	0	0	0	0	1.2	1.2	1.2	1.2	1.2	2.4
Wt. % additive on cellulose	0	0	0	0	11.54	11.54	11.54	11.54	11.54	23.1
Wt. % additive in fiber	0.00	0.00	0.00	0.00	10.34	10.34	10.34	10.34	10.34	18.75
Solid (wt % of total)	10.18	10.18	10.18	10.18	11.30	11.30	11.30	11.30	11.30	12.32

TABLE 2-continued

Processing and Fiber Properties Of Meltblown Fibers Containing Antimicrobial Agents										
Air pressure (psi)	8-30	10.00	10.00	20.00	5.00	10.00	20.00	35.00	mixed	20.00
Diameter (micron)		17.5	19.9	8.3	44.5	32.3	16.3	9.6		14
Zinc ppm			154			77000				141000
Ag, ppm										
Xylan, % by wt. in fiber	4.8	4.82	5.02	4.76	4.45			4.2	4.5	4.34
Mannan, % by wt. in fiber	4.7	4.61	4.72	4.59	4.26			4.1	4.3	3.77
Brightness ISO		70								
Birefringence		0.026	0.026	0.034		0.026	0.026	0.026		0.026

					Sample No.			
					7	8	9	10
			97% NMMO g		66.4	66.4	66.4	66.4
			50% NMMO g		24.7	24.7	24.7	24.7
			Propyl gallate g		0.1	0.1	0.1	0.1
			Pulp g		10.4	10.4	10.4	10.4
			SILVIO ® (5%) (g)					0.2
			Cellulose %		10.25	10.25	10.25	10.25
			Additive		ZnO	ZnO	ZnO	ZnO
			Additive g		2.4	3.6	3.6	1.1
			Wt. % additive on cellulose		23.1	34.6	34.6	14.4
			Wt. % additive in fiber		18.75	25.71	25.71	9.57
			Solid (wt % of total)		12.32	13.32	13.32	11.21
			Air pressure (psi)		30.00	20.00	30.00	20.00
			Diameter (micron)		10.8	15.6	8.8	15.2
			Zinc ppm		146000	188000		80300
			Ag, ppm					130
			Xylan, % by wt. in fiber		3.84	3.95		4.81
			Mannan, % by wt. in fiber		3.48	3.47		4.07
			Brightness ISO		51.2		72.4	55.7
			Birefringence		0.026	0.026	0.026	

TABLE 3

	Antimicrobial Test Results													
	Control				Sample No.									
	A	B	C	D	1	2	3	4	5	6	7	8	9	10
<i>E. coli</i> at zero time (CFU/ml)*	17500									17500	17500	17500		17500
4 hour	4150									8400	5550	10150		<100
24 hour	8800									<100	<100	<100		<100
<i>C. Albicans</i> at zero time (CFU/ml)	2000									2000	2000	2000		2000
4 hour	2350									<100	<100	<100		<100
24 hour	3500									<100	<100	<100		<100

*Colony forming units

TABLE 4

Processing and Fiber Properties of Fibers Containing Antimicrobial Agents											
	Control				Sample No.						
	A	B	C	D	11	12	13	14	15	16	17
97% NMMO g	66.4	66.4	66.4	66.4	66.2	66.2	66.2	66.2	66.2	66.2	66.2
50% NMMO g	25.4	25.4	25.4	25.4	24.5	24.5	24.5	24.5	24.5	24.5	24.5
Propyl gallate g	0.1	0.1	0.1	0.1	0.1	0.1	0.1	0.1	0.1	0.1	0.1
Pulp DP	420	420	420	420	420	420	420	420	420	420	420
Pulp g	10.4	10.4	10.4	10.4	10.4	10.4	10.4	10.4	10.4	10.4	10.4
SILVIO (5%) (g)					0.2	0.2	0.2				0.2
Cellulose %	10.18	10.18	10.18	10.18	10.29	10.29	10.29	10.29	10.29	10.29	10.29
Additive	no	no	no	no	SILVO	SILVIO	Zeolite	Zeolite	CaCO ₃	CaCO ₃	CaCO ₃
Additive g	0	0	0	0	0.2	0.2	0.2	0.2	3	3	1
Wt. % additive on pulp					0.1	0.1	1.9	1.9	28.9	28.9	9.6
% additive (wt) in fiber	0.00	0.00	0.00	0.00	0.095	0.095	1.89	1.89	22.39	22.39	9.77
Solid (wt % of total)	10.18	10.18	10.18	10.18	10.46	10.46	10.46	10.46	12.87	12.87	11.17
Air pressure (psi)	8-30	10.00	10.00	20.00	20.00	30.00	30.00	30.00	20.00	30	20
Diameter, (micron)		17.5	19.9	8.3	15.5	11.2	15.7	12.2	14.5	10.3	14.6
Ag, ppm					216		730				
Ca, ppm							940		80600	83300	33300
Xylan, % by wt. in fiber	4.8	4.82	5.02	4.76	5.19		4.44		4.19		4.83
Mannan, % by wt. in fiber	4.74	4.61	4.72	4.59	4.4		3.92		3.59		4.01
Brightness, ISO		70			37.89					79.3	48.1
Birefringence		0.026	0.026	0.034							

