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A B O G A D O S

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SUPERINTENDENCIA DE INDUSTRIA Y COMERCIO
DIVISIÓN DE NUEVAS CREACIONES
E. S. D.

Re: Expediente No. 00016906
Solicitud de Patente en nombre de
HUMATRO CORPORATION

Yo, JIMENA ESCOBAR URIBE, identificada como aparece al pie de mi firma, domiciliada en Santafé de Bogotá, en la Carrera 11 A No. 94-76 Of. 305, obrando como apoderada de la sociedad HUMATRO CORPORATION solicitante de la patente de la referencia, anexo al presente memorial el siguiente documento:

Copia certificada de la solicitud de Patente Número 09/264,401 presentada en los Estados Unidos de América el día 8 de marzo de 1999, con la debida traducción de lo pertinente, incluyendo el capítulo reivindicatorio.

Señor Superintendente,

JIMENA ESCOBAR URIBE
C.C.39.779.452 de usaquén
T.P. 68228
Cód. 5376

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ESTADOS UNIDOS DE AMERICA
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DEPARTAMENTO DE COMERCIO DE LOS ESTADOS UNIDOS
Oficina de Patentes y Marcas de los Estados Unidos

Marzo 28, 2000

La presente es para certificar que el anexo es una copia fiel de los registros de la Oficina de Patentes y Marcas de los Estados Unidos, de los documentos de la solicitud de patente identificada más adelante, que cumplieron con los requerimientos para concederse una fecha de presentación de acuerdo con 35 USC 111.

Número de Solicitud: 09/264/401

Fecha de Presentación: Marzo 08,1999

Por la autoridad del
Comisionado de Patentes y Marcas

(fdo) P. SWAIN
Oficial Certificador
Sello oficial de la Oficina de Patentes y Marcas de los Estados Unidos de América.

Reivindicaciones:

1. Una estructura flexible absorbente que comprende fibras de almidón pseudo-termoplásticas.

2. La estructura de la reivindicación 1, en donde la estructura tiene una densidad aparente que va desde aproximadamente 0.02 gr/cm³ hasta aproximadamente 0.20 gr/cm³ .

3. La estructura de la reivindicación 2, en donde la estructura tiene una densidad aparente que va desde aproximadamente 0.04 gr/cm³ hasta aproximadamente 0.15 gr/cm³ .

4. La estructura de la reivindicación 3, en donde la estructura tiene una densidad aparente que va desde aproximadamente 0.04 gr/cm³ hasta aproximadamente 0.12 gr/cm³ .

5. La estructura de la reivindicación 1, en donde las fibras de almidón tienen un tamaño que va desde aproximadamente 0.01 dtex hasta aproximadamente 135 dtex.

6. La estructura de la reivindicación 5, en donde las fibras de almidón tienen un tamaño que va desde aproximadamente 0.02 dtex hasta aproximadamente 30 dtex.

7. La estructura de la reivindicación 6, en donde las fibras de almidón tienen un tamaño que va desde aproximadamente 0.02 dtex hasta aproximadamente 5 dtex.

8. La estructura de la reivindicación 1, en donde la estructura tiene una resistencia a la tracción seca media geométrica que va desde aproximadamente 10 gr/cm hasta aproximadamente 1200 gr/cm.

9. La estructura de la reivindicación 8, en donde la estructura tiene una resistencia a la tracción seca media geométrica que va desde aproximadamente 30 gr/cm hasta aproximadamente 600 gr/cm.

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10. La estructura de la reivindicación 9, en donde la estructura tiene una resistencia a la tracción seca media geométrica que va desde aproximadamente 40 gr/cm hasta aproximadamente 475 gr/cm.

11. La estructura de la reivindicación 1, en donde la estructura tiene una resistencia a la tracción húmeda media geométrica inicial que va desde aproximadamente 2 gr/cm hasta aproximadamente 400 gr/cm.

12. La estructura de la reivindicación 11, en donde la estructura tiene una resistencia a la tracción húmeda media geométrica inicial que va desde aproximadamente 2 gr/cm hasta aproximadamente 200 gr/cm.

13. La estructura de la reivindicación 1, en donde la estructura tiene una resistencia a la tracción seca media geométrica por peso básico básico unitario la cual va desde aproximadamente 200 mt hasta aproximadamente 2500 mt.

14. La estructura de la reivindicación 13, en donde la estructura tiene una resistencia a la tracción seca media geométrica por peso básico unitario la cual va desde aproximadamente 200 mt hasta aproximadamente 1500 mt.

15. La estructura de la reivindicación 14, en donde la estructura tiene una resistencia a la tracción seca media geométrica por peso básico unitario la cual va desde aproximadamente 200 mt hasta aproximadamente 1200 mt.

16. La estructura de la reivindicación 1, en donde la estructura tiene una resistencia a la tracción húmeda media geométrica por peso básico unitario la cual va desde aproximadamente 10 mt hasta aproximadamente 1500 mt.

17. La estructura de la reivindicación 16, en donde la estructura tiene una resistencia a la tracción húmeda media geo-

métrica por peso básico unitario la cual va desde aproximadamente 10 mt hasta aproximadamente 1000 mt.

18. La estructura de la reivindicación 17, en donde la estructura tiene una resistencia a tracción húmeda media geométrica por peso básico unitario la cual va desde aproximadamente 10 mt hasta aproximadamente 500 mt.

19. La estructura de la reivindicación 1, en donde la estructura tiene una resistencia a la tracción húmeda decaída media geométrica que va desde aproximadamente 0 gr/cm hasta aproximadamente 20 gr/cm.

20. La estructura de la reivindicación 19, en donde la estructura tiene una resistencia a la tracción húmeda decaída media geométrica que va desde aproximadamente 0 gr/cm hasta aproximadamente 10 gr/cm.

21. La estructura de la reivindicación 1, en donde la estructura tiene un peso básico que va desde aproximadamente 10 gr/mt^2 hasta aproximadamente 450 gr/mt^2.

22. La estructura de la reivindicación 21, en donde la estructura tiene un peso básico que va desde aproximadamente 12 gr/mt^2 hasta aproximadamente 150 gr/mt^2.

23. La estructura de la reivindicación 1, la cual comprende además un plastificante seleccionado entre el grupo constituido por sorbitol, monosacáridos, disacáridos, glicerol, polivinilo alcohol y polietileno glicol.

24. La estructura de la reivindicación 23, en donde dicho plastificante comprende desde aproximadamente 5% de peso hasta aproximadamente 70% de peso con respecto al peso total de la estructura.

25. La estructura de la reivindicación 24, en donde el mencionado plastificante comprende desde aproximadamente 15% de

peso hasta aproximadamente 60% de peso con respecto al peso total de la estructura.

26. La estructura de la reivindicación 1, la cual comprende además agentes reticulantes seleccionados entre el grupo constituido por resinas de poliamida-epiclorohidrina, resinas de urea-formaldehido, resinas de poliacrilamida glioxilatada, resinas de melamina formaldehido, resinas de polietilenimina, resina Caldas 10, resina CoBond 1000.

27. La estructura de la reivindicación 26, en donde los mencionados agentes reticulantes están presentes en cantidades que van desde aproximadamente 0,1% de peso hasta aproximadamente 10% de peso con respecto al peso total de la estructura.

28. La estructura de la reivindicación 27, en donde los mencionados agentes reticulantes están presentes en cantidades que van desde aproximadamente 0,1% de peso hasta 3% de peso con respecto al peso total de la estructura.

29. La estructura de la reivindicación 1, en donde la estructura tiene una absorbencia que va desde aproximadamente 1 gr de agua/gr de estructura seca hasta aproximadamente 15 gr de agua/gr de estructura seca.

30. La estructura de la reivindicación 29, en donde la estructura tiene una absorbencia que va desde aproximadamente 2 gr de agua/gr de estructura seca hasta aproximadamente 14 gr de agua/gr de substancia seca.

31. La estructura de la reivindicación 30, en donde la estructura tiene una absorbencia que va desde aproximadamente 3 gr de agua/gr de estructura seca hasta aproximadamente 13 gr de agua/gr de estructura seca.

32. La estructura de la reivindicación 1, en donde la estructura tiene una flexibilidad total que va desde aproximadamente 1.0 gr/cm hasta aproximadamente 75 gr/cm.

33. La estructura de la reivindicación 32, en donde la estructura tiene una flexibilidad total que va desde aproximadamente 2.0 gr/cm hasta aproximadamente 50 gr/cm.

34. La estructura de la reivindicación 33, en donde la estructura tiene una flexibilidad total que va desde aproximadamente 2.0 gr/cm hasta aproximadamente 35 gr/cm.

35. Una estructura que comprende fibras de almidón pseudotermoplásticas tiene una Tg de por lo menos aproximadamente -30°C y la estructura tiene una resistencia a la tracción húmeda decaída media geométrica que va desde aproximadamente 0 gr/cm hasta aproximadamente 20 gr/cm.

36. La estructura de la reivindicación 35, en donde las mencionadas fibras tienen una Tg que va desde aproximadamente -30°C hasta aproximadamente 150°C.

37. La estructura de la reivindicación 36, en donde las mencionadas fibras tienen una Tg que va desde aproximadamente -30°C hasta aproximadamente 100°C.

38. La estructura de la reivindicación 37, en donde la mencionadas fibras tienen una Tg que va desde aproximadamente -30°C hasta aproximadamente 25°C.

39. La estructura de la reivindicación 35, en donde dicha estructura tiene una resistencia a la tracción húmeda decaída media geométrica que va desde aproximadamente 0 gr/cm hasta aproximadamente 10 gr/cm.

40. La estructura de la reivindicación 36, en donde dicha estructura tiene una resistencia a la tracción húmeda decaída media geométrica que va desde aproximadamente 0 gr/cm hasta aproximadamente 10 gr/cm.

41. La estructura de la reivindicación 35, en donde la mencionadas fibras tienen un tamaño que va desde aproximadamente 0.01 dtex hasta aproximadamente 135 dtex.

42. La estructura de la reivindicación 41, en donde las mencionadas fibras tienen un tamaño que va desde aproximadamente 0.02 dtex hasta aproximadamente 30 dtex.

43. La estructura de la reivindicación 42, en donde las mencionadas fibras tienen un tamaño que va desde aproximadamente 0.02 dtex hasta aproximadamente 5 dtex.

44. Una estructura absorbente que comprende uno o más capas, en la cual por lo menos una capa comprende fibras de almidón pseudo-termoplásticas, y en la cual por lo menos una capa tiene un peso básico que va desde aproximadamente 10 gr/mt^2 hasta aproximadamente 100 gr/mt^2, una resistencia a la tracción seca media geométrica que va desde aproximadamente 40 gr/cm hasta aproximadamente 475 gr/cm, una densidad aparente que va desde aproximadamente 0.04 gr/cm^3 hasta aproximadamente 0.12 gr/cm^3, y una resistencia a la tracción húmeda media geométrica inicial que va desde aproximadamente 2 gr/cm hasta aproximadamente 200 gr/cm.

45. La estructura de la reivindicación 44, en donde por lo menos una capa tiene una resistencia a la tracción húmeda decaída media geométrica que va desde aproximadamente 0 gr/cm hasta aproximadamente 20 gr/cm.

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March 28, 2000

THIS IS TO CERTIFY THAT ANNEXED HERETO IS A TRUE COPY FROM THE RECORDS OF THE UNITED STATES PATENT AND TRADEMARK OFFICE OF THOSE PAPERS OF THE BELOW IDENTIFIED PATENT APPLICATION THAT MET THE REQUIREMENTS TO BE GRANTED A FILING DATE UNDER 35 USC 111.

