

1

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WOOD FINISHING COMPOSITION AND METHOD OF MAKING

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This invention relates to a wood finishing composition, more particularly a composition of the penetrating seal type adapted for the finishing of wood flooring and a method of making the finish.

The penetrating seal finish in accordance with this invention uniformly penetrates the surface and the immediate sub-surface of hardwoods usable for flooring and thus provides a high degree of durability. Further, due to the uniformity of its penetration, it tends to prevent worn areas in flooring and obviates the necessity of removing any excess of the finish which has failed to penetrate.

Further, the finish in accordance with this invention provides a uniform finish for wood having a pore structure varying from large to small in the same piece. Further, it gives a uniform finish to a floor or unit of floor manufacture formed of different woods. This is of great utility, since it enables the flooring manufacturer and the floor layer to use mixed floor wood, such as hackberry, elm, pecan, sycamore, ash, beech, persimmon, holly, locust and southern maple indiscriminately within the same unit. The customary hardwoods such as oak, maple, birch, and the like can be used either by themselves or also indiscriminately in the same unit.

The finish according to this invention lends itself to use in factory finishing where heat for curing or drying the finish is available, since it will dry tack-free in less than two minutes when exposed to conventionally used infrared lamp banks.

The finish when used in the field will air-dry in about four hours and may be combined with a filler-pigment so that on application of the finish the floor is at once filled, colored and finished.

The penetrating seal composition in accordance with this invention comprises a resin which is formed by reacting drying oil fatty acids, a pentaerythritol ester of rosin, dilinoleic acid, pentaerythritol and phthalic anhydride.

By way of more specific example, the drying oil fatty acids may be, for example, drying oil fatty acids free of both elaeostearic acid and licanic acid, such as linseed oil, soybean oil or perilla oil. A number of drying oil fatty acids containing elaeostearic acid and licanic acid, for example, oiticica oil or China-wood oil, may be substituted provided that the total of the elaeostearic acid and the licanic acid taken together does not exceed 40% by weight of the total oil fatty acids used.

The drying oil fatty acids referred to herein, including the specific examples, are the fatty acids in the normal commercial proportions derived from the specified oils.

The proportions can be modified but must not contain over 16% saturated fatty acids or over 40% oleic acid. Dilinoleic acid is readily available commercially.

The pentaerythritol ester of rosin will be the reaction product of pentaerythritol and rosin reacted to have an acid number of less than 20. This starting material is well known to the art and readily available commercially

2

as, for example, "Pentalyn A," which is manufactured by the Hercules Powder Company.

More specifically, in the formation of the resin used in the penetrating seal finish in accordance with this invention, the drying oil fatty acids will be present in an amount from about 30 to 50% by weight of the resin. As set forth above, the drying oil fatty acids may contain a total of from 0 to 40% elaeostearic acid and licanic acid. The pentaerythritol ester of rosin will be present in an amount of from about 18 to 35% by weight of the resin. The dilinoleic acid will be present in an amount of from about 5 to 15% by weight of the resin. The pentaerythritol will be present in an amount of from about 10 to 15% by weight of the resin. The ingredients in the amounts described will be batched together and a major portion reacted to completion to have an acid number of about 40 or less.

Phthalic anhydride will be used in an amount of from about 11 to 20% by weight of the resin and reacted with the above mentioned ingredients, the resinous product thus formed being reacted to substantial completion.

Preferably the actual acid number of the resin thus constituted will be from about 40 to 60, the products in this range having many advantages over the broader range products. In any event, the acid number must not be lower than 20 or higher than 85. The difference between the actual and calculated acid numbers is attributable to impurities found in commercially available products and to slight losses during manufacture and not to any substantial incompleteness of reaction, the reaction being preferably over 97% complete and, in all events, must be over 95% complete.

In order to avoid the confusion resulting from theoretical reactions, reactions with pure materials approaching the theoretical, and reactions with commercial materials, the calculated acid numbers used herein were determined by using the reacting purity of the commercial materials. By taking the reacting purity of pentaerythritol as 88%, we automatically compensate for the impurity of phthalic anhydride and the fatty acids. The following are the theoretical acid numbers used:

Drying oil fatty acids	200
Dilinoleic acid	200
Pentaerythritol ester of rosin	14
Phthalic anhydride	756

1 gram of pentaerythritol has the neutralizing effect of 1645 milligrams of KOH. Thus, taking the reaction purity of pentaerythritol as 88%, 1 gram pentaerythritol has the neutralizing effect of 1447.6 milligrams of KOH. When the resin is prepared by fusion the acid number may be slightly less than the calculated acid number due to a slight volatilization of the phthalic anhydride or to the fact that the pentaerythritol was slightly purer than assumed.

