

UNITED STATES PATENT OFFICE.

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FIBER-PRODUCING PROCESS AND RESULTING FIBER MATERIAL.

996,225.

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No Drawing.

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To all whom it may concern:

Be it known that I, VIGGO DREWSSEN, a citizen of the United States, and resident of the borough of Brooklyn, county of Kings, city and State of New York, have made certain new and useful Inventions Relating to Fiber-Producing Processes and Resulting Fiber Material, of which the following is a specification.

10 This invention relates to fiber producing processes and resulting fibrous material and relates especially to processes of producing fiber suitable for paper manufacture and other purposes from vegetable fiber material such as wood, straw, bagasse, bamboo, cornstalk, cotton, flax, ramie and other material as for example by boiling the same under pressure, preferably in the presence of free lime or similarly acting earthy alkali, in suitable strength treating solutions of loosely combined sulfur such as soluble polysulfids of the alkaline earths to decompose the vegetable material and form a sulfureted fibrous transition compound, and then if desired by eliminating organic sulfur components from this transition compound by treatment with a suitable sulfur eliminating agent.

30 The polysulfids treating solution may be formed by adding to water several per cent. or so of alkaline earth such as caustic lime which should preferably be present in sufficient amounts to maintain free alkali in the treating solution throughout the process. Finely divided sulfur to the extent of about twenty to forty per cent. of the incorporated lime may then be added to the treating solution and the vegetable fiber material boiled therein in a digester. As an illustrative method of carrying out this process spruce wood chips for example may be boiled under pressure in a digester for the desired time with a solution of calcium pentasulfid (CaS_5) in the presence of lime. A suitable treating solution for this purpose may be formed by incorporating caustic lime to the extent of fifty per cent. or so of the weight of the wood chips to be treated in about five hundred per cent. of their weight of water and adding thereto sulfur in finely divided condition to the extent of about ten to twenty per cent. of the weight of the wood chips. The wood chips may then be boiled in this treating solution for five or six hours or so at a pressure of about

six or seven atmospheres which seems to decompose the woody substance and form from the original wood a water insoluble fibrous sulfur transition compound of a complex organic nature. The treated chips may then be thoroughly washed with water which has a quicker action if hot in removing such of the organic fiber material as has been rendered soluble or removable in this way, the washed product being of a brown color, varying from a light to a dark chocolate brown, and containing a considerable proportion of combined sulfur. This treated material may then be readily disintegrated or separated into its individual fibers in any desired way as by running it through a beating engine or edge runners, producing strong brown fiber in which the strength of the original cellulose fibers is little if any impaired and which may be used without further treatment in many ways as for the manufacture of wrapping paper, cardboard, and so forth. The complex organic fibrous sulfur compound formed, is referred to as combined with the disintegrated fibrous material having readily separable fibers which is produced in this first stage process and which is for similar reasons spoken of as sulfureted, because the sulfur compound is probably chemically combined with the fibrous material, or if not is apparently intimately mechanically incorporated or physically combined therewith. This is indicated by the fact that it cannot be eliminated or dissolved out of the disintegrated fibrous transition compound even by the action of hot water, while it is eliminated therefrom by boiling with a suitable solution of sulfur eliminating agents, such as sodium carbonate which seems to render soluble or eliminate a considerable amount of organic sulfur compound from the material in the form of a dark brown liquor from which complex sulfureted compounds akin apparently to the sulfur dyes may be recovered. The organic character of the eliminated sulfur compound is also indicated by the fact that ordinary spruce wood gave yields of this fibrous transition product containing about 2½ to 3 per cent. of sulfur and suitable for paper making, as high as about 70 or 75 per cent. of the dry weight of the spruce wood, this fibrous transition compound being apparently intermediate between the raw

wood and the cellulose recoverable by ordinary processes to the extent of 50 per cent. or so.

