

***E. J. BEVAN,
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***A TEXT-
BOOK
OF PAPER-
MAKING***

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A Text-book of Paper-making

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PAPER-MAKING.

INTRODUCTORY NOTE.

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The raw materials of the paper-maker are primarily the vegetable fibrous substances; in addition to these there are various articles which are employed as auxiliaries, either in the preparatory or finishing processes to which these fibrous materials, or the web of paper are subjected. The latter class are of subsidiary importance, more especially from our present point of view.

In insisting upon the recognition of first principles, we cannot overrate the importance of a thorough grasp of the constitution of the plant fibres, as the necessary foundation for the intelligent conduct of paper-making, and to this subject we will at once proceed.

Careful study of a mature plant will show that it is made up of structural elements of two kinds, viz. fibres and cells, which, to use a rough parallel, we may liken in function to the bricks and mortar of a house. It is the former which admit of the many extended uses, with which we are familiar, in the arts of spinning and weaving, and which constitute the fabrics which are the most indispensable to our civilised life. For the

most part, as we know, fibres and cells are aggregated together into compound tissues, and a process of separation is therefore a necessary preliminary to the utilisation of the former. The cotton fibre is the only important exception to this general condition of distribution. Here we have the seed envelope or perisperm, converted into a mass {2} of fibres, and these by a spontaneous process accompanying the ripening, so isolated as to be immediately available. Next in order in point of simplicity of isolation, are those fibrous masses, or tissues, which, although components of complex structures, exhibit a greater cohesion of their constituent fibres than adhesion to the contiguous cellular tissues with which they go to build up the plant. Into such a tissue the "bast," or inner bark layer of shrubs and trees, more especially those of tropical and sub-tropical regions, frequently develops, and it is, in fact, this bast tissue, graduating in respect of cohesion of its constituent fibres, from a close network such as we have spoken of, to a collection of individual fibres or fibre-bundles disposed in parallel series, which supplies the greater part of the more valuable of the textile and paper-making fibres; we may instance flax, hemp, and jute, each of which is the basis of an enormous industry. According to the degree of adhesion of the bast to the contiguous tissues, or, in another aspect, according to its lesser aggregate development, so is the difficulty of isolation and the necessity of using processes

auxiliary to the mechanical separation of the tissue.

It is worthy of note here that the Japanese paper with which we are in these times so familiar, is prepared by the most primitive means from the bast of a mulberry (*Broussonetia papyrifera*); the isolated tissue, consisting of a close network of fibres, is simply cut and hammered to produce a surface of the requisite evenness, and the production of a web of paper is complete. In isolating the bast fibres employed in the textile industries, a preliminary partial disintegration of the plant stem is brought about by the process of steeping or retting, by which the separation of fibre from flesh or cellular tissue is much facilitated.

Last in order of simplicity of distribution, we have the fibres known to the botanist as the fibro-vascular bundles of leaves and monocotyledonous stems, these bundles being irregularly distributed through the main cellular mass, and consequently, by reason of adhesion thereto, much more {3} difficult of isolation. For this and other reasons, more or less in correlation with natural function, we shall find this class of raw material lowest in value to the paper-maker.

It is necessary at this stage to point out that the work of the paper-maker and that of the textile manufacturer are complementary one to the other, and the supply of fibrous raw material is correspondingly divided: it may be said, indeed, that the paper industry subsists upon the

rejecta of the textile manufactures. The working up of discontinuous fibre elements into thread, which is the purpose of the complicated operations of the spinner, is conditioned by the length and strength of these ultimate fibres. Paper-making, on the other hand, requires that the raw material shall be previously reduced to the condition of minute subdivision of the constituent fibres, and therefore can avail itself of fibrous raw material altogether valueless to the spinner, and of textile materials which from any cause have become of no value as such. To the raw materials of the paper-maker, which we have briefly outlined above, we must therefore add, as a supplementary class, textiles of all kinds, such as rags, rope, and thread.

