

.4 Serviceability and Utilization

General stability and precautions in use. Applications.

.5 History

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.6 Additional Notes**.7 Additional References**

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PART I

DATA ON INDIVIDUAL POLYMERS

TABLE 1

CELLULOSE

PRINCIPALLY AS FIBRES

Synonyms and trade names

Polyanhydroglucose, poly-1,4-anhydro- β -pyranose.

Natural sources (of high cellulose content): Pure cotton, flax, ramie; purified wood pulp.

Regenerated cellulose: Viscose and cuprammonium rayons. Cellophane.

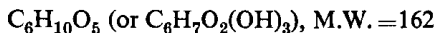
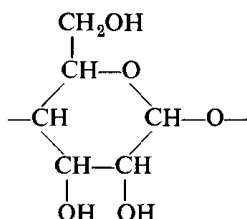
For additional form of cellulose, and names of common fibres, see §§1.61 and 1.62.

General characteristics

Cellulose is the chief structural material of the vegetable world. The purest forms occur naturally in a fibrous state, as exemplified by cotton, flax, ramie, and hemp; extracted filter paper, and regenerated forms such as viscose rayon, also consist of almost pure cellulose (although reduced in molecular weight).

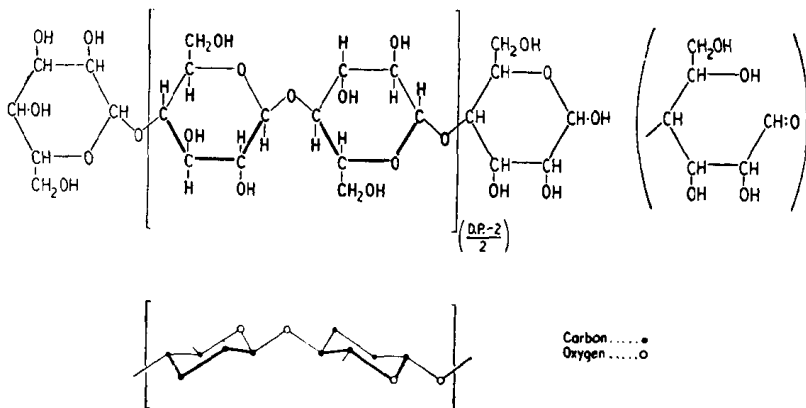
1.1 Structure

Simplest fundamental unit:

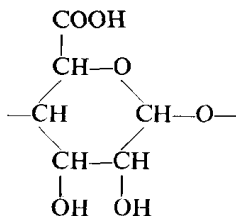


N.B. The structure of a cellulose molecule is better represented if the recurring unit is depicted as two anhydroglucose units mutually rotated through 180° about the chain axis, i.e. as 1,4'-anhydrocellobiose. This is indicated below, with anhydrocellobiose shown (diagrammatically) in perspective.

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One of the two terminal units in each cellulose molecule can exhibit aldehydic properties (as shown in brackets to the right), and each terminal unit possesses four —OH groups compared with three such groups per intrachain anhydroglucose unit. In addition, and particularly in regenerated cellulose, the chain can contain a small number of modified units, such as those composed of glucuronic acid residues, e.g.



Molecular weight: By viscosimetric or sedimentation methods: native cellulose, 300,000 to 500,000 ($>10^6$, by sedimentation and diffusion); regenerated do., 100,000 or more.

Degree of polymerization: Native cellulose, 2,000 to 3,000; regenerated do., up to 600 or more.

X-ray data: Polycrystalline. There are 4 anhydroglucose units to the unit cell, in which the identity period is constant at 10.3 \AA (cellobiose unit) but the lateral and angular dimensions can vary, those of native cellulose (Cellulose I) being different from those of mercerized cellulose and regenerated forms (Cellulose II); additional specialized modifications (Cellulose III and IV) are of academic interest. In native fibres the crystalline regions are *ca.* 60 \AA in diameter $\times 600 \text{ \AA}$ axially (i.e. embracing some 100 to 150 molecular chains), and intermicellar cavities range from 10 to 15 \AA , with

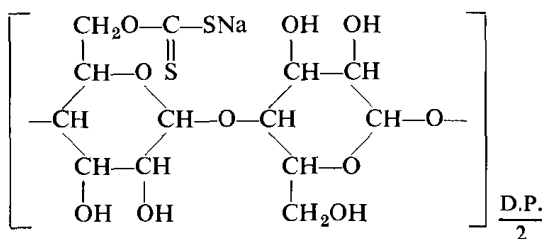
larger regions 100 Å or more, in diameter. In regenerated fibres molecular orientation increases on stretching.

