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REDWOOD BARK COOKING PROCESS AND RESULTING PRODUCTS

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The present invention relates generally to the treatment of redwood bark or its components made up of redwood bark fibrous material and redwood bark dust. More specifically, the present invention relates to an improved cooking process and includes within its scope the resulting modified redwood bark products.

Bark from the California redwood, i. e. a soft wood or conifer known as *Sequoia sempervirens*, has for the most part been considered a waste material of little economic value. The bark contains a relatively high percentage of complex chemicals (as evidenced by the resistance of the redwood tree to decay) and is made up of about equal proportions of redwood bark fibrous material and redwood bark dust containing unique high molecular weight phenolic acids characterized by their relatively high content of acidic carboxyl groups and negligible content of methoxyl groups. While the disposal of the bark has been a problem in the redwood lumber industry for many years, up until the time of the present invention no proposed solution providing for the use of both the bark fibrous material and bark dust had proven commercially practical.

The principal object of the present invention is to provide the art with novel products having utility in various fields by a commercially practical process involving the treatment of redwood bark.

Other objects of the present invention will be apparent as the description proceeds hereinafter.

We have discovered a commercially practical process for treating redwood bark in which all of the materials in the bark are converted to products or compositions having utility in various fields. The process comprises the cooking of the redwood bark, preferably in the form of chips or other comminuted form, in an aqueous alkali sulfite (e. g. sodium sulfite or bisulfite) cooking liquor until the bark acids are solubilized and the separation of the resulting insoluble fibrous material from the cooking liquor. The fibrous material made up of the "insolubles" contains along with the bark fiber short cells insoluble in the cooking liquor together with lignin and pectin-like material which has been modified by the sulfite cooking treatment, as well as other insoluble chemicals. The cooking liquor made up of the "solubles" contains the soluble chemicals which have been modified by the sulfite cooking treatment including alkali salts of sulfonated redwood bark organic acids.

We have discovered that the fibrous material which contains all of the insolubles, i. e. the alkali sulfite digested redwood bark pulp, can be readily converted by standard means and without the addition of resins, adhesives, etc. into insulation board possessing very desirable strengths and other properties. A board two inches thick may be made from the bark pulp, for example, which will weigh less than two pounds per square foot, i. e. have a density of only about ten pounds per cubic foot. A board of this type will have a transverse breaking strength of about 40–80 pounds per square inch and will show a deformation of not more than 5 per cent under a loading of 200 pounds per square foot and will regain, on release of pressure, all but about 1.5 per cent. The heat transmission of a board of this type is also of the low value desired in the insulation field, as the coefficient of thermal conductivity or *k* factor (B. t. u. in./hr. ft.² deg. F.) is around 0.285. (For a reference to *k*, see "Low Temperature Insulation, Physical Properties—Test Data," published by the Cork Insulation Man-

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ufactures Association, A. I. A. File No. 37–B–4, page 8, 1937).

We have also discovered that the soluble material in the cooking liquor, i. e. the alkali sulfite digested redwood bark solubles, is particularly adaptable for use as an additive for oil well drilling muds. Investigations have shown, for example, that the soluble material in the liquor may be used to control viscosity, water loss and gel strength in ordinary muds as well as in lime-base muds, e. g. muds with a pH around 11–12. This latter characteristic is unexpected as quebracho is not adaptable for use in lime-base muds and redwood bark chemicals which have not been sulfonated, i. e. subjected to the sulfite cooking treatment, are not generally adaptable for use at pH values above 10. While concentrates of the cooking liquor may be used, for most purposes it is preferred to isolate the soluble solid material by evaporating the cooking liquor and then spray drying. Where the free acids are desired in place of the salts, they may be readily obtained by running an aqueous solution of the salts through an ion exchange column.

The following example of a preferred method of operation will serve to illustrate the present invention.

Example

About 100 parts by weight of redwood bark chips is first placed in a digester. About 500 parts of cooking liquor made up of water containing about 15 parts of sodium sulfite in solution is then added to the bark. The resulting mixture is then cooked at a temperature of about 160° C. for about 1.5 hours. At the end of the cooking period the digester is discharged into the blow pit where the sodium sulfite digested redwood bark pulp is separated from the cooking liquor. The cooking liquor containing the sodium sulfite digested redwood bark solubles and made up for the most part of sodium salts of the sulfonated redwood bark acids, is evaporated to a solid content of about 20–25 per cent and then spray dried. Before use the fibrous pulp is preferably washed with water and the washings recovered and used to make up the liquor for the next cooking operation.

It will be understood in the above example, where the ratio of liquor to solids is about 5:1, that other ratios may be employed. Sufficient water should, of course, be used to bring the cooking liquor containing the sulfite salt into contact with the bark. It will also be understood in the above example where the ratio of bark to the sulfite salt is around 6.6:1 that other ratios may be employed. Investigations, however, show that this ratio should not be greater than about 20:1, i. e. that at least about 5 per cent sulfite salt should be used based on the weight of the bark. In most cases the use of 10–20 per cent sodium sulfite based on the weight of the bark is preferred. Larger amounts of sulfite salt, however, may be used if desired, although the use of excessive material over and above about 25 per cent sulfite salt based on the weight of the bark does not result in an improvement in the properties of the reaction products.