Having thus acquired a general idea of the sources of our raw materials, we must study more closely the substances themselves, and first of all we must investigate them as we should any other chemical substance, i.e. we must get to understand the nature and properties of the matter of which the vegetable fibres are composed. While these exhibit certain variations, which are considerable, the substances present a sufficient chemical uniformity to warrant their being designated under a class name: this name is cellulose. The prototype of the celluloses is the cotton fibre.

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CHAPTER I.

CELLULOSE: THE CHEMICAL PROPERTIES OF TYPICAL MEMBERS OF THE CELLULOSE GROUP, WITH REFERENCE TO THEIR NATURAL HISTORY.

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Plants are so far built up of cellulose that it may be called the material basis of the vegetable world. Plant tissues, however, seldom, if ever, consist of pure cellulose, but contain besides, other products of growth either chemically combined with the cellulose or mechanically bound up with the tissue, which are, according to the nature of their union, removable either by means of fundamental chemical resolution or by the application of simple solvents. A general method for the isolation of cellulose consists in exposing the moist tissue to the action of chlorine gas or of bromine water in the cold, and subsequently boiling in dilute ammonia; repeating this treatment until the alkaline solution no longer dissolves anything from the tissue or fibre. The cellulose is then washed with water, alcohol, and ether, and dried. Obtained in this way, or in the form of bleached cotton, or of Swedish filter paper, it is a white substance, more or less opaque, retaining the microscopic features of the tissue or fibre from which it has been isolated. Its sp. gr. is 1.25-1.45. Its elementary composition is expressed by the percentage numbers (Schulze)

C	44·0	44·2
H	6·3	6·4
O	49·7	49·4

or by the corresponding empirical formula, viz. $C_6H_{10}O_5$.

These numbers represent the composition of the ash-free cellulose. Nearly all celluloses contain a certain proportion, {5} however small, of mineral constituents, and the union of these with the organic portion of the fibre or tissue is of such a nature that the ash left on ignition preserves the form of the original. It is only in the growing point of certain young shoots that the cellulose tissue is free from mineral constituents. (Hofmeister.)

As already indicated, cellulose is insoluble in all simple solvents; it is, however, dissolved by certain reagents, but only by virtue of a preceding chemical modification. An exception to this is to be found, perhaps, in the ammoniacal solution of cupric oxide (Schweitzer's reagent), in which it dissolves without essential modification, being recovered by precipitation, in a form which is chemically identical with the original, though differing, of course, in being structureless, or amorphous. This reagent may be employed in a variety of forms, but the following method of using it is to be recommended as the most certain in its results. The substance to be operated upon is intimately mixed with copper

turnings in a tube which is narrowed below and provided with a stopcock. Strong ammonia is poured upon the contents of the tube and, after being allowed to stand for some minutes, is drawn off and returned to the tube; the operation is several times repeated until the solution of the substance is effected. In order to facilitate the oxidation of the copper by the atmospheric oxygen, a current of air may be aspirated through the apparatus. The solution of the oxide prepared in this way is more effective in its action on cellulose than that obtained by dissolving the precipitated hydrate in ammonia. Cellulosic tissues in contact with this reagent are seen to undergo a disaggregation of their fibres, which swell up, become gelatinous, and disappear in solution. On adding an acid to the viscous solution, a precipitate of the amorphous cellulose is obtained in the form of a jelly resembling hydrated alumina; after washing and drying, it forms a brownish, brittle, horny mass. The cellulose is also precipitated upon simply diluting the viscous solution with water and allowing it to stand {6} 8-10 days in a closed vessel. From this observation it was inferred by Erdmann that the cellulose could not be considered as dissolved in the strict sense of the word, but the experiments of Cramer upon the osmotic properties of the solution proved this inference to be unfounded, and that cellulose is actually dissolved by the ammoniacal solution of copper oxide.

On treating the ammonio-cupric solution of cellulose with metallic zinc, this metal precipitates the copper, replacing it in the solution, and producing the corresponding ammonio-zincic solution of cellulose, which is colourless. Some of these solutions are lævo-gyrate.