APPLICATION NUMBER: *09/264,401*

FILING DATE: *March 08, 1999*

By Authority of the
COMMISSIONER OF PATENTS AND TRADEMARKS

P. SWAIN
Certifying Officer

Please type a plus sign (+) inside this box → [+]

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UTILITY PATENT APPLICATION TRANSMITTAL

(Only for new nonprovisional applications under 37 CFR 1.53(b))

Attorney Docket No.	7456
First Inventor or Application Identifier	Larry Neil Mackey
Title	Absorbent, Flexible, Structure Comprising Starch Fibers
Express Mail Label No.	EE829903065US

APPLICATION ELEMENTS

See MPEP Chapter 600 concerning utility patent application contents.

ADDRESS TO: Assistant Commissioner for Patents
Box Patent Application
Washington, D.C. 20231

1. [X] * Fee Transmittal Form (e.g., PTO/SB/17)
(Submit an original, and a duplicate for fee processing)
2. [X] Specification Total Pages [32]
(preferred arrangement set forth below)
- Descriptive Title of the Invention
- Cross References to Related Applications
- Statement Regarding Fed sponsored R&D
- Reference to Microfiche Appendix
- Background of the Invention
- Brief Summary of the Invention
- Brief Description of the Drawings *(if filed)*
- Detailed Description
- Claim(s)
- Abstract of the Disclosure

3. [X] Drawing(s) (35 USC 113) Total Sheets [5]
4. Oath or Declaration Total pages [3]
a. [X] Newly executed (original or copy)
b. [] Copy from a prior application (37 CFR 1.63(d))
(for continuation/divisional with Box 16 completed)
i. [] DELETION OF INVENTORS
Signed statement attached deleting inventor(s) named in the prior application, see 37 CFR §§1.63(d)(2) and 1.33(b).

5. [] Microfiche Computer Program (Appendix)
6. Nucleotide and/or Amino Acid Sequence Submission
(if applicable, all necessary)
a. [] Computer Readable copy
b. [] Paper Copy *(identical to computer copy)*
c. [] Statement verifying identity of above copies

ACCOMPANYING APPLICATION PARTS

7. [] Assignment Papers (cover sheet & document(s))
8. [] 37 CFR 3.73(b) Statement [] Power of Attorney
(when there is an assignee)
9. [] English Translation Document *(if applicable)*
10. [] Information Disclosure Statement (IDS)/PTO-1449 [] Copies of IDS Citations
11. [] Preliminary Amendment
12. [X] Return Receipt Postcard (MPEP 503)
(Should be specifically itemized)
13. [] *Small Entity Statement(s) [] Statement filed in prior application Status still proper and desired
14. [] Certified Copy of Priority Document(s)
(if foreign priority is claimed)
15. [] Other: .....
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NOTE FOR ITEMS 1 & 13: IN ORDER TO BE ENTITLED TO PAY SMALL ENTITY FEES, A SMALL ENTITY STATEMENT IS REQUIRED (37. C.F.R. §1.27), EXCEPT IF ONE FILED IN A PRIOR APPLICATION IS RELIED UPON (37 C.F.R.§1.28).

16. If a CONTINUING APPLICATION, check appropriate box and supply the requisite information below and in the preliminary amendment:
[] Continuation [] Divisional [] Continuation-in-part (CIP) of prior application No. /
Prior application information: Examiner: _____ Group/Art Unit: _____
For CONTINUATION or DIVISIONAL only: The entire disclosure of the prior application, from which a copy of the oath or declaration is supplied under Box 4b, is considered a part of the disclosure of the accompanying continuation or divisional application and is hereby incorporated by reference. The incorporation can only be relied upon when a portion has been inadvertently omitted from the submitted application parts.

17. CORRESPONDENCE ADDRESS

[ ] Customer Number or Bar Code Label (Insert Customer No. or Attach bar code label here) or [X] Correspondence address below

NAME	Jay A. Krebs				
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CITY	Cincinnati	STATE	Ohio	ZIP CODE	45224
COUNTRY	U.S.A.	TELEPHONE	513/634-1151	FAX	513/634-3612

Name (Print/Type)	Jay A. Krebs	Registration No. (Attorney/Agent)	41,914
Signature		Date 3-8-99	March 8, 1999

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FEE TRANSMITTAL FORM

CLAIMS	(1) FOR	(2) NUMBER FILED	(3) NUMBER EXTRA	(4) RATE	(5) CALCULATIONS
	TOTAL CLAIMS (37 CFR 1.16 (c))	45 - 20	25	x $18.00 =	$450.00
	INDEPENDENT CLAIMS (37 CFR 1.16 (c))	3 - 3	0	x $78.00 =	$-0-
	MULTIPLE DEPENDENT CLAIMS (if applicable) (37 CFR 1.16(d))			+ $270.00 =	$
				BASIC FEE (37 CFR 1.16(a))	$760.00
				Total of above Calculations -=	$1210.00
	Reduction by 50% for filing by small entity (Note 37 CFR 1.9, 1.27, 1.28).				
				TOTAL =	$1210.00

19. The Commissioner is hereby authorized to charge payment of the following fees associated with this communication or credit any overpayment to Deposit Account No. 16-2480. A duplicate copy of this sheet is enclosed.

    a. [X] Any patent application filing fees required under 37 CFR 1.16.
    b. [X] Any patent application processing fees under 37 CFR 1.17.

20. The Commissioner is hereby authorized to charge payment of the following fees during the pendency of this application or credit any overpayment to Deposit Account No. 16-2480. A duplicate copy of this sheet is enclosed.

    a. [X] Any patent application processing fees under 37 CFR 1.17.
    b. [ ] The issue fee set in 37 CFR 1.18 at or before mailing of the Notice of Allowance, pursuent to 37 CFR 1.311(b).
    c. [X] Any filing fees under 37 CFR 1.16 for presentation of extra claims.

21. The total number of duplicate copies enclosed is 5. The Commissioner is hereby authorized to make any additional copies of this sheet needed to accomplish the purposes provided for herein and to charge any fee for such copies to Deposit Account No. 16-2480.

11. SIGNATURE OF APPLICANT, ATTORNEY, OR AGENT REQUIRED	
NAME	Jay A. Krebs
SIGNATURE	Jay Krebs
DATE	March 8, 1999

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Date of Deposit March 8, 1999

I hereby certify that this paper/fee is being deposited with the United States Postal Service "Express Mail Post Office to Addressee" service under 37 CFR 1.10 on the date indicated above and is addressed to Assistant Commissioner for Patents, Washington, D.C. 20231

Jay A. Krebs — 41,914
Attorney/Agent mailing application — Reg No.

Jay Krebs
Signature of Attorney/Agent mailing application

Case 7456

ABSORBENT, FLEXIBLE, STRUCTURE COMPRISING STARCH FIBERS

Larry Neil Mackey
James Daniel Miller II
Mark Ryan Richards
John Gerhard Michael
David William Cabell
Valerie Ann Bailey

FIELD OF THE INVENTION

The present invention relates to pseudo-thermoplastic starch extruded in the form of fibers. Starch may be extruded and either meltblown or spunbonded to form fibrous low density structures.

BACKGROUND OF THE INVENTION

Cellulosic fibrous webs such as paper are well known in the art. Low density fibrous webs are in common use today for paper towels, toilet tissue, facial tissue, napkins, wet wipes, and the like. The large demand for such paper products has created a demand for improved versions of the products and the methods of their manufacture. In order to meet such demands, papermaking manufacturers must balance the costs of machinery and resources with the total cost of delivering the products to the consumer.

For conventional papermaking operations wood cellulosic fibers are repulped, beaten or refined to achieve a level of fiber hydration in order to form an aqueous pulp slurry. Processes for the making of paper products for use in tissue,

toweling, and sanitary products generally involve the preparation of the aqueous slurry and then subsequently removing the water from the slurry while contemporaneously rearranging the fibers therein to form a paper web. Subsequent to dewatering, the web is processed into a dry roll or sheet form and eventually converted into a consumer package. Various types of machinery must be employed to assist in the dewatering process and converting operations requiring a significant investment in capital.

Another aspect of the conventional papermaking operation involves the incorporation of additives into the pulp in order to achieve specific end properties. For instance, additives such as strength resins, debonding surfactants, softening agents, pigments, lattices, synthetic microspheres, fire-retardants, dyes, perfumes, etc., are often employed in the manufacture of paper. The efficient retention of these additives at the wet end of a papermaking process presents difficulty to the manufacturer since that portion which is not retained creates not only an economic loss but also significant pollution problems if it becomes part of a plant effluent. Additives can also be added to the paper web subsequent to dewatering via coating or saturation processes commonly known in the art. These processes usually require that excess heating energy be consumed to redry the paper after coating. Moreover, in some instances, the coating systems are required to be solvent based which increases capital costs and requires recovery of volatile materials to meet regulatory requirements.

Various natural fibers other than cellulose as well as a variety of synthetic fibers have been employed in making paper, however, all these replacements have failed to provide a commercially acceptable substitute for cellulose due to their high cost, poor bonding properties, chemical incompatibilities, and handling difficulties in papermaking systems. Starch fibers have been suggested as a substitute for cellulose in various aspects of the papermaking process, however, commercial attempts to use such fibers have been unsuccessful. As a result, paper products are still being manufactured almost exclusively from wood base cellulosic ingredients.

Extrusion is a mature technology for production of non-woven webs and films from synthetic thermoplastic polymers. U.S. Pat. Nos. 3,802,817 issued to

Matsuki et al. 1974 and 4,064,605 issued to Akiyama, et al. Dec 27, 1977 provide examples of extrusion and spinbonding techniques used in the formation of non-woven fabrics. Synthetic thermoplastic polymers are not suitable materials for tissue, towel, facial products because they are expensive and most are neither biodegradable, flushable, nor absorbent. However, naturally occurring starch is a relatively inexpensive biodegradable polymer which can be made to be thermoplastic in the presence of a solvent such as water.

In the foods industry, starch is extruded in a thermoplastic form to make cereals and snack foods. Also, starch extrusion has been used in the packaging industry to produce low-density, foam starch structures to compete with non-biodegradable polystyrene packaging materials.

U.S. Patent No. 5,316,578 issued to Buehler et al. May 31, 1994 discloses a process for extruding a chemically modified starch in combination with synthetic thermoplastic polymers to produce a thermoplastic melt which can be pressed either into an injection mold or extruded into the open where it can be optionally further shaped. U.S. Patent No. 5,516,815 issued to Buehler et al. May 14, 1996 discloses melt spun fibers comprising chemically modified starch and synthetic thermoplastic polymers. Although U.S. Patent No. 5,516,815 suggests that the melt spun fibers can be used for biodegradable fabrics and nonwovens, such fabrics and nonwovens made of fibers which include synthetic thermoplastic polymers are not flushable. In addition, U.S. Patent No. 5,516,815 does not disclose how to produce low density, flexible and absorbent structures composed of starch fibers. Furthermore, the costs associated with the chemically modified starch and the synthetic thermoplastic polymers limits the economic benefit desired.

The present invention provides a low density flexible structure comprising starch fibers utilizing extrusion and fiber spinning techniques. Particularly, the present invention provides a low density flexible structure comprising starch fibers, wherein the structure has improved tensile strength, softness, and absorbency properties relative to cellulosic pulp fiber structures while maintaining biodegradability and flushability.

SUMMARY OF THE INVENTION

The present invention provides an absorbent flexible structure comprising starch fibers. Naturally occurring starch in the presence of water, plasticizers and other additives is melt extruded and spun into fibers to form an absorbent flexible structure having an apparent density ranging from 0.02 g/cm^3 to 0.20 g/cm^3 and a basis weight ranging from 10 g/m^2 to 450 g/m^2.

The starch fibers making up the structure can have a size ranging from about 0.01 decitex to about 135 decitex. In a preferred embodiment the fibers can have a size ranging from about 0.02 decitex to about 30 decitex, and most preferably ranging from about 0.02 to about 5 decitex. In addition, fibers making up the structure of the present invention can have a glass transition temperature ranging from about -30 °C to about 150 °C, more preferably from about -30 °C to about 100 °C, and most preferably from about -30 °C to about 25 °C.