The cooking temperatures and times may be varied with the lower cooking temperatures requiring longer cooking periods and the higher cooking temperatures requiring shorter cooking periods. Investigations in this connection show that for most commercial operations a temperature range of about 90–170° C. is satisfactory for carrying out the cooking operation. The use of temperatures below 90° C. requires relatively long cooking periods before the chemicals to be solubilized are solubilized (at which time the bark structure disintegrates upon working), while the use of temperatures above 170° C. tends to deleteriously affect the desired properties of the reaction products.

In place of sodium sulfite used in the above example, other alkali sulfite salts such as the alkali metal, e. g. potassium, and ammonium sulfites or bisulfites may be used. When a bisulfite salt is used as the alkali sulfite salt in place of a salt such as sodium sulfite, more salt is required to sulfonate and solubilize the bark chemicals, i. e. achieve the same degree of cooking. The solubility of the alkali sulfite salts in water is an essential characteristic of the sulfite reaction product. The alkali sulfite salts may also be used in combination with alkali metal

hydroxides, one of the preferred reaction mixtures being made up of 5 per cent sodium hydroxide and 20 per cent sodium sulfite based on the weight of the bark.

The bark pulp produced by the present invention, and which contains in addition to the fiber all of the materials remaining insoluble after the sulfite cook, may be made into insulation board as indicated above or be combined with other materials. A satisfactory board, for example, may be made by mixing about 50 per cent of bark pulp with 50 per cent of plastic redwood pulp produced in accordance with the process described in U. S. Patent 2,392,162. A board made up of this combination has just as good a *k* factor and compression property as a 100 per cent alkali sulfite digested redwood bark pulp board and will have a somewhat better transverse strength. Sawdust without any added resins, etc. may also be used in place of the plastic pulp. A board made up of this combination exhibits a better compression but has a somewhat reduced transverse strength and the *k* factor goes up to about 0.32. A blending agent of loose fill insulation made up of redwood bark fiber separated mechanically from redwood bark slabs may also be used in place of the plastic pulp or sawdust. A board made up of this combination (50 per cent alkali sulfite digested redwood bark pulp and 50 per cent uncooked redwood bark fiber) has a *k* factor around 0.285, a transverse strength between the sawdust and plastic pulp boards, but a somewhat poorer compression property. Where the redwood bark fiber is mechanically separated from the bark and used as above, the remaining bark dust which makes up about 50 per cent of the bark, may be cooked in the alkali sulfite cooking liquor as described in the above example. The preferred cooking conditions (160° C. for about 1.5 hours) are the same except that about 20 per cent sodium sulfite based on the weight of the dust is employed due to the relatively high concentration of chemicals (phenolic acids) in the dust. The cooking liquor obtained is substantially the same as the liquor obtained on cooking the whole bark and may be used as indicated above in the drilling mud additive field. As in the example, after the bark dust chemicals are solubilized, the cooking liquor is separated from any insoluble material and then preferably evaporated to a solid content of about 20–25 per cent and spray dried.

The insulation boards produced with the alkali sulfite digested redwood bark pulp of the present invention may be sized with wax emulsions or a variety of petroleum sizes or with rosin sizes. Investigations have shown a sized board after fifteen days immersion in water to pick up less than 25 per cent by volume of water. With a ten per cent rosin size the water pickup on immersion for fifteen days has been found to be not greater than 20 volume per cent.

The present invention provides a commercially practical process for treating redwood bark. It solves the bark disposal problem and provides the art with novel redwood

bark derivatives of value in the insulation and drilling mud additive fields.

We claim:

1. A process which comprises cooking redwood bark in an aqueous alkali sulfite liquor containing at least about 5 per cent sulfite salt based on the weight of the bark, at a temperature of about 90–170° C. until the redwood bark acids are solubilized.

2. A process which comprises cooking comminuted redwood bark in an aqueous sodium sulfite liquor containing about 10–20 per cent sulfite salt based on the weight of the bark, at a temperature of about 90–170° C., and separating the sodium sulfite digested redwood bark pulp containing all of the insoluble material from the cooking liquor containing the sodium sulfite digested redwood bark solubles.

3. In a process of preparing alkali sulfite digested redwood bark pulp and alkali sulfite digested redwood bark solubles, the steps which comprise cooking redwood bark chips in an aqueous sodium sulfite liquor in which the liquor to solid ratio is about 5:1 and the bark to sodium sulfite ratio is about 6.6:1, for about 1.5 hours at about 160° C., and then separating the bark pulp containing the insoluble material from the cooking liquor containing the soluble material.

4. A process of making alkali sulfite digested redwood bark solubles consisting essentially of alkali salts of sulfonated redwood bark acids, which comprises cooking redwood bark dust in an aqueous alkali sulfite liquor containing at least about 20 per cent sulfite salt based on the weight of the dust at a temperature of about 90–170° C. until the bark dust acids are solubilized.

5. As an article of manufacture, an insulation board comprising consolidated alkali sulfite digested redwood bark pulp.

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