

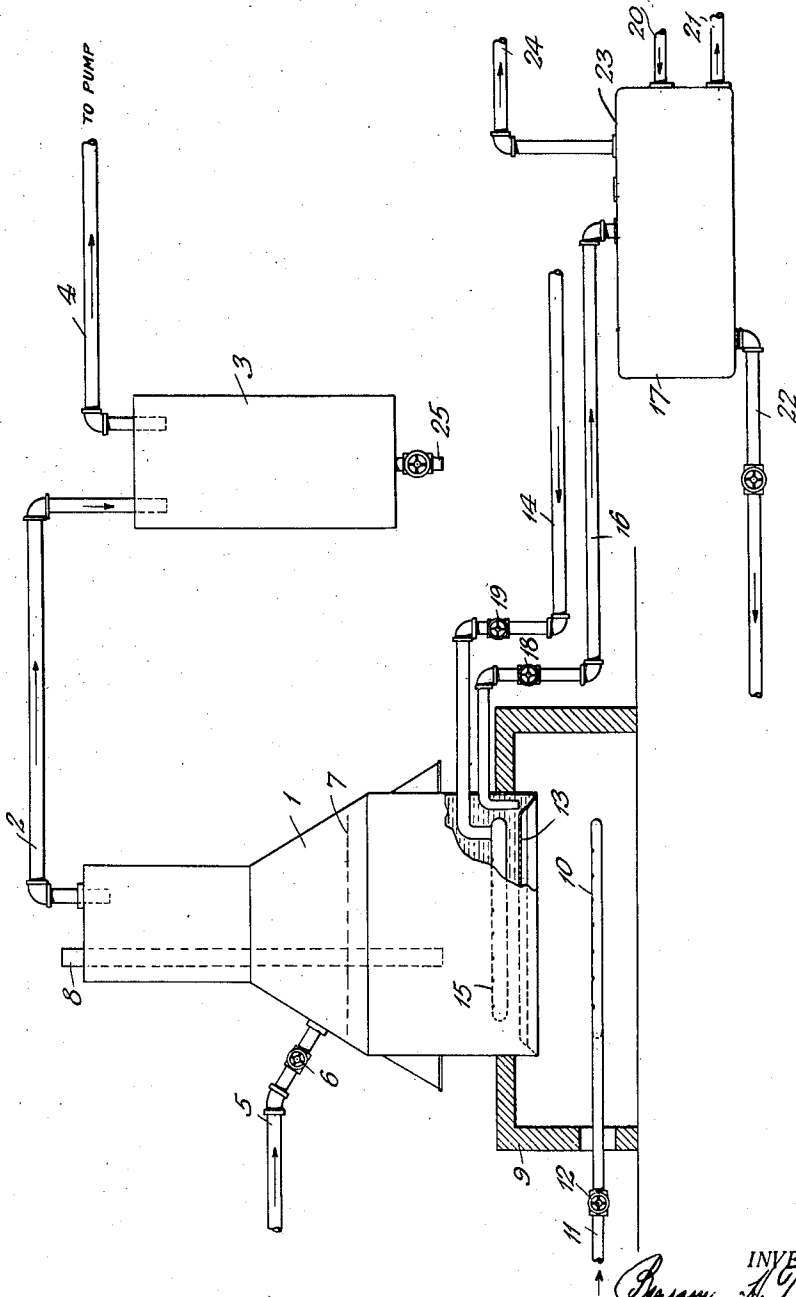
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METHOD OF TREATING LINSEED OIL

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METHOD OF TREATING LINSEED OIL

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My invention relates to a method of treating linseed oil. The method is applicable to any vegetable, animal or marine oil capable of polymerization when heated. The following description will be limited to linseed oil as an example, it being understood that the same method applies to other oils with corresponding results.

Linseed oil is used in many of the arts but is particularly useful in the manufacture of high grade paints and varnishes.

Some of the requirements for the ideal linseed oil in the paint and varnish art are good color, low acid number, high viscosity and quick bodying power. An oil possessing all of the above qualities has been impossible of attainment by any prior method known to me. Some of the difficulties encountered in the production of an oil possessing all of the above qualities are that in the ordinary method of bodying the fatty acids are formed very rapidly and it has heretofore been necessary to stop the bodying in order to keep the acid number low enough for use in the making of paints and varnishes. It has also been necessary for the varnish maker to limit the bodying in order to prevent too great discoloration of the oil. It has also been necessary for the varnish maker to accept a lower viscosity than desired because of discoloration if the viscosity is carried beyond a certain point.