Cellulose, in those forms to which the application of the term has been hitherto restricted, is a comparatively inert substance, and its reactions are consequently few. One of these is available for the identification of cellulose, and is chiefly used in the microscopical examinations of tissues: this is its reaction with iodine. Cellulose is not coloured blue by a solution of iodine excepting under the simultaneous influence of hydriodic acid, potassium iodide, sulphuric acid, phosphoric acid, or zinc iodide or chloride. The solution is prepared in the following way: zinc is dissolved to saturation in hydrochloric acid, and the solution is evaporated to sp. gr. 2.0; to 90 parts of this solution are added 6 parts potassium iodide dissolved in 10 parts of water; and in this solution iodine is dissolved to saturation. By this solution cellulose is coloured instantly a deep-blue or violet. For the identification of cellulose in the gross, mere inspection is usually sufficient; confirmatory evidence is afforded by an observation of the action of the ammonio-copper reagent, and of the absence of reaction with chlorine water. (See p. [18](#).)

Cellulose in its earlier stages of elaboration has no action upon light; but with age it acquires the property of double refraction, not, as has been shown by experiment, by virtue of its state of aggregation, but of its molecular constitution (Sachs). {7}

Animal Cellulose.

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—The mantles of many of the mollusca, e.g. the *Pyrosomidæ*, *Salpidæ*, and *Phallusia mammillaris*, contain a resistant substance which, after isolation by chemical treatment, has been found to be identical both in composition and properties with vegetable cellulose. Cellulose has also been stated to occur in degenerated human spleen and in certain parts of the brain.

Compounds of Cellulose.

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—The chemical inertness of cellulose is a matter of everyday experience in the laboratory, where it fulfils the important function of a filtering medium in the greater number of separations of solids from liquids. Its combinations with acids and with basic oxides are, as might be expected, few and of little stability. It has been shown by Mills that cellulose (cotton) in common with certain other organic fibrous substances, when immersed in dilute solutions of the acids or basic oxides, condenses these bodies within itself at the expense of the surrounding solution, which is proportionately weakened. This effect of concentration is sufficiently uniform and constant

to lead us to assign it to a chemical cause, and the view is strengthened by a consideration of the relative effects upon the various acids and bases which have been investigated, and brought to the following numerical expression:—
 Weight of cotton 3 gram. (with 6·9 per cent. H_2O and 0·05 per cent. ash)—i.e. 2·893 anhydrous fibre. Strength of solution about 0·5 gram. of the reagent in 250 cc.

	Temp.	Time.	Weight absorbed.
H_2SO_4	4° C. (39° F.)	3 min	0·00495
HCl	„	„	0·00733
NaOH	„	„	0·02020

The molecular ratio of the absorption, in the two latter, is $3\text{HCl} : 10\text{NaOH}$, and it is noteworthy that the same ratio was observed for silk.

Cellulose removes barium hydrate from its solution in wafer to form with it an insoluble compound. On adding lead acetate to the solution of cellulose in the ammonio-copper reagent, so prepared as to contain no carbonate, a {8} precipitate is obtained consisting of a compound of cellulose with lead oxide, but in variable proportions. The compound $\text{C}_6\text{H}_{10}\text{O}_5\text{PbO}$ is formed by the action of finely-divided lead oxide upon the above solution. Quite recently it has been shown (O'Shea, *Chem. News*, May 28th, 1886) that when dilute solutions of lead are passed through ordinary filter paper, a certain

amount is retained which cannot be removed by washing.

Cellulose does not combine with metallic salts, a fact which has been established incidentally to researches upon the mode of action of mordants.

The combinations of cellulose with acid radicles (ethereal salts) are both definite and stable.

Triacetyl Cellulose $[C_6H_7(C_2H_3O)_3O_5]$ is formed by treating cellulose with six times its weight of acetic anhydride at $180^\circ C.$ ($356^\circ F.$). The product of the reaction is a syrupy solution from which the compound in question separates on dilution with water as a white flocculent precipitate.

Triacetyl cellulose is insoluble in alcohol and in ether, but soluble in glacial acetic acid. It is easily saponified by boiling with a solution of potassium hydrate, the cellulose being regenerated. No derivative containing more than three acetyl groups has been obtained; but a mixture of the mono-and di-acetyl cellulose is formed in treating cellulose with only twice its weight of acetic anhydride, the formation of these bodies being unattended by their solution.