It is important to select the reaction times and temperatures to obtain completeness of reactions as indicated above. The times and temperatures will vary somewhat depending upon the selection of ingredients. A temperature range of 400° F. to 450° F. will be satisfactory. The reaction time will be determined by close watching of the acid number or by azeotropic distillation and weighing the water, both means giving satisfactory indication of the completeness of reaction.

The drying oil fatty acids, the pentaerythritol ester of rosin, the dilinoleic acid and the pentaerythritol will be first reacted in, for example, a conventional jacketed reactor at a temperature of from about 400 to 450° F. and the initial reaction carried to a completeness of over 50%. A time of about one hour is required.

The phthalic anhydride will then be added and the

3

temperature raised to within about the range of 400 to 450° F. The reaction will go to the completion of 97 to 98% in from two to three hours.

The resin product thus formed is added to a solvent in a cooling tank in order to give the final product a viscosity of A1 to A3 (Gardner scale). In order to achieve this viscosity the product at this stage will comprise from about 25 to 50% resin solids. Solvents conventionally used in forming floor finishes may be used. Thus, for example, conventional floor finishing solvents such as aromatic petroleum solvents having a kauri butanol range of 50 to 80; mineral spirits or naphtha each having a kauri butanol number of about 37; coal tar solvents, such as benzene, toluene, xylene; methyl acetone, butyl Cellosolve and other solvents which are well known to the art as floor finishing solvents, may be used.

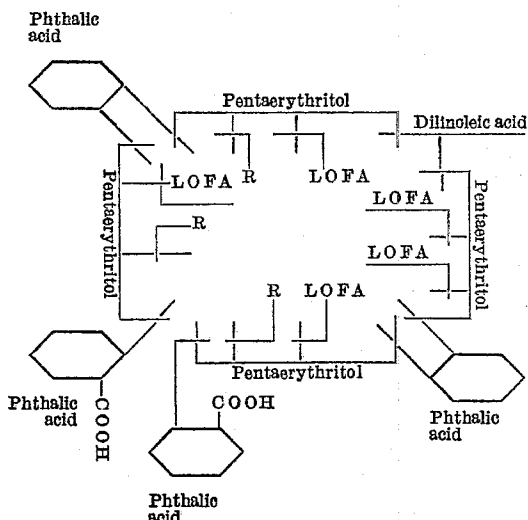
One or more driers will then be added to the preparation. Conventional cobalt driers and manganese driers are satisfactory. Mixed cobalt and manganese driers are satisfactory. Thus, for example, cobalt naphthenate, cobalt tallate, manganese naphthenate and manganese tallate, or mixtures thereof, are satisfactory. It is desirable to have calcium present in the drier.

By way of more specific example, the drier may be a combination drier having a cobalt content of .06 to .12% by weight, a manganese content of .15 to .3% by weight and calcium .05 to .15% by weight of the resin.

Where it is desired, a filler such as, for example, silica, asbestine, gypsum, diatomaceous earth, kieselguhr, calcium carbonate and talc may be included in conventional amounts.

Further, pigment such as, for example, raw sienna, burnt umber, iron oxide, titanium dioxide, burnt sienna, lamp black, Prussian blue, precipitated lakes, zinc chromate and chrome yellow may be added.

There is given below a schematic representation of the resin product of this invention as used in the penetrating seal finish where linseed oil fatty acids are used. It will be understood that the schematic representation is of only one of the many isomeric forms which the product can take. In the schematic representation R equals rosin, LOFA equals linseed oil fatty acid, the references to the constituents of the product being made to the starting material and the crossing of lines indicating linking of the starting materials through condensation of hydroxyl and carboxyl groups. The phthalic acid is derived from the phthalic anhydride which is used as a starting material, the phthalic anhydride acting as a dehydrating agent forming phthalic acid and promoting overall reaction.