1.2 Chemistry

1.21 Preparation

Cellulose is present, as the chief constituent of the cell wall, in all plants (and, in association with lignin, composes the structural material of wood), but it has not been synthesized artificially. For regenerative purposes (i.e. dissolving and then precipitating from solution) the chief commercial sources are cotton linters, wood-pulp freed from lignin by acid bisulphite or alkaline extraction, and spinners' waste.

The *Viscose Process* is most commonly used for the manufacture of fibres (viscose rayon), and the principle employed is as follows: Soda cellulose (from high-grade α -cellulose treated with 17% sodium hydroxide), 'aged' under controlled conditions (whereby oxidative and degradative changes beneficial to the production of a spinnable solution occur), is treated with carbon disulphide, with which it forms sodium cellulose xanthate:



The xanthate is dissolved in dilute sodium hydroxide to give a viscous orange-coloured solution (viscose) which is filtered and allowed to 'ripen' (partial decomposition of the unstable xanthate) until thickened to a viscosity suitable for spinning into a coagulating bath (the principal components of which are dil. H_2SO_4 , NaHSO_4 , and ZnSO_4). When the extruded viscose contacts the acid bath, cellulose is regenerated by decomposition of the xanthate solution and is wound off as fine threads.

The *Cuprammonium Process* utilizes a solution of purified cotton linters in cuprammonium hydroxide, from which cellulose is regenerated by spinning into a setting bath of warm water, followed by an acid wash.

In all regenerative processes filaments are stretched while still wet and ductile, in order to orient the chain molecules in the direction of the fibre axis and improve the tensile strength of the product; and, for high-tenacity fibres, additional stretching is given during passage through hot water or 60% sulphuric acid. An alternative process for a high-strength product consists in deacetylating filaments

of cellulose acetate while they are in a swollen and stretched state (see Fortisan, §1.62).

For the production of films of regenerated cellulose (e.g. Cellophane), the spinneret or multi-holed jet used for spinning fibres is replaced by an extrusion head having a linear or annular slit-shaped orifice.

1.22 Properties

Solvents: No simple solvents. Soluble in sodium xanthate (see viscose process, above), hot zinc chloride solution (or $\text{ZnCl}_2 \cdot 2\text{HCl}$ solution in the cold), ice-cold alkalis, alkalis containing dissolved metallic oxides (e.g. sodium zincate), cuprammonium hydroxide (Schweizer's reagent), cupriethylenediamine solution, beryllium perchlorate or $\text{KI} \cdot \text{HgI}_2$ solutions, quaternary ammonium bases (e.g. tetramethylammonium hydroxide), calcium thiocyanate, and similar solutions containing large ions. **Plasticized by:** Water, steam; and by polyhydric alcohols (e.g. glycerol), which act as humectants. **Relatively unaffected by:** Non-polar liquids, such as hydrocarbons and oils. **Swollen by:** Non-solvent but polar liquids, such as water, and more especially by moderately concentrated aqueous alkalis. N.B. At 20°C maximum swelling of cotton occurs in 10 to 12% sodium hydroxide, but mercerization (i.e. swelling to form a more reactive structure) requires ca. 17.5% sodium hydroxide (this is reduced to ca. 6.5% NaOH at -10°C). **Decomposed by:** Oxidizing agents, and all hot acids of sufficient strength. Some degradation occasioned, in the presence of alkaline solvents, by atmospheric oxygen. Ordinary regenerated cellulose is less resistant to chemical attack than the stretch-spun fibres or natural forms like cotton.