The treated fiber, may, however, be further refined by treatment with a suitable sulfur eliminating agent as for instance by boiling the disintegrated fiber material with a suitable solution of sulfur eliminating agent such as hydrate, carbonate or sulfid of sodium or other suitable compound or salt preferably having a slight alkaline tendency and capable of eliminating the combined sulfur from the sulfureted fibrous compound without undesirably weakening the fiber. For example, sodium carbonate to the extent of about five to ten per cent. of the weight of the sulfureted fiber thus produced from spruce wood chips, may be dissolved in water to form a fairly concentrated solution and the fiber boiled therein until a dark brown liquor is produced which seems to contain such of the organic sulfur compound as has been eliminated, while the fiber is given a much lighter color and is refined so that when thoroughly washed and dried it contains about .35 or .40 per cent. of sulfur. It is of course unnecessary for many purposes that the sulfur absorbed during the treating process should be completely removed by the sulfur eliminating material, fiber containing considerable amounts of organic sulfur compound being available for many grades of paper for instance. The refined fibrous material which may of course be thoroughly washed and which if desired may be bleached in any desired way is a very strong fiber capable of utilization for high grade paper and other purposes and the yield of fiber by this process is unusually large. It is of course understood that it is unnecessary to form the polysulfids in the digester itself during the original treating process, since polysulfids of calcium or other similar earthy alkali may first be prepared separately and then the wood or other material treated in a suitable strength solution thereof, preferably having present enough free or available lime or similar earthy alkali to maintain a decidedly alkaline reaction throughout the treating process, which seems to minimize the formation of fast dyeing organic compounds that otherwise tend to form under some circumstances and permanently stain the fiber to an extent undesirable for some purposes.

Having described this invention in connection with a number of illustrative ingredients, proportions, processes, methods of preparation and products, to the details of which disclosure the invention is not of course to be limited, what is claimed as new and what is desired to be secured by Letters Patent is set forth in the appended claims.

1. The fiber producing process which con-

sists in boiling wood chips under pressure in an aqueous polysulfid treating solution formed by adding to water approximately ten per cent. of caustic lime and two to four per cent. of sulfur to decompose the woody material and form a brown sulfur transition compound, in washing and in mechanically disintegrating said transition compound and in boiling the same with an aqueous sulfur eliminating compound comprising sodium carbonate and in washing the refined fiber material.

2. The fiber producing process which consists in heating vegetable fiber material under pressure in an aqueous treating solution comprising a few per cent. at least of caustic lime and of sulfur combined therewith to form a polysulfid to decompose the vegetable fiber material and form a water insoluble fibrous sulfur transition compound and in eliminating the sulfur therefrom by treating with aqueous sulfur eliminating material.

3. The fiber producing process which consists in boiling woody fiber material under pressure in an aqueous treating solution formed by adding to water approximately two to four per cent. of sulfur and ten per cent. or more of alkaline earth capable of combining therewith to decompose the woody material and form a fibrous organic compound and in treating said fibrous compound with sulfur eliminating material.

4. The fiber producing process which consists in boiling woody fiber material under pressure in an aqueous treating solution formed by adding to water and combining caustic lime and sulfur to the extent of about twenty per cent. of the lime incorporated to decompose the woody material and form a fibrous organic compound and in treating said fibrous compound with sulfur eliminating material.

5. The fiber producing process which consists in heating vegetable fiber material under pressure in an aqueous treating solution comprising a few per cent. at least of caustic lime and sulfur to the extent of about twenty to forty per cent. of the lime incorporated to decompose the fiber material and form a fibrous sulfur transition compound and in eliminating sulfur therefrom by treating with aqueous sulfur eliminating material.

6. The fiber producing process which consists in heating vegetable fiber material under pressure in an aqueous treating solution comprising a few per cent. at least of alkaline earth capable of combining with sulfur and sulfur to the extent of twenty to forty per cent. of the alkaline earth incorporated for sufficient time to decompose the fiber material and form a sulfur transition compound therefrom and in eliminating sulfur from said sulfur transition compound by

treating the same with sulfur eliminating material.

7. The fiber producing process which consists in heating vegetable fiber material under pressure in an aqueous treating solution comprising sufficient earthy alkali capable of combining with sulfur to maintain an excess of alkali therein and a few per cent. of sulfur to decompose the fibrous material and form a water insoluble sulfur transition compound therefrom and eliminating sulfur from said sulfur transition compound by treating the same with sulfur eliminating salt solution.

8. The fiber producing process which consists in heating vegetable fiber material under pressure in an aqueous treating solution formed by adding to water caustic lime sufficient to maintain an excess of lime during the treating process and adding to the water sulfur to the extent of about twenty to forty per cent. of the lime incorporated to decompose the fiber material and form a fibrous organic compound and in treating said fibrous compound with sulfur eliminating material.

9. The fiber producing process which consists in boiling wood chips under pressure in an aqueous polysulfid treating solution formed from alkaline earth and sulfur to decompose the woody material and form a sulfur transition compound, in washing and disintegrating said transition compound and in heating the same with an aqueous sulfur eliminating compound comprising an alkali salt.

10. The fiber producing process which consists in heating vegetable fiber material under pressure in an aqueous polysulfid treating solution comprising an excess of free alkaline earth to decompose the fiber material and form a water insoluble fibrous sulfur transition compound and in eliminating sulfur therefrom by treating with aqueous sulfur eliminating material.

