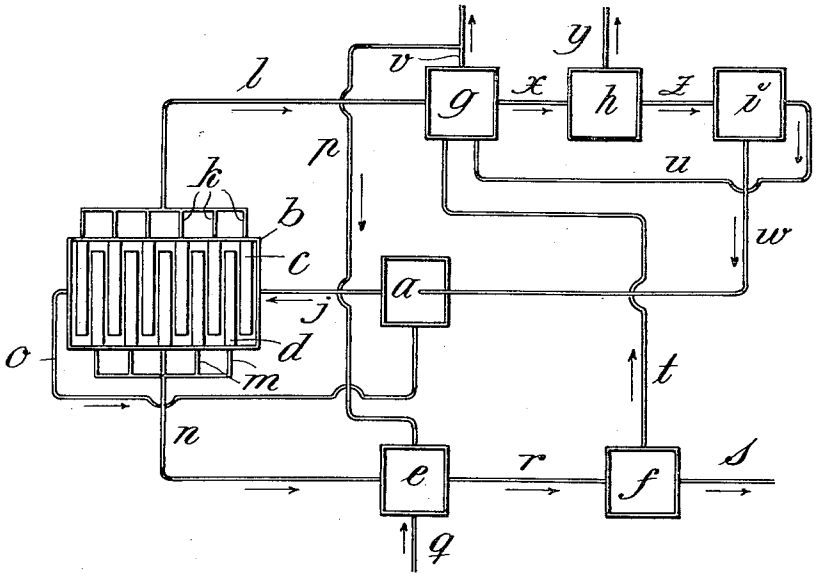
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BY-PRODUCTS RECOVERY IN THE DIGESTION OF CELLULOSE MATERIALS

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BY-PRODUCTS RECOVERY IN THE DIGESTION OF CELLULOSE MATERIALS

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My invention has for its object to facilitate the production of cellulose by a more complete utilization than heretofore of the raw material, such as wood, by conducting the process in such a manner that the component parts of the raw material, which are dissolved during boiling, are collected in a simple and economical way, while at the same time the chemicals used during the boiling operation are recovered from the lye, whereby the production of waste lyes, injurious to rivers and lakes, is prevented.

It has been suggested to effect a similar result by electrolysis of waste lyes from the production of sulphite cellulose directly or at the production of sulphate cellulose first after the neutralization by means of an acid of the excess alkali, in such a manner that the organic matters dissolved first are partially precipitated.

My process on the contrary is based upon an electrolysis of only a soluble sulphate of an alkali, especially sodium sulphate and the use in the subsequent boiling of cellulose or treatment of the lye of the solutions of alkaline hydrate or sulphuric acid formed.

The solution of the alkaline hydrate formed in the electrolysis of a solution of alkaline sulphate is utilized in the boiling operation for the production of sulphite cellulose, after having been transformed into bisulphite of alkali by addition of sulphurous acid.

For the production of sodium cellulose the solution of alkaline hydrate as such or after a partial transformation into sulphohydrate is used.

The solution of sulphuric acid formed at the electrolysis of the solution of alkaline sulphate is supplied to the lye after the boiling of the cellulose, when that product has been separated from the lye. The alkali, which has taken part in the boiling operation and formed organic combinations, is hereby again transformed into sulphate, as also the alkali remaining in the lye in the form of bisulphite or sulphite. The quantity of the solution of sulphuric acid from the electrolysis corresponds to the alkali, which as alkaline hydrate has been formed during that process and been brought over to the boiling operation, so that, when the boiling lye and the solution of sulphuric acid obtained at the electrolysis after the boiling are mixed, the same quantity of alkaline sulphate is generated as was decomposed at the electrolysis. The alkaline sulphate thus regenerated in the lye after the boiling is crystallized (for the purpose of being utilized if desired), but first after having been subjected to the treatment described hereinafter for separation of the organic substances contained in same.

A lye of the composition stated above, used for sulphite boiling, and to which has been added the electrolytically separated solution of sulphuric acid, does not have a concentration presenting any essential difference from another sulphite lye, resulting directly from the boiling process, although in the former one the alkaline combinations have been transformed into sulphates, and the lye moreover contains free combinations of sulfonic acids. If to such a lye (which already contains a solution of sulphuric acid from the electrolysis) such a quantity of sulphuric acid is added, that the solution will contain an excess of free sulphuric acid, the organic substances of the lye will become decomposed by a concentration of the lye by heating, as soon as the free sulphuric acid has reached a certain degree of concentration. The said organic substances hereby will be precipitated in a form, facilitating an easy filtration and a washing of the precipitate. When these organic precipitated substances thus are separated from the filtrate, the latter contains besides free sulphuric acid also alkaline sulphate and organic substances, the quantity of which depends upon the degree to which such substances have been previously brought. From this solution the alkaline sulphate is crystallized, removed and brought back to the electrolysis, whereupon the mother lye containing sulphuric acid is added to a new quantity of lye from the boiling operation for the purpose of precipitation of the organic substances contained therein.

