

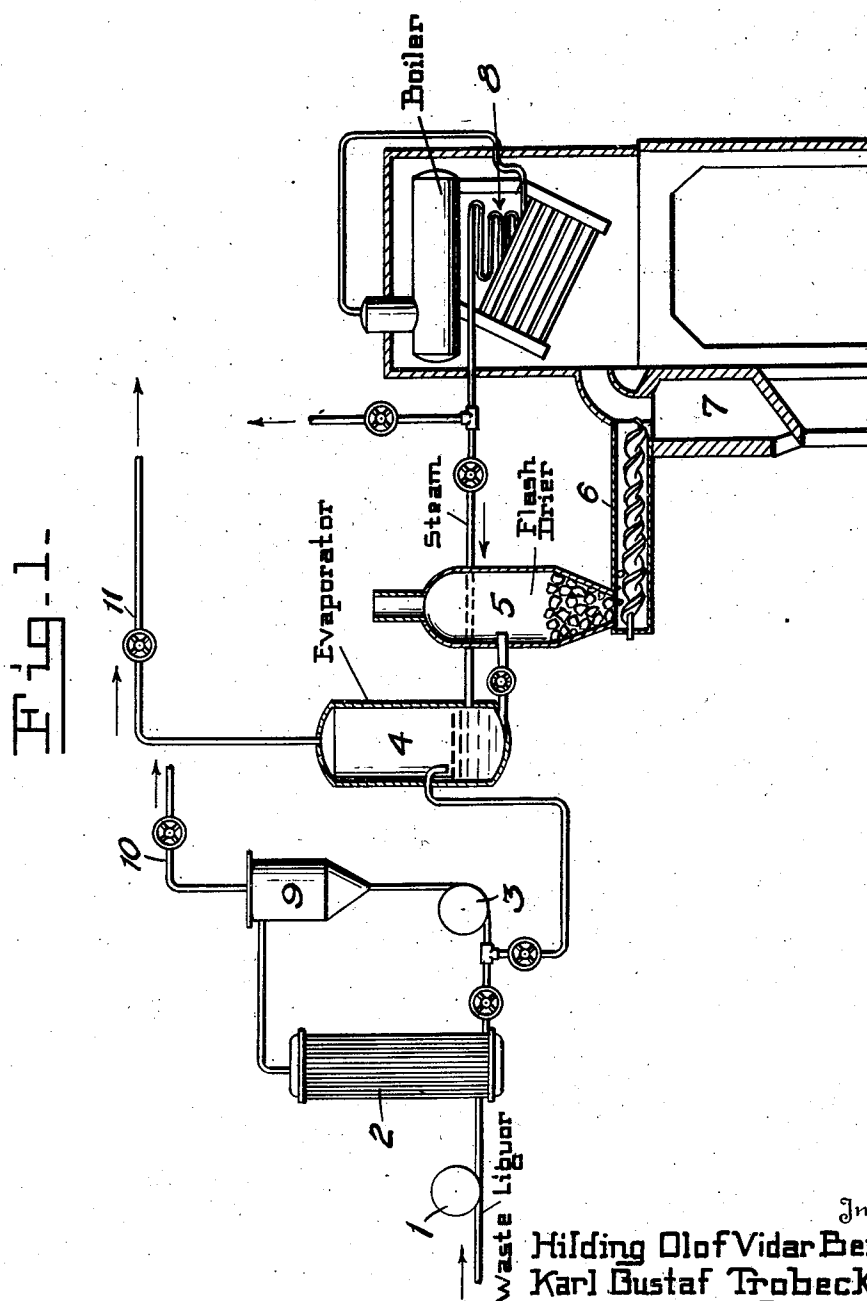
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PROCESS OF UTILIZING WASTE LIQUOR