TABLE 5

Antimicrobial Test Results											
	Control				Sample No.						
	A	B	C	D	11	12	13	14	15	16	17
<i>E. Coli</i> at zero time (CFU/ml)*	17500				17500					17500	17500
4 hour	4150				<100					<100	<100
24 hour	8800				<100					<100	<100
<i>C. Albicans</i> at zero time (CFU/ml)	2000				2000	2000	2000				
4 hour	2350				<100	<100	<100				
24 hour	3500				<100	<100	<100				

*Colony forming units

Birefringence of Fibers by Polarized Light Microscopy

[0036] In theory, fibers can be characterized as having an index of refraction parallel (axial) to the fiber axis and an index of refraction which is perpendicular to the fiber axis. The birefringence for purposes of this method is the difference between these two refractive indices. The convention is to subtract the perpendicular R.I. (refractive index) from the axial R.I. The axial R.I. is typically represented by the Greek letter ω , and the perpendicular index by the letter ϵ . The birefringence is typically represented as

$$\Delta = (\omega - \epsilon)$$

Refractive Index Oils

[0037] Oils are manufactured with known refractive index at a given wavelength of exciting light and at a given temperature. The fibers were compared to Cargile refractive index oils.

Polarized Light

[0038] Using transmitted light in the light microscope, the refractive index is measured using a polarizing filter. When

the exciting light is polarized in a direction parallel to the axis of the fiber the axial refractive index can be measured. Then the polarizing filter can be rotated 90 degrees and the refractive index measured perpendicular to the fiber axis.

Measurement Using the Light Microscope

[0039] When the refractive index of the fiber matches the refractive index of the oil in which it is mounted, the image of the fiber will disappear. Conversely, when the fiber is mounted in an oil which greatly differs in refractive index, the image of the fiber is viewed with high contrast.

[0040] When the R.I. of the fiber is close to the R.I. of the oil, a technique is used to determine whether the fiber is higher or lower in refractive index. First the fiber, illuminated with the appropriately positioned polarizing filter, is brought into sharp focus in the microscope using the stage control. Then the stage is raised upward slightly. If the image of the fiber appears brighter as the stage is raised, the fiber is higher in refractive index than the oil. Conversely if the fiber appears darker as the stage is raised, the fiber is lower in refractive index than the oil.

[0041] Fibers are mounted in R.I. oils and examined until a satisfactory match in refractive index is obtained. Both the axial and the perpendicular component are determined and the birefringence is calculated.

Sugar Analysis

[0042] This method is applicable for the preparation and analysis of pulp and wood Samples for the determination of the amounts of the following pulp sugars: fucose, arabinose, galactose, rhamnose, glucose, xylose and mannose using high performance anion exchange chromatography and pulsed amperometric detection (HPAEC/PAD).

Summary of Method

[0043] Polymers of pulp sugars are converted to monomers by hydrolysis using sulfuric acid.

[0044] Samples are ground, weighed, hydrolyzed, diluted to 200-mL final volume, filtered, diluted again (1.0 mL+8.0 mL H₂O) in preparation for analysis by HPAEC/PAD.

