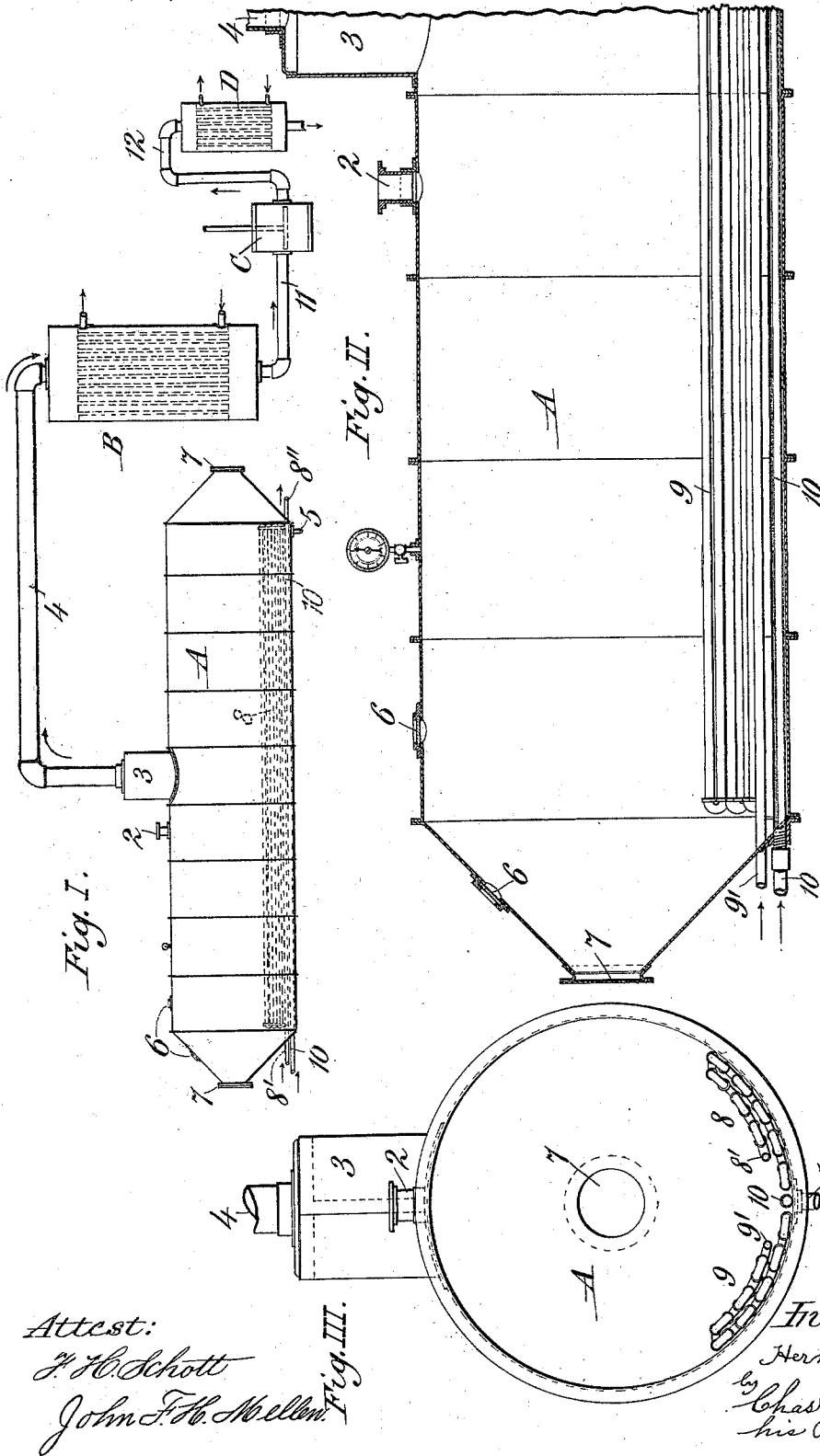


(No Model.)

H. FRASCH.
PURIFYING SOLVENT EXTRACTED OILS.

No. 550,716.

Patented Dec. 3, 1895.



Attest:

J. H. Schott

John F. H. Mellen.

Fig. III.

