Depolymerized lignins contained within black liquor are transferred to an organic solvent, soluble in organic matter and insoluble of aqueous sodium compounds contained within the black liquor. Upon separation of the aqueous sodium compounds from the organic solvent, saturated with contained depolymerized lignins, depolymerized lignins are removed from the organic solvent to result in depolymerized lignins and a separated organic solvent suitable for recycling. Resulting aqueous sodium compounds are concentrated by a multiple effect evaporator to provide liquor to digest wood chips. Whereby depolymerized lignins, substantially free from sodium compounds, are separated from a black liquor. Accordingly this invention modifies black liquor without employing a lime kiln and a recovery boiler, producing a molten smelt.
LIGNINS DERIVED FROM BLACK LIQUOR

BACKGROUND OF THE INVENTION

0001 Liquor containing sodium hydroxide is employed to digest wood chips to produce a pulp and, depolymerize, disperse and dissolve lignins from the wood to yield a black liquor containing sodium hydroxide and depolymerized lignins. A recovery boiler is used to recover the sodium compounds. Lignins contained in concentrated black liquor, upon combustion, furnishes energy contained in the lignins to provide steam.

0002 A state of the art Kraft pulping digestion process employs white liquor containing sodium hydroxide and sodium sulfide and creates a black liquor. After concentration of the black liquor, by means of a multiple-effect evaporator, the concentrated black liquor is sent to a recovery boiler to produce steam and a molten smelt containing sodium carbonate. The smelt of melted sodium compounds flows to a vat where it dissolves into green liquor. Green liquor, removed from the vat, is slaked by calcium oxide to form the sodium carbonate to produce sodium hydroxide. Calcium oxide, as a result, is converted to calcium carbonate which settles at the bottom of a white liquor classifier tank. The resulting calcium carbonate, after substantial separation of adhering white liquor, is transported to a lime kiln where the calcium carbonate is converted to calcium oxide. Thus the black liquor recovery cycle is completed. The pulping operation can be accomplished in batch form or continuous form, for example, a Kamyr continuous digester. Accordingly, it is believed, that only a well defined recovery method has been developed.

0003 Therefore, an object of this invention is to obviate many of the limitations and disadvantages of the prior art to digest wood chips and form a black liquor containing sodium hydroxide.

0004 This invention relates to a method of recycling a solution containing sodium hydroxide without using a lime kiln and a recovery boiler producing a smelt.

0005 A significant object of this invention is to produce lignins significantly free of sodium compounds from the black liquor.

0006 Another object of this invention is to utilize a multiple-effect evaporator, previously required to concentrate black liquor, to remove water from liquor containing sodium hydroxide.

0007 With the above and other objects in view, this invention relates to the novel features and alternatives and combinations presently described in the brief description of the invention.

PHRASEOLOGY APPLIED IN THE INVENTION.

0008 Wood chips in the presence of white liquor, containing sodium hydroxide at an elevated temperature, will digest wood chips to result in cellulose pulp and depolymerize, disperse and dissolve lignins to form black liquor. Lignins are not well structured polymers. When a lignin polymer is fractured the term “depolymerized lignin” is often utilized even though no definite “lignin monomer” exists. The term “depolymerized lignin” is used interchangeably to designate lignins. The term lignins is commonly applied to in a black liquor containing sodium hydroxide and depolymerized lignins. Sodium sulfide, anhydroquinone and polysulfide polymers, provide catalysis to delignify wood chips.

0009 Black liquor, when extracted by an organic solvent to extract depolymerized lignin from the black liquor will form an extractate of an organic solvent of depolymerized lignin and an insoluble raffinate. The term “extractate” establishes a function and the ending ate, such as filtrate. Lignins are prone to self condensation under acidic conditions. Lignins removed from the previously formed extractate, with bound sodium compounds, and mixed with an acidic solution will produce a sodium salt solution or another from the lignins to produce lignins free of sodium and a solution of sodium salts. The solution containing sodium salts is treated in a salt splitting procedure to remove salts and form a sodium hydroxide solution and an acidic solution. The sodium hydroxide solution, combined with the raffinate, containing sodium hydroxide, is evaporated to remove water to concentrate the solution. The concentrated solution, referred to as an “active liquor,” regularly contains about 40% sodium hydroxide to about 10% sodium hydroxide. A salt splitting operation often utilizes a bipolar membrane to remove sodium from the salt and form a sodium hydroxide solution. As a result sodium hydroxide is created from the salt. Concentrated liquor thus formed is skimmed off tall oil. Tall oil resembles a soap having chemically bound sodium. A multiple-effect evaporator, previously required to concentrate black liquor, is capable of being applied to remove water from the solution containing sodium hydroxide to produce “white liquor” for recycle.