Sampling, Sample Handling and Preservation

[0045] Wet Samples are air-dried or oven-dried at 25±5 C

Equipment Required

[0046] Autoclave, Market Forge, Model # STM-E, Serial # C-1808

[0047] 100×10 mL Polyvials, septa, caps, Dionex Cat # 55058

[0048] Gyrotory Water-Bath Shaker, Model G76 or some equivalent.

[0049] Balance capable of weighing to ±0.01 mg, such as Mettler HL52 Analytical Balance.

[0050] Intermediate Thomas-Wiley Laboratory Mill, 40 mesh screen.

[0051] NAC 1506 vacuum oven or equivalent.

[0052] 0.45-μ GHP filters, Gelman type A/E, (4.7-cm glass fiber filter discs, without organic binder)

[0053] Heavy-walled test tubes with pouring lip, 2.5×20 cm.

[0054] Comply SteriGage Steam Chemical Integrator

[0055] GP 50 Dionex metal-free gradient pump with four solvent inlets

[0056] Dionex ED 40 pulsed amperometric detector with gold working electrode and solid state reference electrode

[0057] Dionex autoSampler AS 50 with a thermal compartment containing the columns, the ED 40 cell and the injector loop

[0058] Dionex PC10 Pneumatic Solvent Addition apparatus with 1-L plastic bottle

[0059] 3 2-L Dionex polyethylene solvent bottles with solvent outlet and helium gas inlet caps

[0060] CarboPac PA1 (Dionex P/N 035391) ion-exchange column, 4 mm×250 mm

[0061] CarboPac PA1 guard column (Dionex P/N 043096), 4 mm×50 mm

[0062] Millipore solvent filtration apparatus with Type HA 0.45 μ filters or equivalent

Reagents Required

[0063] All references to H₂O is Millipore H₂O

[0064] 72% Sulfuric Acid Solution (H₂SO₄)—Transfer 183 mL of water into a 2-L Erlenmeyer flask. Pack the flask in ice in a Rubbermaid tub in a hood and allow the flask to cool. Slowly and cautiously pour, with swirling, 470 mL of 96.6% H₂SO₄ into the flask. Allow solution to cool. Carefully transfer into the bottle holding 5-mL dispenser. Set dispenser for 1 mL.

[0065] JT Baker 50% sodium hydroxide solution, Cat. No. Baker 3727-01, [1310-73-2]

[0066] Dionex sodium acetate, anhydrous (82.0±0.5 grams/1 L H₂O), Cat. No. 59326, [127-09-3].

[0067] Standards

[0068] Internal Standards

[0069] Fucose is used for the kraft and dissolving pulp Samples. 2-Deoxy-D-glucose is used for the wood pulp Samples.

[0070] Fucose, internal standard. 12.00±0.005 g of Fucose, Sigma Cat. No. F 2252, [2438-80-4], is dissolved in 200.0 mL H₂O giving a concentration of 60.00±0.005 mg/mL. This standard is stored in the refrigerator. 2-Deoxy-D-glucose, internal standard. 12.00±0.005 g of 2-Deoxy-D-glucose, Fluka Cat. No. 32948 g [101-77-9] is dissolved in 200.0 mL H₂O giving a concentration of 60.00±0.005 mg/mL. This standard is stored in the refrigerator.

Kraft Pulp Stock Standard Solution

[0071]

KRAFT PULP SUGAR STANDARD CONCENTRATIONS

Sugar	Manufacturer	Purity	g/200 mL
Arabinose	Sigma	99%	0.070
Galactose	Sigma	99%	0.060
Glucose	Sigma	99%	4.800
Xylose	Sigma	99%	0.640
Mannose	Sigma	99%	0.560

Kraft Pulp Working Solution

[0072] Weigh each sugar separately to 4 significant digits and transfer to the same 200-mL volumetric flask. Dissolve sugars in a small amount of water. Take to volume with water, mix well, and transfer contents to two clean, 4-oz. amber bottles. Label and store in the refrigerator. Make working standards as in the following table.