I have discovered a method by which an oil having all of the above mentioned characteristics may be produced and at a very much lower cost than by any method known to me.

My invention will be better understood by reading the following description in connection with the accompanying drawing which illustrates one type of apparatus in which my process may be carried out.

It is understood of course that my invention is not in any manner limited to the exact apparatus shown or to apparatus of the type of that shown in the accompanying draw-

ings, the process being entirely independent of the apparatus.

The apparatus shown comprises a closed vessel 1 into the top of which enters a pipe 2 leading to a drum 3 to which is connected a pipe 4 leading to a vacuum pump (not shown) of any well-known character.

A supply pipe 5 enters the vessel 1 and is provided with a valve 6. Normally the liquid level in the vessel 1 is maintained approximately at the dotted line 7. A thermometer well 8 is placed in the vessel 1 so that the temperature may be observed. The vessel 1 is supported upon a structure 9 and a gas burner 10 or other suitable heating means is placed underneath the vessel 1 and is supplied with fuel through the pipe 11 which is controlled by the valve 12. The vessel 1 has a raised or dished bottom 13 for a purpose which will appear later.

A steam supply pipe 14 enters the vessel 1 and connects with a perforated pipe or series of pipes 15 so that steam may be passed up from 15 through the body of the oil and taken off through the pipe 2 as will be more fully explained later.

A drain pipe 16 carries the treated oil from the vessel 1 to a cooler 17. Because of the dished bottom of the vessel 1 a trough is created into which the end of the pipe 16 is placed so that practically all of the oil may be removed from the vessel 1. A valve 18 is placed in the pipe 16 and a valve 19 in the steam pipe 14.

The cooler 17 may be of any desired design but as shown consists of a container into which pipe 20 leads a cooling fluid which is discharged through a pipe 21. The oil from the vessel 1 flows into the cooler and is cooled and discharged therefrom through the pipe 22 to a suitable storage container. A thermometer well 23 is placed in the cooler 17 so that the temperature may be observed. The temperature maintained in the cooler 17 is preferably about 250° F. or lower. A pipe 24

opens into the upper part of the cooler and may be connected with a pump for the purpose of creating a vacuum and removing any volatile products given off by the oil. A drain pipe 25 is provided at the lower end of the drum 3 for the purpose of discharging condensates from the system.

My preferred method is as follows: Ordinary varnish oil, or any vegetable, animal or marine oil capable of polymerization when heated, is put into the vessel 1 up to the preferred liquid level 7. The vessel is then closed by closing the valve 6 and a vacuum of 29 inches or more placed upon the oil, by using any high grade vacuum pump, such as that made by the E. H. Wheeler Mfg. Co. Heat is applied to the vessel 1 by the heater 10 until the oil is heated up to a temperature of about 600° F. This temperature and vacuum are maintained for a period of about six hours. If desired, traces of metal such, for instance, as cobalt, lead or manganese, may be used as a catalyst to shorten the period of polymerization. In the following examples the linseed oil is the varnish makers' ordinary grade of linseed oil, which is a well-known article on the market. I have successfully used in my process:

1. Linseed oil containing .01 per cent cobalt by weight.
2. Linseed oil containing .20 per cent lead by weight.
3. Linseed oil containing .20 per cent manganese.
4. Linseed oil containing .20 per cent of a 50-50 mixture of lead and manganese.

The catalyst used is commonly known in the trade as drier metals and causes polymerization in substantially direct ratio to the amount used up to the limit above specified. The rate of polymerization depends somewhat upon the type of oil used. For instance, if dryer metals are added to linseed oil, as above stated, polymerization will take place much faster than if the same drier metals, and in the same proportions, are added to soya bean oil. The rate of polymerization in soya bean oil is greater under the given conditions than it would be in cotton seed oil. By the use of the catalyst the period of polymerization may be as low as 4 hours instead of 6. During this period the products split off during polymerization such as water, fatty acids, acrolein and products non-condensable at the receiver temperature specified, such as CO₂, are quickly removed by the vacuum pump and are caught in part in the receiver 3 and removed through the discharge pipe 25 and may be sold as linseed oil substitute or used in the making of driers. The temperature in the receiver 3 is not allowed to rise above the point necessary to preserve a vacuum of 29 inches in the container 1 and is preferably between 90° and 110° F. The temperature in the drum 3 is controlled by

any suitable means, such as a water bath, not shown.