The following examples will be further illustrative of the above described resin and a penetrating seal finish including said resin:

4

EXAMPLE 1

Resin

Percent by weight

Linseed oil fatty acids	33.3
Pentaerythritol ester of rosin (Acid No. 14)	22.4
Pentaerythritol	12.0
Dilinoic acid	15.0
Phthalic anhydride	17.3
	100.0

Additional ingredients

Mineral spirits—kauri butanol No. 37, 150% by weight of resin

15 Drier:

Cobalt naphthenate—cobalt content of .08% by weight of resin

Manganese naphthenate—manganese content of .20% by weight of resin

Calcium naphthenate—calcium content of .10% by weight of resin

The linseed oil fatty acids, pentaerythritol, dilinoic acid and pentaerythritol ester of rosin of the above formula were heated at 420° F. for one hour to react to 62% of completion. The phthalic anhydride was then added and the temperature again raised to 420° F. and maintained for two hours. The resulting product had an acid number of 55, and a calculated acid number of 56.8.

The thus formed resin was run into the solvent contained in a cooling tank and the drier combination added. The resulting product had a viscosity of A2.

EXAMPLE 2

Resin

Percent by weight

Soybean oil fatty acids	41.0
Pentaerythritol ester of rosin (Acid No. 14)	21.8
Pentaerythritol	11.7
Dilinoic acid	14.5
Phthalic anhydride	11.0
	100.0

Additional ingredients

45 Petroleum aromatic solvent—kauri butanol No. 74 to produce solution of 40% resin solids, 150% by weight of resin

Drier:

50 Cobalt naphthenate—cobalt content of .06% by weight of resin

Manganese naphthenate—manganese content of .30% by weight of resin

Calcium naphthenate—calcium content of .10% by weight of resin

55 The soybean oil fatty acids, pentaerythritol, dilinoic acid and pentaerythritol ester of rosin of the above formula were heated at 410° F. for 70 minutes to react to 70% of completion. The phthalic anhydride was then added and the temperature again raised to 420° F. and maintained for two hours. The resulting product had an acid number of 28 and a calculated acid number of 27.8.

60 The thus formed resin was run into the solvent contained in a cooling tank and the drier combination added. The resulting product had a viscosity of A2.

EXAMPLE 3

Resin

Percent by weight

Perilla oil fatty acids	31.0
Pentaerythritol ester of rosin (Acid No. 14)	26.8
Pentaerythritol	11.7
Dilinoic acid	14.5
Phthalic anhydride	16.0
	100.0

75

5

Additional ingredients

Mineral spirits—B. P. 330° F. to 345° F.—kauri butanol No. 37 to produce solution of 40% resin solids, 150% by weight of resin

Drier:

Cobalt naphthenate—cobalt content of .12% by weight of resin

Manganese naphthenate—manganese content of .20% by weight of resin

Calcium naphthenate—calcium content of .05% by weight of resin

The perilla oil fatty acids, pentaerythritol, dilinoleic acid and pentaerythritol ester of rosin of the above formula were heated at 440° F. for one hour to reach to 72% completion. The phthalic anhydride was then added and the temperature again raised to 410° F. and maintained for 100 minutes. The resulting product had an acid number of 48 and a calculated acid number of 46.4.

The thus formed resin was run into the solvent contained in a cooling tank and the drier combination added. The resulting product had a viscosity of A3.

EXAMPLE 4*Resin*

	Percent by weight
Linseed oil fatty acids.....	20.7
China-wood oil fatty acids.....	13.0
Pentaerythritol ester of rosin (Acid No. 14).....	21.8
Pentaerythritol	13.5
Dilinoleic acid.....	11.0
Phthalic anhydride.....	20.0
	100.0

Additional ingredients

Xylene to produce a solution of 40% resin solids, 150% by weight of resin

Drier:

Cobalt naphthenate—cobalt content of .08% by weight of resin

Manganese naphthenate—manganese content of 20% by weight of resin

Calcium naphthenate—calcium content of .05% by weight of resin

The linseed oil fatty acids, China-wood oil fatty acids, pentaerythritol, dilinoleic acid and pentaerythritol ester of rosin of the above formula were heated at 420° F. for one hour to react to 63% of completion. The phthalic anhydride was then added and the temperature again raised to 420° F. and maintained for two and one-half hours. The resulting product had an acid number of 51 and a calculated acid number of 48.2.