0010 For supplementary particulars on lignin, for example, refer to Pulp And Paper Technology, pages 33-36. For information on tall oil, for example, refer to Kirk-Othmer, Encyclopedia Of Chemical Technology, Fifth Edition, Volume 23, pages 615-622. Water splitting is described, in a bulletin provided by Aqualytics, in which a salt solution is converted to an acid and a hydroxide.

BRIEF DESCRIPTION OF THE INVENTION

0011 The present invention, in its broadest aspect, is a method to yield lignins separated from a black liquor containing sodium hydroxide. This invention defines a method to produce a lignin, substantially free of sodium compounds, and a liquor made suitable for reuse to depolymerize, disperse and dissolve additional lignins. The method comprises: providing a black liquor, and an organic solvent. The method, depicted herein, originates from a black liquor containing lignins dispersed and dissolved in a liquor containing sodium hydroxide. Upon subjecting black liquor to extraction by an organic solvent, an extractate of lignins contained within the organic solvent is formed and a raffinate containing sodium hydroxide is also formed. Consequent to separating the extractate, with lignins of bound sodium compounds from the raffinate, and adding an acidic solution, such as one derived from carbon dioxide contained in a flue gas, to the extractate forms sodium salts and renders the lignins insoluble within the extractate. By means of separating the lignins insoluble within the organic solvent and separating the sodium salts contained within the organic solvent, an organic solvent for recycle is obtained. The organic solvent is often selected from the group of hydro-
carbons, organic halogens and alcohols including an individual or a combination thereof. The neutralized extractate forms a phase of a solution containing sodium salts and an insoluble phase of lignins. Separation of the salt phase will provide lignins substantially free of sodium salts. The salt phase is subjected to a salt splitting technique to convert to a solution of sodium hydroxide and an acidic solution.

[0012] Key Features of this Invention are: 
[0013] Separating lignins from sodium compounds contained within a black liquor. Extraction of a black liquor by an organic solvent creates an extractate containing depolymerized lignins.
[0014] Producing depolymerized lignins, substantially free of sodium, from a black liquor. Depolymerized lignins, substantially free of sodium, is suitable to fire boilers to avoid explosions due to the presence of sodium.
[0015] Recycle substantially all of the sodium compounds as sodium hydroxide and sodium sulfide.
[0016] Concentrated active liquor, containing sodium hydroxide, is subjected to recycle for employment to digest wood chips, depolymerize, disperse and dissolve lignin from wood chips to create black liquor.

BRIEF DESCRIPTION OF THE DRAWINGS

[0017] Features that are considered characteristic of this invention are set forth in the appended claims. This invention, however, both as to its origination and method of operations as well as additional advantages will best be understood from the following description when read in conjunction with the accompanying drawings in which:

[0018] FIG. 1 is a flow sheet denoting the invention as set forth in the appended claims.
[0019] FIG. 2 is a flow sheet denoting a method to separate a small amount from a concentrated liquor containing sodium hydroxide employing a multiple-effect evaporator for concentration.
[0020] FIG. 3 is a flow sheet denoting a method for acidifying lignins to form condensed lignins.
[0021] FIG. 4 is a flow sheet denoting a method of settling the extractate to form lignins.
[0022] FIG. 5 is a flow sheet denoting a method of centrifugal division of the extractate to form lignins.
[0023] FIG. 6 is a flow sheet denoting a method for gasification of lignins.

DETAILED DESCRIPTION OF THE PREFERRED EMBODIMENT

[0024] The flow diagram of FIG. 1 illustrates the preferred embodiment of the present invention. In the diagram, rectangles represent stages or functions of the present invention and not necessarily separate components. Arrows indicate direction of flow of material in the method.

[0025] Referring to FIG. 1, black liquor 10, containing depolymerized lignins and sodium hydroxide, is conveyed to counter flow extraction stage 12, within the extraction is achieved by recycled organic solvent 26A to produce a raffinate 16, and an extractate 14. Extractate 14, is conveyed to a heat exchanger 20 for exchanging heat from extractate 14 to establish heated recycled organic solvent 26A and cooled extractate 14A to exchange heat to cooled organic solvent 26. Cooled extractate 14A, containing insoluble lignins is conveyed to separation of lignins stage 22 to form cooled recycled organic solvent 26 and lignins 24. Recycled organic solvent 26A readily dissolves depolymerized lignins from black liquor 10 and forms extractate 14, containing depolymerized lignins, transferred within recycled organic solvent 26A, and a raffinate 16, previously employed as an aqueous sodium hydroxide liquor for depolymerization of lignins. Accordingly recycled organic solvent 26A, is utilized to extract lignins from black liquor 10.

[0026] Separation of lignins stage 22 can be a supplied centrifuge, settling tank or any equipment utilized to separate insoluble lignins and form recycled organic solvent. Organic solvent 26 is ordinarily selected from the group of hydrocarbons, organic cyclic compounds, organic halogens and alcohols including an individual or a combination thereof. Pulp washing, composed of weak black liquor, is customarily combined with black liquor 10.