11. The fiber producing process which consists in boiling woody fiber material under pressure in an aqueous polysulfid treating solution to decompose the woody material and form a brown sulfur transition compound and in eliminating the sulfur therefrom by heating with an aqueous sulfur eliminating compound comprising an alkali salt.

12. The fiber producing process which consists in boiling vegetable fiber material under pressure in an aqueous treating solution comprising a polysulfid of an alkaline earth to decompose the fiber material and form a water insoluble fibrous sulfur transition compound and in eliminating the sulfur from said transition compound by heating with aqueous sulfur eliminating material.

13. The fiber producing process which

consists in boiling vegetable fiber material under pressure in an aqueous treating solution comprising a polysulfid to decompose the fiber material and form a water insoluble sulfur organic compound and in eliminating the sulfur from said organic compound by heating with aqueous sulfur eliminating material.

14. The fiber producing process which consists in heating wood fiber material under pressure in a polysulfid treating solution to decompose the fiber material and form a water insoluble sulfur compound and in eliminating the sulfur compound from said fiber by treating with sulfur eliminating material.

15. The fiber producing process which consists in heating vegetable fiber material under pressure in an aqueous treating solution comprising considerable proportions of sulfur and of alkaline earth capable of combining therewith to decompose the vegetable fiber material and form a water insoluble sulfur transition compound and in eliminating the sulfur therefrom by treating with sulfur eliminating material.

16. The fiber producing process which consists in heating vegetable fiber material under pressure in an aqueous treating solution comprising sufficient alkaline earth capable of forming polysulfids in combination with sulfur to have free alkali present during the treating process and incorporated sulfur to the extent of about twenty to forty per cent. of the alkaline earth incorporated to decompose the vegetable fiber material and form a fibrous organic compound therefrom, and in eliminating sulfur therefrom.

17. The fiber producing process which consists in treating vegetable fiber material under pressure in an aqueous treating solution formed by adding to water a considerable proportion of sulfur and sufficient alkaline earth to maintain an excess of earthy alkali throughout the treating process to decompose the fiber material and form a fibrous sulfur transition compound and in eliminating sulfur therefrom by treating with sulfur eliminating material.

18. The fiber producing process which consists in boiling vegetable fiber material under pressure in an aqueous treating solution formed by adding to water approximately ten per cent. of caustic lime and two to four per cent. of sulfur to decompose the fibrous material and form a fibrous organic compound.

19. The fiber producing process which consists in heating vegetable fibrous material under pressure in an aqueous treating solution comprising a considerable proportion of lime and sulfur to the extent of about twenty to forty per cent. of the lime to decompose the vegetable fiber material.