Exemplary physical properties of the flexible structure of the present invention include dry tensile strength and wet tensile strength. The dry tensile strength of the structure, which is measured as a geometric mean tensile strength, can range from about 10 g/cm to about 1200 g/cm, more preferably from about 30 g/cm to about 600 g/cm, and most preferably from about 40 g/cm to about 475 g/cm. The wet tensile strength of the structure, which is also measured as a geometric mean tensile strength, can range from about 2 g/cm to about 400 g/cm, and more preferably from about 2 g/cm to about 200 g/cm.

BRIEF DESCRIPTION OF THE DRAWINGS

These and other features, aspects and advantages of the present invention will become better understood with regard to the following description, appended claims, and accompanying drawings where:

Figure 1a illustrates a torque rheometer assembly used to produce starch fibers.

Figure 1b illustrates the twin screw elements attached to the drive unit and disposed within the barrel of the torque rheometer assembly illustrated in Figure 1a.

Figure 2a illustrates a vented twin screw extruder assembly.

Figure 2b illustrates the screw and mixing element configuration for the extrusion assembly depicted in Figure 2a.

Figure 3a illustrates a non-vented twin screw extruder assembly.

Figure 3b illustrates the screw and mixing element configuration for the extrusion assembly depicted in Figure 3a.

Figure 4 illustrates a spinneret and a drawing unit used for pseudo-thermoplastic starch melt fiber spinning.

Figure 5 illustrates the sample rack and cover used for determining absorbency of the starch fiber structures.

Figure 6 illustrates the cross section of the frames for the sample rack and cover illustrated in Figure 5.

DETAILED DESCRIPTION OF THE INVENTION

Definitions

As used herein, the following terms have the following meanings:

Pseudo-thermoplastic composition is intended to denote materials which by the influence of elevated temperatures may be softened to such a degree that they can be brought into a flowable state, and in this condition may be shaped as desired. Pseudo-thermoplastic materials may be formed under simultaneous influence of heat and pressure. Pseudo-thermoplastic compositions differ from thermoplastic compositions in that the softening or liquefying of the pseudo-thermoplastic is caused by softeners or solvents present without which it would be impossible to bring them by any temperature or pressure into a soft or flowable condition necessary for shaping since pseudo thermoplastics do not melt as such. The influence of water content on the glass transition temperature and melting temperature of starch can be measured by differential scanning calorimetery as described by Zeleznak and Hoseny "Cereal Chemistry", Vol. 64, No. 2, pp. 121-124, 1987.

Pseudo-thermoplastic melt is a pseudo-thermoplastic material in a flowable state.

Glass transition temperature, T_g, is the temperature at which the material changes from a viscous or rubbery condition to a hard and relatively brittle condition.

Basis weight is the weight (in grams) per unit area (in square meters) of a sample reported in grams per square meter.

Caliper is the macroscopic thickness of a sample measured as described below.

Apparent density is the basis weight of the sample divided by the caliper with appropriate unit conversions incorporated therein. Apparent density used herein has the units of grams / centimeters cubed (g/cm^3).

Machine direction, designated MD, is the direction parallel to the flow of the starch fiber structure through the product manufacturing equipment.

Cross machine direction, designated CD, is the direction perpendicular to the machine direction in the same plane of the starch fiber structure.

Geometric Mean Dry Tensile Strength (GMDT) is the square root of the product of the machine and cross-machine dry tensile strengths (in grams/cm). The value of GMDT is reported in grams/cm.

Geometric mean wet tensile strength (GMWT) is the square root of the product of the machine and cross-machine wet tensile strengths (in grams/cm). The value of GMWT is reported in grams/cm.

Structure is an arrangement of one or more parts forming a substance or body.

Absorbency is the ability of a material to take up fluids by various means including capillary, osmotic, solvent or chemical action and retain such fluids.

Flexibility indicates the capability of being deformed under a given load without being broken and with or without returning of itself to its former shape.

A fiber is a slender object having a major axis which is very long compared to the two orthogonal axes and having an aspect ratio of at least 4/1, preferably at least 10/1.

Decitex ,dtex, is a unit of measure for a fiber expressed in $\frac{\text{grams}}{10{,}000 \text{ meters}}$

Flushability is determined by the geometric mean decayed wet tensile strength (GMDWT) (defined below). A flushable structure has a geometric mean decayed wet tensile of less than about 20 g/cm and more preferably less than about 10 g/cm.

The specification contains a detailed description of (1) exemplary materials of the present invention, (2) exemplary processes for producing the present invention, (3) material properties of the present invention, and (4) analytical procedures for measuring properties of the present invention

(1) Exemplary Materials

For the present invention a starch polymer is mixed with water, plasticizers and other additives and melt extruded to produce fibers . Standard meltblowing or spunbonding techniques are used to produce starch fiber structures. Such structures may be absorbent and flexible. These structures may be used as substitutes for paper products such as paper towels, napkins, toilet tissues, facial tissues, place mats and wet wipes.

The starch fibers of the present invention may be useful for forming fibrous structures and also forming absorbent materials, as described above. The absorbent structures/fibrous materials comprising the starch fibers of the present invention may have from a trace amount to one hundred percent (100%) starch fibers, or a blend of starch fibers and other suitable fibers. Other suitable fibers for the blend include cellulose fibers, synthetic fibers, and a combination thereof.

Starch polymers can include any naturally occurring starch, physically modified starch or chemically modified starch. Suitable naturally occurring starches can include, but are not limited to, corn starch, potato starch, sweet potato starch, wheat starch, sago palm starch, tapioca starch, rice starch, soybean starch, arrow root starch, bracken starch, lotus starch, waxy maize starch, high amylose corn starch, and commercial amylose powder. Naturally occurring starches particularly, corn starch and wheat starch, are the preferred starch polymers of choice due to their economy and availability.

Physically modified starch is formed by changing the dimensional structure. Physically modified starch can include α starch, fractionated starch, moisture and heat treated starch.

Chemically modified starch may be formed by reaction of its OH groups with alkylene oxides, and other ether-, ester-, urethane-, carbamate-, or isocyanate-forming substances. Hydroxyalkyl, acetyl, or carbamate starches or mixtures thereof are preferred chemically modified starches. The degree of substitution of the chemically modified starch is 0.05 to 3.0, preferably 0.05 to 0.2.

The starch desirably has a native water content of about 5% to 16% by weight. A water content of 8% to 12% is particularly preferred. The amylose content of the starch is 0% to 80%, preferably 20% to 30%.

A plasticizer is typically added to the starch polymer in order to lower the glass transition temperature of the starch fibers thereby enhancing the flexibility of the fibers. In addition, the presence of the plasticizer lowers the melt viscosity which in turn facilitates the melt extrusion process. The plasticizer is advantageously an organic compound having at least one hydroxyl group, preferably a polyol. Sorbitol, mannitol, D-glucose, polyvinyl alcohol, ethylene glycol, polyethylene glycol, propylene glycol, polypropylene glycol, sucrose, fructose, glycerol and mixtures thereof have been found especially suitable. For the present invention, the preferred plasticizers include sorbitol, sucrose, and fructose in quantities ranging from about 5% by weight to about 70% by weight, more preferably from about 15% by weight to about 60% by weight, most preferably from about 30% by weight to about 40% by weight.

Other additives are typically included with the starch polymer as a processing aid and to modify physical properties such as elasticity, dry tensile strength, and wet strength of the extruded fibers. Additives are typically present in quantities ranging from 0.1% to 70% by weight on a non-volatiles basis. Preferred additives are urea, urea derivatives, cross-linking agents, emulsifiers, surfactants, lubricants, proteins and their alkali salts, biodegradable synthetic polymers, waxes, low melting synthetic thermoplastic polymers, tackifying resins, extenders, and mixtures thereof. Preferred biodegradable synthetic polymers include

polycaprolactone, polyhydroxybutyrates, polyhydroxyvalerates, polylactides, and mixtures thereof. Other preferred additives and associated properties include optical brighteners, antioxidants, flame retardants, dyes, pigments, and fillers. For the present invention, a preferred additive is urea in quantities ranging from 20% to 60% by weight.

Suitable extenders for use herein include gelatin, vegetable proteins such as sunflower protein, soybean proteins, cotton seed proteins, and water soluble polysaccharides; such as alginates, carrageenans, guar gum, agar, gum arabic and related gums, pectin, water soluble derivatives of cellulose, such as alkylcelluloses, hydroxyalkylcelluloses, carboxymethylcellulose, etc. Also, water soluble synthetic polymers, such as polyacrylic acids, polyacrylic acid esters, polyvinylacetates, polyvinylalcohols, polyvinylpyrrolidone, etc., may be used.

Lubricant compounds may further be added to improve the flow properties of the starch material during the processes used for producing the present invention. The lubricant compounds can include animal or vegetable fats, preferably in their hydrogenated form, especially those which are solid at room temperature. Additional lubricant materials include mono-glycerides and di-glycerides and phosphatides, especially lecithin. For the present invention, a preferred lubricant compound includes the mono-glyceride, glycerol mono-stearate.

Further additives including inorganic fillers such as the oxides of magnesium, aluminum, silicon, and titanium may be added as inexpensive fillers or processing aides. Additionally, inorganic salts, including alkali metal salts, alkaline earth metal salts, phosphate salts, etc., may be used as processing aides.

Other additives may be desirable depending upon the particular end use of the product contemplated. For example, in products such as toilet tissue, disposable towels, facial tissues and other similar products, wet strength is a desirable attribute. Thus, it is often desirable to add to the starch polymer cross-linking agents known in the art as "wet strength" resins.

A general dissertation on the types of wet strength resins utilized in the paper art can be found in TAPPI monograph series No. 29, Wet Strength in Paper and Paperboard, Technical Association of the Pulp and Paper Industry (New York, 1965).

The most useful wet strength resins have generally been cationic in character. Polyamide-epichlorohydrin resins are cationic polyamide amine-epichlorohydrin wet strength resins which have been found to be of particular utility. Suitable types of such resins are described in U.S. Patent Nos. 3,700,623, issued on October 24, 1972, and 3,772,076, issued on November 13, 1973, both issued to Keim and both being hereby incorporated by reference. One commercial source of a useful polyamide-epichlorohydrin resin is Hercules, Inc. of Wilmington, Delaware, which markets such resins under the mark Kymene™.

Glyoxylated polyacrylamide resins have also been found to be of utility as wet strength resins. These resins are described in U.S. Patent Nos. 3,556,932, issued on January 19, 1971, to Coscia, et al. and 3,556,933, issued on January 19, 1971, to Williams et al., both patents being incorporated herein by reference. One commercial source of glyoxylated polyacrylamide resins is Cytec Co. of Stanford, Connecticut, which markets one such resin under the mark Parez™ 631 NC.

Still other water-soluble cationic resins finding utility in this invention are urea formaldehyde and melamine formaldehyde resins. The more common functional groups of these polyfunctional resins are nitrogen containing groups such as amino groups and methylol groups attached to nitrogen. Polyethylenimine type resins may also find utility in the present invention. In addition, temporary wet strength resins such as Caldas 10 (manufactured by Japan Carlit) and CoBond 1000 (manufactured by National Starch and Chemical Company) may be used in the present invention.

For the present invention, the preferred cross-linking agent is the wet strength resin Kymene , in quantities ranging from about 0.1% by weight to about 10% by weight, more preferably from about 0.1% by weight to about 3% by weight.

(2) Exemplary Processes

Extruder Apparatus

The apparatus for carrying out the process of the invention consists of an extruder having

a. first inlet chamber containing at least one conveying element,

b. a heated receiving chamber downstream of said first chamber and containing at least one conveying element;

c. a heated destructurization chamber, downstream of said second chamber, containing kneading and retaining elements;

d. a heated degassing chamber under reduced pressure downstream of said destructurization chamber and said degassing chamber containing at least one conveying element, and

e. a heated extrusion chamber downstream of said degassing chamber being under elevated pressure and having at least one conveying element.