1.23 Identification Reactions [Non-thermoplastic. Cl, N, S, and P nominally absent. (See Table 57.)]

Combustion: Chars, burns without melting. When pure and with free access of air, leaves practically no ash. **Pyrolysis:** Chars without melting, 'woody' smell, black residue. Chief products, water, carbon dioxide, acetic acid, and phenolic tars.

Colour reactions: The following sensitive tests serve to distinguish cellulose, cellulose derivatives, and related polymers from practically all other types:

Anthrone test. To a small sample suspended in 0.5 ml water, add some 2 ml of a 0.2% solution of anthrone (9,10 dihydro-9-ketoanthracene) in concentrated sulphuric acid, until the precipitate first formed redissolves. Formation of a distinctive

sea-green or dark bluish-green colour, together with a greenish-yellow precipitate on careful dilution with water, occurs in the presence of cellulose, alginates, starch, or furfural, and to a less extent with cellulose esters and ethers (but not with cellulose nitrate, unless previously saponified).

Molisch test. Shake a small sample with 1 ml water and 2 drops of a 10% solution of α -naphthol in chloroform, then add slowly at least 2 ml concentrated sulphuric acid so as to form a lower layer. When carbohydrates are present, a violet ring is produced at the interface, and on careful mixing a purple solution is obtained that yields a violet precipitate on dilution with water. The colour is due to formation of ω -hydroxymethyl-furfural and its interaction with α -naphthol, and is given by all carbohydrates (inc. wood, paper, regenerated cellulose), alginates, and furfural. Ester and ether derivatives of cellulose respond less readily, but the purple colour usually appears on heating the mixed liquids; cellulose nitrate does not respond to the test at all.

Distinctions between natural and regenerated cellulose:

Iodine test. Cellulose is deeply stained when brought into contact with a 10 to 15% solution of iodine in aqueous potassium iodide, but on prolonged immersion in water natural cellulose (cotton, linen) washes clear while regenerated cellulose (viscose and cuprammonium rayons) assumes a deep blue colour. Mercerized cellulose behaves intermediately; secondary cellulose acetate is coloured brown; partial ethers (methylcellulose, hydroxyethyl-cellulose) also stain deeply with iodine.

Cross-section of fibres. In natural cellulose fibres the outline of the transverse section and the shape of the central canal, or lumen, are often characteristic, and in many of the bast fibres the interstices in the fibre bundles are filled with lignin (stains red-violet with acidic phloroglucinol). In fibres of regenerated cellulose, lumen and lignin are absent, but the cross-sectional outline is usually characteristic, e.g. as commonly produced, cuprammonium rayon is smooth and circular but viscose rayon is deeply corrugated.

1.3 Physics

1.31 General Properties

Specific gravity: Varies with moisture content and method of measurement, e.g. cotton (dry, in He), *ca.* 1.56; do. (in water), *ca.* 1.60. Typical values, room-conditioned samples (measured

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for example, in toluene): jute, hemp, 1.48–1.49; flax, 1.50; viscose rayon, 1.52; cotton, ramie, highly-stretched rayon, 1.53–1.54; regenerated film, 1.53.

Refractive index and birefringence: Typical mean values,

	n	n_γ	n_α	$n_\gamma - n_\alpha$
Cotton	1.555	1.577	1.532	0.045
Flax, ramie	1.563	1.595	1.531	0.064
Viscose rayon	1.534	1.547	1.521	0.026†

† Higher when stretched.

Optical activity: Cellulose is not optically active in solution, but a complex that it forms with cuprammonium hydroxide exhibits laevorotation. **Fluorescence in u.v. light:** Bluish-white (bright); some samples (e.g. lignified fibres) show also a yellow tinge. **Moisture relations:** (i) *Water absorption (%)*: Regenerated foil (air-dry), over 100; do. (undried gel), 300. Vulcanized fibre, ca. 25. (ii) *Water retention of fibres*: Cotton and high-tenacity (saponified acetate) fibres, ca. 45%; mercerized cotton, ca. 50%; hemp, ca. 65%; flax, jute, ca. 70%; ordinary regenerated cellulose fibres, 80 to >100%. (iii) *Moisture regain of fibres*: Typical values, % at 25°C (desorption values in brackets):