Inventor
Herman Frasch
by Chas. J. Hedrick
his Attorney

UNITED STATES PATENT OFFICE.

HERMAN FRASCH, OF CLEVELAND, OHIO, ASSIGNOR TO THE CLEVELAND
LINSEED OIL COMPANY, OF SAME PLACE.

PURIFYING SOLVENT-EXTRACTED OILS.

SPECIFICATION forming part of Letters Patent No. 550,716, dated December 3, 1895.

Application filed November 27, 1894. Serial No. 530,146. (No specimens.)

To all whom it may concern:

Be it known that I, HERMAN FRASCH, a citizen of the United States, residing at Cleveland, in the county of Cuyahoga and State of Ohio, have invented certain new and useful Improvements in Purifying Solvent-Extracted Oils; and I do declare the following to be a full, clear, and exact description of the same.

This invention relates to the purification of oils which have been extracted by a solvent, such as naphtha, from ground linseed or similar substances and which contain matters coagulable by heat or heat and moisture that have been extracted with the fatty constituents of the seed. In the purification of such oils it has been found essential to pass steam through the solvent-containing oil in order effectively to get rid of the solvent, since dry heat alone does not accomplish that object. As heretofore practiced, the steaming has brought about not only the removal of the naphtha, but also the coagulation of some of the albuminous or mucilaginous constituents of the oil and the formation of an intimate admixture or emulsion of such coagulated constituents—oil and condensed moisture. It has consequently been necessary to submit the steamed and emulsified oil to a further treatment to clarify it and rid it of the suspended matters. It will be perceived, therefore, that this old process involves a further treatment after the removal of the solvent, and also changes the composition of the oil by the removal of the mucilaginous or albuminous constituents. The further treatment is, of course, an item of expense; but the change of composition is, as I have discovered, a much more serious objection, since to it must be attributed (as I believe) the well-known unfitness of solvent extracted oil as heretofore made for certain industrial uses—such as painting, for example.

I have discovered that by having the oil at a suitably low temperature and introducing steam of a suitably reduced tension and temperature the removal of the naphtha may be effected without coagulating the mucilaginous or albuminous constituents which give body to the oil and which are extracted with the fatty matters from the ground linseed. The temperature of the oil and of the steam must

be sufficiently below 212° Fahrenheit to be non-coagulative of the mucilaginous or albuminous constituents, and is preferably at about or below 140° Fahrenheit, for there is no practical difficulty in securing such reduced temperature and obtaining excellent results thereby. Temperatures above 140° Fahrenheit may be used, (and still be within the invention;) but there is believed to be no substantial advantage in employing such higher temperatures, and it is evident, also, that the higher the temperatures the greater the risk of causing injurious coagulation.

In accordance with the invention, therefore, I keep the solvent-containing mucilaginous or albuminous oil at a non-coagulative temperature, (preferably at about or below 140° Fahrenheit,) and I pass through such oil low-tension steam of a non-coagulative temperature, (preferably at about 140° Fahrenheit during the principal steaming operation,) thereby removing the naphtha or solvent, while leaving a substantial body of coagulable matter in the oil. Low-tension steam of proper temperature may be secured by suitably reducing the vapor-pressure in the separator—that is, the vessel which holds the oil to be purified—and delivering steam from an ordinary boiler below the surface of the oil through a pipe of a bore to deliver sufficient steam rapidly to drive off the naphtha without unduly raising the vapor-pressure. In passing through the pipe the steam is relieved more and more of the pressure which confined it in the boiler, so that at its entrance into the oil its tension corresponds with the back-pressure in the separator, (due both to the resistance of the overlying body of oil and to the vapor-pressure above it.) Its temperature will then, also, be low, in correspondence with the reduced tension.

Practically I find it best to carry on the solvent removing operation in two stages, in the first of which the temperature of the oil is kept lower and a less volume of steam introduced than in the second or later stage, when the solvent or naphtha has been largely removed. This mode constitutes a special improvement, although I do not confine all my claims thereto, but only the third of those hereinafter written.

The accompanying drawings illustrate what is considered the best form of apparatus for use in carrying the invention into effect.