Furthermore, the extruder preferably has at least one delivery device for solids for process step a, a liquid metering device for process step b, a degassing fitting for process step d, and a die for process step e. A twin screw extruder having closely meshing screws which run in the same direction is preferred.

For the present invention, the starch material can have a total water content, i.e. water of hydration plus added water, in the range of about 5 to about 40%; preferably in the range of about 10 to about 20%. The starch material is heated to elevated temperatures sufficient to form a pseudo-thermoplastic melt. Such temperature is typically higher than the glass transition and/or melting temperature of the formed material. For the present invention, the glass transition temperatures are at least about minus -30°C, preferably in the range of about -30°C to about 150°C, more preferably in the range of about -30°C to about 100°C, and most preferably in the range of about -30°C to about 25°C. The melting temperature is preferably in the range of about 100°C to about 180°C. The pseudo-thermoplastic melts of the invention are polymeric fluids having a shear rate dependent viscosity, as known in the art. The viscosity decreases with increasing shear rate as well as with increasing temperature.

The starch material is heated preferably in a closed volume in the presence of a low concentration of water to convert the starch material to a pseudo-thermoplastic melt. A closed volume can be a closed vessel or the volume created by the sealing action of the feed material as happens in the screw of extrusion equipment. Pressures created in a closed vessel will include pressures due to the vapor pressure

of water as well as pressures generated due to compression of materials in the screw-barrel of the extruder.

A chain scission catalyst, which reduces the molecular weight by splitting the glycosidic bonds in the starch macromolecules resulting in a reduction of the average molecular weight of the starch, may be used to reduce the viscosity of the pseudo-thermoplastic melt. Suitable catalysts include inorganic and organic acids. Suitable inorganic acids include hydrochloric acid, sulfuric acid, nitric acid, phosphoric acid, and boric acid as well as the partial salts of polybasic acids, e.g. $NaHSO_4$ or NaH_2PO_4 etc. Suitable organic acids include formic acid, acetic acid, propionic acid, butyric acid, lactic acid, glycolic acid, oxalic acid, citric acid, tartaric acid, itaconic acid, succinic acid, and othef organic acids known in the art, including partial salts of the polybasic acids. For the present invention, the preferred catalysts are hydrochloric acid, sulfuric acid, and citric acid, including mixtures thereof.

The reduction of the molecular weight of the non-modified starch used is by a factor of 2 to 5000, preferably by a factor of 4 to 4000. The concentration of catalysts is in the range of 10^{-6} to 10^{-2} mole of catalyst per mole of anhydro-glucose unit, preferably between $0.1x10^{-3}$ to $5x10^{-3}$ mole of catalyst per mole of anhydro-glucose unit of starch.

The following examples illustrate the type of extrusion equipment and operating parameters for producing starch fibers.

Example 1

The purpose of this example is to illustrate starch fibers extruded at a particular cross section and subsequently drawn to a reduced cross section. Drawn pseudo-thermoplastic starch fibers were produced using a torque rheometer assembly 100 illustrated in Figure 1a. The torque rheometer assembly 100 includes a drive unit 110 (manufactured by Haake GmbH, model Rheocord 90), a barrel 120 partitioned into four temperature zones 122, 124, 126 and 128, a feed port 121, a single capillary die assembly 130, and a simple mandrel winder 140. Twin screw elements 160 (model TW100, from Haake GmbH), depicted in Figure 1b, are

attached to the drive unit 110 and disposed within the barrel 120. A capillary die was made to fit the die assembly 130, with an orifice diameter of 0.5 mm and a length of 5.6 mm. The mandrel rewinder 140 comprises a 3 inch core mounted to a simple DC driven 3 inch diameter shaft. The 3 inch core can achieve surface speeds from 150 to 2000 fpm.

Raw materials utilized included the following:

45% by weight Durabond A Corn Starch from National Starch
25% by weight Water
15% by weight Urea available from Aldrich Chemicals
15% by weight Sorbitol available from Aldrich Chemicals

All raw materials were mixed off-line until a slurry was formed. The slurry was then manually fed into the feed port 121 of the torque rheometer assembly 100.

The settings on the torque rheometer were as follows:

RPM	50
Barrel Temperature	110 °C
Die Temperature	105 °C
Feed Rate	1.7 grams/minute

After running the rheometer for approximately 20 minutes, the process stabilized and a single pseudo-thermoplastic starch fiber 150 exited the die 130. The single fiber 150 was manually wound around the mandrel winder 140. The winder 140 was then slowly sped up to 900 feet/minute surface speed in order to draw the fiber 150 increasing the fiber length and decreasing the cross sectional area. The diameter of the wound fiber 150 was between 70 and 90 microns.

Example 2

The purpose of this example is to illustrate how starch fibers can be arranged to form a starch fiber structure. The pseudo-thermoplastic starch fibers of Example 1 were cut into 8 mm length staple fibers. The starch staple fibers at a basis weight of 55 g/m^2 were air laid onto a papermaking forming fabric as described in U.S. Pat. No. 4,637,859 issued to Trokhan, January 20, 1987, incorporated herein by reference. The fibers were misted with water at a level of 20%, based on the weight of the fibers, and then dried at an elevated temperature to produce a bonded starch fiber structure.

Example 3 :Starch Fibers Extruded Using A Vented Twin Screw Extruder

The purpose of this example is to illustrate a vented twin screw extruder configuration, depicted in Figure 2a, used to make starch fibers for the present invention. Starch fibers are made using a an APV Baker (Peterborough, England) twin screw extruder 200, a capillary die 212, and a winder (not shown).

As show in Figure 2a, the twin screw extruder comprises a barrel 202 that is separated into five zones. The barrel 202 encloses the extrusion screws and mixing elements and serves as a containment vessel during the extrusion process. A solid feed port 204 is disposed in zone 1 and liquid feed ports 206 and 208 are disposed in zone 1 and zone 2. A vent 210 is included in zone 4 for venting the pseudo-thermoplastic melt to decrease the water content of the mixture prior to extrusion through the die 212.

The screw and mixing element configuration 300 for the twin screw extruder 200 is illustrated in Figure 2b. The twin screw extruder comprises a plurality of twin lead screws (TLS) and single lead screws (SLS) installed in series. Screw elements are characterized by the number of continuous leads and the pitch of these leads.

A lead is a flight (at a given helix angle) which wraps the core of the screw element. The number of leads indicates the number of flights wrapping the core at any given location along the length of the screw. Increasing the number of leads

reduces the volumetric capacity of the screw and increase the pressure generating capability of the screw.

The pitch of the screw is the distance needed for a flight to complete one revolution of the core. It is expressed as the number of screw element diameters per one complete revolution of a flight. Decreasing the pitch of the screw increase the pressure generated by the screw and decreases the volumetric capacity of the screw.

The length of a screw element is reported as the ratio of length of the element divided by the diameter of the element.

This example uses TLS and SLS. Screw element 310 is a TLS with a 1.0 pitch and a 1.5 length ratio. Screw element 320 is a TLS with a 1.0 pitch and a 1.0 L/D ratio. Screw element 340 is a SLS with a ¼ pitch and a 1.0 length ratio. Screw element 350 is a SLS and a ¼ pitch and a ½ length ratio.

Bilobal paddles 360 serving as mixing elements are also included in a series with the SLFS and TLFS screws in order to enhance mixing. Various configurations of bilobal paddles 360 and reversing elements 340 and 350 are used in order to control flow and corresponding mixing time.

In zone 1, Durabond A starch and sorbitol are fed into the solid feed port 204 and urea is fed into the liquid port 208 forming a mixture with a 60/20/20 weight ratio. These materials are combined inside the extruder with water added at the liquid feed port 206 to form a pseudo-thermoplastic melt. The temperature, pressure, and corresponding function of each zone are provided in Table I.

Table I

Zone	Temperature (deg F)	Pressure (gauge PSI)	Description of Screw
1	70	0	Feeding
2	193	34	Mixing
3	268	0	Mixing
4	210	0	Pressure Decreasing Conveying
5	205	0-10	Pressure Generating
Die	194	430	Shaping

Example 4 :Starch Fibers Extruded With A Non-Vented Twin Screw Extruder

The purpose of this example is to illustrate a non-vented twin screw extruder configuration, depicted in Figure 3a, used to make starch fibers for the present invention. Starch fibers are made using an APV Baker (Peterborough, England) twin screw extruder 200, a capillary die 212, and a winder (not shown).

The non-vented twin screw extruder configuration is illustrated in Figure 3a. The twin screw extruder comprises a barrel 202 that is separated into five zones. The barrel 202 encloses the extrusion screws and mixing elements and serves as a containment vessel during the extrusion process. A solid feed port 204 is disposed in zone 1 and liquid feed ports 206, and 208 are disposed in zone 1 and zone 2.

The screw and mixing element configuration for the twin screw extruder is illustrated in Figure 3b.

In zone 1, Durabond A starch and sorbitol are fed into the solid feed port 204 and urea is fed into the liquid port 208 forming a mixture with a 60/20/20 weight ratio. These materials are combined inside the extruder with water added at the liquid feed port 206 to form a pseudo-thermoplastic melt. The temperature, pressure, and corresponding function of each zone are provided in Table II.

Table II

Zone	Temperature (deg F)	Pressure (gauge PSI)	Description of Screw
1	70	0	Feeding
2	180	0	Mixing
3	260	0	Mixing
4	215	0	Pressure Decreasing Conveying
5	193	30	Pressure Generating
Die	172	150	Shaping

Example 5 :Foamed Starch Fibers Extruded With A Non-Vented Twin Screw Extruder

The purpose of this example is to illustrate the various zones of a twin screw extruder without a vent and the operating parameters associated with each zone for producing foamed starch fibers which are lower in density and having a higher absorbent capacity relative to non-foamed starch fibers. Foamed starch fibers are made using a fiber making apparatus comprising the twin screw extruder configuration depicted in Figures 3a and 3b.

In zone 1, Durabond A starch and sorbitol are fed into the solid feed port 204 and urea is fed into the liquid port 208 forming a mixture with a 60/20/20 weight ratio. These materials are combined inside the extruder with water added at the liquid feed port 206 to form a pseudo-thermoplastic melt. The temperature, pressure, and corresponding function of each zone are provided in Table III.

Table III

Zone	Temperature (deg F)	Pressure (gauge PSI)	Description of Screw
1	70	0	Feeding
2	180	0	Mixing
3	260	0	Mixing
4	240	0	Pressure Decreasing Conveying
5	220	30	Pressure Generating
Die	225	150	Shaping

Pseudo-thermoplastic Starch Melt Fiber Spinning

The production of fibers according to the invention from the pseudo-thermoplastic melt compositions occurs by the usual melt spinning processes. Devices for producing non-woven thermoplastic fabric structures from extruded polymers are well known in the art. Extruded polymers under pressure, are forced through a spinneret forming a vertically oriented curtain of downward advancing fibers. The fibers are quenched with air in conjunction with a suction-type drawing or attenuating air slot. U.S. Pat. No. 5,292,239 issued to Zeldin, et al., March 8, 1994, discloses a device that reduces significant turbulence in the air flow in order to uniformly and consistently apply a drawing force to the fibers.

For the present invention, structures are produced from a mixture comprising starch, water, plasticizers, and other optional additives. As shown in Figure 4, the mixture is converted to a pseudo-thermoplastic melt in an extruder and conveyed through a spinneret 10 to a drawing unit 20 forming a vertically oriented curtain of downward advancing fibers F.

The spinneret 10 comprises an assembly which is known in the art. The spinneret 10 includes a plurality of nozzle bores 12 with hole diameters customary for fiber production. The spinneret assembly 10 can be adapted to the fluidity of the melt so that every nozzle bore 12 has the same rate of flow.

