

No. 711,596.

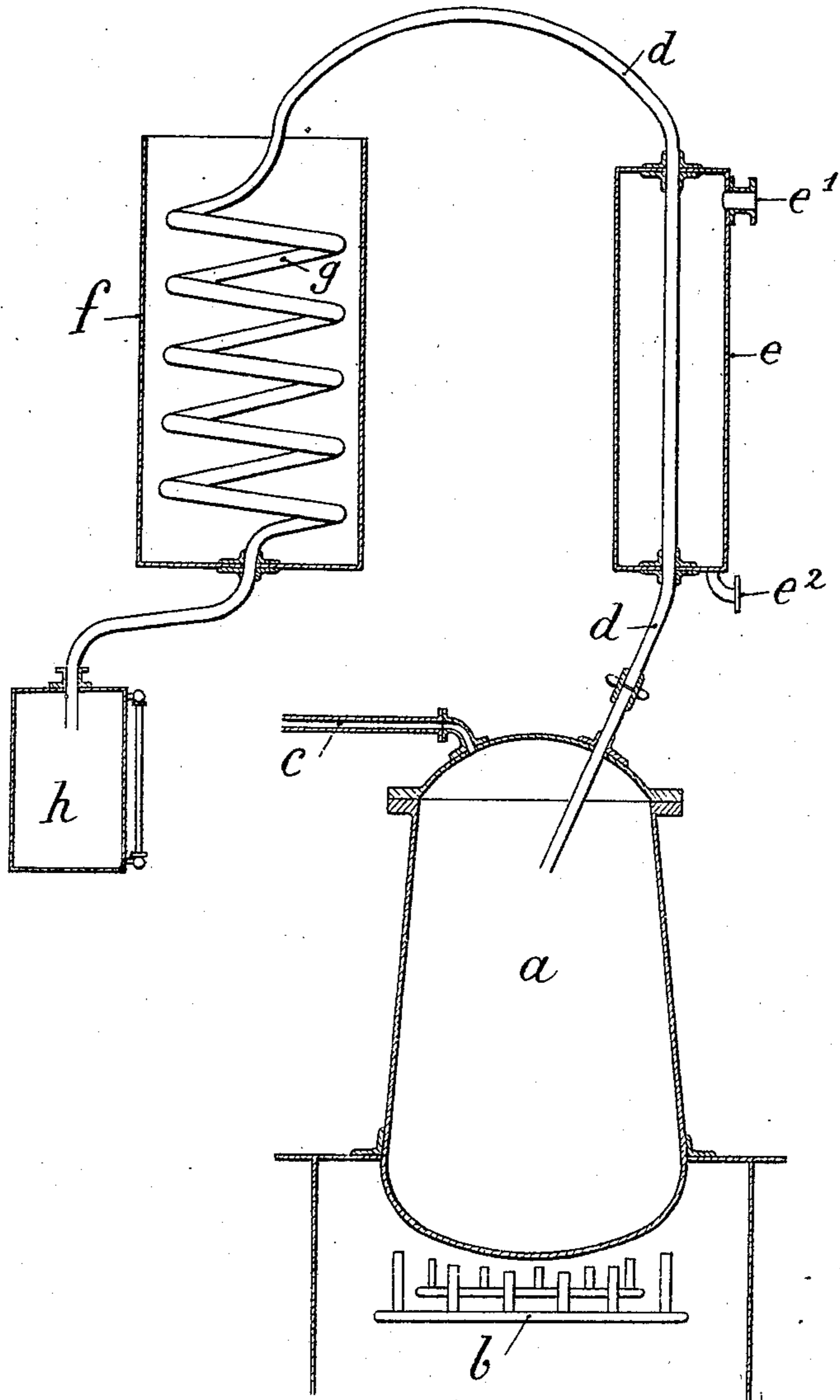
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A. L. TÊDESCO.

PROCESS OF MANUFACTURING VARNISH.

(Application filed Feb. 27, 1902.)

(No Model.)