Figure I is a general view, in elevation, of such apparatus. Fig. II is a view in longitudinal section of the left-hand portion of the separator or vessel to hold the oil, and Fig. III is an end view of such separator with the head removed.

The separator A is provided with a fitting 2 to receive the filling-pipe, (not shown,) by which the oil is introduced into the separator. It also has a dome 3, with which the vapor-outlet 4 is connected, and a draw-off 5, provided with a suitable cock or valve, (not shown,) for removing the purified oil.

At 6 are bull's eyes or glass-covered holes in the walls of the separator, through which the interior can be inspected. At the ends are manholes 7.

The coils 8 and 9 are dry-heating coils—that is to say, they are imperforate coils—through which steam is passed for raising the temperature of the oil by dry heat. The inlets 8' and 9' are shown at one end and the outlets (8'' only is shown) at the opposite end. The pipe 10 is perforated along the bottom and serves to admit steam into the oil through such perforations.

The vapor-outlet pipe 4 leads to a condenser B, which in turn is connected by the pipe 11 with an aspirating-pump C, (or exhaustor of any suitable description,) the discharge of said pump being connected by the pipe 12 with a second condenser D.

The oil extracted, say, at or slightly above atmospheric temperature from linseed by naphtha by means of one of the well-known percolators and containing solvent and coagulable matter is introduced into the separator A, so as to fill it, say, two-thirds full. The oil may come from the percolator containing all the solvent or with some of the solvent already evaporated off, or, generally, it may have been subjected to any treatment not inconsistent with the purposes of the present invention. Preferably it is introduced into the separator just as it comes from storage-tanks, in which it is pumped from the percolator.

When the separator has been charged, the filling pipe or opening is closed and the pump C started. Steam is introduced into the dry coils 8 and 9, and steam is also injected into the oil through the pipe 10. Proper conditions at this stage are a temperature of about 125° Fahrenheit, a pressure of about seventeen inches by the vacuum-gage, (or, in other words, about seventeen inches below the ordinary barometrical pressure,) and the injection of enough steam to prevent foaming. Practically suitable conditions can be secured by keeping the dry coils 8 and 9 (having suitably proportioned heating-surface) full of steam from a boiler at, say, sixty pounds pressure per square inch (the boiler connection being left open and the

water from condensation of the steam in the coils being drawn off through a cock sufficiently closed to let off the water, while preventing any considerable escape of steam) and maintaining a suitably rapid removal of vapor by means of the condenser B and pump C. Assuming a conical-ended cylindrical separator A eight feet in diameter and thirty-nine feet extreme length provided with coils 8 and 9, each composed of five hundred and twenty-seven feet of two-inch pipe, the condenser B may contain fifteen hundred copper tubes each three-fourths of an inch in outside diameter and ten feet in length and be supplied with enough condensing water at ordinary temperature (say 67° Fahrenheit) to be not above 110° Fahrenheit at its outlet, and the pump C may be of the reciprocating type, of thirty-six inches diameter of cylinder and thirty-six inches stroke, and make about forty-five revolutions (or complete reciprocations) a minute. By observation through a bull's-eye 6 the attendant can regulate the supply of injected steam through the pipe 10, so as to correct any tendency of the oil in the separator to foam. The pipe 10 may be three inches in diameter. The perforations in this pipe are preferably small and close together along the bottom line of the pipe—say of one-eighth inch diameter of aperture and an inch and a third apart. The naphtha, vapor, and steam from the separator together pass through pipe 4 to the condenser B, wherein the steam and most of the naphtha are condensed. The condensed liquids and uncondensed vapor pass through the pump C and condenser D, wherein a higher pressure is retained and wherein the remaining solvent vapor is condensed. The liquids need not enter the pump C and condenser D, as they could be trapped off or separated by well-known means. From the condenser D the water and liquefied solvent would flow into a suitable receiver. As the evaporation proceeds, it is not necessary to alter the power of the condenser B, nor the speed of the pump C, nor the volume of steam injected through the pipe 10, although as to the latter it is to be observed that the tendency to foam diminishes, and the volume of injected steam could therefore be reduced if thought desirable. Under these conditions the vacuum-gage gradually rises, (in other words, the pressure in the separator becomes less.) The temperature also gradually rises. When the solvent has nearly disappeared, which will be in about three-quarters of an hour and which will be shown by the height of the vacuum-gage, (say when such gage reaches about twenty-five and one-half inches, the thermometer standing about 140° Fahrenheit,) the steam is shut off from the dry coils and is injected freely through the pipe 10, so as to maintain the oil in thorough ebullition until the water of condensation from the condenser B shows no more naphtha, which should be the case in about five minutes. On passing through the pipe 10 the tension of the steam and its temperature