PULP SUGAR STANDARD CONCENTRATIONS FOR KRAFT PULPS

Fucose Sugar	mg/mL	mL/200 mL 0.70 ug/mL	mL/200 mL	mL/200 mL	mL/200 mL	mL/200 mL
			1.40 ug/mL	2.10 ug/mL	2.80 ug/mL	3.50 ug/mL
Fucose	60.00	300.00	300.00	300.00	300.00	300.00
Arabinose	0.36	1.2	2.5	3.8	5.00	6.508
Galactose	0.30	1.1	2.2	3.30	4.40	5.555

-continued

<u>PULP SUGAR STANDARD CONCENTRATIONS FOR KRAFT PULPS</u>						
Sugar	mg/mL	mL/200 mL 0.70 ug/mL	mL/200 mL 1.40 ug/mL	mL/200 mL 2.10 ug/mL	mL/200 mL 2.80 ug/mL	mL/200 mL 3.50 ug/mL
Fucose	24.0	84	168.0	252.0	336.0	420.7
Xylose	3.20	11	22.0	33.80	45.00	56.05
Mannose	2.80	9.80	19.0	29.0	39.0	49.07

Dissolving Pulp Stock Standard Solution

[0073]

<u>DISSOLVING PULP SUGAR STANDARD CONCENTRATIONS</u>			
Sugar	Manufacturer	Purity	g/100 mL
Glucose	Sigma	99%	6.40
Xylose	Sigma	99%	0.120
Mannose	Sigma	99%	0.080

Dissolving Pulp Working Solution

[0074] Weigh each sugar separately to 4 significant digits and transfer to the same 200-mL volumetric flask. Dissolve sugars in a small amount of water. Take to volume with water, mix well, and transfer contents to two clean, 4-oz. amber bottles. Label and store in the refrigerator. Make working standards as in the following table.

<u>PULP SUGAR STANDARD CONCENTRATIONS FOR DISSOLVING PULPS</u>						
Sugar	mg/mL	mL/200 mL 0.70 ug/mL	mL/200 mL 1.40 ug/mL	mL/200 mL 2.10 ug/mL	mL/200 mL 2.80 ug/mL	mL/200 mL 3.50 ug/mL
Fucose	60.00	300.00	300.00	300.00	300.00	300.00
Glucose	64.64	226.24	452.48	678.72	904.96	1131.20
Xylose	1.266	4.43	8.86	13.29	17.72	22.16
Mannose	0.8070	2.82	5.65	8.47	11.30	14.12

Wood Pulp Stock Standard Solution

[0075]

<u>PULP SUGAR STANDARD CONCENTRATIONS FOR KRAFT PULPS</u>						
Sugar	mg/mL	mL/200 mL 0.70 ug/mL	mL/200 mL 1.40 ug/mL	mL/200 mL 2.10 ug/mL	mL/200 mL 2.80 ug/mL	mL/200 mL 3.50 ug/mL
2-Deoxy-D-glucose	60.00	300.00	300.00	300.00	300.00	300.00
2-DG	0.300	1.05	2.10	3.15	4.20	6.50
Fucose	0.36	1.2	2.5	3.8	5.00	6.508
Arabinose	0.30	1.1	2.2	3.30	4.40	5.555
Galactose	0.3500	1.225	2.450	3.675	4.900	6.125
Rhamnose	24.00	84	168.0	252.0	336.0	420.7
Glucose	3.20	11	22.0	33.80	45.00	56.05
Xylose	2.80	9.80	19.0	29.0	39.0	49.07
Mannose						

WOOD PULP SUGAR STANDARD CONCENTRATIONS

Sugar	Manufacturer	Purity	g/200 mL
Fucose	Sigma	99%	12.00
Rhamnose	Sigma	99%	0.0701

[0076] Dispense 1 mL of the fucose solution into a 200-mL flask and bring to final volume. Final concentration will be 0.3 mg/mL.

Wood Pulp Working Solution

[0077] Use the Kraft Pulp Stock solution and the fucose and rhamnose stock solutions. Make working standards as in the following table.

Procedure

Sample Preparation

[0078] Grind 0.2 ± 0.05 g Sample with Wiley Mill 40 Mesh screen size. Transfer ~200 mg of Sample into 40-mL Teflon container and cap. Dry overnight in the vacuum oven at 50°C .

