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IMPREGNATION OF FIBROUS MATERIALS

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This invention relates to improvements in the art of impregnating fibrous materials. The invention is especially applicable for impregnating and hardening of sheets, plates, blocks, etc., such as used in clutches, brakes, etc., when thorough oxydation by air is not possible.

It has always been a problem to impregnate fibrous materials, such as asbestos, with a binder which will withstand exposure to high temperatures, particularly when the fibrous material consists of thick sheets or plates. Impregnation by drying oils, unless especially prepared, does not give satisfactory results under such conditions, as in the case of thick plates the oxygen of the air cannot penetrate into the center of such plates, in order to effect drying by oxydation. Therefore, the center will remain softer than the outside.

A typical example for such conditions is a molded brake block compounded from asbestos fiber and other solids with an oil as binder. These brake blocks are cured by exposure to a temperature of 350 to 450 degrees Fahrenheit for many hours. Linseed oil, bodied under vacuum to a high viscosity with very low acidity has been found to be the most suitable material for the purpose, also in combination with phenolic resins.

The present invention utilizes principally cheap raw materials having a high degree of polymerization and a high acidity, such as still bottoms from the distillation of fatty acids.

Such materials consist principally of polymerized triglycerides and fatty acids, the latter also mostly polymerized. The presence of these free acids prevents the coagulation of the still bottoms at the high temperatures prevailing in the fatty acid stills. The fact that the free fatty acids are present in the form of dimers or higher polymers explains the impossibility to distill them off under the vacuum and temperature under which the corresponding monomeric acids distill over.

He discovered that such still bottoms, when heated with polyhydric alcohols, e. g. glycerin, in a quantity sufficient to esterify the free fatty acids in the still bottoms to a point, where all the alcohol is combined to form mono or diesters, the resulting material forms an excellent impregnating and coating base, which upon baking at the usual temperatures of 350 to 450 degrees Fahrenheit solidifies completely due to the completion of esterification, and becomes insoluble and infusible by heat.

He also discovered that such partially esterified

still bottoms are excellent solvents for phenolic resins, probably due to the free hydroxyl groups. In cases where greater hardness of the polymerized binder is required, a solution of such resins in the partially esterified compound was found to be very suitable.

Not only still bottoms, but drying and semi-drying oils can be utilized according to the present invention. As such oils he mentioned linseed oil, perilla oil, China-wood oil, oiticia oil, raw or dehydrated castor oil, walnut oil, poppy seed oil, rapeseed oil, soya bean oil, sunflower oil, fish oils, tall oil, and oils of similar chemical composition.

In utilizing these oils, they are first bodied under heat with or without the addition of fatty acids to a high degree of polymerization and high viscosity. Then a polyhydric alcohol, such as glycerin, sorbitol, pentaerithritol, is added in quantities sufficient to substantially esterify the free fatty acids present in the bodied oil, and heating continued until no more free glycerin is present, but short of gelation.

Mixtures of still bottoms and such oils can also be used. Inasmuch as still bottoms are dark in color, the use of oils or a combination of oils with still bottoms is preferable in cases where light color of the coating or impregnating material is desired.

The esterification may be conducted under atmospheric, increased or preferably reduced, pressure, without, or preferably with mechanical agitation, under cover of inert gases, such as carbon dioxide or nitrogen, in vessels heated by direct fire, electrically or through a jacket, or in any other manner known to the industry of varnish manufacture, oil bodying or resin manufacture.

While a temperature of 400 degrees F. is generally used in the examples cited, a lower temperature is usable particularly when vacuum is applied and low viscosities are desired. Higher temperatures may be applied especially when less polymerized raw materials are used and when higher viscosities are desired in the finished product.

Compounds made according to this invention are suitable for many purposes, e. g. impregnation of gaskets, brake lining and clutch facing, asbestos shingles and tile, etc., also as coating material, priming and finishing coat on asbestos materials, on steel, cast iron and other metals, in the form of sheets, wire, cast or machined parts. Particularly waterproof finishes are obtained by incorporation of suitable phenolic resins or com-

ponents, which form resins during the baking process. Other resins, such as natural gums, urea-formaldehyde-, styrene-, acrylic-, and alkyd-resins may be combined to obtain certain desired effects.

It is not desired that the invention be limited to the specific embodiment described, as many modifications may be resorted to without departing from the spirit of this invention.

Example 1

100 parts of still bottoms from the distillation of linseed-oil fatty acids with an acid value of 90 are heated with 5 parts of glycerin to a temperature of 400 degrees F. under carbon dioxide until the acid value is below 20. Then the product is cooled rapidly. The oil thus obtained has a viscosity of 500 poises or more (depending on the degree of polymerization of the raw material), is soluble in hydrocarbon solvents, and when coated on a metal sheet and baked at 350 degrees F. for 2 hours forms a hard, tough film of good elasticity, infusible by heat and sparingly soluble in all organic solvents.

Example 2

100 parts of soya bean oil pitch (still bottoms from the distillation of soya bean fatty acids) with an acid value of 100 are heated with 15 parts of castor oil to a temperature of 450 degrees F. for 1 hour, then for one half hour at 550 degrees F., then cooled to 400 F., and 5½ parts of glycerin are added and heating at 400 degrees F. continued, until the acid value of the product is below 25.