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2 Sheets-Sheet 1



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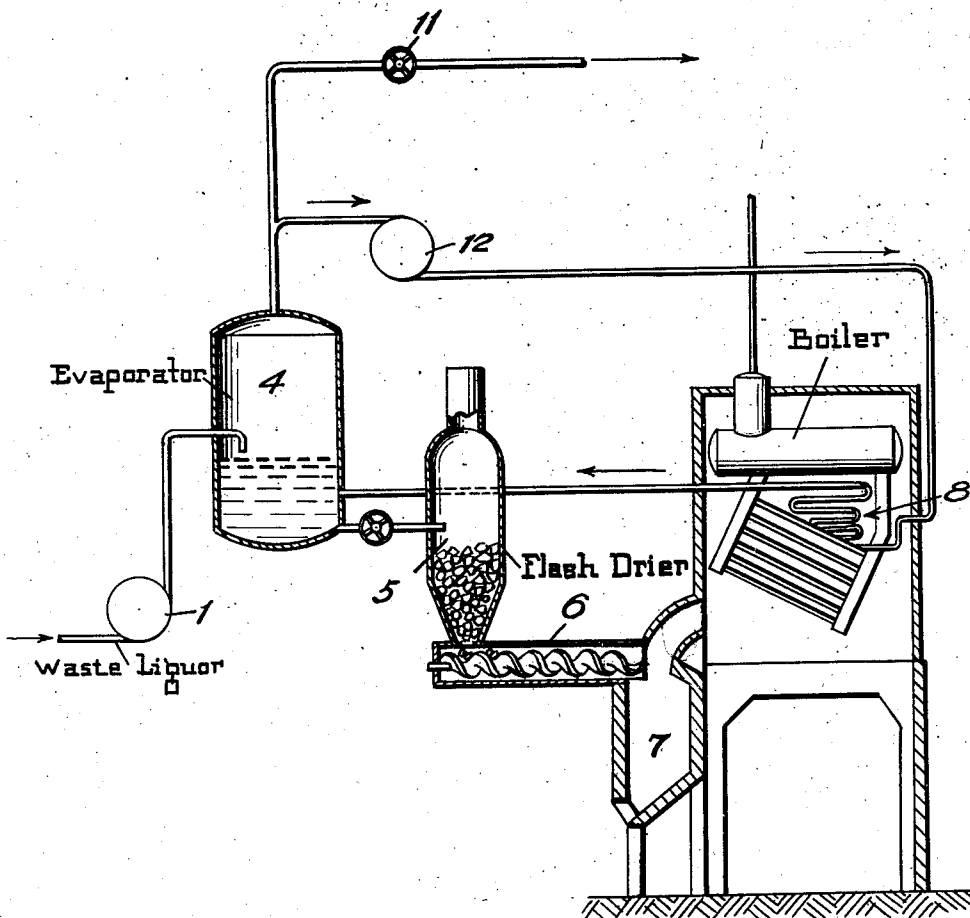
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Fig-2-



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PROCESS OF UTILIZING WASTE LIQUORS

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The present invention relates to a process of utilizing waste cellulose liquors and other fluids by means of a particular system of concentration and treatment.

Highly concentrated waste liquors from e. g. the sulfate, sulfite or soda pulp processes have a pitch-like consistency, for which reason they are unsuitable as a fuel as well as for treatments such as dry distillation.

The present invention, which has for its object to transform liquors of this character into substances suitable for combustion, dry distillation etc., is based on our discovery that highly concentrated liquors, when ejected in a suitable manner, will yield a more or less porous or finely divided dry product.

The ejection, which is made under pressure, is effected by means of nozzles of suitable size and shape, so that a development of steam will take place in the material when this is released into a room with lower pressure, e. g. the free air or a suitable chamber. The pressure should thus during the evaporation of the liquid to suitable concentration, e. g. corresponding to 75-95 per cent by wt. of dry substance, previous to the ejection be kept sufficiently high to produce the effect of "flash-heat" evaporation or flash drying with steam-expansion within the material when this is ejected.

The ejection may be effected in various ways and from different depths of the liquor in the evaporator (the final evaporator) through nozzles of various types, as this influences upon the structure and consistency of the dry product. The nozzle or nozzles may thus have a round or oval cross section or may resemble some other geometrical figure. They may further be of different length and be made with cut out edges or shaped as a connecting piece, having an inward or outward projecting conicity, or be provided with a central core so that the liquor will be ejected in the form of a hollow cylinder, etc. The nozzle may also be shaped as an injector so that air will be brought into intimate contact with the ejected material.