Whenever cellulose, in any form, is brought into contact with strong nitric acid at a low temperature, a nitro product, or a nitrate, is formed. The extent of the nitration depends upon the concentration of the acid, on the time of contact of the cellulose with it, and on the state of the physical division of the cellulose itself.

Knop, and also Kamarsch, and Heeren, found that a mixture of sulphuric acid and nitric acid also formed nitrates of cellulose; and still later (1847), Millon and Gaudin employed a mixture of sulphuric acid and nitrates of soda or potash, which they found to have the same effect. {9}

Several well characterised nitrates have been formed, but it is a very difficult matter to prepare any one in a state of purity, and without admixture of a higher or lower nitrated body.

The following are known:—

Hexa-nitrate, $C_{12}H_{14}O_4(NO_3)_6$,* gun cotton. In the formation of this body, nitric acid of sp. gr. 1.5, and sulphuric acid of sp. gr. 1.84 are mixed, in varying proportions, about 3 of nitric to 1 of sulphuric (sometimes this proportion is reversed), and cotton is immersed in this at a temperature not exceeding 10° C. (50° F.) for 24 hours: 100 parts of cellulose yield about 175 of cellulose nitrate. The hexa-nitrate so prepared is insoluble in alcohol, ether, or mixtures of both, in glacial acetic acid or in methyl alcohol. Acetone dissolves it very slowly. This is the most explosive gun-cotton. It ignites at 160°–170° C. (320°–338° F.). According to Eder the mixtures of nitre and sulphuric acid do not give this nitrate. Ordinary gun cotton may contain as much as 12 per cent. of nitrates soluble in ether-alcohol. The hexa-nitrate seems to be the only one quite insoluble in ether-alcohol.

* To represent the series of cellulose nitrates so as to avoid fractional proportions, the ordinary empirical formula is doubled and the nomenclature has reference to this double molecule.

Penta-nitrate, $C_{12}H_{15}O_5(NO_3)_5$. This composition has been very commonly ascribed to gun-cotton. It is difficult, if not impossible, to prepare it in a state of purity by the direct action of the acid on cellulose. The best method is the one devised by Eder, making use of the property discovered by de Vrij, that gun-cotton (hexa-nitrate) dissolves in nitric acid at about 80° – 90° C. (176° – 194° F.) and is precipitated, as the pentanitrate, by concentrated sulphuric acid after cooling to 0° C. (32° F.); after mixing with a larger volume of water, and washing the precipitate with water and then with alcohol, it is dissolved in ether-alcohol, and again precipitated with water, when it is obtained pure.

This nitrate is insoluble in alcohol, but dissolves readily {10} in ether-alcohol, and slightly in acetic acid. Strong potash solution converts this nitrate into the di-nitrate, $C_{12}H_{18}O_8(NO_3)_2$.

The tetra- and tri-nitrates (collodion pyroxyline) are generally formed together when cellulose is treated with a more dilute nitric acid, and at a higher temperature, and for a much shorter time (13 to 20 minutes), than in the formation of the hexa-nitrate. It is not possible to separate them, as they are soluble to the same

extent in ether-alcohol, acetic ether, acetic acid or wood spirit.

On treatment with concentrated nitric and sulphuric acids, both the tri- and tetra-nitrates are converted into penta-nitrate and hexa-nitrate. Potash and ammonia convert them into di-nitrate.

Cellulose di-nitrate, $C_{18}H_{13}O_8(NO_3)_2$ always results as the final product of the action of alkalis on the other nitrates, and also from the action of hot, somewhat dilute, nitric acid on cellulose. The di-nitrate is very soluble in ether-alcohol, acetic ether, and in absolute alcohol. Further action of alkalis on the di-nitrate results in a complete decomposition of the molecule, some organic acids and tarry matters being formed. The reactions and resolution products of this body have, however, been but slightly studied, and apparently not at all with the view to elucidate anything respecting the constitution of cellulose itself.

Cellulose and Chlorine.

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—Dry chlorine gas has no action upon cellulose, but the presence of water determines an indirect oxidising action. This oxidising action results from the decomposition of water by chlorine with formation of hydrochloric acid and liberation of oxygen. The reaction, however, can only take place in the presence of light.

