

# UNITED STATES PATENT OFFICE.

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## IMPROVEMENT IN RECOVERING WASTE ALKALIES USED IN TREATING PAPER-PULP.

Specification forming part of Letters Patent No. **132,452**, dated October 22, 1872.

*To all whom it may concern:*

Be it known that I, CYPRIAN MARIE TESSIÉ DU MOTAY, of the city of Paris, in France, have invented a certain new and Improved Process for the Treatment of Lyes resulting from the preparation of woody and other similar fibers, and of wash-waters after the washing or cleansing of filaments and fabrics.

My invention has for its object the separation and precipitation of the impurities contained in liquors after the boiling of woody fiber, or its treatment for the reduction to pulp suitable for paper-makers, with a view to recover the liquor so that it can be again used for treating fresh fiber.

In all previous processes it has been the practice to boil or evaporate the liquor which was treated as waste until it was so reduced in quantity that very little else than the impurities and the alkali which it contained were left, and these have been burnt or, as it is termed, "incinerated," to destroy the organic bodies, in order that the soda or alkali could be recovered from among the ashes by an after treatment. It has been proposed, also, to act upon the used or spent liquor by an injection of carbonic-acid gas for the purpose of precipitating the impurities, and thus to save the liquor; but this has been found in practice not to answer the purpose, as it only produced a flocculent and thick muddy appearance in the liquor without in any way causing a separation or a precipitation of the impurities. Now, it is this most important part of the process which my invention is intended to perfect.

According to the first process forming part of my invention I run the hot liquor, after it has been used for treating woody fibers, into an open tank, and then inject a current of carbonic-acid gas into the liquor, which causes the alkali to free itself from the dissolved impurities, which partly combine and become suspended in the liquor, the complete combination being effected by the admixture of a quantity of sulphuret of sodium, in a liquid state, still continuing the injection of the gas. The consequence is that, through the combination or the separation of the resinous and the ulmic particles, the liquor assumes a green yellowish tint, with the particles still in suspense. So soon as the proper

tint shows itself, which is determined by practice and according to the description of fiber under treatment, I stop the injection of the gas and run the tinted liquor into an open boiler or tank, and subject it to fresh heat until the liquor boils, which boiling I allow to go on for from fifteen to twenty minutes; and it is during this period that the particles, both resinate and ulmate, precipitate as one mass, and the liquor assumes a dark ruby transparent color. The heat is then discontinued and the liquor is drawn off into another tank and recaustified, when it can be stored for after use for boiling down a fresh quantity of fiber. In this state the liquor contains all, or nearly all, of the alkali which it at first possessed, because the quantity of alkali absorbed by the fiber in boiling, and by the lime in causticising, is compensated for by the "sulphide of sodium" which was added. The mass (resinates and ulmates) which has been precipitated is then scooped or shoveled out of the precipitating-tank, and can be broken up and used for generating heat, when mixed with coal or other fuel, or it can be converted for useful purposes, such as in the manufacture of gas, resin, oil, &c. Its character and color vary with its constituents. In some cases it is very hard and solid, and almost black; in others it is comparatively soft and honey-combed, and of a dark-amber brown.

The woods to be treated as above described are pine, spruce, fir, and woods of a similar nature known to be rich in resins and gums; but for harder woods, which are less rich in resins, a different and somewhat tedious treatment is necessary, which I hereinafter describe.

It will be understood that pulp—in fact, all pulps—treated according to my invention retain the color corresponding to the liquor first run from the boiling-down vessel into the open tank, and it is deprived of this color by one or more washings in clear water, and then submitted to a bleaching process to render it white and fit for the paper-maker. I, however, make no claim to the bleaching process, because it is not essential in all cases to bleach the fiber, that being dependent upon the kind of paper to be made.

I have, for the sake of clearness only, referred to the employment of carbonic-acid gas

and sulphuret of sodium for the treatment by my process, but I do not limit myself to their employment, as other gases or bodies—such as bicarbonates, which have the property of fixing alkaline liquor from resinous and other matter—can be used; nor do I limit myself to using sulphide of sodium to complete the division or freeing action, as other sulphurets, such as those of potassium, barium, or calcium can be used in lieu thereof.

I employ the term “woody” in its broadest sense, and intend thereby to include stalks, leaves, and roots of plants; also fruits and flowers which are of a fibrous or stringy character, whether they contain an admixture of gum (ulmates) and resin, or only one of these ingredients, in which case I dispense with the use of the particular agent which would free the alkaline from the particles for which it has no affinity, but rather a reaction.

According to another process, in lieu of freeing the alkaline liquor and precipitating the ulmates and resinates by any specific period of boiling after the injection of the gas or the bicarbonate, and the admixture of the sulphuret, I heat the liquor nearly to boiling-point, maintaining that heat until a complete coagulation has taken place, when I filter the liquor to effect the perfect separation of the impregnated liquor from the impurities. In this particular process an excess of carbonic-acid gas is necessary, which is afterward removed and the clarified liquor brought into a complete caustic state again by the addition of lime, which acts upon the bicarbonate and produces potash or soda. The impregnated liquor thus heated can also have a quantity of sulphydric acid produced in it by the addition of sulphuret of potassium, sulphuret of sodium, sulphuret of barium, or sulphuret of cal-

cium in the presence of a current of carbonic-acid gas. By this means any portion of the liquor containing ulmic matters which have not been operated upon in the previous stage become separated or freed. In this treatment I prefer using sulphuret of sodium or sulphuret of potassium, because the alkaline carbonates formed by them are added to the alkaline liquor and return the fraction lost, so that the liquor is reconstituted for the next boiling down.

I do not claim reclaiming the soda from the spent solution of caustic soda after its action upon reeds, straws, &c., by charging the solution with carbonic-acid gas in a vessel, so that the organic matter will be precipitated, as such is not my invention; but

I claim as my invention—

1. The process, hereinbefore described, for recovering for reuse the lyes and waters after the boiling down of woody fibers and the washing of fabrics and filaments, as herein set forth.

2. I claim the boiling of spent lyes or washwaters after their impregnation with a gas, or a bicarbonate and a sulphuret, substantially as described, for producing a precipitation of the impurities contained in such lyes and washwaters.

3. I claim the precipitation of the resinates or the ulmates after they, or one of them, have been acted upon by a gas or a sulphuret in the manner described, whether the precipitation be assisted by the adding of sulphydric-acid to the heated liquor, or not.

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Witnesses:

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