R.h., %	10	30	65	90
<i>Cotton:</i>				
Grey	2.1 (2.5)	3.9 (4.5)	6.8 (8.2)	11.1 (14.3)
Kier-boiled	1.9 (2.6)	3.8 (4.7)	7.1 (9.0)	12.3 (15.3)
Kiered and bleached	2.0 —	3.7 (4.6)	6.8 (8.4)	11.8 (14.9)
Mercerized	2.4 (3.0)	4.6 (5.4)	8.5 (10.2)	14.3 (17.6)
<i>Viscose rayon:</i>				
Normal	3.5–4.0 (4.5–5.0)	6.5–7.5 (8.0–9.0)	12.0–13.5 (14–16)	20–23 (24.5–27.5)
High-tenacity	3.5 (4–5)	6.5–7.5 (7.5–8.5)	11.5–14.5 (13–15)	18.5–22.5 (22–26)
<i>Regenerated film:</i>				
Washed	4 (5)	7 (8.5)	11.5–13. (15)	22 (25)
<i>Bast and related fibres:</i>				
Flax	—	—	9.0	—
Hemp	—	—	9.8	—
Jute	2.8 (3.1)	5.3 (6.1)	10.5 (12.0)	> 18
Manila hemp	—	—	10.3 (11.3)	—
Sunn hemp	—	—	8.8 (9.9)	—

(iv) *Heat of wetting*: See §1.32. **Permeability**: Regenerated cellulose foil, unless specially coated, is permeable to water vapour, moderately permeable to alcohol vapour, but highly impermeable to hydrocarbon vapours (e.g. petrol, benzene). See Table 64.

1.32 Thermal Properties

Specific heat: 0.32 to 0.35. **Conductivity**: Cotton (compressed), *ca.* $5.5 \cdot 10^{-4}$ cal/cm/sec/°C; 'cotton wool' (loose), *ca.* $1 \cdot 10^{-4}$ cal/cm/sec/°C. **Effects of temperature**: Loses strength when maintained at elevated temperatures.† Time, in air at 150°C, for tendering to half-strength (typical values): Cotton, 70 hours, viscose rayon, 50 to 120 hours; hemp and manila ropes begin to char about this temperature. Decomposes in air (chars and ignites), without melting, *ca.* 270°C. **Heat of wetting**: Average results for fibres (cal/g dry material),

Initial Moisture Regain, %	0	2	7	10	20
Cotton, scoured and bleached	11.0	7.1	2.4	—	—
Do., mercerized	18	13.3	6.2	3.8	—
Viscose rayon	20-25	18-21	—	6-10	—
High-tenacity rayon, stretched	23	18	12	8	2.5
Do., saponified acetate	18.2	13.9	6.9	4.1	0.9
Flax	13	8	2.5	—	—

See also J. C. GUTHRIE, *J. Text. Inst.* 40 (1949) T489-T504.

The heat of adsorption of all cellulosic fibres at zero moisture regain is approx. 297 cal/g of liquid water absorbed.

1.33 Electrical Properties

Resistivity: Fibres, variable according to moisture content and purity of sample; typical values (not necessarily representative), ohm cm at 25°C,

R.h., %	65	90
<i>Cotton:</i>		
Grey yarn	<i>ca.</i> $5 \cdot 10^8$	$3 \cdot 10^4$
Purified yarn	<i>ca.</i> 10^9	$5-20 \cdot 10^4$
<i>Viscose rayon:</i>		
Filament yarn	$7 \cdot 10^6$	$2.5 \cdot 10^4$
Do., purified	$13 \cdot 10^6$	$5-20 \cdot 10^4$

† Hemp ropes lose moisture when exposed to hot dry conditions, and suffer temporary embrittlement with loss of strength.

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Dielectric strength (volts per mil): Cotton (cotton-covered wire), *ca.* 100 (declines with increase of humidity); vulcanized fibre (electrical grade), 175–300 (other grades, under 200).