20. The fiber producing process which consists in heating vegetable fiber material under pressure in an aqueous treating solution comprising several per cent. at least of
5 alkaline earth capable of combining with sulfur and sulfur to the extent of about twenty to forty per cent. of the alkaline earth incorporated to decompose the fibrous material and form a fibrous organic compound.
- 10 21. The fiber producing process which consists in heating raw vegetable fiber material under pressure in a polysulfid treating solution comprising an excess of alkaline earth
15 to decompose the fiber material and render its fibers readily separable and form a water insoluble fibrous organic sulfur compound.
- 20 22. The fiber producing process which consists in boiling raw wood fiber material under pressure in a treating solution comprising a polysulfid of an alkaline earth to decompose the fiber material and render its
25 fibers readily separable and form a fibrous organic sulfur compound.
- 30 23. The fiber producing process which consists in boiling vegetable fiber material under pressure in an aqueous treating solution comprising an alkaline earth polysulfid to decompose the fiber material and form a water
35 insoluble fibrous organic sulfur compound.
- 40 24. The fiber producing process which consists in boiling wood fiber material under pressure in an aqueous treating solution comprising a polysulfid of an alkaline earth
45 to decompose the woody material and form a fibrous organic sulfur compound.
- 50 25. The fiber producing process which consists in boiling raw wood fiber material under pressure in an aqueous treating solution comprising a polysulfid of lime to decompose the woody material and form a
55 brown fibrous sulfur organic compound.
- 60 26. The fiber producing process which consists in boiling wood fiber material under pressure in an alkaline aqueous treating solution comprising a polysulfid to decompose the woody material and form a fibrous
65 brown sulfur organic compound.
27. The fiber producing process which consists in boiling wood fiber material under pressure in an alkaline polysulfid treating solution formed by adding to water caustic lime and sulfur to decompose the fiber material and form a brown fibrous sulfur organic compound.
28. The fiber producing process which consists in heating raw vegetable fiber material under pressure in an aqueous treating solution comprising a polysulfid of an alkaline earth to decompose the fiber material and form a water insoluble sulfureted fibrous organic compound.
29. The fiber producing process which consists in boiling vegetable fiber material under pressure in an aqueous treating solution comprising calcium polysulfid to decompose the vegetable fiber material and form a water insoluble sulfureted fibrous organic compound.
30. The fiber producing process which consists in heating vegetable fiber material under pressure in an aqueous treating solution comprising considerable proportions of sulfur and of alkaline earth capable of combining therewith to decompose the vegetable
70 fiber material and form a water insoluble sulfureted organic compound.
31. The fiber producing process which consists in boiling wood fiber material under pressure in a polysulfid of lime treating solution until a sulfureted fibrous organic compound is formed and the material is disintegrated and the fibers rendered readily
75 separable and in thereby eliminating not more than 30 per cent. of the dry weight of the wood fiber material therefrom.
32. The fiber producing process which consists in boiling wood fiber material under pressure in a polysulfid treating solution until a sulfureted fibrous organic compound is formed and the material disintegrated and its fibers rendered readily separable and in eliminating not more than 40 per cent. of the
80 dry weight of the wood fiber material therefrom.
33. The fiber producing process which consists in heating raw wood fiber material under pressure in an alkaline polysulfid treating solution until the material is disintegrated and the fibers sulfureted and rendered readily separable and in eliminating
85 not more than 40 per cent. of the dry weight of the wood fiber material therefrom.
34. The sulfureted wood fiber material whose fibers are strong and readily separable and which is made from wood by disintegrating the same by treatment in a polysulfid treating solution and by eliminating not more than forty per cent. of the dry weight
90 of the wood therefrom.
35. The sulfureted wood fiber material containing a few per cent. of sulfur and comprising a brownish sulfur organic compound largely insoluble in hot water, but which may be largely removed from the fiber material by treating with an aqueous solution of sulfur eliminating salt, said sulfureted wood fiber material having strong readily
95 separable fibers and representing more than 70 per cent. of the dry weight of the wood from which it was made.
36. The sulfureted wood fiber material having a brownish color and containing a considerable proportion of sulfur combined therewith, the individual fibers of said wood fiber material being strong and readily separable and said wood fiber material representing more than 65 per cent. of the dry weight of the wood from which it was made.
37. The brownish sulfureted wood fiber

material whose individual fibers are strong and readily separable and available for paper making, and which contains a few per cent. of combined sulfur which cannot be eliminated therefrom by washing with hot water but which may be largely removed therefrom by aqueous sulfur eliminating material, said sulfureted wood fiber material representing more than 65 per cent. of the dry weight of the wood from which it was made.

38. The sulfureted wood fiber material whose fibers are strong and readily separable and which is made from wood by disintegrating the same by boiling under pressure in a strong polysulfid of lime treating solution in the presence of excess lime and by eliminating sulfur organic material therefrom by treating with an aqueous solution of sulfur eliminating salt.

39. The brownish sulfureted wood fiber material whose fibers are readily separable and possess a large proportion of the strength of the original cellulose fibers and which contains more than 2 per cent. of combined sulfur which cannot be eliminated therefrom by washing with hot water but which may be largely removed therefrom by treating with aqueous sodium carbonate sulfur eliminating material.

40. The sulfureted wood fiber material in which the individual fibers may be readily separated and possess a large proportion of the strength of the original cellulose fibers, said wood fiber material representing more than 65 per cent. of the dry weight of the wood from which it was made and containing a considerable proportion of sulfur combined therewith which may be largely removed therefrom by boiling with aqueous sodium carbonate sulfur eliminating material.

41. The sulfureted vegetable fiber material whose fibers are strong and readily separable and suitable for paper making and which represents more than 70 per cent. of the dry weight of the vegetable fiber from which it was made.

42. The brownish vegetable fiber material containing a considerable amount of combined sulfur which cannot be eliminated therefrom by washing with hot water but which may be eliminated therefrom by heating with aqueous sulfur eliminating material, the component fibers of said vegetable fiber material being readily separable and possessing considerable strength.

43. The brownish vegetable fiber material containing a considerable proportion of combined sulfur which cannot be eliminated therefrom by washing with hot water but which may be substantially eliminated therefrom by boiling with aqueous sulfur eliminating material, the component fibers of said vegetable fiber material being readily

separable and possessing a large proportion of the strength of the original cellulose fibers.