Cellulose and Oxygen.

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—When cotton is exposed for some time to the action of a solution of bleaching powder, in the cold, and with access of air, e.g. when a piece of white calico is moistened with the solution, squeezed out and exposed to the air, it is found to undergo gradual disintegration accompanied by a change in composition, and an elementary {11} analysis of the product shows it to contain less carbon and more oxygen than the original cellulose. The following percentage numbers indicate the composition of these oxidised derivatives of cellulose, and the progress of the oxidation:—

	12 hours' exposure.	24 hours' exposure.
C	43·78 43·47	43·00 42·90
H	5·85 6·13	6·28 6·18
O	50·37 50·40	50·72 50·92

Other oxidising agents produce similar results; even by exposure to air and light, cellulose is slowly converted into these oxidised derivatives.² From their mode of formation, they have been termed oxycelluloses, and to distinguish them from a series of more highly oxidised derivatives, produced by the action of nitric acid upon cellulose, which they nevertheless resemble in many of their characteristics, the prefix α is employed. The following are the distinguishing features of the α oxycelluloses as represented by the more extreme of the above mentioned

products. It reduces Fehling's solution at the boiling temperature, and the cuprous oxide is deposited upon the fibre in a state of intimate union, producing the effect of an orange dye. It attracts the basic colouring matters from their solutions and is dyed to a full shade, the depth of colour being proportionate to the amount of oxidation to which the cellulose has been subjected. See also p. [43](#). Treated with a warm solution of phenylhydrazine salts in water, it is coloured a bright lemon-yellow. Its most remarkable property is its attraction for the vanadium compounds, which is so powerful that combination may be proved to take place when this element in the form of chloride is presented to the oxycellulose in an aqueous solution containing not more than 1 in 1,000,000,000,000 parts.

[2](#) Witz. Bull. Soc. Ind. Rouen, X. 416, and XI. 189.

The β oxycellulose resulting, as already indicated, from the action of dilute nitric acid upon cellulose, will be subsequently considered, under the head of the decompositions of cellulose, to which the reaction which we have been considering may be regarded as transitional. {12}

Decomposition of Cellulose.

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—*Acids.*—Of the decomposition of cellulose by artificial means, i.e. by the action of reagents, the most important are those which disclose the

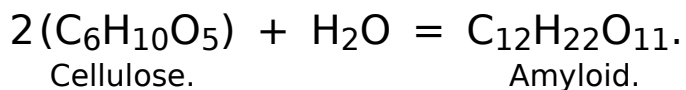
relationship which it bears to the group of so-called carbo-hydrates. Cellulose is, indeed, a highly *elaborated* carbo-hydrate, differing from dextrin and starch, not so much in its essential constitution as in the molecular arrangement which has been impressed upon it in and through the life of the plant. There are numerous observations in biological science which demonstrate the close connection, and in many cases the physiological equivalence of the members of this natural group, which includes the sugars, dextrin, starches, and cellulose. The following facts show the constitutional or chemical ties which underlie their interdependence.

Amyloid.

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—When cotton is treated with 30 times its weight of sulphuric acid somewhat diluted (1 part H_2O , 4 parts H_2SO_4) it quickly dissolves, the solution being at first gelatinous, becoming afterwards syrupy. On pouring this solution into water, a white flocculent precipitate is formed, consisting of a substance closely resembling the cellulose from which it is derived, differing, indeed, only in being amorphous, and in being coloured blue by iodine. This substance, from its resemblance to starch, has been called *amyloid*. When sufficiently dehydrated it constitutes a swollen starchy mass, which dries to a brittle transparent solid. This transformation appears to consist in a

simple hydration of the cellulose molecule, thus:—



The modification of cellulose, which occurs on the conversion of unsized paper into the so-called parchment paper, by exposure for a short time to the action of strong sulphuric acid, and subsequent washing and drying, consists doubtless in a superficial conversion of the cellulose into *amyloid*, or a body closely resembling it.

Hydracellulose.