[0079] Add 1.0 mL 72% H_2SO_4 to test tube with the Brinkman dispenser. Stir and crush with the rounded end of a glass or Teflon stirring rod for one minute. Turn on heat for Gyrotory Water-Bath Shaker. The settings are as follows:

[0080] Heat: High

[0081] Control Thermostat: 7°C .

[0082] Safety thermostat: 25°C .

[0083] Speed: Off

[0084] Shaker: Off

[0085] Place the test tube rack in gyrotory water-bath shaker. Stir each Sample 3 times, once between 20-40 min, again between 40-60 min, and again between 60-80 min. Remove the Sample after 90 min. Dispense 1.00 mL of internal standard (Fucose) into Kraft Samples.

[0086] Tightly cover Samples and standard flasks with aluminum foil to be sure that the foil does not come off in the autoclave.

[0087] Place a Comply SteriGage Steam Chemical Integrator on the rack in the autoclave. Autoclave for 60 minutes at a pressure of 14-16 psi (95-105 kPa) and temperature $>260^\circ\text{F}$. (127°C).

[0088] Remove the Samples from the autoclave. Cool the Samples. Transfer Samples to the 200-mL volumetric flasks. Add 2-deoxy-D-glucose to wood Samples. Bring the flask to final volume with water.

[0089] For Kraft and Dissolving pulp Samples:

[0090] Filter an aliquot of the Sample through GHP 0.45 μm filter into a 16-mL amber vial.

[0091] For Wood pulp Samples:

[0092] Allow particulates to settle. Draw off approximately 10 mL of Sample from the top, trying not to disturb particles and filter the aliquot of the Sample through GHP 0.45 μm filter into a 16-mL amber vial. Transfer the label from the volumetric flask to the vial. Add 1.00 mL aliquot of the filtered Sample with to 8.0 mL of water in the Dionex vial.

[0093] Samples are run on the Dionex AS/500 system. See Chromatography procedure below.

Chromatography Procedure

[0094] Solvent Preparation

[0095] Solvent A is distilled and deionized water (18 meg-ohm), sparged with helium while stirring for a minimum of 20 minutes, before installing under a blanket of helium, which is to be maintained regardless of whether the system is on or off.

[0096] Solvent B is 400 mM NaOH. Fill Solvent B bottle to mark with water and sparge with helium while stirring for 20 minutes. Add appropriate amount of 50% NaOH.

[0097] $(50.0\text{ g NaOH}/100\text{ g solution}) \times (1\text{ mol NaOH}/40.0\text{ g NaOH}) \times (1.53\text{ g solution}/1\text{ mL solution}) \times (1000\text{ mL solution}/1\text{ L solution}) = 19.1\text{ M NaOH}$ in the container of 50/50 w/w NaOH.

[0098] $0.400\text{ M NaOH} \times (1000\text{ mL H}_2\text{O}/19.1\text{ M NaOH}) = 20.8\text{ mL NaOH}$

[0099] Round 20.8 mL down for convenience:

[0100] $19.1\text{ M} \times (20.0\text{ mL} \times \text{mL}) = 0.400\text{ M NaOH}$

[0101] $x\text{ mL} = 956\text{ mL}$

[0102] Solvent D is 200 mM sodium acetate. Using 18 meg-ohm water, add approximately 450 mL deionized water

to the Dionex sodium acetate container. Replace the top and shake until the contents are completely dissolved. Transfer the sodium acetate solution to a 1-L volumetric flask. Rinse the 500-mL sodium acetate container with approximately 100 mL water, transferring the rinse water into the volumetric flask. Repeat rinse twice. After the rinse, fill the contents of the volumetric flask to the 1-L mark with water. Thoroughly mix the eluent solution. Measure $360 \pm 10\text{ mL}$ into a 2-L graduated cylinder. Bring to $1800 \pm 10\text{ mL}$. Filter this into a 2000-mL sidearm flask using the Millipore filtration apparatus with a 0.45 μm , Type HA membrane. Add this to the solvent D bottle and sparge with helium while stirring for 20 minutes.

[0103] The postcolumn addition solvent is 300 mM NaOH. This is added postcolumn to enable the detection of sugars as anions at $\text{pH} > 12.3$. Transfer $15 \pm 0.5\text{ mL}$ of 50% NaOH to a graduated cylinder and bring to $960 \pm 10\text{ mL}$ in water.