44. The vegetable fiber material having a brownish color and containing a considerable proportion of sulfur combined therewith, the individual fibers of said vegetable fiber material being readily separable and possessing substantially the full strength of the original cellulose fibers.

45. The vegetable fiber material in which the individual fibers may be readily separated and possess a large proportion at least of the strength of the original cellulose fibers, said vegetable fiber material containing an appreciable amount of sulfur combined therewith that may be eliminated therefrom by treating with aqueous sulfur eliminating material.

46. The sulfureted vegetable fiber material having a brownish color and whose fibers possess substantially the full strength of the original cellulose fibers and comprising a considerable proportion of combined sulfur that may be eliminated therefrom by heating with aqueous sulfur eliminating material, such vegetable fiber material being produceable from raw vegetable fiber material by heating the same in an aqueous treating solution comprising polysulfid of lime and an excess of caustic lime until decomposition is effected.

47. The sulfureted vegetable fiber material having a brownish color and whose fibers possess a large proportion of the strength of the original cellulose fibers and containing a considerable proportion of combined sulfur that may be eliminated therefrom by heating with aqueous sodium carbonate sulfur eliminating material, such fibrous material being produceable by heating raw vegetable fibrous material in an aqueous treating solution comprising polysulfid of lime and an excess of caustic lime until decomposition is effected.

48. The vegetable fiber material whose fibers possess a large proportion of the strength of the original cellulose fibers and comprising a considerable proportion of combined sulfur which cannot be eliminated therefrom by washing with hot water, such vegetable fibrous material being produceable from raw vegetable fiber material by heating the same in an aqueous treating solution comprising a polysulfid until decomposition is effected and a fibrous organic sulfur compound produced and sulfur eliminated therefrom by treatment with aqueous sulfur eliminating material.

49. The sulfureted vegetable fiber material comprising an appreciable amount of sulfur in the form of an organic compound which cannot be eliminated therefrom by washing with hot water, the individual fibers of the vegetable fiber material being

separable and possessing a large proportion of the strength of the original cellulose fibers, such vegetable fiber material being produceable from raw vegetable fiber material heated in an aqueous treating solution comprising a polysulfid of an alkaline earth to cause decomposition.

50. The brownish sulfureted vegetable fiber material containing a considerable portion of sulfur in the form of a brownish organic compound, the individual fibers of the vegetable fiber material being readily separable and possessing a large proportion of the strength of the original cellulose fibers, said vegetable fiber material being produceable from raw vegetable fiber material heated in an aqueous treating solution comprising a polysulfid of an alkaline earth to cause decomposition.

51. The brownish vegetable fiber material whose fibers possess a large proportion of the strength of the original cellulose fibers and which contains an appreciable amount of combined sulfur which may be eliminated therefrom, said vegetable fibrous material being formed from raw vegetable fiber material heated in an aqueous treating solution comprising a polysulfid until decomposition is effected.

52. The vegetable fiber material whose fibers are readily separable and possess a large proportion of the strength of the original cellulose fibers and containing an appreciable amount of sulfur combined therewith and which cannot be eliminated therefrom by washing with hot water, said vegetable fiber material being formed from raw vegetable fiber material by heating the same in an aqueous treating solution comprising polysulfid of lime and an excess of caustic lime until decomposition is effected.

53. The vegetable fiber material whose

fibers are readily separable and possess a larger part at least of the strength of the original cellulose fibers and formed from vegetable fiber material by heating the same in an aqueous treating solution comprising polysulfid of an alkaline earth until decomposition is effected and a fibrous organic compound produced and the sulfur at least largely eliminated therefrom by sulfur eliminating material.

54. The brownish sulfureted vegetable fiber material whose fibers possess a large proportion of the strength of the original cellulose fibers and which contains more than two per cent. of combined sulfur which cannot be eliminated therefrom by washing with hot water but which may be largely removed therefrom by treating with aqueous sodium carbonate sulfur eliminating material.

55. The brownish sulfureted vegetable fiber material whose individual fibers are readily separable and possess a large proportion of the strength of the original cellulose fibers and which contains a few per cent. of combined sulfur which cannot be eliminated therefrom by washing with hot water but which may be largely removed therefrom by aqueous sulfur eliminating material.

56. The sulfureted strong vegetable fiber material containing a few per cent. of sulfur and comprising a brownish sulfur organic compound largely insoluble in hot water, but which may be largely removed from the fiber material by treating with an aqueous solution of sulfur eliminating salt.

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Witnesses:

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