As already mentioned, the ejection of the concentrated liquor may be made into the free air or into a so called cyclone apparatus or similar chamber, whereby the heat content of the developed steam may, at least partly, be utilized. In such event it is suitable to operate the cyclone or chamber under a certain elevated pressure. It is not necessary that the ejection be made directly from the evaporator. The liquor may instead be conveyed from this into an intermedi-

ate container from which it is then released in some suitable manner for obtaining a product of the desired quality.

If waste sulfate liquor, previous to the evaporation, is treated with air or gases containing oxygen, e. g. smoke stack gases, the sulfur containing compounds in the sulfate cellulose liquor are transformed in such manner that they will not develop hydrogen sulfide during the evaporation, or at least only minor quantities thereof.

The usual manner in which waste sulfate cellulose liquor is at present evaporated causes great quantities of H_2S to escape. The liquor will thus frequently lose from 2 up to as much as 10 kilograms of sulfur calculated on the basis of one ton of cellulose pulp produced, in the form of H_2S , mercaptanes and organic sulfides. The sulfur loss will partly occur during the evaporation of the waste liquor to form so called thick-lye and partly when this is dried to "black stuff."

For known reasons losses of sulfur are, however, not desired in the process since the regenerated cooking liquor should contain as much Na_2S as possible. If the "black liquor" (the waste liquor from the wood digesting process) is subjected to an oxidizing treatment according to the invention, e. g. by means of air, the sulfur will, however, become "fixed" in the liquor whereby the above mentioned losses are reduced to a negligible quantity. The oxidation may, of course, take place during some stage of evaporation prior to the final evaporating step if the original waste liquor is not previously treated in this manner.

The oxidation may for instance be effected by means of air or other oxidizing gases in towers through which the liquor flows downwards in contact with the oxidizing medium, or it may take place in columns with perforated plates or bell bottoms, or in disk evaporators or drums in which the waste liquor is brought into contact with the oxidizing medium. If the oxidation is carried out in a column the waste liquor should suitably be introduced on one of the lower places since the liquor will, in general, not flow downwards through the column but instead leave this in the upper part, in the form of a foam. This foam may then be collected in some suitable manner. The column may then suitably be provided with a foam-chamber or foam separator from which the air or other gaseous oxidizing medium may escape. From the foam-chamber or separator the liquor and foam is for instance led to a tank in which remaining foam may be abated by being led through a fan or similar arrangement

which causes the foam to circulate within the tank. The foam may also be abated by means of a centrifuge or the like. The oxidation may take place at different temperatures. Increased temperatures will increase the velocity of oxidation. This should, however, not be carried too far since it has been found that a too high degree of oxidation will have an undesired influence upon the organic substance in the liquor. A certain alkalinity will also cause the oxidation to take place more rapidly.

By the oxidation of the waste sulfate liquor an increase in the sulfur content of the digesting liquor and a dry product ("black stuff") more suitable for combustion is thus obtained. The consumption of lime in the causticizing process is furthermore reduced, and the condensate and gases formed in the evaporation will be free or almost free from poisonous substances.

The oxidation of the waste liquor may, as already mentioned, be made prior to or during its primary evaporation, i. e. before it is introduced into the final evaporation step, and may be carried out under ordinary or raised pressure as well as under a vacuum.

Instead of being treated with oxygen containing gases the waste liquor may, prior to or during the evaporation, be treated with SO_2 or SO_2 -containing gases, which have a similar oxidizing effect on the sulfidic and other reducing sulfur compounds as oxygen, so that evolution of hydrogen sulfide will be prevented.