[0104] $(50.0\text{ g NaOH}/100\text{ g Solution}) \times (1\text{ mol NaOH}/40.0\text{ g NaOH}) \times (1.53\text{ g Solution}/1\text{ mL Solution}) \times (1000\text{ mL Solution}/1\text{ L solution}) = 19.1\text{ M NaOH}$ in the container of 50/50 w/w NaOH.

[0105] $0.300\text{ M NaOH} \times (1000\text{ mL H}_2\text{O}/19.1\text{ M NaOH}) = 15.7\text{ mL NaOH}$

[0106] Round 15.7 mL down:

[0107] $19.1\text{ M} \times (15.0\text{ mL}/x\text{ mL}) = 0.300\text{ M NaOH}$

[0108] $x\text{ mL} = 956\text{ mL}$

[0109] (Round 956 mL to 960 mL. As the pH value in the area of 0.300 M NaOH is steady, an exact 956 mL of water is not necessary.)

[0110] Set up the AS 50 schedule.

[0111] Injection volume is 5 μL for all Samples, injection type is "Full", cut volume is 10 μL , syringe speed is 3, all Samples and standards are of Sample Type "Sample". Weight and Int. Std. values are all set equal to 1.

[0112] Run the five standards at the beginning of the run in the following order:

STANDARD A1 DATE

STANDARD B1 DATE

STANDARD C1 DATE

STANDARD D1 DATE

STANDARD E1 DATE

[0113] After the last Sample is run, run the mid-level standard again as a continuing calibration verification

[0114] Run the control Sample at any Sample spot between the beginning and ending standard runs.

[0115] Run the Samples.

CALCULATIONS

Calculations for Weight Percent of the Pulp Sugars

[0116]

$$\text{Normalized area for sugar} = \frac{(\text{Area sugar}) \times (\mu\text{g/mL fucose})}{(\text{Area Fucose})}$$

IS Corrected sugar amount

$$(\mu\text{g/mL}) = \frac{((\text{Normalized area for sugar}) - (\text{intercept}))}{(\text{slope})}$$

-continued

$$\text{Monomer Sugar Weight \%} = \frac{\text{IS} - \text{Corrected sugar amt } (\mu\text{g/mL})}{\text{Sample wt. (mg)}} * 20$$

Example for arabinose:

Monomer Sugar Weight % =

$$\frac{0.15 \mu\text{g/mL arabinose}}{70.71 \text{ mg arabinose}} * 20 = 0.043\%$$

Polymer Weight %=(Weight % of Sample sugar)*(0.88)

Example for arabinan:

Polymer Sugar Weight %=(0.043 wt %)*(0.88)=0.038 Weight

[0117] Note: Xylose and arabinose amounts are corrected by 88% and fucose, galactose, rhamnose, glucose, and mannose are corrected by 90%.

Report results as percent sugars on an oven-dried basis.

Growth Medium Study Procedure

1. Phosphate Buffer Solution

[0118] a. Stock PBS (1 liter):

Na ₂ HPO ₄	12.36 grams
NaH ₂ PO ₄	1.80 grams
NaCl	85.00 grams
H ₂ O	1.0 LITER (Makes stock solution for 6 Samples)

[0119] b. Dilute 9:1 for Working PBS Solution. (Do this into individual, screw-top flasks and sterilize). Need 7,200 mls of working solution for 4 Samples; 9,000 mls for 6 Samples.

[0120] c. Adjust pH to 7.0-7.2, using pH meter probe dipped into working solution; titrate with Dilute HCl (1 or 2N).

2. Minimum Growth Media (MGM) to add to PBS:

[0121] a. Make 100 mls each of 1% and 10% peptone, autoclave.

[0122] b. Make 100 mls 10% (NH₄)₂SO₄ solution, filter sterilize.

[0123] c. Obtain BME vitamins—100x, from Sigma B-6891.

[0124] d. Make 100 mls of Mineral Salts Medium (MSM) Sterilize.