The thus formed resin was run into the solvent contained in a cooling tank and the drier combination added. The resulting product had a viscosity of A3.

EXAMPLE 5*Resin*

	Percent by weight
Linseed oil fatty acids.....	30.0
Pentaerythritol ester of rosin (Acid No. 14).....	35.0
Pentaerythritol	11.7
Dilinoleic acid.....	5.4
Phthalic anhydride.....	17.9
	100.0

Additional ingredients

Toluene and mineral spirits mixture having kauri butanol No. 55 to produce solution of 40% resin solids, 150% by weight of resin

6

Drier:

Cobalt naphthenate—cobalt content of .05% by weight of resin

Manganese naphthenate—manganese content of .20% by weight of resin

Calcium naphthenate—calcium content of .20% by weight of resin

The linseed oil fatty acids, pentaerythritol, dilinoleic acid and pentaerythritol ester of rosin of the above formula were heated at 420° F. for one hour to react to 60% of completion. The phthalic anhydride was then added and the temperature again raised to 440° F. and maintained for two hours. The resulting product had an acid number of 43 and a calculated acid number of 41.6.

The thus formed resin was run into the solvent contained in a cooling tank and the drier combination added. The resulting product had a viscosity of A2.

EXAMPLE 6*Resin*

	Percent by weight
Linseed oil fatty acids.....	36.0
Pentaerythritol ester of rosin (Acid No. 14).....	21.8
Pentaerythritol.....	11.7
Dilinoleic acid.....	14.6
Phthalic anhydride.....	15.9
	100.0

Additional ingredients

Benzene having a kauri butanol No. 60 to provide a solution of 10% resin solids, 150% by weight of resin

Drier:

Cobalt tallate, .43% by weight of resin

The linseed oil fatty acids, pentaerythritol, dilinoleic acid and pentaerythritol ester of rosin of the above formula were heated at 440° F. for 45 minutes. The phthalic anhydride was then added and the temperature again raised to 440° F. and maintained for one hour. The resulting product had an acid number of 54.4 and a calculated acid number of 55.0.

The thus formed resin was run into the solvent contained in a cooling tank and the drier combination added. The resulting product had a viscosity of A1.

EXAMPLE 7*Resin*

	Percent by weight
Linseed oil fatty acids.....	41.0
Pentaerythritol ester of rosin (Acid No. 14).....	21.8
Pentaerythritol.....	11.7
Dilinoleic acid.....	14.5
Phthalic anhydride.....	11.0
	100.0

Additional ingredients

Mineral spirits—kauri butanol No. 37, 150% by weight of resin

Drier:

Cobalt naphthenate—cobalt content of .08% by weight of resin

Manganese naphthenate—manganese content of .20% by weight of resin

Calcium naphthenate—calcium content of .10% by weight of resin

The linseed oil fatty acids, pentaerythritol, dilinoleic acid and pentaerythritol ester of rosin of the above formula were heated at 420° F. for one hour to react to 60% of completion. The phthalic anhydride was then added and the temperature again raised to 420° F. and maintained for two hours. The resulting product had an acid number of 28 and a calculated acid number of 27.8.

The thus formed resin was run into the solvent contained in a cooling tank and the drier combination added. The resulting product had a viscosity of A2.

EXAMPLE 8

Resin	Percent by weight
Linseed oil fatty acids.....	33.7
Pentaerythritol ester of rosin (Acid No. 14).....	21.8
Pentaerythritol.....	14.0
Dilinoleic acid.....	10.5
Phthalic anhydride.....	20.0
	100.0

Additional ingredients

Methyl acetone to produce a solution of 25% resin solids, 300% by weight of resin

Drier:

Manganese naphthenate—manganese content of .30% by weight of resin

The linseed oil fatty acids, pentaerythritol, dilinoleic acid and pentaerythritol ester of rosin of the above formula were heated at 410° F. for 90 minutes to react to 80% of completion. The phthalic anhydride was then added and the temperature again raised to 415° F. and maintained for two hours. The resulting product had an acid number of 40.0 with a calculated acid number of 40.0.

The thus formed resin was run into the solvent contained in a cooling tank and the drier combination added. The resulting product had a viscosity of A3+.