From the above it will be understood, that the excess of free sulphuric acid used is not

lost or consumed but that it only serves as an interagent at the decomposition of the organic substances, a decomposition which can take place at a temperature under 100° C. At the concentration and the decomposition also volatile acids, such as formic acid and acetic acid, escape and also sulphurous acid which is used in the boiling operation for the formation of bisulphite, while the two other acids are collected and utilized per se.

If the boiling lye arises from the boiling of soda cellulose, the solution of alkaline hydrate obtained by the electrolysis of a solution of alkaline sulphate has been utilized either directly at the boiling or after having first been transformed into sulpho-hydrate or been mixed with such hydrate or other sulphur combinations. When to this lye arriving from the boiling, a solution of sulphuric acid from the electrolysis of the alkaline sulphate is added, the alkali in the alkaline combinations of the lye is converted into sulphate, whereby, however, sulphuretted hydrogen can be generated, if the boiling lye contains sulphohydrate or combinations of sulphur. Organic substances which are difficult to filter are also precipitated.

The sulphuretted hydrogen generated is introduced into an alkaline hydrate to be used at the boiling of cellulose, whereby a sulphohydrate for the boiling is obtained. The organic substances, difficult to filter, are allowed to follow the lye to the next operation.

To the soda-cellulose-lyes created as now described there is according to my invention added a quantity of sulphuric acid in excess (as is already stated in the description of the treatment of the sulphite cellulose lye), whereupon the lye is concentrated. At that addition of sulphuric acid in excess and a concentration of the mixture the organic substances are precipitated in such a condition that they can easily be filtered and washed. They are then separated from the mixture. From the remaining solution sulphate of alkali is obtained by crystallization, whereupon the mother lye with its content of sulphuric acid can be used again for a new precipitation of organic substances. The dissolved organic substances in the lyes of sulphite cellulose or of soda cellulose can be precipitated more or less completely or in fractions by a suitable adjustment of their concentration and the quantity of excess of acid used.

The process is schematically illustrated in the accompanying drawing, which represents a plan of an establishment for its execution under presumption that the boiling process is executed with sodium as base and sulphate of soda as electrolyte at the electrolysis. Other bases than sodium, however, may of course be used.

a is a container for a solution of sulphate of soda, which solution also can be prepared in the container by dissolving crystals of sodium, *b* is an electrolytic apparatus connected to that container and enclosing anode cells *c* and cathode cells *d* with porous walls. Especially those walls which face the direction of the electric current are porous. The cells moreover are provided with adjustable outlets for the liquid contained in them. The cathode cells *d* by means of conduits *m* and conduit *n* communicate with a collector *e*, which in its turn communicates with the cellulose boiler *f* by a conduit *r*. The said boiler *f* communicates by means of a conduit *t* with a concentrating apparatus *g* which again by means of an intermediate filtering and washing apparatus *h* is connected to a tank *i*, in which the crystallization can take place. The anode cells *c* are by means of conduits *k* and conduit *l* connected to the concentrating apparatus *g*.

The solution of sulphate of sodium is brought from the container *a* through the conduit *j* to the electrolytic apparatus *b*, in which the said solution is electrolytically decomposed in an anode solution, consisting of a solution of sulphuric acid, and a cathode solution, consisting of hydrate of sodium. The former one flows through the conduits *k* and conduit *l* to the concentrating apparatus *g*, while the cathode solution through the conduits *m* and conduit *n* flows to the collector *e* and thereupon to boiling apparatus *f* through the conduit *r*. That part of the solution of the sulphate of sodium introduced in the electrolytic apparatus, which has not passed through the cells in said apparatus returns to the container *a* by the conduit *o*.

The solution of hydrate of sodium, which has been introduced in the collector *e* is converted to a solution of sulphite of sodium by means of sulphurous acid introduced in the container through the conduits *p* and *q*, if the object of the process is the production of sulphite cellulose. If on the contrary sulphate cellulose shall be produced, sulphuretted hydrogen is introduced through the same conduits.