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—Closely related in composition to the above {13} described amyloid, is the substance called hydracellulose, into which cellulose is converted by exposure for some time to the action of moderately strong sulphuric, or hydrochloric acids (sp. gr. 1.35). This substance is also formed by the action of very weak acids, if the exposure of the cellulose be sufficiently prolonged. Its formation is accompanied by the complete disintegration of the cellulose. If the action of strong sulphuric acid be prolonged, a further modification ensues, dextrin or sugar being formed. These products have not been sufficiently studied to have established their exact identity with any of the substances known under these group-names. The dextrin has a close resemblance to the dextrin obtained from

starch; the sugar is susceptible of fermentation; but beyond this nothing specific is known of these bodies. Their general properties, however, and mode of formation, sufficiently establish the fact that cellulose itself is constituted of similar molecules.

The action of zinc chloride solution upon cellulose is similar to that of sulphuric acid.

Cellulose in contact with iron, and in presence of air and moisture, is converted into a sugar and a gummy substance, which latter is converted into a sugar on boiling with dilute acids. Oxidised by potassium permanganate or bichromate in presence of acetic acid, it is converted into glucose, dextrin, and formic acid.

*Cellulose and nitric acid: β Oxycellulose.**

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—On digesting cellulose with nitric acid (sp. gr. 1.3) at 100° C. (212° F.), a considerable quantity of oxalic acid is formed; but after prolonged digestion, a residue remains, which yields but very slowly to the action of the acid. This substance, which is white and flocculent, when thrown upon a filter and washed with water, combines with the latter to form a gelatinous hydrate. It dissolves in dilute alkalis, but without forming a stable compound with the base. It usually amounts to about 30 per cent. of the cellulose acted upon. Its elementary composition is expressed by the formula {14} $C_{18}H_{26}O_{16}$ (C = 43.4, H = 5.3 per cent.). Treated with a mixture of concentrated sulphuric and nitric acids, it

dissolves, and, on pouring the solution into water, the compound $C_{18}H_{23}O_{13}(NO_3)_3$ separates as a white flocculent precipitate. In its essential properties therefore β oxycellulose exhibits a close resemblance to cellulose itself. This oxycellulose is distinguished from the members of the cellulose group by dissolving in concentrated sulphuric acid with development of a pink colour. It appears to stand in very close relationship to the group of pectic substances, which are oxidised derivatives of cellulose.

* Cross and Bevan, Chem. Soc. Journ., xliii. p. 23.

Alkalis.

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—Concentrated solutions of the caustic alkalis in the cold attack and disintegrate cellulose but slowly. The action is in many cases merely superficial. Dilute solutions of the alkalis are without action upon cellulose even at the boiling temperature, unless the condition of oxidation is super-added; in this case, acid bodies are found. The extreme product of the oxidation of cellulose in presence of the caustic alkalis is oxalic acid; this acid has indeed been prepared on the commercial scale by the “fusion” of cellulosic substances, with mixture of sodium and potassium hydrates. Malic acid has been obtained as an intermediate product of this decomposition. By potassium permanganate in presence of potassium hydrate, both in solution, finely divided cellulose is oxidised to a syrupy

mixture of acids resembling in composition and properties Frémy's meta-pectic acid. A similar decomposition is effected by concentrated solutions of the hypochlorites, employed cold.

Water at High Temperature.

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—Cellulose itself is only slightly affected by exposure to a temperature of 200° C. (392° F.) but in contact with water at this temperature, in sealed glass⁴ most part into dark brown insoluble products, a portion, however, passing into solution in the water, imparting to it a deep yellow colour. A large quantity of carbonic acid is {15} formed, and the solution contains in addition to formic acid an appreciable quantity of pyrocatechol. The formation of this aromatic substance from cellulose is, perhaps, the most noticeable feature of the decomposition.

⁴ The action of alkaline substances dissolved from the glass must be taken into account in this decomposition.

Decomposition by means of ferments, &c.

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—The soluble ferment of the foxglove has been found to convert cellulose into glucose and dextrin. The fluid from the vermiform appendix of the rabbit has also been found to digest cellulose with liberation of marsh gas and formation of a soluble compound which reduces cupric oxide in alkaline solution.

Humus.

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