UNITED STATES PATENT OFFICE.

FRANK E. MARINER, OF GULL POINT, FLORIDA, ASSIGNOR TO THE PENSACOLA TAR & TURPENTINE COMPANY, OF GULL POINT, FLORIDA, A CORPORATION OF FLORIDA.

TREATING ROSIN.

1,114,278. Specification of Letters Patent. **Patented Oct. 20, 1914.**

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To all whom it may concern:

Be it known that I, FRANK E. MARINER, a citizen of the United States, residing at Gull Point, in the county of Escambia and State of Florida, have invented a new and useful Improvement in Treating Rosin, of which the following is a specification.

My invention relates to an improved treatment of rosin, or colophony, meaning the residue obtained by distilling turpentine and from which the turpentine is thus separated, for the purpose of obtaining a product, which may properly be termed a "rosinoid" because of its physical resemblance to rosin, though possessing grease-setting property in an extraordinary degree.

It is the practice in manufacturing axle grease and other similar greases to use cheap petroleum oil set through the medium of a lime soap made by saponifying rosin oil with milk of lime. This rosin oil is the distillate obtained by distilling rosin at atmospheric pressure and is known in the art as "first-run" or "kidney" oil, and it contains a grease-setting acid value of between 40 and 50 per cent., though this value rarely exceeds 45 per cent. This acid content of rosin oil, which is the portion of the latter that combines with the milk of lime to produce the rosin soap for setting the grease, is generally understood to be abietic acid, notwithstanding the fact that the rosin from which the rosin oil is produced, and which is approximately all abietic acid, will not properly set grease, nor does it have necessary lubricating qualities. It would therefore seem that in producing rosin oil by distilling rosin, a re-arrangement of its molecular structure takes place, due to the very material decomposition brought about by the heat; and that in the course of this re-arrangement the saponifying agent therein, believed to be abietic acid, contained in the resultant rosin oil is rendered a grease-set, though the precise molecular structure of this agent is not known. It being evident, therefore, that the only value of rosin oil as a grease set is in its acid content of about 45 per cent., it follows that the remaining 55 per cent., or thereabout, of neutral oil is wasted.

By distilling, under a more or less high vacuum, rosin from which the turpentine has been thoroughly extracted by distillation, or with superheated steam, a peculiar distillate is produced which, as I have discovered, possesses superior grease-setting properties. By distilling such rosin in a vacuum of, say, 22 inches at a temperature between 290° C. and 310° C. the distillate contains an acid value of approximately 90 per cent. and this constitutes approximately 90 per cent. of the rosin treated. This acid, which is apparently an isomer of abietic acid, is probably similar to the acid formed by distilling rosin at atmospheric pressure to produce ordinary rosin oil, containing only about 45 per cent. of acid value, as aforesaid. Consequently, about one-half the quantity of my rosinoid product will suffice for the grease-setting purpose in the manufacture of lubricant greases that is required of regular first-run rosin oil.

If the rosin be distilled by the use of steam, the latter should preferably be not only superheated, but should be conducted into the rosin in the still through a perforated pipe or pipes; and the still may be additionally heated with externally-applied fire, which, however, is not necessary, but preferable because more economical, since it obviates the need of carrying highly superheated steam. Moreover, the rosin can be heated in the still to the comparatively-low boiling point essential to carrying it over in the presence of steam, which then becomes superheated by contact with the mass of hot rosin; and the bulk of the rosin may be distilled by a temperature of about 275° C., though the distillation starts at a much lower temperature, while at that specified the distillation proceeds rapidly and steadily and continues until most of the rosin has been distilled over in the form of this peculiar article of rosinoid. The last 8 to 10 per cent. of the rosin, however, requires the temperature to be raised somewhat to carry it over. Practically all of the rosin may be distilled over by this method.

While the distillate is the same, whether produced by the described practice *in vacuo* or by the use of superheated steam, of obtaining the molecular change of the rosin necessary to convert the abietic acid into my product, thereby to enhance its chemical action with milk of lime in manufacturing grease, the latter method is preferable by reason of its being simpler and more economical, it being less expensive in the mat-

ter of apparatus required and easier of operation.

My improved product, while resembling rosin in appearance, presents the physical differences from the ordinary rosin-product of turpentine-distillation, in being susceptible of chewing like chewing gum, and in remaining solid at ordinary temperatures. To possess this last-named characteristic the abietic acid content should be at least about 80 per cent. However, by agitating it while it is cooling after distillation, whereby it is reduced to the form of crystals, it will remain solid at ordinary atmospheric temperatures even though the abietic acid content be lower than 80 per cent., and this particularly, if from 1 to 2 per cent. of water be added to it while undergoing agitation, to promote the crystallization. This property of remaining solid at ordinary temperatures affords the advantage of enabling my improved article to be transported, like rosin, in slack cooperage without material or undesirable change in its condition, while rosin oil requires to be shipped in tight and more expensive packages.

What I claim as new and desire to secure by Letters Patent is—

1. The process which consists in distilling over rosin, from which the turpentine has previously been separated, at a temperature below its boiling point at atmospheric pressure, condensing the distillate, and continuing the distillation until substantially all the rosin has been distilled over, whereby a grease-set high in abietic-acid content is produced.

2. The process which consists in distilling over, with superheated steam, rosin, from which the turpentine has previously been separated, condensing the distillate, and continuing the distillation until substantially all the rosin has been distilled over, whereby a grease-set high in abietic-acid content is produced.

3. The process which consists in distilling over, with superheated steam at a temperature of about 275° C., rosin, from which the turpentine has previously been separated, raising the temperature of the steam to complete the distillation of substantially all the rosin, and condensing the distillate, whereby a grease-set high in abietic-acid content is produced.

4. As a grease-set for lubricant greases, the distillate of rosin having an abietic-acid content of over 65 per cent.

FRANK E. MARINER.

In presence of—
A. C. FISCHER,
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