[0027] Referring to FIG. 2, a flow sheet is portrayed denoting a method to concentrate a raffinate 16, conveyed to a multiple-effect evaporator stage 18, to remove water 28, and form concentrated raffinate 30A, containing tail oil. Concentrated raffinate 30A, containing tail oil, is conveyed to a skimming tail oil stage 32 to skimming tail oil 34 and form concentrated raffinate 30, substantially devoid of tail oil. Herebefore skimming tail oil 34, can be reacted with an acid to form crude tail oil. Concentrated raffinate 30, regularly contains about 40% sodium hydroxide to about 10% sodium hydroxide. Concentrated raffinate 30 is employed to depolymerize, disperse and dissolve lignins contained within wood chips, to form additional black liquor 10.

[0028] Referring to FIG. 3, lignins 24 is conveyed to lignins condensation stage 36 and combined with acid 38, to transform lignins to self condensation lignins to form condensed lignins mixture 40 for transport to condensed lignins separation stage 42 to form condensed lignins 40B and additionally separate sodium salt solution 40A. Lignins condensation stage 36, regularly releases gaseous hydrogen sulfide 38A which is reacted with sodium hydroxide to form sodium sulfide.

[0029] Referring to FIG. 4, a flow sheet is portrayed denoting a method to transport cooled extractate 14A, containing insoluble lignins, to a settling tank 22A, to settle and provide lignins 24 and cooled organic solvent 26. Cooled extractate 14A is generally cooled from cooled water obtained from a cooling tower.

[0030] Referring to FIG. 5, a flow sheet is portrayed denoting a method to convey cooled extractate 14A, containing insoluble lignins, to a centrifugal separation stage 22B, to separate and provide lignins 24 and cooled organic solvent 26.

[0031] Referring to FIG. 6, a flow sheet is portrayed denoting a method to convert lignins 24 by steam 48 within gasification stage 46, to produce gasification products 50 to function as a fuel.

What is claimed is:

1. A method to produce lignins substantially free from sodium compounds, which comprises:
providing a black liquor containing depolymerized lignins, and
providing an organic solvent for extraction, and
subjecting said black liquor to extraction by said organic solvent to form an extractate of said depolymerized lignins contained in said organic solvent and a raffinate containing sodium hydroxide and, separating said extractate from said raffinate, and
separating said depolymerized lignins from said extractate to provide depolymerized lignins free of said organic solvent, and
recycling the heretofore separated organic solvent for extraction of additional black liquor, and
adding an acidic solution to the heretofore separated depolymerized lignins to form sodium salts and lignins insoluble within the sodium salts, and
separating lignins heretofore derived from the insoluble sodium salts whereby lignins substantially free of sodium compounds are obtained from a black liquor.

2. The method as described in claim 1 wherein said sodium salts are subjected to a bipolar membrane for a salt splitting operation from said sodium salts creates sodium hydroxide and creates an acid.

3. The method as described in claim 3 wherein sodium hydroxide is combined with said raffinate.

4. The method as described in claim 1 wherein said acidic solution added to said extractate forms gaseous hydrogen sulfide.

5. The method as described in claim 5 wherein the gaseous hydrogen sulfide is added to said raffinate and reacted with sodium hydroxide contained within said raffinate to form sodium sulfide.

6. The method as described in claim 1 wherein the acid is derived from carbon dioxide contained in a flue gas.

7. The method as described in claim 1 wherein said raffinate is concentrated within a multiple effect evaporator.

8. The method as described in claim 7 wherein the concentrated raffinate contains about 40% sodium hydroxide to about 10% sodium hydroxide.

9. The method as described in claim 7 wherein the concentrated raffinate is substantially skimmed of tall oil.

10. The method as described in claim 1 wherein said black liquor contains sodium sulfide.

11. The method as described in claim 1 wherein said black liquor contains polysulfide polymers.

12. The method as described in claim 1 wherein said black liquor contains anthraquinone.

13. The method as described in claim 1 wherein black liquor is supplied from kraft pulping digestion.

14. The method as described in claim 1 wherein black liquor is supplied from soda pulping digestion.

15. The method as described in claim 1 wherein said extraction by said organic solvent is accomplished by counter flow of said organic solvent.

16. The method as described in claim 1 wherein said organic solvent is selected from the group of hydrocarbons, organic halogens and alcohols including an individual or a combination thereof.

17. The method as described in claim 1 wherein said extractate creates depolymerized lignins substantially devoid of organic solvent.

18. The method as described in claim 17 wherein the extractate is cooled to form depolymerized lignins.

19. The method as described in claim 1 wherein said extractate is subjected to a centrifuge to obtain organic solvent for recycle, and depolymerized lignins substantially devoid of the organic solvent.

20. The method as described in claim 1 wherein said depolymerized lignins are subject to gasification to produce gasification products which function as a fuel.

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