Dielectric constant and power factor:

	Dielectric constant	Power factor
Pure cotton, dry	7	—
Do., 40% r.h.	—	<i>ca.</i> 0.1 (50 c/s), 0.03 (1 Mc/s)
Acetylated cotton, 40% r.h.	—	<i>ca.</i> 0.005 (50 c/s), 0.01 (1 Mc/s)
Regenerated film, dry	7.7 (50 c/s), 6.7 (1 Mc/s)	0.009 (50 c/s), 0.06 (1 Mc/s)

1.34 Mechanical Properties (at 65% r.h., 20°C, unless otherwise stated)

Elastic modulus and elastic recovery:

	Initial elastic modulus, kg/sq. mm.		Elastic recovery†, %					
			g/den. stress			% strain		
	Range	Measured example†	1	2	3	2	5	10
Cotton	600–1100	750 (55 g/den.)	60	41	34	74	45	—
Flax	2500–5000	2740 (203 g/den.)	78	71	66	65	—	—
Ramie	Do.	2260 (165 g/den.)	76	58	48	52	—	—
Jute	Do.	2600 (195 g/den.)	72	74	75	75	—	—
Hemp	Do.	3260 (245 g/den.)	64	55	50	50	—	—
Viscose rayon	600–900	890 (65 g/den.)	45	32	—	82	52	40
High-tenacity rayon	up to 2500	2360 (170 g/den.)	94	80	72	81	66	—
Regenerated film	400–1200‡	—						

† Values for 1 cm test length.

‡ According to direction.

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Tensile strength and elongation at break:

	Tensile strength, kg/sq. mm.			Extension at break, %	
	Approximate range	Typical values		Typical values	
	65% r.h.	65% r.h.	Wet	65% r.h.	Wet
Cotton	25-80 (1.8-5.8 g/den.)	50 (3.6 g/den.)	60 (4.3 g/den.)	6-8	ca. 10
Flax	50-100 (3.7-7.5 g/den.)	85 (6.3 g/den.)	90 (6.7 g/den.)	2	2.2
Ramie	90-95 (6.5-7 g/den.)	90 (6.5 g/den.)	108 (7.1 g/den.)	3.7†	—
Jute	40-80 (3.0-6.0 g/den.)	45 (3.4 g/den.)	—	1.8†	—
Hemp	—	70 (5.2 g/den.)	—	2.2†	—
Viscose rayon	23-30 (1.7-2.2 g/den.)	30 (2.2 g/den.)	15 (1.1 g/den.)	20-25	ca. 35
High-tenacity rayon	up to 80 (up to 6 g/den.)	60 (4.4 g/den.)	53 (3.9 g/den.)	6-8	ca. 8
Regenerated film‡	—	9	4	15 to 30	30

† Value for 1 cm test length, slightly lower for longer lengths.

‡ Measured along the length; across films strength is reduced by ca. 30%, extension increased up to fourfold.

Tensile strength of vulcanized fibre, 3.5 to 6 kg/sq. mm. **Flexural strength** (kg/sq. mm.): Vulcanized fibre (fabricated from cellulose, see §1.62), ca. 10. **Compressive strength** (kg/sq. mm.): Vulcanized fibre, 15 to 20. **Impact strength** (Izod test, ft lb per inch of notch): Vulcanized fibre, up to ca. 5. **Hardness**: Vulcanized fibre: Brinell 10; Rockwell, R50-80. **Frictional data**: Coefficients of friction (approximate values for yarns): μ_{static} ; cotton/cotton, 0.3 to 0.6; viscose rayon/viscose rayon, 0.2. μ_{dynamic} (mostly at 70 yd/min, 65% r.h.): grey or mildly scoured cotton or linen, or viscose rayon/mild steel, ca. 0.3; do. (lubricant free)/do., 0.6-0.8; do. (lubricated)/do., 0.15 to 0.35. Grey cotton/red vulcanized fibre, ca. 0.25. Cotton/hard glass or glazed porcelain, ca. 0.3; viscose rayon/do., ca. 0.5. For grey cotton yarn, running against

other materials, see also the Tables on: Cellulose acetate, Regenerated proteins, Polyamides (nylon), Rubber, Polyethylene, Polytetrafluoroethylene, Polystyrene, Polyvinyl formal, Polyvinyl chloride, Polyvinylidene chloride, Polymethyl methacrylate, Polyester resins (cross-linking type), Urea- and Phenol-formaldehyde resins, and Glass. **Fabrication:** Although a linear polymer, cellulose does not soften or melt before it chars, and therefore cannot be moulded as a thermoplastic material. It can, however, be fabricated as 'vulcanized fibre' (see §1.62) and sheets of this product can be cut, drilled, and machined satisfactorily. Most forms of cellulose can be stuck with glues of the types employed for wood or paper.