The dry organic substance obtained in the previously described manner is suitable for dry-distillation. In the case of organic matter from the sulfate pulp process, such a dry-distillation will yield oils in an amount corresponding to 100-150 kilograms as calculated per ton of cellulose pulp produced. The dry-distillation is suitably carried out continuously in a stationary retort provided with internal conveying means and may, if desired, be directly combined with a combustion of the residue. The non-condensable gas from the dry-distillation may be used as a fuel for the steam boilers or for other purposes, preferably after removal of hydrogen sulfide, mercaptanes etc., for instance by scrubbing with cooking liquor or other liquors used in the pulping process. The remaining dry-distilled material may be burnt in a calcining furnace, or the alkali in the same be extracted by lixiviation or leaching. In the latter case the remaining carbonaceous matter may be used as a fuel, if desired after briquetting. The carbonaceous matter as well as the undistilled dry product is suitable for powdered fuel firing.

The evaporation of the waste liquor to a very high concentration may be effected in the following manner. The evaporation is divided into several steps. In the first step the evaporation is in a multiple effect evaporating system carried up to for instance 50-60% dry content. The liquor from this step is introduced in the second step evaporator, in which the concentration may be carried up to for instance 70-80%. The temperature in this step is kept at about 110° to 250° C., to permit the liquor to remain sufficiently easy-flowing to ensure an efficient heat transfer through the heating surface, whereby the capacity of the equipment will be sufficiently high. The evaporation in the second step is suitably made in an evaporator in which the liquor is caused to circulate over the heating surfaces by means of a pump. From the second step evaporation the liquor is now introduced into the third

step evaporation system which consists of an evaporating apparatus in which superheated steam is brought in direct contact with the liquor, e. g. by being forced directly through the liquor or by being showered with a spray of the same, whereby it is concentrated to such an extent that the product at normal temperature will assume a solid state.

In the final evaporator a constant pressure is automatically maintained and the level of the liquor also automatically regulated by special means.

The concentrating of the liquids coming from the three stages of evaporation may suitably be regulated so that the vapor arising from the third step will as much as possible be adjusted to the steam requirements for the second step, and the vapor from this similarly adjusted to the steam requirements in the first step, or else so that the vapor quantities from the second and third steps will nearly correspond to the steam requirements in the first step. The vapors from the various steps may, of course, also be utilized in some other manner. The vapors from the second and third steps may thus, by means of heat exchangers, be transformed into pure steam which can be utilized for evaporation or other purposes.

In certain instances the evaporation in the third step may be carried out at a pressure which is insufficient for permitting an employment of the escaping vapors as a heating medium in the second step evaporation. In such cases the vapors coming from the third step are better utilized in some other way, steam of sufficiently high pressure, e. g. 8-30 atmospheres, being used in the second step evaporation.

The process may also be carried out in such a manner that the vapors in the third step evaporation are circulated by means of a fan or blower through a superheater and from there back to the liquid. In this case the second step evaporation may be entirely dispensed with. That part of the evaporation which is carried out by means of direct steam may be distributed on several units, the vapor from the first of these being passed through a superheater and directly from there into the liquid in the second unit, and so forth. In this case the above mentioned circulating fan or blower is dispensed with.

The final evaporator, or the apparatus in which the evaporation is carried out with the aid of superheated steam, must be arranged so that the vapor will leave the same saturated or nearly saturated. The vapor should thus leave at nearly the same temperature as that of the liquor. The superheated steam may be introduced into the liquid in several ways. The steam may be injected tangentially or nearly tangentially or in some other manner whereby the liquid is brought into vigorous motion. The steam introducing means may be placed horizontally or at an angle with the horizontal. It is important that the superheated steam be introduced in such a manner that the means of introduction will not present any heating surfaces in the liquid, as this may cause decomposition by superheating. The intake for the liquid in the final evaporator should be arranged at some point higher than the liquid level in the evaporator, so that the fresh liquid flows onto the top level. The pressure in this evaporator should be kept higher than 1.5 atmospheres overpressure (above the atmospheric) e. g. above about 36 lbs./sq. in. abs. pressure.

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If the concentration in the second step is sufficiently high, the liquor from this may be directly ejected, in which case a third evaporating step may be dispensed with.

