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PROCESS FOR REFINING SULPHATE WOOD TURPENTINE

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This invention relates to the manufacture of sulphate wood turpentine and has for its object the improvement of such processes by the efficient removal of offensive odors and the development of a product of pleasing odor and high quality.

A further object is the provision in a method of the character described of improved steps for eliminating the highly offensive and obnoxious odors otherwise arising from the presence of sulphur compounds developed by the reagents used in the process.

To these and other ends the invention resides in certain improvements and combinations of parts, all as will be hereinafter more fully described, the novel features being pointed out in the claims at the end of the specification.

In the manufacture of wood pulp by the known sulphate process, wood chips are treated under elevated temperatures and pressures in the presence of an alkaline cooking liquor of which the main constituents are generally sodium hydroxide, sodium sulphide and sodium carbonate.

Heat is supplied to the cooking vessel, or digester, either by the direct supply of steam, or by external heating of the cooking liquor which is then circulated through the cooking vessel. During the cooking operation it is common practice to remove a part of the steam generated in the digester, which operation is known in the trade as "gassing the digester". This gassing is usually done as the pressure is being increased on the digester. At the end of the cook there is also a gassing period to partially relieve the pressure before the contents of the digester are blown.

The steam which is drawn off at these two gassing operations carries with it the vapors of turpentine and other volatile compounds liberated from the wood material. These vapors are carried through a suitable condenser which condenses the water and oil vapors. The condensed vapors are led into a separating device where the condensed oil layer is removed and run into a storage tank and the water layer sent to waste. The oil layer is known as crude sulphate wood turpentine.

Due to the presence of sodium sulphide in the cooking liquor, a number of exceedingly malodorous sulphur compounds may be formed. Such compounds may include mercaptans, dimercaptans, thio-ethers, sulphoxides, sulphones, sulphinic acids, sulphonic acids, disulphides, thio-aldehydes, thio-ketones, esters, amides and other complex sulphur or nonsulphur compounds.

Some or all of these compounds may be present in the crude sulphate turpentine, imparting to it an exceedingly offensive odor, the removal of which presents the chief problem in producing sulphate wood turpentine of a pleasing and marketable odor.

It has been found that this problem is efficiently solved by the present process which is a combination of refining and chemical treatment steps. The process may be carried out by the use of two distilling systems of similar construction, together with several washing and storage tanks. Each distilling system preferably comprises a kettle with a steam heating coil, a fractionating column, a condenser and two receivers. Each system is preferably provided with a steam jet for evacuating it to a vacuum of 27 to 29 inches of mercury. Such equipment may be of the usual or any suitable variety for the operations described, as well understood in the art.

The first distilling system is known as the "stripping system". The kettle is charged with crude sulphate turpentine and subjected to a vacuum. Steam is then admitted to the heating coil and the stripping distillation begun. The vapors pass up through the fractionating column which serves to classify them, as well understood in the art. The low boiling compounds pass up through the column and are condensed in the condenser, while the higher boiling compounds are condensed in the column and returned to the kettle.

A part of the distillate from the condenser is returned to the kettle and a part is run to the receiver. About 7 to 12 per cent of the kettle charge is distilled in the receiver and is referred to as "heads".

During this distillation, specific gravity determinations are made and when the so-called heads reach a specific gravity of approximately 0.8630 at 15.5° centigrade, the distillate is diverted to a second receiver until the specific gravity reaches 0.8670 at 15.5° centigrade. The fraction between these specific gravities, 0.8630 and 0.8670, contains most of the turpentine and is called "stripped crude". The higher boiling compounds in the kettle are known as "foots" and their disposal is not a part of the present invention.

The second distilling system is called the "refining system". The kettle is charged with stripped crude turpentine and from substantially 0.2 to 0.5 per cent by weight of ethylene diamine solution of substantially sixty per cent free base, is added to the charge in the kettle. Preferably, steam is admitted to the kettle coil and the mixture boiled preferably at atmospheric pressure

for a period of from 6 to 18 hours, all the vapors being condensed and returned to the kettle. While best results have been obtained in this manner, it is not necessary that this ethylene diamine treatment be carried out with heat. Thus it has been found that this treatment may be satisfactorily performed by agitation with air at lower temperatures. It has also been found that such treatment may be carried on under elevated pressures with good results. After such treatment with ethylene diamine, the mixture is allowed to settle and the ethylene diamine is drawn off.

The refining system is then exhausted to a vacuum of substantially 27 to 29 inches of mercury and distillation is begun. During the first stage of this distillation, a part of the distillate is caught in a heads receiver and a part is returned to the kettle. As soon as the character of the distillate is satisfactory a part of it is diverted to a receiver for refined turpentine.