EXAMPLE 9

Resin	Percent by weight
Linseed oil fatty acids.....	47.2
Pentaerythritol ester of rosin (Acid No. 14).....	21.8
Pentaerythritol.....	10.0
Dilinoleic acid.....	5.0
Phthalic anhydride.....	16.0
	100.0

Additional ingredients

Naphtha having a kauri butanol No. 37 to produce a solution of 40% resin solids, 150% by weight of resin

Drier:

Cobalt naphthenate—cobalt content of .14 percent by weight of resin

The linseed oil fatty acids, pentaerythritol, dilinoleic acid and pentaerythritol ester of rosin of the above formula were heated at 440° F. for one hour to react to 75% of completion. The phthalic anhydride was then added and the temperature again raised to 420° F. and maintained for two hours. The resulting product had an acid number of 85 and a calculated acid number of 82.6.

The thus formed resin was run into the solvent contained in a cooling tank and the drier added. The resulting product had a viscosity of A3.

EXAMPLE 10

Resin	Percent by weight
Linseed oil fatty acids.....	31.5
Oitica oil fatty acids.....	15.7
Pentaerythritol ester of rosin (Acid No. 14).....	21.8
Pentaerythritol.....	10.0
Dilinoleic acid.....	5.0
Phthalic anhydride.....	16.0
	100.0

Additional ingredients

Naphtha having a kauri butanol No. 37 to produce a solution of 40% resin solids, 150% by weight of resin

Drier:

Cobalt naphthenate—cobalt content of .14% by weight of resin

The linseed oil fatty acids and oitica oil fatty acids, pentaerythritol, dilinoleic acid and pentaerythritol ester of rosin of the above formula were heated at 410° F. for one hour to react to 63% of completion. The phthalic anhydride was then added and the temperature again raised to 410° F. and maintained for one hour. The resulting product had an acid number of 83, and a calculated acid number of 83.6.

The thus formed resin was run into the solvent contained in a cooling tank and the drier added. The resulting product had a viscosity of A3.

EXAMPLE 11

Resin	Percent by weight
Perilla oil fatty acids.....	31.0
Pentaerythritol ester of rosin (Acid No. 13).....	26.8
Pentaerythritol.....	11.7
Dilinoleic acid.....	14.5
Phthalic anhydride.....	16.0
	100.0

Additional ingredients

Mineral spirits—B. P. 330° F. to 345° F.—kauri butanol No. 37 to produce solution of 40% resin solids, 150% by weight of resin

Drier:

Cobalt naphthenate—cobalt content of .12% by weight of resin

Manganese naphthenate—manganese content of .20% by weight of resin

Calcium naphthenate—calcium content of .05% by weight of resin

The perilla oil fatty acids, pentaerythritol, dilinoleic acid and pentaerythritol ester of rosin of the above formula were heated at 440° F. for one hour to reach to 72% of completion. The phthalic anhydride was then added and the temperature again raised to 410° F. and maintained for 100 minutes. The resulting product had an acid number of 48 and a calculated acid number of 46.4.

The thus formed resin was run into the solvent contained in a cooling tank and the drier combination added. The resulting product had a viscosity of A3.

EXAMPLE 12

Resin	Percent by weight
Linseed oil fatty acids.....	20.7
China wood oil fatty acids.....	13.0
Pentaerythritol ester of rosin (Acid No. 15).....	21.8
Pentaerythritol.....	13.5
Dilinoleic acid.....	11.0
Phthalic anhydride.....	20.0
	100.0

Additional ingredients

Xylene to produce a solution of 40% resin solids, 150% by weight of resin

Drier:

Cobalt naphthenate—cobalt content of .08% by weight of resin

Manganese naphthenate—manganese content of .20% by weight of resin

Calcium naphthenate—calcium content of .05% by weight of resin

The linseed oil fatty acids, China wood oil fatty acids, pentaerythritol, dilinoleic acid and pentaerythritol ester of rosin of the above formula were heated at 420° F. for one hour to react to 63% of completion. The phthalic anhydride was then added and the temperature again raised to 420° F. and maintained for two and one-half hours. The resulting product had an acid number of 51 and a calculated acid number of 48.2.

The thus formed resin was run into the solvent contained in a cooling tank and the drier combination added. The resulting product had a viscosity of A3.

