

UNITED STATES PATENT OFFICE.

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METHOD OF TREATING FIBROUS VEGETABLE SUBSTANCES TO OBTAIN FIBER SUITABLE FOR PAPER-
MAKING AND FOR TEXTILE AND OTHER PURPOSES.

SPECIFICATION forming part of Letters Patent No. 260,749, dated July 11, 1882.

Application filed June 5, 1882. (No specimens.) Patented in England July 13, 1881, No. 3,062; in France November 10, 1881, No. 145,744, and in Belgium November 12, 1881, No 56,244.

To all whom it may concern:

Be it known that I, CARL DANIEL EKMAN, of the city of London, in the United Kingdom of Great Britain and Ireland, have invented certain Improvements in the Method of Treating Fibrous Vegetable Substances in order to Obtain Fiber Suitable for Paper-Making and for Textile and other Purposes; and I do hereby declare that the following specification is a full, clear, and exact description thereof.

In the Letters Patent heretofore granted to me, dated February 7, 1882, No. 253,357, I have described a method of treating wood in order to obtain fiber suitable for paper-making. My present invention is predicated upon the process described in said Letters Patent, and is subordinate thereto; and it consists in the adaptation of the same to the treatment of vegetable substances other than wood, so as to reduce them to a state useful in the manufacture of paper, or to obtain fiber or filaments suitable to be spun into yarn for the manufacture of textile fabrics or felted goods.

Inasmuch as the treatment required to be given to different plants varies considerably according to their respective peculiarities and the amount of resistance which is encountered in dissolving the incrusting matter which binds the fibers together, and varies also with the uses to which the fiber when obtained is to be applied, it will be convenient to describe the process which I employ, first, in its application to the production of material suitable for paper-making, and, secondly, to the obtaining for the manufacture of textiles the ultimate fibers from plants, or the compound fibers making filaments from the same plants, composed of a number of the ultimate fibers cemented together by the incrusting matter which the plants contain.

As described in the said Letters Patent dated February 7, 1882, the process hereinafter described may be conducted by the aid of a boiler capable of being tightly closed and surrounded by a jacket, within which, and around the outside of the boiler proper, steam can be admitted by means of pipes connecting a steam-generator with the space formed by such surrounding jacket.

The boiler or digester should be lined with lead or a suitable material capable of resisting the corrosive action of chemical agents, and it should also be provided with an escape-valve, to enable from time to time injurious gases or resultants formed from the chemicals to be discharged.

It will generally be found convenient to use a vertical cylindrical boiler of, say, four feet inside diameter and twelve feet between the two ends, and to facilitate the filling and emptying of the same it should be mounted on trunnions, so as to be readily turned on its bearings.

For paper-making, if the incrusting matter in the vegetable plants selected to be reduced to pulp is to be completely dissolved, the treatment is similar to that which is described in my said patent for the treatment of wood; but it is not necessary in general to crush or mechanically disintegrate the plants, though it would be advisable to cut them into short pieces. The pressure, too, at which the boiling is to be conducted and the length of time for which it is to be continued differ from that required when wood is to be dealt with. Neither are so strong chemicals essential, and in proportion as the pressure required is below that necessary for reducing wood to pulp the total duration of the whole boiling process will be shortened.

I will now, by way of illustration, describe the process for treating straw and esparto in order to obtain fiber for paper-making.

After the raw material has been suitably cleansed it is put into the boiler or digester, and completely covered with or submerged in a liquid solution containing sulphurous acid and magnesia or other alkaline substitute for the magnesia—as soda—possessing substantially the equivalent chemical properties both with respect to its reaction upon the sulphurous acid and its effect upon the fiber of the plant. The solution used may contain only about 0.45 per cent. magnesia or equivalent alkali and about 0.85 per cent. of sulphurous acid, although a stronger solution will generally be preferable. The boiler having been closed tight, steam is let into the jacket until the pressure within

the digester or boiler proper has reached, say, fifty pounds to the square inch, when it should be continued at this pressure for about three hours. In case any sulphurous acid has been set free from the magnesia it will become converted under the continued influence of heat and pressure into sulphuric acid, and therefore it should be blown off through the escape-valve of the boiler; otherwise it will blacken or discolor the material under treatment. Below a pressure of fifty pounds, however, there is little liability of the existence of free sulphurous acid. After the boiling has been completed the steam is blown off and the contents of the boiler are turned out. The fiber resulting from the process is then to be washed and treated in the usual or in any other suitable way.