Witnesses

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PROCESS OF MANUFACTURING VARNISH.

SPECIFICATION forming part of Letters Patent No. 711,596, dated October 21, 1902.

Application filed February 27, 1902. Serial No. 96,006. (No specimens.)

To all whom it may concern:

Be it known that I, ALOYS LOUIS TÉDESCO, a citizen of France, residing at 9 Rue Pigalle, Paris, France, have invented an Improved
5 Process for the Manufacture of Varnishes, of which the following is a specification.

The manufacture of oil-varnishes containing gums (resins) comprises, in the first place, the heating of the gums, which are usually
10 included in the generic term of "copal." This operation is indispensable, as natural copal is almost insoluble in oils and in spirits of turpentine. In order to render them soluble, the gum-copal has to be heated to more or less
15 high temperatures for a longer or shorter time, according to its nature, (soft copal, semihard, and hard.) The longer the copal is subjected to the action of heat the greater becomes its capacity for mixing with oils.

20 Hitherto copal has been generally heated in open apparatuses (matrass) or in ordinary still-boilers, (the Violette process.) During this heating operation the volatile products (essential oils of copal) are continually
25 given off, which constitutes a loss to the manufacturer varying from twenty-five per cent. to forty per cent. and sometimes more of the weight of the copal on starting the process.

The copals being always more or less mixed
30 with foreign matter—such as leaves, fragments of wood, and the like, which the most careful sorting cannot entirely eliminate and the charring of which causes the production of particles of carbon, which brown or blacken
35 the final product—that is, the varnish. The processes which have been just mentioned, therefore, place the manufacturer in the difficulty either of having to moderate the heating of the copals, and thus keep them below
40 the temperature desirable for mixing with the oil, or else of having to augment or prolong the heating to avoid the first defect, and thereby infallibly obtain too dark a color.

The present invention relates to a new process for heating copal.
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The copals, which are chemically-condensed terebenthenes, (according to Riban,) undergo under the action of heat a depolymerization or a deconcentration, which brings them back
50 to the state of more simple terebenthenes, among which are easily found ordinary terebenthene and diterebenthene. We have as-

certained that if the heating of these copals takes place in the presence of the volatilized terebenthenes, the said terebenthenes result- 55
ing only from the terebenthenes making integral part of the constitution of the gum and the said terebenthenes falling back after condensation into the mass, the depolymerization takes place much more easily and, moreover, 60
at a lower temperature, whence results much less colored products. The most simple process for attaining this result consists in heating the copal in a boiler surmounted by a top and by a reflux-condenser. In these circum- 65
stances as soon as the portion of copal most in contact with the source of heat begins to give off volatile terebenthenes these retained by the reflux-condenser fall back in a continuous stream on the substance not yet trans- 70
formed, moistening it and by their presence and cooling action rapidly effecting the transformation of the rest of the copal in half the time. There is thus obtained a melted substance having the valuable property of mix- 75
ing in any proportions with the raw, boiled, or thickened oils which are combined with spirits of turpentine in the preparation of oil-varnishes. At the same time as the volatile hydrocarbons aforesaid—namely, tere- 80
benthene and diterebenthene—the depolymerization of the copal produced a certain quantity of acid liquids and some traces of alcohol, (principally methyllic.) Now it is necessary to prevent these products falling 85
on the untransformed copal, as they retard and frequently entirely stop the depolymerizing action which the latter is undergoing. The presence of these acid liquids is certainly the drawback which has caused the heating 90
of copal in a Papin's digester to be abandoned. In order to get rid of these liquids, I place in the condensing-cover substances capable of absorbing them—such as quicklime, chlorid of calcium, &c. Even more econom- 95
ical and practical would be to maintain at 100° centigrade by means of a current of steam in a steam-jacket the temperature of the reflux-condenser, so that the terebenthenes (the most volatile of which boil at 156° 100
centigrade to 160° centigrade) are retained in the retort, while the steam escapes. A second but descending condenser is placed after the reflux-condenser lower than the lat-

ter, and there is collected at its outlet all the acid liquid, as well as a portion of the terebenthene carried over by the steam. Very homogeneous meltings are thus obtained, including all the terebenthene constituents of the copal and excluding the acid-liquid traces of alcohols and moisture.