The distillation is carried on until there is a sharp change in the specific gravity and odor of the distillate. This change occurs at an approximate specific gravity of 0.865 at 15.5 degrees centigrade, there being a sharp change to approximately 0.867 specific gravity at 15.5 degrees centigrade. The residue left in the kettle, known as "refined foots", is drawn off and its treatment is not a part of the present invention. That portion taken off in the refined receiver is pumped to a treatment tank where it is preferably first washed for about two to four hours with about an equal volume of water by agitation with air. The air is then cut off and the water is allowed to settle out after which it is drained off.

The washed distillate is then treated with a bleaching agent, such as a bleaching powder solution containing approximately three per cent available chlorine. About ten to fifteen per cent by volume of this solution is added and the mixture agitated with air for a period of eighteen to twenty-four hours. It is then allowed to settle for from three to five days after which the bleaching powder solution is drawn off from the turpentine. The resulting turpentine is of pleasing odor and high quality and is drawn off and pumped to storage for market.

The "heads" fractions from this treatment are returned to the first distilling system and treated as crude turpentine. This fraction yields a higher percentage of turpentine than the original crude turpentine.

While bleaching powder has previously been employed in the manufacture of sulphate wood turpentine, we are not aware that there has been any prior recognition of the possibility and utility of the employment in the process of the substance ethylene diamine alone or together with a bleaching powder as described above. It is recognized that other substances may be found related and equivalent to ethylene diamine and within the spirit of the present invention. Furthermore, the above illustrative description of the process refers preferentially to various details of the procedure which may be modified by those skilled in the art within the spirit and substance of the present invention.

We claim:

1. The process of making sulphate wood turpentine comprising the treatment of the turpentine material with ethylene diamine or its equivalent to remove offensive odors.

2. The process of making sulphate wood turpentine comprising boiling the turpentine material with ethylene diamine or its equivalent to remove offensive odors.

3. The process of making sulphate wood turpentine comprising the treatment of the turpentine material with from substantially 0.2 to 0.5 per cent by weight of ethylene diamine of substantially sixty per cent free base, to remove offensive odors.

4. The process of making sulphate wood turpentine comprising the treatment of the stripped crude turpentine material with from substantially 0.2 to 0.5 per cent by weight of ethylene diamine of substantially sixty per cent free base, to remove offensive odors.

5. The process of making sulphate wood turpentine comprising the boiling of the stripped crude turpentine material with from substantially 0.2 to 0.5 per cent by weight of ethylene diamine of substantially sixty per cent free base, to remove offensive odors.

6. The process of making sulphate wood turpentine comprising the treatment of the turpentine material with ethylene diamine and with a bleaching powder, to remove offensive odors.

7. The process of making sulphate wood turpentine comprising the treatment of the turpentine material with ethylene diamine and with a bleaching powder solution containing approximately three per cent available chlorine, to remove offensive odors.

8. The process of making sulphate wood turpentine comprising the treatment of the stripped crude turpentine material with ethylene diamine removing the ethylene diamine, distilling the residue to obtain refined turpentine, and treating the distillate with a bleaching powder, to develop said product with a pleasing and marketable odor and quality.

9. The process of making sulphate wood turpentine comprising the treatment of the stripped crude turpentine material with ethylene diamine removing the ethylene diamine, distilling the residue to obtain refined turpentine, washing the distillate with water by agitation with air, removing the water, and treating the washed turpentine material with a bleaching powder, to develop said product with a pleasing and marketable odor and quality.

10. The process of making sulphate wood turpentine comprising the treatment of the stripped crude turpentine material with from substantially 0.2 to 0.5 per cent by weight of ethylene diamine of substantially sixty per cent free base, removing the ethylene diamine, distilling the residue to obtain refined turpentine, treating the distillate with a bleaching powder solution containing approximately three per cent available chlorine, to develop said product with a pleasing and marketable odor and quality.

11. The process of making sulphate wood turpentine comprising the treatment of the stripped crude turpentine material with from substantially 0.2 to 0.5 per cent by weight of ethylene diamine of substantially sixty per cent free base, removing the ethylene diamine, distilling the residue to obtain refined turpentine, washing the distillate with water by agitation with air, removing the water, and treating the washed distillate with a bleaching powder solution containing approximately three per cent available chlorine, to develop said product with a pleasing and marketable odor and quality.

12. The process of making sulphate wood turpentine comprising the boiling of the stripped crude turpentine material with from substantially

0.2 to 0.5 per cent by weight of ethylene diamine of substantially sixty per cent free base, removing the ethylene diamine, distilling the residue to obtain refined turpentine, washing the distillate, removing the water, treating the washed distillate by the addition of from substantially ten to fifteen per cent by volume of a bleaching powder solution containing approximately three per cent available chlorine, and removing the bleaching powder solution, to remove offensive odors and develop a turpentine of marketable odor and quality.

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