The fact that a dry product of suitable quality is directly obtained by flash drying of the waste cellulose liquors makes it possible to improve and simplify their utilization as a fuel. This may e. g. be realized in the following manner. The dry product (the so called black stuff) is burnt in calcining furnaces or combustion rooms which may be built as very large-sized units. The combustion gases are first brought to act on a steam-boiler or a tube-system combined with a steam-generator and are from there conveyed to a superheater for superheating of the steam for the final evaporator. From the superheater they pass to the above mentioned steam-generator which in certain cases is combined with the tube-system, and from there to an economizer or similar apparatus. By this arrangement the superheater will be exposed to the stack gases at the most suitable temperature (from 900° C. at the front down to 500 C. near the outlet end). It is, namely, very important that the superheater is not exposed to excessively high temperatures, which may cause injuries on the equipment at the comparatively low velocities which the steam in the superheater sometimes may assume. A fairly high temperature on the combustion gases at the outlet end will, on the other hand, make it possible to use a superheater with a comparatively small heating surface. Special means should be provided for an automatic injection of steam in the system, in order to prevent the superheater from being burnt if the circulating fan for the vapor from the final evaporator, which is conveyed through the superheater, should break down or stop.

Thanks to the fact that the dry product may be easily formed into briquets of an even size, it is also possible to use this as fuel in a gas-producer in which the combustion may thus be very closely regulated. The briquetting is also of importance when the product is to be dry distilled for the recovery of oils. In this case the calcining furnace may suitably serve for the dry distillation.

In the foregoing the invention has chiefly been described as applied for the utilization of waste cellulose liquors, particularly waste sulfate liquor. It should be evident, however that the method may also be applied for the recovery of the dry matter in other liquors, particularly those which by means of evaporation may be brought up to a high content of solids.

Examples of suitable recovery plants operating according to the invention are shown in the accompanying drawings. The evaporators in the first evaporating step are not shown, as this detail may be arranged according to any known system.

Figure 1 is a plant operating with three evaporating steps, of which the second and third are shown in the drawings. Figure 2 is a plant operating with only two evaporating steps, of which the last is shown in the drawings. Similar parts carry the same numeral in both figures.

In Figure 1 numeral 1 denotes the pump by which the partly evaporated liquor coming from the first step evaporation is conveyed to the second step evaporator 2, which is provided with a circulating pump 3. The final evaporation takes place in a container 4 in which superheated steam from superheater 8 is directly forced

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through the liquor. The highly concentrated liquor is ejected by means of the above described nozzles into the chamber 5 in which it is received as a dry and porous product. From this chamber the dry product is, by means of the screw-conveyor 6, fed into the combustion room 7 in the steam-generating system, comprising the superheater 8.

The system shown in Figure 1 operates as follows.

The waste liquor which is to be worked up, is pre-concentrated in a suitable evaporation system and by means of pump 1 conveyed to the tube-evaporator 2 which is provided with a circulating pump 3. The liquor is further concentrated in evaporator 2 and from there introduced in the final evaporator 4. In the final evaporator 4 the dry content is brought up to about 80-95% by means of blowing superheated steam directly through the liquor. The highly concentrated liquor is by means of suitable nozzles ejected into the chamber 5 in which a porous solid is received with a moisture content of about 5%. The vapors from evaporator 4 escape from this through line 11 with a temperature of about 180-220° C., which corresponds substantially to the temperature of the liquor in the evaporator, and are either led to the boiler system or used as "live" steam at some suitable point in the plant. The saturated vapors from the catch-all 9 in evaporator 2 are by means of line 10 also conveyed to the boiler-system or used as "live" steam for other purposes.

As already mentioned, the second evaporating step may in certain cases be dispensed with. An example of this is shown in Figure 2. The pre-evaporated liquor is in this case by means of pump 1 introduced directly into evaporator 4 in which the final concentration in direct contact with superheated steam takes place. Part of the vapors from evaporator 4 are by means of line 12 conveyed to the superheater 8 from which they are again returned to evaporator 4 in the form of superheated steam with a temperature of about 300°-500° C. The bleeder-line 11 is connected to the boilers or other apparatus, e. g. the primary step evaporators. The quantity of steam through line 11 is adjusted to correspond to the evaporation in evaporator 4, so that a constant pressure will be maintained in this, the steam in line 12 being re-circulated as already described.