The spinning of fibres is dealt with under §1.21.

1.4 Serviceability and Utilization

General stability

Ageing effects under normal conditions—very slight. Degradation of M.W. and loss of strength occur on exposure in moist air to sunlight, the effect being greatly accelerated in the presence of certain dyes (e.g. red and yellow anthraquinonoid vat colours). For ageing or tendering at elevated temperatures, see §1.32; for data on photochemical degradation of fibres, see Table 82.

Fungous (mildew) or bacterial attack is favoured by dampness and requires the presence of appropriate nutrients. The effects are essentially confined to or initiated in surface foreign matter; pure dry cellulose is resistant. Viscose and cuprammonium rayons are attacked by silver-fish insects (*Lepisma saccharina* L.), but not by moth larvae.

Uses

Too widespread for simple enumeration. Natural fibres: Textiles and related materials, paper, fillers for imparting added strength to thermosetting plastics; also largely composing the universal structural material, wood. Regenerated forms: Rayons, for general purposes and industrial uses (e.g. tyre cords); clear films for packaging; and (from cuprammonium solution) a stiffening finish for heavy-duty fabrics. See also Additional Notes on Fibres, §1.61, and Vulcanized Fibre, §1.62.

1.5 History

The utility of flax and hemp twines was recognized in the Stone Ages, and linen (flax) fabrics have been found in the remains of

Neolithic lake dwellings in Switzerland. Later, it was the development of these fibres for sails and rigging (also made from leather) that made possible the maritime exploits and commerce of the ancient world. Flax was woven in ancient Egypt, is frequently mentioned in the books of the Old Testament and in Babylonian records, and both flax (λίνον) and hemp (καρναβίς) are clearly indicated in Greek literature.

The spinning of cotton (and flax) featured in the earliest Egyptian and Indian civilizations (3000 B.C.); cotton is mentioned in Hindu literature as early as 1500 B.C., and is referred to by Herodotus (445 B.C.). Europe was introduced to the fine cotton fabrics of India by Alexander the Great (356–323 B.C.) but the cotton industry of Lancashire, England, did not originate until towards the end of the eighteenth century. In America, fabrics of cotton (and of flax) have been unearthed from the tombs of ancient Mexico and pre-Inca Peru.

References to hemp, sunn hemp, and ramie (fibres cultivated in more temperate latitudes, and unknown to the Egyptians and Hebrews, or to the Greeks until later) occur in ancient Chinese writing as early as 2800 B.C., and in early Sanskrit literature. Jute appears to have been cultivated in Bengal from the most ancient times.

The first scientific approach towards elucidation of the composition of the structural matter in the vegetable kingdom was made by Payen, who investigated the substance over the years 1837–42 and named it cellulose. Some of the more prominent steps from that period are as follows: Solubility in cuprammonium hydroxide noted by Schweizer, 1857. Chain formula of condensed glucose molecules proposed by Tollens, 1883. X-ray diagrams obtained by Herzog and Jancke, 1920, and dimensions of the elementary cell first calculated by Polanyi, 1921. Following Emil Fischer's work on simpler carbohydrates, Haworth, in 1926, extended the ring structure to cellulose. This step, in conjunction with developments in the general concepts on polymeric substances, resulted in the present picture of the cellulose molecule.

On the more technical side Mercer, commencing *ca.* 1844 (first patent 1850), developed a process for treating cotton fabric with alkali to improve the dyeing properties and increase the strength and weight. (N.B. Mercerization under tension, as used today to impart lustrous effects, is an 1889 modification introduced by Lowe.) The earliest regenerated filaments were produced by J. Wilson Swan, in 1878, in connexion with the development of the first practical electric lamps (for which purpose the filaments