also would be reduced, so that on entering the oil its tension would be somewhat above four and one-half inches of mercury, for an indication of twenty-five and one-half inches by the vacuum-gage represents a vapor-pressure of four and one-half inches of mercury when the outside barometer stands at thirty inches, and the entering steam has to overcome the resistance of the oil in addition to such vapor-pressure, and its temperature would be correspondingly above $131\frac{1}{2}^{\circ}$ Fahrenheit, which is the temperature of saturated steam under the pressure of four and one-half inches of mercury. The temperature of the entering steam would therefore be about 140° Fahrenheit. The degree of vacuum in the separator A thus allows such an expansion of the injected steam as that its temperature is lowered below the coagulating point of the mucilaginous or albuminous constituents of the oil. Consequently these constituents are not coagulated and separated from the fatty matter as they have been in all previous processes known to me in which solvent-extracted linseed-oil or an oil extracted by solvent and containing such albuminous or mucilaginous matter as occurs in linseed-oil has been steamed to remove the solvent. When a sample withdrawn shows no trace of naphtha, the steam is shut off, the pump is stopped, and the purified oil drawn off. The oil should then be clear. It contains a substantial body of albuminous and mucilaginous matter and is well adapted to painting and other uses for which an oil with "body" is desired. Should, however, the oil appear cloudy or contain water, it can be heated by the dry coils 8 and 9 alone (the pump C being operated) in order to clarify it, or other means (such as an air-blast through, say, the pipe 10) can be used for that purpose. It is not necessary, however, to do this if the process has been carefully carried out.

While the use of air is no part of the present invention, it may be observed that it can be injected into and allowed to pass through the oil before or simultaneously with or after the injection of steam; but since it has a chemical action its use may be injurious if carried too far.

I claim as my invention or discovery—

1. The process of purifying linseed or similar oil, containing solvent such as naphtha together with the coagulable mucilaginous or

albuminous matter extracted with the fatty matter, by evaporating the naphtha at a temperature suitably below 212° Fahrenheit to be non-coagulative of said mucilaginous or albuminous matter (preferably about or below 140° Fahrenheit) with the aid of low tension steam passed through said oil and having a temperature suitably below 212° Fahrenheit to be non-coagulative of its said mucilaginous or albuminous matter, (preferably about 140° Fahrenheit;) substantially as described.

2. The process of purifying linseed or similar oil, containing solvent such as naphtha together with the coagulable mucilaginous or albuminous matter extracted with the fatty matter, by evaporating the naphtha at a temperature suitably below 212° Fahrenheit to be non-coagulative of said mucilaginous or albuminous matter (preferably about or below 140° Fahrenheit) with the aid of dry heat and of low tension steam passed through said oil and having a temperature suitably below 212° Fahrenheit to be non-coagulative of its said mucilaginous or albuminous matter, (preferably about 140° Fahrenheit;) substantially as described.

3. The process of purifying linseed or similar oil, containing solvent such as naphtha together with the coagulable mucilaginous or albuminous matter extracted with the fatty matter, by evaporating the naphtha with the aid of dry heat and low tension steam in two stages, in both of which the oil is kept at a temperature suitably below 212° Fahrenheit to be non-coagulative of said mucilaginous or albuminous matter (preferably about or below 140° Fahrenheit) and in both of which low tension non-coagulative steam is passed through said oil, the temperature of the oil being lower and less volume of steam being passed through the oil in the first than in the second or later stage, wherein the temperatures of the oil and of the injected steam are preferably about 140° Fahrenheit; substantially as described.

In testimony whereof I affix my signature in presence of two witnesses.

HERMAN FRASCH.

Witnesses:

F. W. LOTHMAN,
N. J. WORLEY.