We claim:

1. A process of utilizing waste cellulose liquors comprising subjecting the liquor to evaporation in at least two steps, the concentration of the liquor in the last step being conducted under superatmospheric pressures and being carried to a solids content of about 80 to 95% so that the liquor will assume a solid state at normal temperature, ejecting said liquor in a finely divided state from the last step of evaporation, while at a temperature of about 180° to 220° C. and while under superatmospheric pressure, under release of pressure so that flash drying takes place and collecting the resulting dry porous product thus received.

2. A process of utilizing waste cellulose liquors, comprising subjecting the liquor to evaporation in at least two steps, superheated steam being brought in direct contact with the liquor in the last step to effect concentration of said liquor, said concentration being conducted under superatmospheric pressures and being carried to a solids content of about 80 to 95% so that the

liquor will assume a solid state at normal temperature, ejecting said concentrated liquor in a finely divided state, while at a temperature of about 180° to 220° C. and while under superatmospheric pressure, under release of pressure so that flash drying takes place and collecting the dry porous product thus received.

3. A process according to claim 2 in which part of the vapors from the last evaporation step are circulated through a superheater and again brought in direct contact with the liquor in said step to serve as a means of evaporation.

4. A process of utilizing waste sulfate liquor, comprising subjecting the liquor to evaporation in at least two steps, concentrating the liquor in the last step under superatmospheric pressures to a solids content of about 80 to 95% so that it will assume a solid state when cooled to normal temperature, ejecting said concentrated liquor in a finely divided state, while at a temperature of about 180° to 220° C. and while under superatmospheric pressure, under reduction of pressure so that flash drying takes place, collecting the dry porous product received and dry-distilling the same.

5. A process for the recovery of waste cellulose liquors comprising concentrating the waste liquor by a multi-stage evaporation, the last evaporation stage consisting in bringing superheated steam in direct contact with a pre-concentrated liquor containing at least 70 per cent solids, bringing the concentration up to at least 80 per cent solids while maintaining at least 36 lbs. per sq. in. absolute pressure in the evaporator and a temperature of at least 110° C., ejecting the concentrated liquor in a finely divided state under a sudden pressure drop so that flash drying takes place within the ejected material, and collecting the dry porous product thus obtained.

6. A process of utilizing waste sulfate liquor which comprises concentrating the liquor while contacting it with an oxidizing gas in quantity

sufficient to substantially eliminate evolution of hydrogen sulfide during subsequent evaporation to dryness, then concentrating the oxidized liquor to a solids content of from about 80 to 95 per cent while under superatmospheric pressures and at a temperature of about 180° to 220° C., ejecting said concentrated liquor in a finely divided state into a space maintained at substantially atmospheric pressure, so that flash drying to a porous solid takes place, without substantial evolution of hydrogen sulfide, and collecting said solids.

7. A process of utilizing waste sulfate liquor which comprises concentrating the liquor while contacting it with air in quantity sufficient to substantially eliminate evolution of hydrogen sulfide during subsequent evaporation to dryness, then concentrating the oxidized liquor to a solids content of from about 80 to 95 per cent while under superatmospheric pressures and at a temperature of about 180° to 220° C., ejecting said concentrated liquor in a finely divided state into a space maintained at substantially atmospheric pressure, so that flash drying to a porous solid takes place, without substantial evolution of hydrogen sulfide, and collecting said solids.

8. A process of utilizing waste sulfate liquor which comprises concentrating the liquor while contacting it with sulfur dioxide in quantity sufficient to substantially eliminate evolution of hydrogen sulfide during subsequent evaporation to dryness, then concentrating the oxidized liquor to a solids content of from about 80 to 95 per cent while under superatmospheric pressures and at a temperature of about 180° to 220° C., ejecting said concentrated liquor in a finely divided state into a space maintained at substantially atmospheric pressure, so that flash drying to a porous solid takes place, without substantial evolution of hydrogen sulfide, and collecting said solids.

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