The drawing unit 20 comprises an open upper end 22, an open lower end 24, and an air supply manifold 26 supplying compressed air to internal nozzles (not shown) oriented in a downward direction. As compressed air flows through the internal nozzles, air is drawn into the open upper end 22 of the drawing unit 20 forming a rapidly moving stream of air flowing in the downward direction. The air stream produces a drawing force on the fibers causing them to be attenuated or stretched before exiting the open lower end 24 of the drawing unit 20.

For the present invention, the fibers exiting the drawing unit 20 can have a size ranging from about 0.01 decitex to about 135 decitex. Preferably, the fibers exiting the drawing unit 20 have a size ranging from about 0.02 decitex to about 30 decitex. Most preferably, the fibers exiting the drawing unit 20 have a size ranging from about 0.02 decitex to about 5 decitex.

Upon exiting the drawing unit 20, the fibers are deposited on a moving conveyor belt 30 to form flexible, low density structure comprising fibers. The fibers are then joined to each other through conventional techniques. A preferred process for producing structures of the present invention is described in U.S. Pat. No. 5,688,468 issued to Lu, November 18, 1997, which is incorporated herein by reference.

In addition to spunbonded structures, mono-fibers, multi-fibers, staple fibers, hollow fibers, shaped fibers, such as multi-lobal fibers and multi-component fibers can all be produced by using the compositions and methods of the present invention. The process for the production of these fibers may be in one stage with a compounding extruder producing a pseudo-thermoplastic starch melt and conveying it without cooling through a melt filter to a spinneret. Staple starch fibers may also be converted to flexible, low density structures by carding, air laying, and similar processes known in the art.

(3) Material Properties

Products such as disposable towels, toilet tissue, facial tissue, napkins and wet wipes manifest various physical characteristics which include basis weight and apparent density, both of which have been previously defined. For the present invention, the structure comprising pseudo-thermoplastic starch fibers can have a basis weight ranging from about 10 g/m^2 to about 450 g/m^2. More preferably, the structure can have a basis weight ranging from about 12 g/m^2 to about 150 g/m^2. Moreover, the structure of the present invention can have an apparent density ranging from about 0.02 g/cm^3 to about 0.20 g/cm^3; more preferably, an apparent density ranging from about 0.04 g/cm^3 to about 0.15 g/cm^3 and most preferably, an apparent density ranging from about 0.04 g/cm^3 to about 0.12 g/cm^3.

The products listed above also exhibit certain mechanical properties, particularly, strength, flexibility, and absorbency. Measures of strength include geometric mean dry tensile strength (GMDT), and geometric mean wet tensile strength (GMWT) wherein GMWT includes initial wet tensile strength and decayed wet tensile strength. Flexibility is related to stiffness and can attribute to softness.

Absorbency relates to the products' ability to take up fluids as well as the capacity to retain them.

Geometric mean dry tensile strength (GMDT), previously defined, provides a measure of the dry tensile strength of the structure. The method used to determine this measure is described below. For the present invention, the structure comprising pseudo-thermoplastic starch fibers can have a GMDT ranging from about 10 g/cm to about 1200 g/cm. More preferably, the structure can have a GMDT ranging from about 30 g/cm to about 600 g/cm. Most preferably, the structure can have a GMDT ranging from about 40 g/cm to about 475 g/cm.

Geometric mean wet tensile strength (GMWT), previously defined, provides a measure of the wet tensile strength of the structure. The initial geometric mean tensile strength is the wet tensile strength of a structure after it has been immersed in water for five seconds. The method used to determine this measure is described below. For the present invention, the structure comprising pseudo-thermoplastic starch fibers can have a GMWT ranging from about 2 g/cm to about 400 g/cm. More preferably, the structure can have a GMWT ranging from about 2 g/cm to about 200 g/cm.

Geometric mean decayed wet tensile strength (GMDWT) is a measure of the wet tensile strength of the structure after being immersed in water for thirty minutes. For the present invention, the structure comprising pseudo-thermoplastic starch fibers can have a GMDWT ranging from about 0 g/cm to about 20 g/cm. More preferably, the structure can have a GMDWT ranging from about 0 g/cm to about 10 g/cm.

Softness has been described as a physiologically perceived attribute which is generally measured by expert or non-expert panel evaluations. Perceived softness can be broken down into two components; bulk softness and surface softness. Bulk softness has been correlated to sheet stiffness and flexibility while surface softness has been related to surface texture and smoothness. High softness requires flexibility. The method used for determining the total flexibility of a structure is defined below. For the present invention, the structure has a total flexibility ranging

from about 1.0 g/cm to about 75 g/cm; preferably from about 2.0 g/cm to about 50 g/cm; and more preferably from about 2.0 g/cm to about 35 g/cm.

Products such as disposable towels, toilet tissue, facial tissue, napkins, and wet wipes require a certain level of absorbency. Herein, absorbency means absorbent capacity which is a measure of the amount of distilled water absorbed and retained by the structure. The method used for determining the absorbency of a structure is defined below. For the present invention, the structure has an absorbency ranging from about 1 $g_{Water}/g_{Dry\ Structure}$ to about 15 $g_{Water}/g_{Dry\ Structure}$; preferably from about 2 $g_{Water}/g_{Dry\ Structure}$ to about 14 $g_{Water}/g_{Dry\ Structure}$; more preferably from about 3 $g_{Water}/g_{Dry\ Structure}$ to about 13 $g_{Water}/g_{Dry\ Structure}$.

(4) Analytical Methods

(a) Sample Conditioning And Preparation:

Prior to testing, samples are conditioned at a relative humidity of 48% to 50% and within a temperature range of 22°C to 24°C until a moisture content of from about 5% to about 16% by weight as measured by TGA (Thermo Gravimetric Analysis) is achieved. For Thermo Gravimetric Analysis, a Hi-res. TGA2950 Termogravimetric analyzer from TA Instruments is used. Approximately 20 mg of sample is weighed into a TGA pan. Following the manufacturer's instructions, the sample and pan are inserted into the unit and the temperature is increased at a rate of 10°C/minute to 250°C. The % moisture in the sample is determined using the weight lost and the initial weight as follows:

$$\%\ \text{Moisture} = \frac{\text{Start Weight} - \text{Weight @ 250°C}}{\text{Start Weight}} * 100\%$$

where all weights are in milligrams.

(b) Basis Weight

One stack of 8 plies is made from the preconditioned samples. The stack of 8 plies is cut into a 4 inch by 4 inch square. A rule die from Acme Steel Rule Die Corp. (5 Stevens St. Waterbury Conn., 06714) is used to accomplish this cutting.

For the actual measurement of the weight of the sample, a top loading balance with a minimum of 0.01 g readability is used. The stack of 8 plies is laid on the pan of the top loading balance. The balance is protected from air drafts and other disturbances using a draft shield. Weights are recorded when the readings on the balance become constant. Weights are measured in grams.

The weight reading is divided by the number of plies tested. The weight reading is also divided the weight reading by the area of the sample which is normally 16 square inches, which is approximately equal to 0.0103 square meters.

The unit of measure for basis weight as used herein is grams/square meter. This is calculated using the 0.0103 square meter area noted above.

(c) Caliper

Preconditioned samples are cut to a size greater than the size of the foot used to measure the caliper. The foot to be used is a circle with an area of 3.14 square inches.

The sample is placed on a horizontal flat surface and confined between the flat surface and a load foot having a horizontal loading surface, where the load foot loading surface has a circular surface area of about 3.14 square inches and applies a confining pressure of about 15 g/square cm (0.21 psi) to the sample. The caliper is the resulting gap between the flat surface and the load foot loading surface. Such measurements can be obtained on a VIR Electronic Thickness Tester Model II available from Thwing-Albert, Philadelphia, Pa. The caliper measurement is repeated and recorded at least five times. The result is reported in millimeters.

The sum of the readings recorded from the caliper tests is divided by the number of readings recorded. The result is reported in millimeters (mm).

(d) Dry Tensile Strength

The dry tensile strength is determined on one inch wide strips of sample using a Thwing-Albert Intelect II Standard Tensile Tester (Thwing-Albert Instrument Co., 10960 Dutton Rd., Philadelphia, Pa., 19154). This method is intended for use on finished paper products, reel samples, and unconverted stocks.

Two stacks of 8 plies are made from the preconditioned samples. From one of these stacks of 8 plies, four strips are cut 1 inch by 7 inch with the long 7 inch dimension running parallel to the machine direction. Note these samples are machine direction samples. An additional four strips 1 inch by 7 inch with the long 7 inch dimension running parallel to the cross direction. All cuts are made using a cutter (JDC-1-10 or JDC-1-12 with safety shield from Thwing-Albert Instrument Co., 10960 Dutton Road, Philadelphia, Pa., 19154). A total of eight samples are produced: four 1 inch by 7 inch strips, 8 plies thick, with the 7 inch dimension running parallel to the machine direction and four 1 inch by 7 inch strips, 8 plies thick, with the 7 inch dimension running parallel to the cross direction.

Each of the four eight ply stacks of machine direction and cross machine direction sample tensile strips are measured in the Thwing-Albert Intelect II Standard Tensile Tester. The four measurements of the 8 ply stacks of machine direction sample tensile strips are summed and divided by four, which is the number of machine direction strips tested. The sum is also divided by eight, which the number of usable units per tensile strip. The calculation is repeated for the cross machine direction measurements.

All results are in units of grams/inch. Appropriate unit conversions may be made to achieve units of grams/cm as reported herein.

(e) Initial Wet Tensile Strength

For the initial wet tensile strength determination, a portion of the test sample is immersed in water for five seconds prior to the tensile strength measurement. The wet tensile strength is determined on one inch wide strips of sample using a Thwing-Albert Intelect II Standard Tensile Tester (Thwing-Albert Instrument Co., 10960 Dutton Rd., Philadelphia, Pa., 19154) and a Finch Wet Strength Device, Catalog

Number 731D (Thwing-Albert Instrument Co., 10960 Dutton Rd., Philadelphia, Pa., 19154).

Prior to sample preparation and wet tensile testing, the samples should be cured in a forced draft oven at 105 ± 3 degree Celsius for a period of 5 minutes ± 10 seconds. The samples should be suspended in the oven such that the forced air can circulate between them.

Sample preparation and all aspects of the wet tensile testing should take place within the confines of the constant temperature and humidity room. Two stacks of 5 plies each are made from the cured samples after conditioning. From one of these stacks of 5 plies, four strips are cut 1 inch by 4 inch with the long 4 inch dimension running parallel to the machine direction for machine direction samples. An additional four strips are cut 1 inch by 4 inch with the long 4 inch dimension running parallel to the cross direction for cross direction samples. All cuts are made using a paper cutter (JDC-1-10 or JDC-1-12 with safety shield from Thwing-Albert Instrument Co., 10960 Dutton Road, Philadelphia, Pa., 19154). There are a total of eight samples: four 1 inch by 4 inch strips which are 5 plies thick with the 4 inch dimension running parallel to the machine direction and four 1 inch by 4 inch strips which are 5 plies thick with the 4 inch dimension running parallel to the cross direction.

Each of the four five ply stacks of machine direction and cross machine direction sample tensile strips are measured in the Thwing-Albert Intelect II Standard Tensile Tester. The four measurements of the 5 ply stacks of machine direction sample tensile strips are summed and divided by four, which is the number of machine direction strips tested. The sum is also divided by five, which the number of usable units per tensile strip. The calculation is repeated for the cross machine direction measurements.

All results are in units of grams/inch. Appropriate unit conversions may be made to achieve units of grams/cm as reported herein.

(f) Decayed Wet Tensile (Soaked for 30 minutes)

Same as the Initial Wet Tensile Strength except the samples are allowed to soak in the water for 30 minutes (± 30 seconds) prior to Wet Tensile Strength Testing.