The strength of the solution of sulphurous acid and magnesia or equivalent alkaline agent, the degree of pressure in the boiler, and the duration of the boiling operation are to be varied according to the necessities of the particular case, for the reason that plants which are compact in structure or resinous in character require more vigorous treatment than those which are soft or free from resinous matter.

I have discovered that there is a special value in the employment of a solution composed of sulphurous acid and magnesia in obtaining from plants fibers or filaments to be used in the manufacture of textiles, from the fact that the action of the magnesia is specifically to render the fiber soft and pliable, and quite the opposite of the effect of the lime hitherto used, which leaves the fiber harsh and difficult to be spun or worked by machinery. In applying my process for this purpose the raw material employed must be such as yields fibers of sufficient length—as, for example, flax or hemp—and the process can be so adapted to the end sought that there can be obtained either the ultimate fibers or filaments composed of many ultimate fibers cemented together. If fibers are to be obtained which are wholly freed from incrusting matter, the solution of sulphurous acid and magnesia, or alkaline substitute having like properties as magnesia, should be of greater strength, and the boiling operation be conducted for a greater length of time and under a greater pressure than will be proper in case filaments are to be obtained composed of a number of fibers. If only long filaments are desired, it will be well to use a more basic solution by adding magnesia in a finely-divided state, or a more basic compound of sulphurous acid and magnesia. Otherwise stated, although the general process is in all cases the same and the plants are to be boiled under pressure in a close boiler while submerged in a solution of the character described, the action of which is to dissolve the incrusting matter which binds together the component fibers, and also to render soft and pliable the body of the fibers themselves, the treatment should have regard to the structure

of the plant, to the nature of the gummy incrustation, and to the product that is desired to be obtained, and the strength of the solution, the duration of the boiling operation, and the degree of pressure to which by heat the solution is raised should be adapted to the requirements of the case. In treating flax, for example, either fine, white, soft, silky, but short fibers can be obtained, or coarser, darker, stiffer, but longer fibers or filaments, with the different gradations lying between the extremes, all depending on the treatment being varied and adapted in the manner referred to. If the solution used contains 1.4 per cent. of magnesia and 4.2 per cent. of sulphurous acid, and the boiling is carried on with about fifteen pounds pressure for about two hours and a half, coarser filaments of a darker color are obtained; but if the boiling is carried on with thirty pounds pressure for about three hours the filaments obtained are finer, more flexible, of improved appearance, and rather lighter in color. A good result can even be obtained by using a solution containing only 0.25 per cent. of magnesia and 0.8 per cent. of sulphurous acid and boiling about three hours with forty pounds pressure. Even sulphite of magnesia (the single salt) may be used; but since that salt is little soluble in water a salt is preferred containing so much sulphurous acid that it dissolves easily in the requisite or a less quantity of water. When this neutral salt is used it is strewn on the flax as it is placed in the boiler, so that it may be pretty evenly spread, and then the boiler is afterward filled up with water to the same height as when a solution is used. Two per cent. of single sulphite of magnesia and boiling for three hours with fifty pounds pressure will yield fiber of a fair quality.

For obtaining filaments that are soft, silky, lustrous, and of pale color, the boiling is or may be carried on with seventy pounds pressure for about three hours with a solution containing 1.6 per cent. of magnesia and 3.8 per cent. of sulphurous acid, and (mixed with the flax) about one per cent. of weight of the water of caustic magnesia finely divided.

When only a small proportion of the incrusting matter is required to be dissolved very small quantities of the chemicals may be used. Even 0.3 per cent. of the water's weight of neutral sulphite of magnesia has a perceptible effect; but as a rule it is preferred to have no less than one per cent. of this salt. A solution of 0.035 per cent. of magnesia and 0.1 per cent. of sulphurous acid can be used; but a stronger solution is preferred. Even as small a pressure as three pounds will have some effect; but it is advisable to work with no less pressure than ten pounds.

The foregoing example for the treatment of flax will serve as a guide for the treatment of other plants containing fibers suitable for textile purposes, especially hemp.

I do not claim in this patent the invention

set forth and claimed in the said Letters Patent heretofore granted to me, dated February 7, 1882, No. 253,357; but

What I do claim herein is—

- 5 The method, substantially as hereinbefore described, of treating fibrous vegetable plants for wholly or partially resolving their fibrous constituents, which consists in boiling such

substances under pressure in a solution containing sulphurous acid and magnesia or other 10 alkaline equivalent having the properties of magnesia, as set forth.

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Witnesses:

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