The product thus obtained is a viscous, brown oil suitable as binder for asbestos fibers in the manufacture of brake linings and clutch facings in place of vacuum bodied linseed oil. The drying of the impregnated lining is effected by baking at 300 degrees F. or more. Lining of one inch or more thickness is thoroughly cured in the center.

Example 3

20 parts of p-tertiary-butyl-phenol formaldehyde resin are dissolved by heating to 300 degrees F. in 80 parts of cotton seed pitch. Then 4 parts of glycerin are added and the mass heated at 400 degrees F. until a sample upon cooling has a viscosity of 500 poises. Then the mass is cooled rapidly to avoid coagulation.

The resulting product gives a harder drying coating or impregnation of even better water resistance than the products from Examples 1 and 2, and is completely infusible and almost insoluble in any solvent.

Example 4

20 parts of oiticia oil, 50 parts of linseed pitch, 20 parts of tall oil and 10 parts of phthalic anhydride are heated with 16 parts of sorbitol (85%) to 410 degrees F. with good agitation and after one half hour also under vacuum, until the acid value of the mixture is below 30, then the mass is cooled to 250 degrees F. and 50 parts of toluol are added.

The resulting varnish is suitable as insulating varnish for wire coating of excellent hardness and flexibility.

Example 5

80 parts of raw linseed oil are heated while carbon dioxide is passed through the oil with 20 parts of fatty acids, obtained by saponification of dehydrated castor oil to a temperature of 580 degrees F. until the viscosity of a sample is 35

poises at 70 degrees F. Then the acid value is obtained by titration of a sample and after the mass is allowed to cool to 400 degrees F., glycerin is slowly added in an amount of 1.2 times the theoretical amount calculated from the acid value. Heating is continued without reflux until the acid value is below 25.

A medium light colored oil is obtained of high viscosity, which dries to a very durable film when baked at temperatures above 250 degrees F. and is suitable as coating and impregnating material.

What is claimed is:

1. In the art of impregnating of fibrous materials those steps which consist in adding to a mixture of polymerized fatty acids and polymerized fatty acid esters an amount of polyhydric alcohols substantially sufficient to esterify all the free fatty acids contained in said mixture; in esterifying at first only a part of said free fatty acids with said polyhydric alcohols to obtain a viscous oil; in impregnating the fibrous materials to be treated with said viscous oil; in solidifying said oil by subjecting the impregnated materials to a baking process to substantially finish the esterification of the free acids contained in said mixture.

2. In the art of impregnating of fibrous materials those steps which consist in adding to a mixture of polymerized fatty acids and polymerized fatty acid esters an amount of polyhydric alcohols substantially sufficient to esterify all the free fatty acids contained in said mixture; in esterifying at first only a part of said free fatty acids with said polyhydric alcohols to obtain a viscous oil; in dissolving an oil-soluble resin in the raw mixture or in the product resulting from the partial esterification of said mixture; in impregnating the fibrous materials to be treated with said viscous oil; in solidifying said oil by subjecting the impregnated material to a baking process to substantially finish the esterification of the free acids contained in said mixture.

3. In the art of impregnating of fibrous materials, the process consisting in esterifying a part of the free acids contained in still bottoms from the distillation of fatty acids with polyhydric alcohols; in applying the liquid product resulting from the partial esterification to the material to be impregnated and in substantially finishing the esterification of the free acids contained in said still bottoms, by subjecting said material to a baking process.

4. In the manufacture of brake linings and clutch facings the process of thoroughly impregnating and binding together the asbestos fibers of the linings, comprising the heating of still bottoms from the distillation of fatty acids with polyhydric alcohols to partially esterify said still bottoms; impregnating the asbestos fibers of said linings with the viscous oil resulting from said partial esterification of said still bottoms and subjecting the linings, to a baking process.

5. In the manufacture of brake linings and clutch facings the process of thoroughly impregnating and binding together the asbestos fibers of the linings, comprising the heating of still bottoms from the distillation of soya bean fatty acids with castor oil and glycerin to partially esterify said soya bean oil pitch; impregnating the asbestos fibers of said linings with the viscous oil resulting from said partial esterification of the soya bean oil pitch and subjecting the linings, to a baking process.

6. In the art of impregnating of fibrous materials, the process consisting in polymerizing drying oils in the presence of fatty acids to high viscosity; in partially esterifying the free acids contained in said polymerized drying oils with polyhydric alcohols; in applying the product resulting from the partial esterification to the material to be impregnated and in substantially finishing the esterification of the free acids contained in said polymerized drying oils by subjecting said material to a baking process.

7. The process of impregnating of fibrous materials, consisting in polymerizing raw linseed oil

5 in the presence of fatty acids obtained by the saponification of dehydrated castor oil, in adding an amount of glycerine substantially sufficient to esterify all the free fatty acids contained in said polymerized products; in partially esterifying the free acids contained in said polymerized products; in applying the resulting oil to the material to be impregnated and in substantially finishing the esterification by subjecting said material to a baking process.

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