(g) Flexibility

Flexibility as used herein is defined as the slope of the secant of the graph-curve derived from force vs. stretch % data which secant passes through the origin (zero % stretch, zero force) and through the point on the graph-curve where the force per centimeter of width is 20 grams. For example, for a sample which stretches 10% (i.e., 0.1 cm/cm of length) with 20 grams of force per cm of sample width, the slope of the secant through (0%, 0) and (10%, 20) is 2.0 using the formula:

$$Slope = \frac{Y_2 - Y_1}{X_2 - X_1}$$

Total Flexibility as used herein means the geometric mean of the machine-direction flexibility and cross-machine-direction flexibility. Mathematically, this is the square root of the product of the machine-direction flexibility and cross-machine-direction flexibility in grams per cm.

(h) Absorbency

Absorbency herein is defined as the amount (grams) of distilled water at 73 ± 2 °F per gram of sample held by the sample after it has been submerged in a water bath for a period of 30 ± 3 seconds and then allowed to sit in a horizontal position for 120 ± 5 seconds followed by 60 ± 5 seconds sitting at a 75° angle (as measured off of horizontal).

A preconditioned sample is cut to a size of 11 inches by 11 inches. The Machine Direction of the sample is marked and the sample is weighed on a torsion balance to ± 0.01 grams and recorded. This is known as the Sample Dry Weight. After weighing the sample, the dry sample rack (further described below) is placed

on the balance and the weight is recorded to ± 0.01 grams. This is known as the Rack Dry Weight.

The sample is placed on a rack and covered with a rack cover, further described below. The sample, contained by the rack and rack cover, is gently and completely submerged (to a depth of 2 to 3 inches) horizontally in a bath of distilled water at a temperature of 73 ± 2 °F for 30 ± 3 seconds.

After being submerged for 30 ± 3 seconds, the sample is gently raised (horizontally), the rack cover is gently removed, and the sample and rack are allowed to sit for a period of 120 ± 5 seconds in order to drain. While the sample is sitting in the horizontal position, water sitting on the rack is gently wiped off without touching the sample.

Following the drying of the rack and the completion of the horizontal sitting period, the rack and sample together are gently raised so that the Machine Direction is at an angle of 75° from horizontal and allowed to sit in this position for a period of 60 ± 5 seconds. After this sitting period is completed, the rack and sample are returned to a horizontal position and once again the rack is dried of standing water. The rack and sample are gently placed on the balance and the weight ± 0.01 grams is recorded. This is known as the Sample and Rack Wet Weight.

The absorbency measurement is made and recorded for three (3) machine direction samples and three (3) cross machine direction samples. During the cross machine direction measurement, the cross-machine direction of the sample is placed at an angle of 75° from horizontal.

An illustration of the sample rack and cover is shown in Figure 5. Both include frames 400 constructed of 16 GA Aluminum (Teflon Coated after Fabrication) with a cross-section shown in Figure 6. The outside dimensions 405, 410 of the frames 400 are about 13.75 inches by about 16.75 inches. Nylon thread 420 (0.3mm diameter) is tightly strung across the Aluminum frames 400 in a pattern shown in Figure 5. All diagonal threads go over those threads running perpendicular and/or parallel to the frames 400.

For each of the 6 tests, the following calculation is made (all units are grams):

Sample Wet Weight = Sample and Rack Wet Weight - Rack Dry Weight

$$\text{Absorbency} = {}^{(\text{Sample Wet Weight} - \text{Sample Dry Weight})}\!\big/_{\text{Sample Dry Weight}}$$

The calculation is repeated for each of the 6 measurements and all 6 absorbency numbers are averaged together and reported as ${}^{g_{Water}}\!/_{g_{Dry\ Structure}}$ (grams of water / grams of sample dry weight).

While particular embodiments of the present invention have been illustrated and described, it would be obvious to those skilled in the art that various other changes and modifications can be made without departing from the spirit and scope of the invention. It is intended to cover in the appended claims all such changes and modifications that are within the scope of the invention.

What is claimed is:

1. An absorbent, flexible structure comprising pseudo-thermoplastic starch fibers.

2. The structure of Claim 1, wherein the structure has an apparent density ranging from about 0.02 g/cm^3 to about 0.20 g/cm^3.

3. The structure of Claim 2, wherein the structure has an apparent density ranging from about 0.04 g/cm^3 to about 0.15 g/cm^3.

4. The structure of Claim 3, wherein the structure has an apparent density ranging from about 0.04 g/cm^3 to about 0.12 g/cm^3.

5. The structure of Claim 1, wherein the starch fibers have a size ranging from about 0.01 dtex to about 135 dtex.

6. The structure of Claim 5, wherein the starch fibers have a size ranging from about 0.02 dtex to about 30 dtex.

7. The structure of Claim 6, wherein the starch fibers have a size ranging from about 0.02 dtex to about 5 dtex.

8. The structure of Claim 1, wherein the structure has a geometric mean dry tensile strength ranging from about 10 g/cm to about 1200 g/cm.

9. The structure of Claim 8, wherein the structure has a geometric mean dry tensile strength ranging from about 30 g/cm to about 600 g/cm.

10. The structure of Claim 9, wherein the structure has a geometric mean dry tensile strength ranging from about 40 g/cm to about 475 g/cm.

11. The structure of Claim 1, wherein the structure has an initial geometric mean wet tensile strength ranging from about 2 g/cm to about 400 g/cm.

12. The structure of Claim 11, wherein the structure has an initial geometric mean wet tensile strength ranging from about 2 g/cm to about 200 g/cm.

13. The structure of Claim 1, wherein the structure has a geometric mean dry tensile strength per unit basis weight ranging from about 200 m to about 2500 m.

14. The structure of Claim 13, wherein the structure has a geometric mean dry tensile strength per unit basis weight ranging from about 200 m to about 1500 m.

15. The structure of Claim 14, wherein the structure has a geometric mean dry tensile strength per unit basis weight ranging from about 200 m to about 1200 m.

16. The structure of Claim 1, wherein the structure has a geometric mean wet tensile strength per unit basis weight ranging from about 10 m to about 1500 m.

17. The structure of Claim 16, wherein the structure has a geometric mean wet tensile strength per unit basis weight ranging from about 10 m to about 1000 m.

18. The structure of Claim 17, wherein the structure has a geometric mean wet tensile strength per unit basis weight ranging from about 10 m to about 500 m.

19. The structure of Claim 1, wherein the structure has a geometric mean decayed wet tensile strength ranging from about 0g/cm to about 20 g/cm.

20. The structure of Claim 19, wherein the structure has a geometric mean decayed wet tensile strength ranging from about 0g/cm to about 10 g/cm.

21. The structure of Claim 1, wherein the structure has a basis weight ranging from 10 g/m2 to about 450 g/m2.

22. The structure of Claim 21, wherein the structure has a basis weight ranging from 12 g/m2 to about 150 g/m2.

23. The structure of Claim 1, further comprising a plasticizer selected from the group consisting of sorbitol, monosaccharides, disaccharides, glycerol, polyvinyl alcohol, and polyethylene glycol.

24. The structure of Claim 23, wherein said plasticizer comprises, based on total weight of the structure, from about 5 wt % to about 70 wt %.

25. The structure of Claim 24, wherein said plasticizer comprises, based on total weight of the structure, from about 15 wt % to about 60 wt %.

26. The structure of Claim 1, further comprising cross-linking agents selected from the group consisting of, polyamide-epichlorohydrin resins, urea-formaldehyde resins, glyoxylated polyacrylamide resins, melamine formaldehyde resins, polyethylenimine resins, Caldas 10 resin, CoBond 1000 resin.

27. The structure of Claim 26, wherein said cross-linking agents are present in amounts ranging from about 0.1 wt % to about 10 wt. %, based on the total weight of the structure.

28. The structure of Claim 27, wherein said cross-linking agents are present in amounts ranging from about 0.1 wt % to about 3 wt. %, based on the total weight of the structure.

29. The structure of Claim 1, wherein the structure has an absorbency ranging from about 1 $g_{Water}/g_{Dry\ Structure}$ to about 15 $g_{Water}/g_{Dry\ Structure}$.

30 The structure of Claim 29, wherein the structure has an absorbency ranging from about 2 $g_{Water}/g_{Dry\ Structure}$ to about 14 $g_{Water}/g_{Dry\ Structure}$.

31. The structure of Claim 30, wherein the structure has an absorbency ranging from about 3 $g_{Water}/g_{Dry\ Structure}$ to about 13 $g_{Water}/g_{Dry\ Structure}$.

32. The structure of Claim 1, wherein the structure has a total flexibility ranging from about 1.0 g/cm to about 75 g/cm.

33. The structure of Claim 32, wherein the structure has a total flexibility ranging from about 2.0 g/cm to about 50 g/cm.

34. The structure of Claim 33, wherein the structure has a total flexibility ranging from about 2.0 g/cm to about 35 g/cm.

35. A structure comprising pseudo-thermoplastic starch fibers wherein the fibers have a Tg of at least about -30 °C and the structure has a geometric mean decayed wet tensile strength ranging from about 0g/cm to about 20 g/cm.

36. The structure of Claim 35, wherein said fibers have a Tg ranging from about -30 °C to about 150 °C.

37. The structure of Claim 36, wherein said fibers have a Tg ranging from about -30 °C to about 100°C.

38. The structure of Claim 37, wherein said fibers have a Tg ranging from about -30 °C to about 25°C.

39. The structure of Claim 35, wherein said structure has a geometric mean decayed wet tensile strength ranging from about 0g/cm to about 10 g/cm.

40. The structure of Claim 36, wherein said structure has a geometric mean decayed wet tensile strength ranging from about 0g/cm to about 10 g/cm.

41. The structure of Claim 35, wherein said fibers have a size ranging from about 0.01 dtex to about 135 dtex.

42. The structure of Claim 41, wherein said fibers have a size ranging from about 0.02 dtex to about 30 dtex.

43. The structure of Claim 42, wherein said fibers have a size ranging from about 0.02 dtex to about 5 dtex.

44. An absorbent structure comprising one or more plies wherein at least one ply comprises pseudo-thermoplastic starch fibers, and wherein the at least one ply has a basis weight ranging from about 10 g/m^2 to about 100 g/m^2, a GMDT ranging from about 40 g/cm to about 475 g/cm, an apparent density ranging from about 0.04 g/cm^3 to about 0.12 g/cm^3, and an initial GMWT ranging from about 2 g/cm to about 200 g/cm.

45. The structure of Claim 44, wherein the at least one ply has a geometric mean decayed wet tensile strength ranging from about 0 g/cm to about 20 g/cm.

Abstract

A flexible absorbent structure is produced comprising starch fibers. Naturally occurring starch in the presence of water, plasticizers and other additives is melt extruded and spun bonded to form low density, absorbent, flexible structures. The structures exhibit properties matching those of consumer paper products such as paper towels, toilet tissue, facial tissue, napkins, wet wipes, and the like.

DECLARATION COMBINED WITH POWER OF ATTORNEY

Attorney Docket No. 7456

As a below named inventor, I hereby declare that:

My residence, post office address and citizenship are as stated below next to my name.

I believe I am the original, first and sole inventor (if only one name is listed below) or an original, first and joint inventor (if plural names are listed below) of the subject matter which is claimed and for which a patent is sought on the invention entitled

Absorbent, Flexible, Structure Comprising Starch Fibers

the specification of which

[X] is attached hereto.

[ ] was filed on _____ as United States Application No. or PCT International Application Number _____ and was amended on _____ (if applicable)

I hereby state that I have reviewed and understand the contents of the above identified specification, including the claims, as amended by any amendment referred to above.

I acknowledge the duty to disclose information which is material to patentability as defined in Title 37 Code of Federal Regulations §1.56.