1.	MgSO ₄	5.0 grams
2.	ZnSO ₄	0.01 gram
3.	FeSO ₄	0.05 gram
4.	MnSO ₄	0.01 gram
5.	Con HCl	1.5 ml
6.	H ₂ O	100 ml

3. Culture Organism Prep:

[0125] *S. aureus*: Prep Culti-Loop in 1.0 ml sterile TSB for 10 min.

[0126] ATCC 6538 Streak onto TSA slant and grow for 24 hrs at 35 C

[0127] *E. coli*: Prep Culti-Loop in 1.0 ml sterile TSB for 10 min.

[0128] ATCC 8739 Streak to TSA slant and grow for 24 hours @ 35 C

[0129] *C. albicans*: Prep Culti-Loop in 1.0 ml of sterile dilute PBS

[0130] ATCC 10231 Solution for 10 min. Streak onto SDA and grow for 24 hours @ 35 C

4. Other media:

[0131] SDA: 400 ml, Sabourand Dextrose Agar: Prep slants and flasks.

[0132] TSA: 1,800 ml Trypticase Soy Agar: Prep Slats and flasks.

5. Dilute culture:

[0133] a. Prepare 9.0 ml dilute PBS tubes. Remove (wash) each culture organism from slants and add to PBS tubes.

[0134] Adjust so the turbidity of the culture matches a 0.5 McFarland solution (In BAM Media Pages). This is the stock culture solution. (*S. aureus* & *E. coli*, *C. albicans*.)

[0135] b. For *S. aureus* and *E. coli*, make 1:100 dilution of stock culture solution, and add 1.0 ml of the diluted solution to 1.0 liter of MGM. (about 103 final population)

[0136] c. For *C. albicans* add 1.0 ml stock culture solution to 1.0 liter of MGM. (about 103 final population)

6. Specialized MGM solutions for each Organism:

[0137] a. *S. aureus*: 94.4 ml stock PBS+849.6 DI water; adjust pH & sterilize. (or 994 ml pre-diluted PBS Stock solution). (2X+2)200=2000 ml for 4.

[0138] ADD: 1.0 ml MSM

[0139] 50 ml BME vitamins.

[0140] 5 ml 1% Peptone.

[0141] 1.0 ml diluted Bacterial Culture (pre-made)

[0142] b. *E. coli*: 94.9 ml stock PBS+854.1 ml DI water; adjust pH & sterilize. (or 949 ml pre-diluted PBS Stock solution). (2X+2)200=2000 ml for 4.

[0143] ADD: 1.0 ml MSM

[0144] 50 ml BME vitamins.

[0145] 1.0 ml diluted Bacterial Culture (pre-made)

[0146] c. *C. albicans*: 92.9 ml stock PBS+836.1 ml DI water; adjust pH & sterilize. (or 929 ml pre-diluted PBS Stock solution). (2X+2)200=2000 ml for 4.

[0147] ADD: 1.0 ml MSM

[0148] 50 ml BME vitamins.

[0149] 20 ml 10% Peptone.

[0150] 10 ml 10% (NH₄)₂SO₄

[0151] 1.0 ml of Undiluted Bacterial Culture (pre-made)

7. Product to test:

[0152] a. Prep enough sterile 250 Plastic Bottles to handle 2 repetitions of each organism plus pos. and neg. control. (45 flasks are needed to test 6 products.)

[0153] B. Label all Plastic Bottles with organism (*Staph*, *E. coli*, *C. alb.*), plus Sequential Number (W-1, W-2 . . . , W-45).

[0154] c. Add 2.0 gm of each product to be tested to each Plastic Bottle (or a sterile blender if Sample is not soft), then add 200 ml of inoculated MGM. Incubate, with shaking (in wire milk case—attached to shaker water-bath—plus one or two plastic cases) for 4 and 24 hours at 37 C Run duplicate tests. Sample for micro at time zero (blank and first Weyerhaeuser Sample), at time 4 hours, and at time 24 hours.

[0155] d. Repeat item #3 above for each organism and each product.