During the period above mentioned steam is admitted to the body of the oil through the pipe or pipes 15 and passes up through the oil and acts to help vaporize the fatty acids and facilitate the removal of same as in distillation. By properly co-ordinating the steam with the temperature and vacuum on the oil the acids originally present in the oil and those formed during the polymerization may be removed almost completely so that an oil is produced which is substantially neutral. Of course the acid number may be any desired amount by properly regulating the steam, temperature and vacuum.

The minimum acid number of the oil is attained by having the vacuum at its maximum, and ample steam feed. It is not necessary, however, to use steam for producing new and improved oil. This can be done by heat and vacuum alone. As an example of the treatment without the use of steam, I give the following: 1800 parts of varnish makers' linseed oil was heated for five hours at 600° F. under an absolute pressure of 5/8 inches of mercury. At the end of this time the viscosity of the oil was 39 minutes as measured in the ordinary varnish makers' tube. The acid number of the finished oil was 3.4 and its color 30 yellow, 2.5 red, as measured on the Lovibond scale, using a 5 1/4 inch column of oil. A sufficiently low acid number or free fatty acid content can be obtained by maintaining the vacuum between 28" and 29" and a fraction and the proper temperature of 545° F. to 600° F. If the vacuum in this case without steam is allowed to drop more fatty acids are formed. If, of course, steam is used, the vacuum could be lowered and additional steam used to distill off the acids.

As an example of the proper co-ordination of these elements it may be stated that with a vessel 1 containing 500 gallons of linseed oil maintained under a vacuum of 29 inches or better and at a temperature of approximately 600° F. for a period of 6 hours, there should be about 500 pounds of steam passed through the oil to give the best results. This process produces an oil having a color of 35 yellow and 4 red, as measured on the Lovibond scale, using a 5 1/4 inch column of oil, an acid number of one or less, a viscosity of 40 to 50 minutes and much more rapid bodying properties than oils as heretofore produced. During the carrying on of my method samples are withdrawn at suitable intervals and tested for viscosity. When the desired viscosity for any required oil is reached the process is discontinued. The viscosity of the finished product is determined by trade conditions and varies anywhere between the viscosity of the original starting oil, which is ordinary varnish makers' oil having a viscosity not accurately measurable on the var-

nish makers' viscosity tube, to a viscosity of 40 to 50 minutes.

The viscosimeter used consists of a glass vial $\frac{3}{8}$ " in diameter and 6" long which is filled with the oil to be tested and corked up so as to allow a small air bubble which when resting at the side of the tube measures one-third the diameter of the tube. The viscosity is determined by inclining the tube 60° from the horizontal and measuring with the stop watch the length of time required for the passage of the air bubble from one end of the tube to the other or a distance of 6 inches. The viscosity is determined at a temperature of 72° F.

It may be necessary to slightly modify the vacuum, temperature and amount of steam with some grades of oil, but those skilled in the art, after reading the foregoing example and understanding my invention as expressed herein, will have no difficulty in making the modifications necessary to adapt the process to different oils.

While I do not know exactly what takes place in the above treatment, my opinion is that the high vacuum removes the products that might be split off during polymerization and which, if not removed, would remain in the oil and act to prevent rapid polymerization. The vacuum also eliminates or prevents oxidation in the oil and thereby prevents the glycerides from splitting up and forming free fatty acids, thus assisting in keeping the acid number low. The vacuum also lowers the boiling point of some of the products and thus makes it possible to remove same without breaking them down, thus aiding in this manner to keep the acid number low and also aiding in the prevention of discoloration usually caused by air.

By the use of the steam the products are removed which ordinarily produce a high acid number. Thus the combination of the steam with the vacuum makes it possible to keep a low acid number in the oil. The acid number of drying and semi-drying oils which have been bodied by the usual method for use in paints, driers, enamels, varnishes, lithographic inks and the like, range from 2 to 14 according to the degree of bodying and other factors, such as the skill of the operator will determine. Other gases than steam may be used, such as hydrogen, nitrogen, carbon-dioxid or any gas non-oxidizing toward the oil under specified conditions to the oil.

The advantage of a low acid number is apparent to all those skilled in the art. An oil having a high acid number when used in the manufacture of paint or enamel, causes with certain pigments what is known in the art as thickening or livering which frequently renders it impossible to use such an oil in the manufacture of a high grade paint or enamel.

