UNITED STATES PATENT OFFICE.

DORRIS WHIPPLE, OF NEW YORK, N. Y., ASSIGNOR TO STANDARD CHEMICAL COMPANY, OF BAYONNE, NEW JERSEY, A CORPORATION OF NEW JERSEY.

PROCESS OF PRODUCING A RESINOUS COMPOUND.

1,083,168. Specification of Letters Patent. Patented Dec. 30, 1913.

No Drawing. Application filed November 6, 1909. Serial No. 526,660.

To all whom it may concern:

Be it known that I, DORRIS WHIPPLE, a citizen of the United States, residing at New York, in the county of New York and State of New York, have invented new and useful Improvements in Processes of Producing a New Resinous Compound, (Case A,) of which the following is a specification.

This invention relates to the process of producing a new resinous compound, the product being in the form of a soft, non-oxidizing, non-drying, permanently plastic mass of very high specific electrical resistance factor, which properties admirably adapt the same to the insulation of electrical cables and the like, and to various other uses, said product forming the basis of my co-pending application, Serial No. 526,661, filed of even date herewith.

In carrying out the process any resin which consists essentially of abietic anhydrid or abietic acid, preferably rosin, is placed in an iron still and heated to a temperature of approximately 135° C., which has the effect, of course, of melting the rosin. While the rosin is still in molten condition, and not overheated, moist air is forced through the molten mass for the purpose of oxidizing the abietic acid of the rosin, and until the contents of the still shows a high percentage of what is apparently tetra-oxy-abietic acid. This time will, of course, probably vary with each particular batch of rosin treated, and can be regulated and determined by testing samples from time to time to ascertain the oxygen content, as for example by treating a chloroform solution of the compound with iodin solution and titrating back the uncombined iodin, the proportion of iodin consumed giving the percentage of rosin still left unchanged, or by subjecting the selected specimens to electrical voltage or puncture tests; the conditions of the latter being that two hemispherical poles say 1 c. m. in diameter placed one tenth inch apart with a portion of the specimens filling the gap will require at least 20,000 volts to show a puncture of the specimen; or, in the alternative, twenty sheets of pure Manila paper, each six mils. gage, and previously saturated with the material, are placed between flat disks and compressed to a working distance of about one tenth inch and placed between the terminals of a generator will not be ruptured or punctured at a voltage less than 30,000 calculated on the mean square reading of a Thomson voltmeter.

The chemical changes which take place in the foregoing process cannot be definitely specified, but probably consist in the formation, from abietic anhydrid and abietic acid, of tetra-oxyabietic acid, or more properly, tetra-oxyabietin, by the combined action of the moisture and the oxygen introduced with the moist air, with the further possibility of many subsidiary compounds being formed which are removed in the later oxidation process. The oxidation of the abietic oxid and abietic anhydrid to produce the product cannot be effected by air alone or by steam alone but only by the conjoint action of the two. A further effect of treating the molten rosin, at the temperature indicated, with a blast of moist air is the elimination by oxidation, of all the turpentine products and organic acids which may have been present as impurities in the original rosin.

After the foregoing operation has been conducted for the necessary period of time, as determined by the tests aforesaid, the compound in the still is preferably heated to a temperature of approximately 140° C. and dry air is passed through the same, for the purpose of expelling any uncombined watery vapor, resulting from the previous operation, and to effect a further oxidation of the abietic acid. During this latter stage of the process, great care and considerable skill on the part of the operator is necessary in order to avoid an excessive heat or an excessive oxidation which would tend to carbonize the entire mass and render it unsuitable for the purposes for which it is intended. Too high temperature will be evidenced by the coming over of comparatively large volumes of turpentine vapors, and as all such products in the original rosin were eliminated by the first steps of the process, their presence during the second stage of the operation indicates their formation at the expense of the products resulting from the first step. Resulting from the last step mentioned is the final product, which may be best defined by its peculiar physical characteristics and also by comparison with the rosin from which it is derived. The product is a soft gummy

material, having a specific gravity of approximately .98, and which melts at a temperature about 20° C. lower than the melting point of the rosin from which it was made, and contains many hydroxyl groups.

As is well known, rosin which constitutes the initial substance, is a transparent or translucent resin having a faint turpentine odor, and shows a definite fracture when broken. The specific gravity of rosin is between 1.07 and 1.1, and it is, as is well understood soluble in alcohol, more or less soluble in petroleum spirits, is not soluble in petroleum oils, and on oxidation, either in the air or by artificial means, results in a pulverulent mass. The product of the herein described process is less soluble in petroleum spirits but is freely soluble in petroleum oils. The composition of the resin acid in rosin is approximately abietic acid.

From the foregoing, the essential differences between the initial resin or rosin and the resulting product of the process, as described, are apparent. The ultimate product may be further described as non-drying, incapable of further oxidation under any circumstances or conditions of ordinary use, and retains its soft plastic condition indefinitely; it is free from carbonaceous particles and contains no rosin oil, no pinene derivatives and no volatile organic acids.

Although the product finds many useful applications in the arts, its non-oxidizing and non-drying character, its perfect and permanent plasticity and its high specific electrical resistance factor, to wit, 2.8×10^8 megohms per cubic centimeter, which as compared with rosin is about one to five in favor of the product, render it essentially valuable as an insulating medium for electrical cables, and it is in this particular field that the invention finds its widest application.

What I claim is:

1. The process of producing a new resinous compound, which consists in heating a resin in the presence of moisture to a temperature sufficient to break up the complex turpentine products and volatilize the same together with the volatile organic acids, increasing the temperature and supplying an oxidizing agent.

2. The process of producing a new resinous compound, which consists in heating a resin in the presence of moisture to a temperature sufficient to break up the complex turpentine products and volatilize the same together with the volatile organic acids, increasing the temperature and passing dry air through the molten mass.

3. The process of producing a new resinous compound, which consists in heating moist resin to a temperature of approximately 135° C., increasing the temperature and subjecting the mass to the action of an oxidizing agent.

4. The process of producing a new resinous compound which consists in heating a resin to a temperature of approximately 135° C., passing moist air through the molten mass, heating the residue to approximately a temperature of 140° C. and passing dry air through the mass.

5. The process of producing a new resinous compound which consists in heating a resin at a temperature of approximately 135° C. in the presence of moisture, increasing the temperature and passing air through the mass.

In testimony whereof I have hereunto set my hand in presence of two subscribing witnesses.

DORRIS WHIPPLE.

Witnesses:
MERLE F. ST. JOHN,
HARRIETT E. REESE.