What is claimed is:

1. A penetrating seal finish having a resin comprising the reaction product of drying oil fatty acids in an amount from about 30 to 50% by weight of the resin, and containing not in excess of 16% saturated fatty acids and not in excess of 40% oleic acid, a pentaerythritol ester of rosin having an acid number of less than 20 and being in an amount from about 18 to 35% by weight of the resin, pentaerythritol in an amount from about 10 to 15% by weight of the resin, dilinoleic acid in an amount from about 5 to 15% by weight of the resin, a major portion of all said ingredients reacted to completion and further reacted to substantial completion with phthalic anhydride in an amount from about 11 to 20% by weight of the resin, said resin having an acid number within the range of about 20 to 85 and a solvent, the penetrating seal finish having a viscosity of about A1 to A3 Gardner scale.

2. A penetrating seal finish having a resin comprising the reaction product of drying oil fatty acids in an amount from about 30 to 50% by weight of the resin, and containing not in excess of 16% saturated fatty acids and not in excess of 40% oleic acid, a pentaerythritol ester of rosin having an acid number of less than 20 and being in an amount from about 18 to 35% by weight of the resin, pentaerythritol in an amount from about 10 to 15% by weight of the resin, dilinoleic acid in an amount from about 5 to 15% by weight of the resin, a major portion of all said ingredients reacted to completion and further reacted to substantial completion with phthalic anhydride in an amount from about 11 to 20% by weight of the resin, said resin having an acid number within the range of about 20 to 85, a solvent and a metallic drier, the penetrating seal finish having a viscosity of about A1 to A3 Gardner scale.

3. Method of making a penetrating seal finish resin which comprises heating a mixture of drying oil fatty acids in an amount from about 30 to 50% by weight of the resin, and containing not in excess of 16% saturated fatty acids and not in excess of 40% oleic acid, a pentaerythritol ester of rosin having an acid number of less than 20 and being in an amount from about 18 to 35% by weight of the resin, pentaerythritol in an amount from about 10 to 15% by weight of the resin and dilinoleic acid in an amount from about 5 to 15% by weight of the resin, said heating being carried out at

a temperature within the range of from about 400 to 450° F. to react a major portion of all said ingredients to completion, reacting the thus reacted mixture with phthalic anhydride in an amount from about 11 to 20% by weight of the resin at a temperature of from about 400 to 450° F., said reaction being carried to substantial completion and providing a resin having an acid number within the range of about 20 to 85 and adding said resin to a solvent in a cooling tank.

4. A penetrating seal finish having a resin comprising the reaction product of drying oil fatty acids containing not in excess of 40% by weight of elaeostearic acid and licanic acid, said drying oil fatty acid being in an amount from about 30 to 50% by weight of the resin, and containing not in excess of 16% saturated fatty acids and not in excess of 40% oleic acid, a pentaerythritol ester of rosin having an acid number of less than 20 and being in an amount from about 18 to 35% by weight of the resin, pentaerythritol in an amount from about 10 to 15% by weight of the resin, dilinoleic acid in an amount from about 5 to 15% by weight of the resin, a major portion of all said ingredients reacted to completion and further reacted to substantial completion with phthalic anhydride in an amount from about 11 to 20% by weight of the resin, said resin having an acid number within the range of about 20 to 85 and a solvent, the penetrating seal finish having a viscosity of about A1 to A3 Gardner scale.

5. Method of making a penetrating seal finish resin which comprises heating a mixture of drying oil fatty acids containing a total content of elaeostearic acid and licanic acid not in excess of 40% by weight of the drying oil fatty acids, said drying oil fatty acids being in an amount of from 30 to 50% by weight of the resin, and containing not in excess of 16% saturated fatty acids and not in excess of 40% oleic acid, a pentaerythritol ester of rosin having an acid number of less than 20 and being in an amount from about 18 to 35% by weight of the resin, pentaerythritol in an amount from about 10 to 15% by weight of the resin and dilinoleic acid in an amount from about 5 to 15% by weight of the resin, said heating being carried out at a temperature within the range of from about 400 to 450° F. to react a major portion of all said ingredients to completion, reacting the thus reacted mixture with phthalic anhydride in an amount from about 11 to 20% by weight of the resin at a temperature of from about 400 to 450° F., said reaction being carried to substantial completion and providing a resin having an acid number within the range of about 20 to 85 and adding said resin to a solvent in a cooling tank.

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