- [0156] e. Run two bottles for each organism containing 200 ml of inoculated MGM, without Weyerhaeuser Sample. These are Blanks.
- [0157] f. Run positive controls, TSB in place of Sample (see below) for each organism type. 7
8. Incubation and counting.
- [0158] a. After prepping all bottles, microbially plate each "Blank" control and product bottle on TSB or SDA depending on organism. Time Zero.
- [0159] b. Incubate all culture and product inoculated flasks for 4 and 24 hours at 37 C on a shaker. Use milk case attached to shaker; in room-incubator adjusted to 37 C
- [0160] c. After 4 and 24 hours, remove aliquot from all flasks and plate for CFU present (inc. "no-product" controls). USE TSA: for *S. aureus* and *E. coli*, (Incubate plates at 35 C-48 hours) USE SDA: for *C. albicans*. (Incubate plates at 20 C-5 days)
- [0161] d. Prep dilutions expecting 10^3 to 10^4 organisms on tested product plates and blanks. Positive controls will be higher.
9. Positive Controls:
- [0162] Prepare one flasks with ~100 ml of modified MGM solution for each organism (three flasks total) and incubate at 37 C for 4 and 24 hours.
- [0163] a. *S. aureus*: 100 ml of TSB
- [0164] 0.1 ml of MSM
- [0165] 5.0 ml of BME Vitamins.
- [0166] 0.5 ml of 1% Peptone.
- [0167] 0.1 ml of diluted *Staph* Bacterial Culture
- [0168] b. *E. coli*: 100 ml of TSB
- [0169] 0.1 ml of MSM
- [0170] 5.0 ml of BME vitamins.
- [0171] 0.1 ml of diluted *E. coli* Bacterial Culture
- [0172] c. *C. albicans*: 100 ml of TSB
- [0173] 0.1 ml of MSM
- [0174] 5.0 ml of BME vitamins.
- [0175] 2.0 ml of 10% peptone
- [0176] 1.0 ml of $(\text{NH}_4)_2\text{SO}_4$
- [0177] 0.1 ml of Undiluted *C. albicans* Bacterial Culture
- [0178] d. Inoculate *S. aureus* and *E. coli*+ controls with 0.1
- [0179] ml of $1/100$ dilution of stock culture.
- [0180] e. Inoculate *C. albicans*+ control with 0.1 ml of stock culture solution.

What is claimed is:

1. Meltblown lyocell fibers comprising at least one antimicrobial agent, said antimicrobial agent uniformly distributed throughout the cross section of said fiber, said fibers further comprising four percent to eighteen by weight hemicellulose in said fibers and, an ISO brightness of at least 35.
2. The fibers of claim 1 wherein said antimicrobial agent is an inorganic compound.
3. The fibers of claim 2 wherein the antimicrobial agent is selected from the group consisting of compounds containing one or more of copper, silver, zinc, potassium, magnesium, calcium or combinations thereof.
4. The fibers of claim 3 wherein the antimicrobial agent is a compound containing zinc.
5. The fibers of claim 3 wherein the antimicrobial agent is a compound containing silver.
6. The fibers of claim 3 wherein the antimicrobial agent is a compound containing calcium.
7. The fibers of claim 1 wherein said antimicrobial agent is an organic compound.
8. The fibers of claim 7 wherein said antimicrobial agent is an organic compound containing silver.
9. The fibers of claim 8 wherein the fibers contain from about 5 to about 1000 ppm silver.
10. The fibers of claim 2 wherein the fibers contain from about 0.1 percent to about 40 percent by weight of the antimicrobial agent.
11. The fibers of claim 1 wherein the fibers contain from about 10 percent to about 25 percent by weight of the antimicrobial agent.
12. The fibers of claim 1 wherein the fibers contain from about 15 percent to about 20 percent by weight of the antimicrobial agent.
13. The fibers of claim 1 wherein the birefringence is at least 0.020.
14. The fibers of claim 1 wherein the fiber diameter is from about 2 to about 50 microns.
15. The fibers of claim 1 wherein the fiber diameter is from about 5 to about 35 microns.
15. The fibers of claim 1 wherein the fiber diameter is from about 10 to about 20 microns.
16. The fibers of claim 1 wherein the reduction in *E. Coli* colony forming units is at least ninety five percent relative to a control at twenty four hours.
17. The fibers of claim 1 wherein the reduction in *C. Albicans* colony forming units is at least ninety five percent relative to a control at four hours.

* * * * *