When the boiling of wood by a boiling fluid in the boiler *f* is finished, the cellulose is separated and removed through the conduit *s*, while the lye is brought to the concentrating or evaporating apparatus *g*, where it is mixed with the solution of sulphuric acid introduced from the anode cells *c* by the conduits *k* and conduit *l* and also with an additional quantity of sulphuric acid, which is introduced through the conduit *u*.

During the concentration of the boiling fluid in the apparatus *g* after the introduction of the acid solutions just mentioned, steam, volatile acids (acetic acid and formic acid) and sulphurous acid escape from the said apparatus *g*, if the boiling lye consists of sulphite lye. The two gases first stated are

carried away by conduits v, while the sulphurous acid is returned to the collector e through the conduit p and utilized again in the boiling process, if desired.

If the lye in the concentrating apparatus results from a boiling of sulphate cellulose organic acids (acetic acid and formic acid) are also set free, but no sulphurous acid. In place of the latter sulphuretted hydrogen is generated which gas is returned through the conduit p to e and facilitates the generation of the sulphohydrates for a new boiling of sulphate cellulose.

When the concentration in the apparatus g has proceeded a sufficient time and the free sulphuric acid therein has thus become concentrated to a certain degree the organic substances of the lye are precipitated in such a form that they can easily be filtered and washed. The lye with these substances is brought to the filtering and washing apparatus h through the conduit x and is filtered and washed in the apparatus, whereby the precipitate is separated. For the washing of the precipitate a nonconcentrated boiling lye is used which at the last step of the washing operation is replaced by water. The cleaned precipitate is removed through the conduit y and the filtrate first separated is conducted through the conduit z to the crystallizing apparatus i. The crystallized sulphate of sodium may eventually be returned through the conduit w to the container a, whereby the sodium used in the boiling operation is again introduced in the process. The mother lye containing very concentrated sulphuric acid is conducted back to the concentrating apparatus g, where it serves for the decomposition of further quantities of organic substances.

Having now particularly described the nature of my invention and the manner of its operation, what I claim is:

1. A process for the digestion of cellulose wherein, alkaline sulphate is electrolytically decomposed into a cathode solution of alkaline hydrate and an anode solution of sulphuric acid, said alkaline hydrate is treated by sulphur compounds to form a suitable lye for boiling, the cellulosic raw material is boiled in said lye, the cellulose is separated from said lye after boiling and said anode solution of sulphuric acid is added to said lye.

2. A process for the digestion of cellulose wherein, alkaline sulphate is electrolytically decomposed into a cathode solution of alkaline hydrate and an anode solution of sulphuric acid, said alkaline hydrate is treated by sulphur compounds to form a suitable lye for boiling, the cellulosic raw material is boiled in said lye, the cellulose is separated from said lye after boiling, said anode solution of sulphuric acid is added to said lye, free sulphuric acid is added to said lye in excess to precipitate organic substances therein and the volatile products of the precipitation are collected.

3. A process for the digestion of cellulose wherein, alkaline sulphate is electrolytically decomposed into a cathode solution of alkaline hydrate and an anode solution of sulphuric acid, said alkaline hydrate is treated by sulphur compounds to form a suitable lye for boiling, the cellulosic raw material is boiled in said lye, the cellulose is separated from said lye after boiling, said anode solution of sulphuric acid is added to said lye, free sulphuric acid is added to said lye in excess to precipitate organic substances therein, the volatile products of the precipitation are collected, said precipitated organic substances are removed from said lye, said lye is crystallized to produce akaline sulphate, said alkaline sulphate is dissolved and again electrolytically decomposed and the mother lye is returned for the concentration of other boiling lye.

4. A process for the digestion of cellulose wherein, alkaline sulphate is electrolytically decomposed into a cathode solution of alkaline hydrate and an anode solution of sulphuric acid, said alkaline hydrate is treated by sulphur compounds to form a suitable lye for boiling, the cellulosic raw material is boiled in said lye, the cellulose is separated from said lye after boiling, said anode solution of sulphuric acid is added to said lye, free sulphuric acid is added to said lye in excess to precipitate organic substances therein, the volatile products of the precipitation are collected, the degree of concentration of said lye being varied by variation of the quantity of said free sulphuric acid added to give the desired quality of precipitate and said precipitated organic substances are removed from said lye in fractions to obtain precipitates of different composition, said lye is crystallized to produce alkaline sulphate, said alkaline sulphate is dissolved and again electrolytically decomposed and the mother lye is returned for the concentration of other boiling lye.

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