The heated masses resulting from this process differ from those obtained in retorts or in the well-known distilling apparatuses, not only by their lighter color, but also by their special property (doubtless due to the elimination of the acid liquid) of mixing with oils in any proportions. The oil-varnishes thus obtained are distinguished, moreover, by remarkable elasticity and gloss.

If by means of a pump or other suitable means a more or less complete vacuum be produced in the apparatus, the transformation of natural copal is much easier and takes place at a lower temperature, according to the degree of the vacuum. The products obtained are consequently much more discolored, while preserving their full power of absorbing oil. It is necessary in making use of the vacuum to regulate the temperature of the reflux-condenser, according to the elastic force of the aqueous vapor, to the pressure existing in the apparatus, so as to eliminate the injurious liquid, while retaining almost the whole of the terebenthenes, which are volatile at a higher temperature.

By means of this process the loss is very inconsiderable, generally from five to six per cent. of the weight of copal treated. It never exceeds seven per cent. Moreover, this loss is only apparent, as will be proved.

The adoption of the second condenser allows of collecting, mixed with water, all the terebenthene which has been carried over by the water. This aqueous mixture is allowed to stand for twenty-four hours, and the floating terebenthene, which represents about half of the total volume, is removed. It is deodorized by means of one to two per cent. of quicklime and can be used afterward. It therefore follows that this process, while providing varnishes which are very much less colored, more resistant, and having remarkable properties of elasticity and gloss, never involves a loss of more than two and one-half per cent. to three per cent. of the copal treated.

As a rule the products obtained by this process are much more drying than the varnishes produced by heating in the open air or in the ordinary distilling apparatuses.

The accompanying drawing illustrates, by way of example and diagrammatically, an apparatus suitable for the operation above described.

In this drawing, *a* indicates the boiler,

which is heated by a gas-jet *b* or any other convenient heating apparatus. A communication-pipe *c* connects to a vacuum-pump when such is made use of. A tube *d* is provided for the removal of the volatile products. It is surrounded by a reflux-condenser *e*, which is filled with steam passing by the tube *e'* and discharged by the tube *e''*. This steam at 100° centigrade condenses the terebenthenes, which fall into the boiler, and allows the acid-vapors to pass. The tube *d* is connected to a coil *g*, placed in a cooling vessel *f* with water circulating therein. The acid-vapors and the terebenthenes are condensed and collected in the receiver *h*.

What I claim is—

1. The process for the depolymerization of copals which consists in heating the copals, in condensing the volatilized terebenthenes, and in returning the same to the mass under treatment for the purpose of facilitating its depolymerization without addition of terebenthenes derived from other sources.

2. The process for the depolymerization of copals which consists in heating the copals, in condensing the volatilized terebenthenes with separation and elimination therefrom of the acid aqueous liquid simultaneously volatilized and in returning the separated and condensed terebenthenes to the heated mass.

3. The process for the depolymerization of copals which consists in heating the copals (at the atmospheric pressure or in a more or less complete vacuum) undercondensing the volatilized terebenthenes in a reflux-condenser maintained at 100° centigrade and forcing these condensed terebenthenes to fall back continuously in the smelting mass and in simultaneously separating the condensed terebenthenes from the acid liquids and alcoholic vapors.

4. In the process for the depolymerization of copals, by the act of presence and favored by the cooling action of condensed and purified terebenthenes derived from the mass by the aid of heat, collecting the acid and alcoholic liquids in a second condenser, allowing the mixed liquid to stand until the terebenthene carried over collects at the top, removing the terebenthene by decantation, deodorizing it by means of one per cent. to two per cent. of quicklime in order to be utilized.

In testimony whereof I have signed my name to this specification in the presence of two subscribing witnesses.

ALOYS LOUIS TÉDESCO.

Witnesses:

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