A high acid number is not objectionable in an oil used for lead grinding but only a small

amount of oil is used for that purpose. It is impossible to use satisfactorily an oil with a high acid number in zinc grinding because of the livering action produced by the acids in the oil. Since a properly bodied low acid number oil was impossible of production by the prior methods the enamel maker was forced to either sacrifice quick drying time or thick enamels because, if the oils were bodied sufficiently to get a quick drying time their acid number was so high that they produced equal objections. The oil produced by my method enables the enamel maker to secure quick drying thick enamel without any of the difficulties heretofore experienced.

It is necessary in the manufacture of high grade white paints and enamels that the color of the oil be kept light and brilliant. In the ordinary method the color increases in direct proportion to viscosity and acid number. Heretofore it has therefore been necessary for the manufacturer to compromise on viscosity, color and acid number. By my method a substantially colorless oil may be produced. With viscosity of from 33 to 50 minutes as compared with an ordinary oil having a viscosity of about 72 seconds and an acid number of less than 1, as compared with an acid number of 2 to 14.

According to the prior processes, the working time to produce the best oils, none of which approach my oil as to color, viscosity and acid number, it was necessary to work them for a period of from 10 to 70 hours. By my method a much superior product is produced in 6 hours or less.

By the use of the apparatus heretofore described the losses of volatile products are reduced by my method as compared with the open kettle method usually employed. For example, the losses in the open kettle method are from about 4% to 8%, whereas with my method these losses are 1% or less.

Tests have shown that the oil produced by my method has a greater index of refraction than the best oils as heretofore produced. This property is of advantage in the production of gloss or brilliance of enamels made with my oil.

As is well known in the art, a high viscosity enables the user to employ a greater amount of thinner than a low viscosity. Perhaps china wood oil is the varnish maker's ideal as far as this property is concerned. It has been definitely shown that the higher viscosity the oil has, and especially if it has been polymerized, the greater its covering power is and therefore the greater amount of thinner used.

For example, an ordinary kettle bodied linseed oil which is commonly of about 72 seconds viscosity and high acid number, as above explained, may be diluted with more than 30 parts thinner to each 100 parts oil to produce a suitable consistency, whereas a mixture of

equivalent consistency and covering power may be made by mixing 100 parts of my linseed oil, treated as above described, with 300 parts thinner.

5 Oils as heretofore made also have the property of yellowing in the dark after drying, but tests have shown that with my oil there is such a slight yellowing that it is not noticeable.

10 Changes may be made in details without departing from the spirit of my invention.

This application is a continuation in part of my application filed May 7, 1925, for linseed oil and method of making same, Ser.

15 No. 28,554.

I claim:

1. A process of polymerizing vegetable and animal drying and semi-drying oil capable of polymerization when heated, which consists in heating the oil to a temperature between 500° F. and 600° F. under an absolute pressure of about 2 inches mercury or less, and simultaneously passing steam through the body of the oil.

25 2. A process of polymerizing vegetable and animal drying and semi-drying oil capable of polymerization when heated, which consists in heating the oil to a temperature between 500° F. and 600° F. under an absolute pressure of about 2 inches mercury or less, and simultaneously passing a non-oxidizing gas through the oil to remove free fatty acids therefrom.

3. A process of polymerizing vegetable and animal drying and semi-drying oil capable of polymerization when heated, which consists in heating the oil to a temperature between 500° F. and 600° F. under an absolute pressure of about 2 inches mercury or less, and simultaneously passing steam through the oil to remove free fatty acids.

4. A process of polymerizing vegetable and animal drying and semi-drying oils capable of polymerization when heated, which consists in heating the oil to a temperature of approximately 600° F. under an absolute pressure of approximately 1 inch of mercury and simultaneously passing a non-oxidizing gas through the body of the oil.

5. A process of polymerizing linseed oil, which consists in heating oil to a temperature between 500° F. and 600° F. under an absolute pressure of about 2 inches mercury or less, and simultaneously passing a non-oxidizing gas therethrough.

6. A process of polymerizing linseed oil, which consists in heating oil to a temperature between 500° F. and 600° F. under an absolute pressure of about 2 inches mercury or less, and simultaneously passing a non-oxidizing gas through the oil to remove free fatty acids therefrom.

7. A process of polymerizing linseed oil, which consists in heating oil to a temperature between 500° F. and 600° F. under an

absolute pressure of about 2 inches mercury or less, and simultaneously passing steam through the oil.

8. The process of polymerizing linseed oil, which consists in heating the oil to a temperature approximately 600° F. while under an absolute pressure of approximately 1 inch mercury and simultaneously passing steam through the body of the oil to remove free fatty acids.

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