I hereby claim foreign priority benefits under Title 35 United States Code §119(a)-(d) or §365(b) of any foreign application(s) for patent or inventor's certificate, or §365(a) of any PCT International application which designated at least one country other than the United States of America, listed below and have also identified below any foreign application for patent or Inventor's certificate, or of any PCT international application having a filing date before that of the application on which priority is claimed:

Prior Foreign Application(s)			Priority Claimed	
_____	_____	_____	[ ]	[ ]
(Number)	(Country)	(Day/Month/Year Filed)	Yes	No
_____	_____	_____	[ ]	[ ]
(Number)	(Country)	(Day/Month/Year Filed)	Yes	No

I hereby claim the benefit under Title 35, United States Code §119(e) of any United States provisional application(s) listed below.

_____	_____	_____	_____
Application Serial No.	Filing Date	Application Serial No.	Filing Date

I hereby claim the benefit under Title 35, United States Code §120 of any United States application(s), or §365(c) of any PCT International application designating the United States of America, listed below and, insofar as the subject matter of each of the claims of this application is not disclosed in the prior United States or PCT International application in the manner provided by the first paragraph of Title 35, United States Code §112, I acknowledge the duty to disclose information which is material to patentability as defined in Title 37, Code of Federal Regulations §1.56 which became available between the filing date of the prior application and the national or PCT international filing date of this application:

U.S. Parent Application Number	PCT Parent Number	Parent Filing Date (MM/DD/YYYY)	Parent Patent Number (If applicable)

As named inventor, I hereby appoint the following registered practitioner(s) to prosecute this application and to transact all business in the Patent and Trademark Office connected therewith:

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E. Kelly Linman	26,759	Donald E. Hasse	29,387
Larry L. Huston	32,994	Julia A. Glazer	41,783
Vladimir Vitenberg	42,204	Jacobus C. Rasser	37,043
Roddy M. Bullock	37,290	T. David Reed	32,931
Kevin D. Hogg	31,839	Timothy B. Guffey	41,048

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The Procter & Gamble Company — Facsimile No.: (513) 634-3612
Winton Hill Technical Center, 6100 Center Hill Avenue, Cincinnati, Ohio 45224

I hereby declare that all statements made herein of my own knowledge are true and that all statements made on information and belief are believed to be true; and further that these statements were made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under Section 1001 of Title 18 of the United States Code and that such willful false statements may jeopardize the validity of the application or any patent issued thereon.

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150

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Date

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FIGURE 1a:

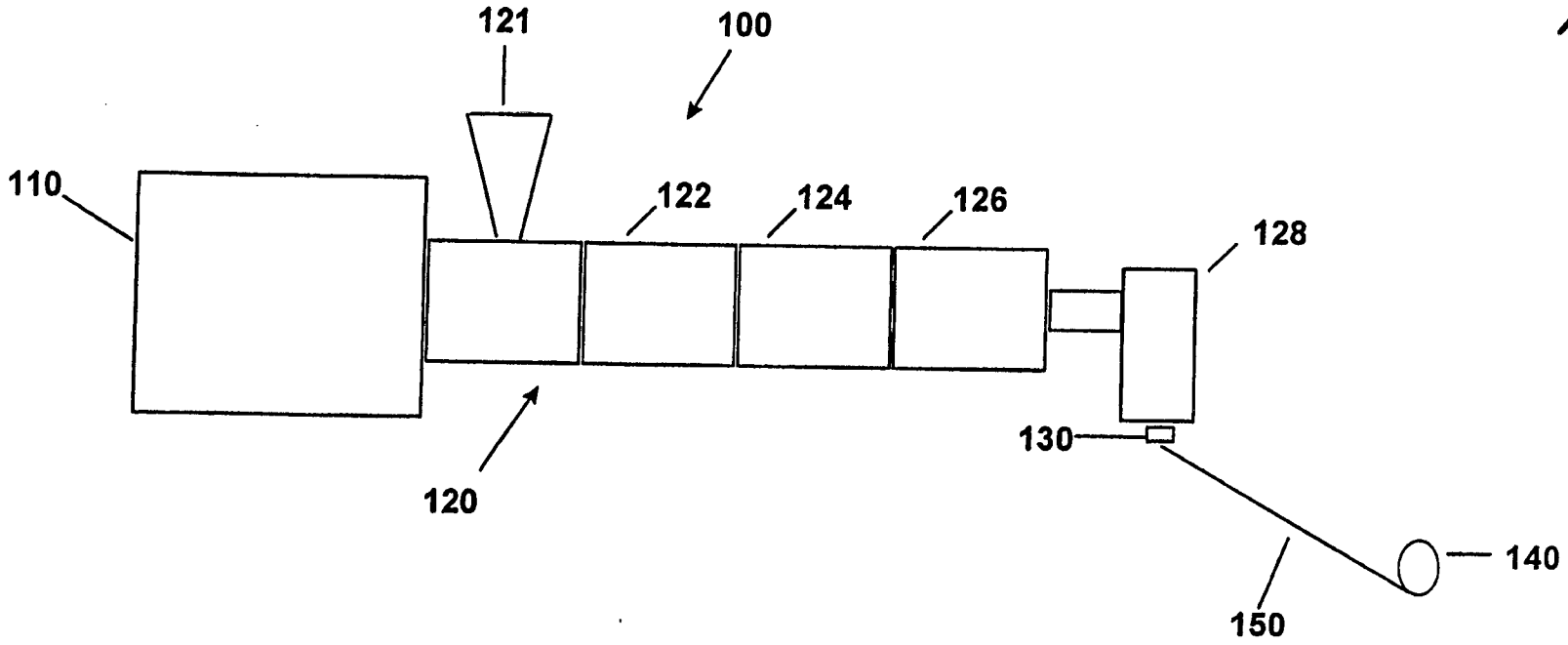
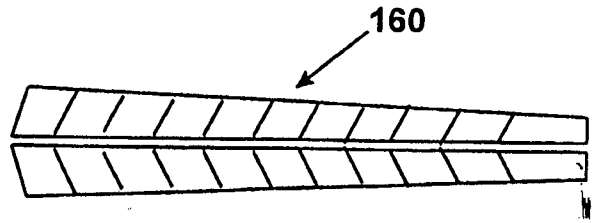


FIGURE 1b:



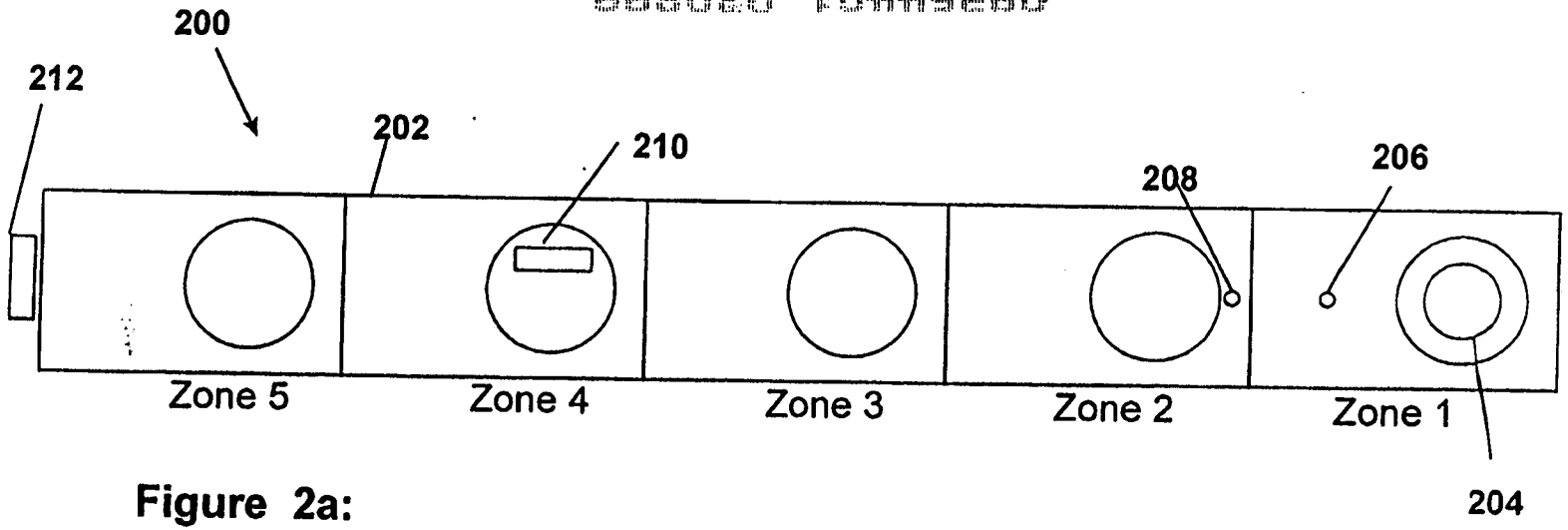


Figure 2a:

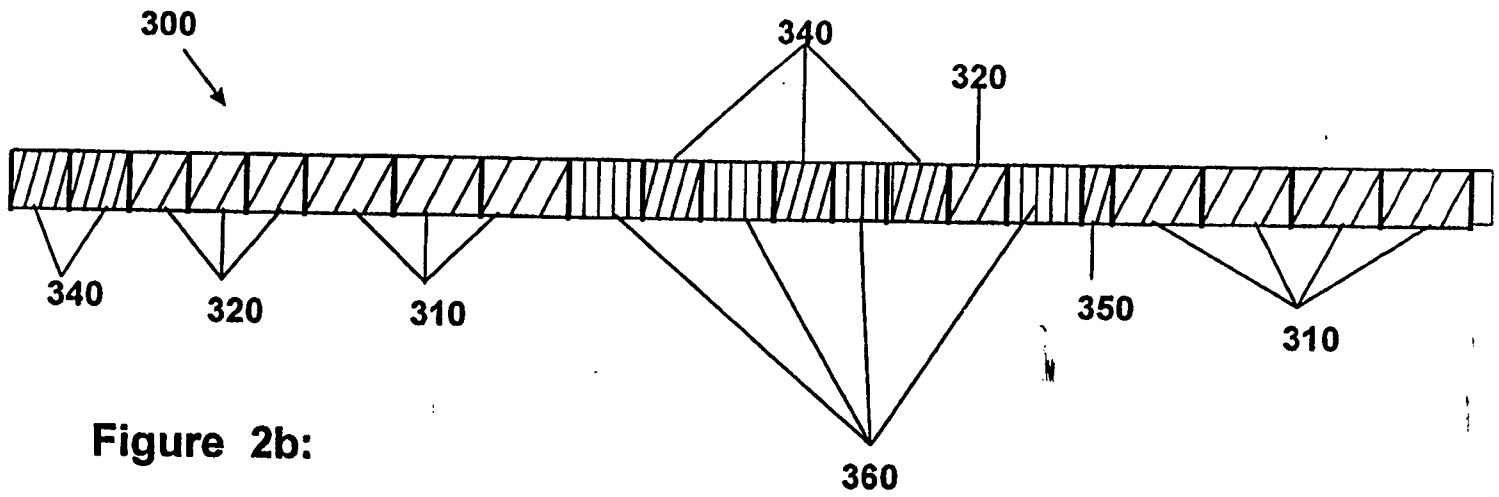


Figure 2b:

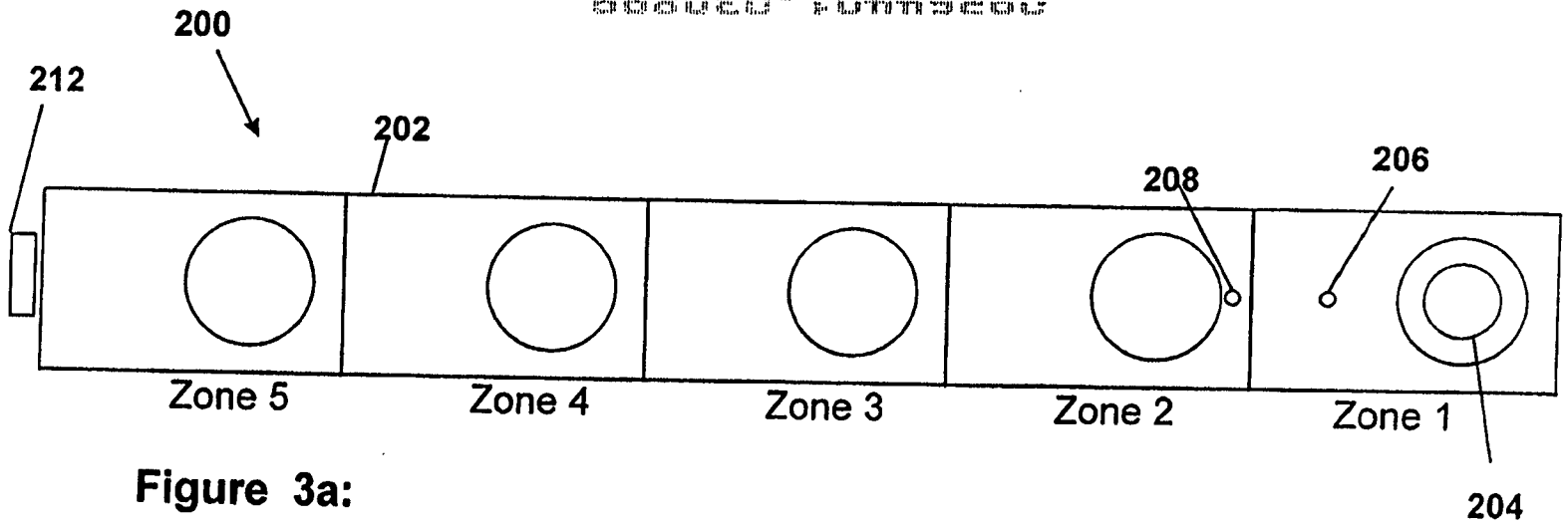


Figure 3a:

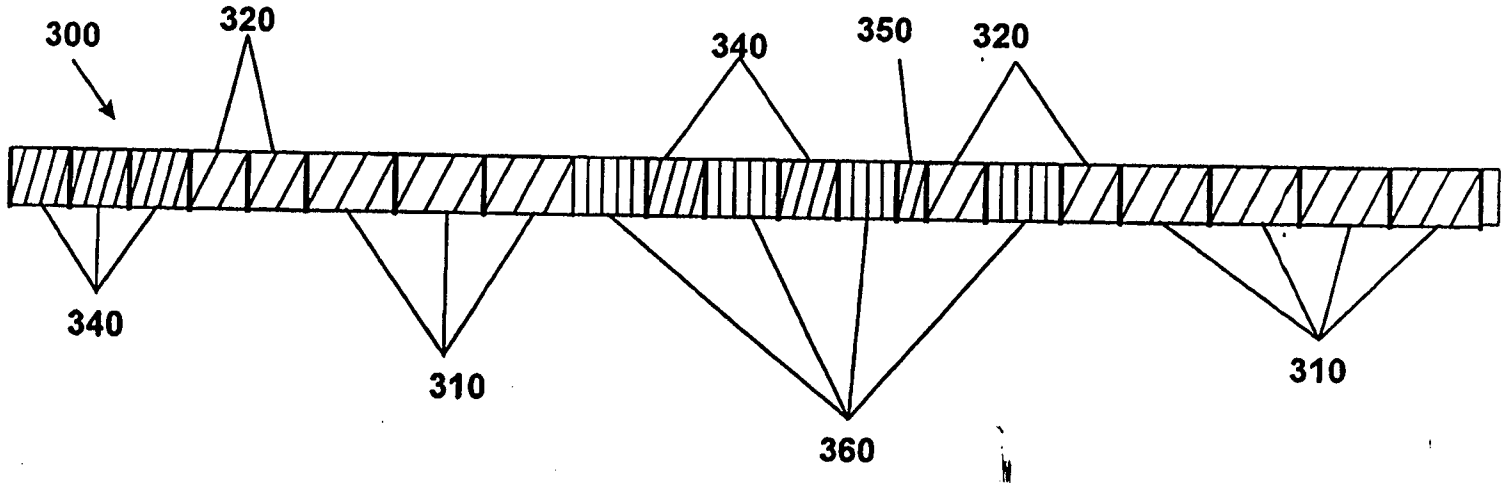


Figure 3b:

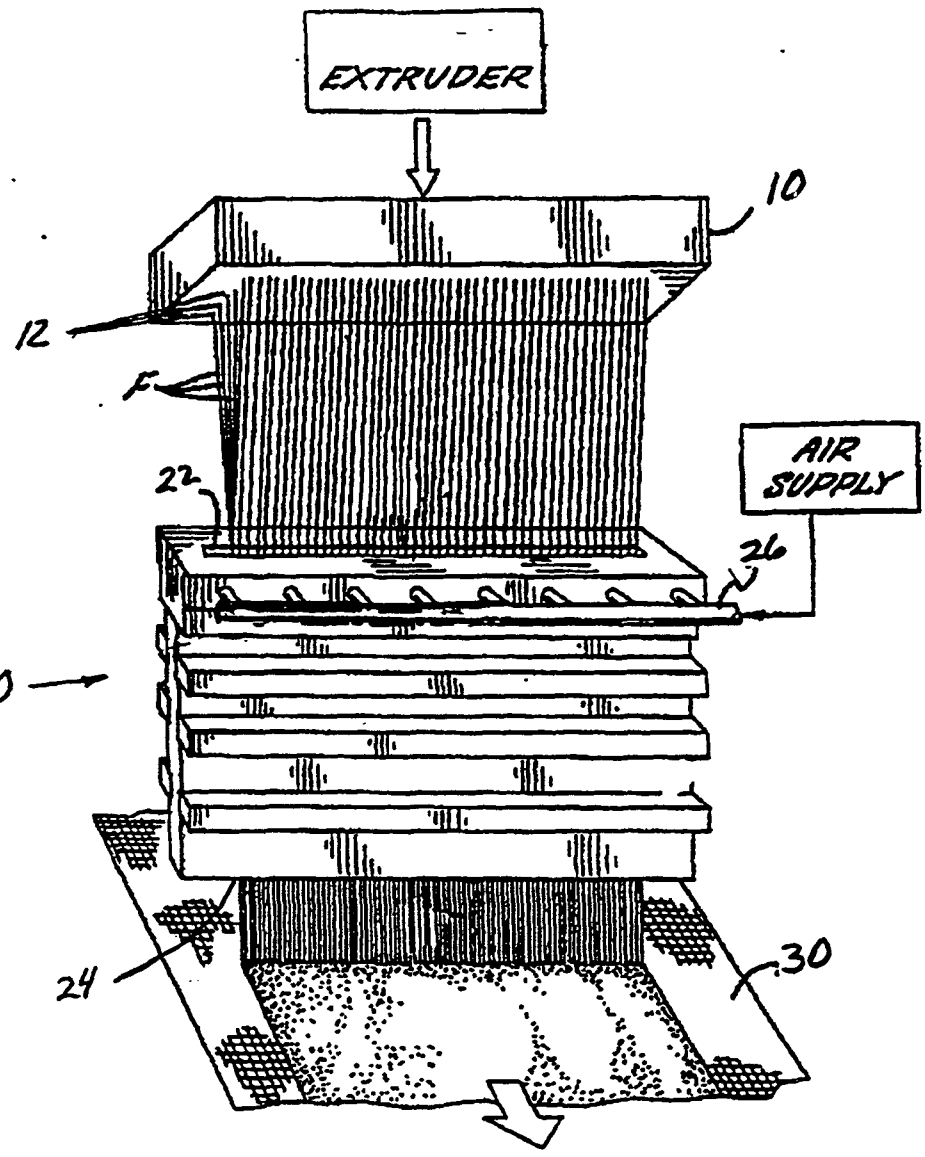


FIGURE: 4

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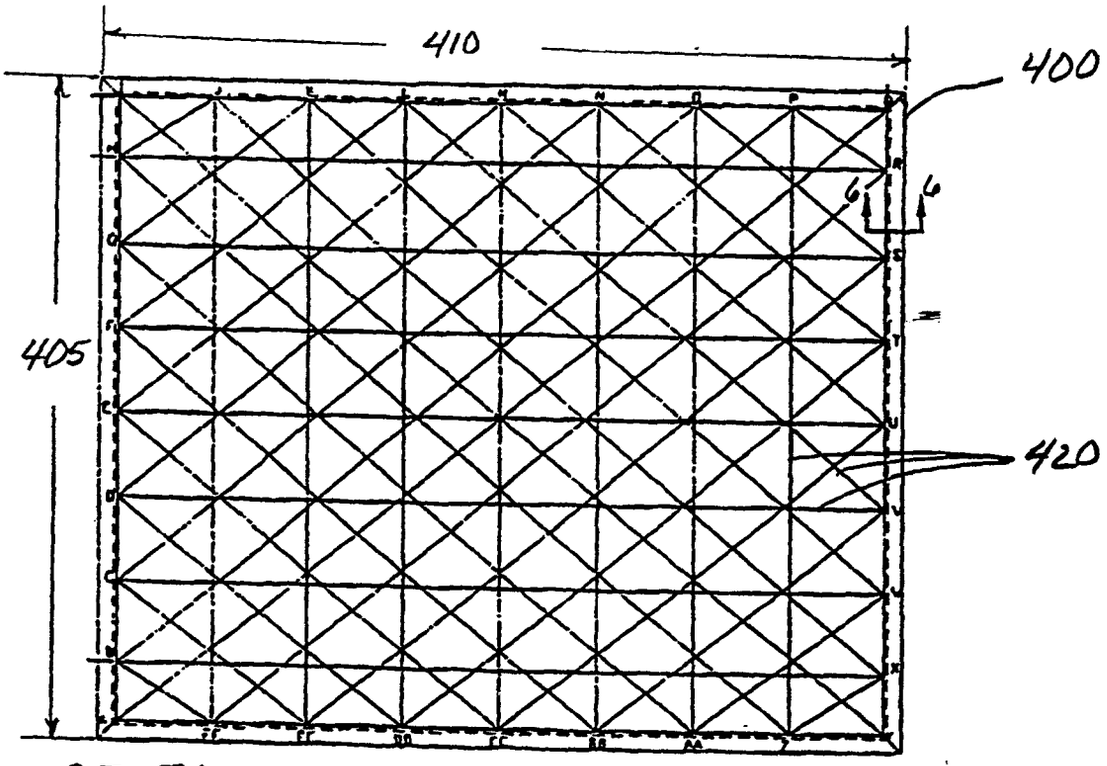

FIGURE 5:

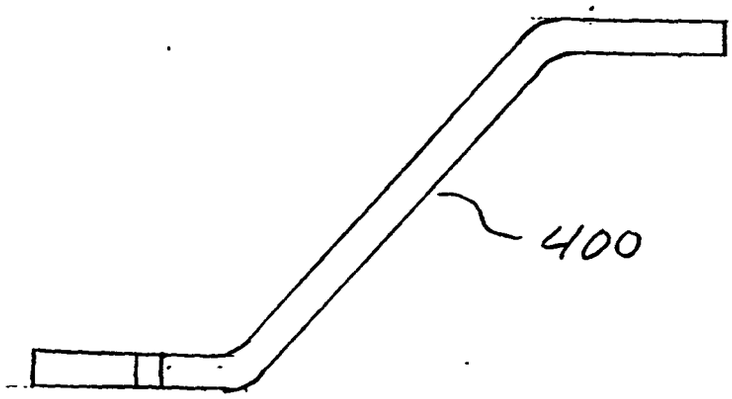

FIGURE 6: