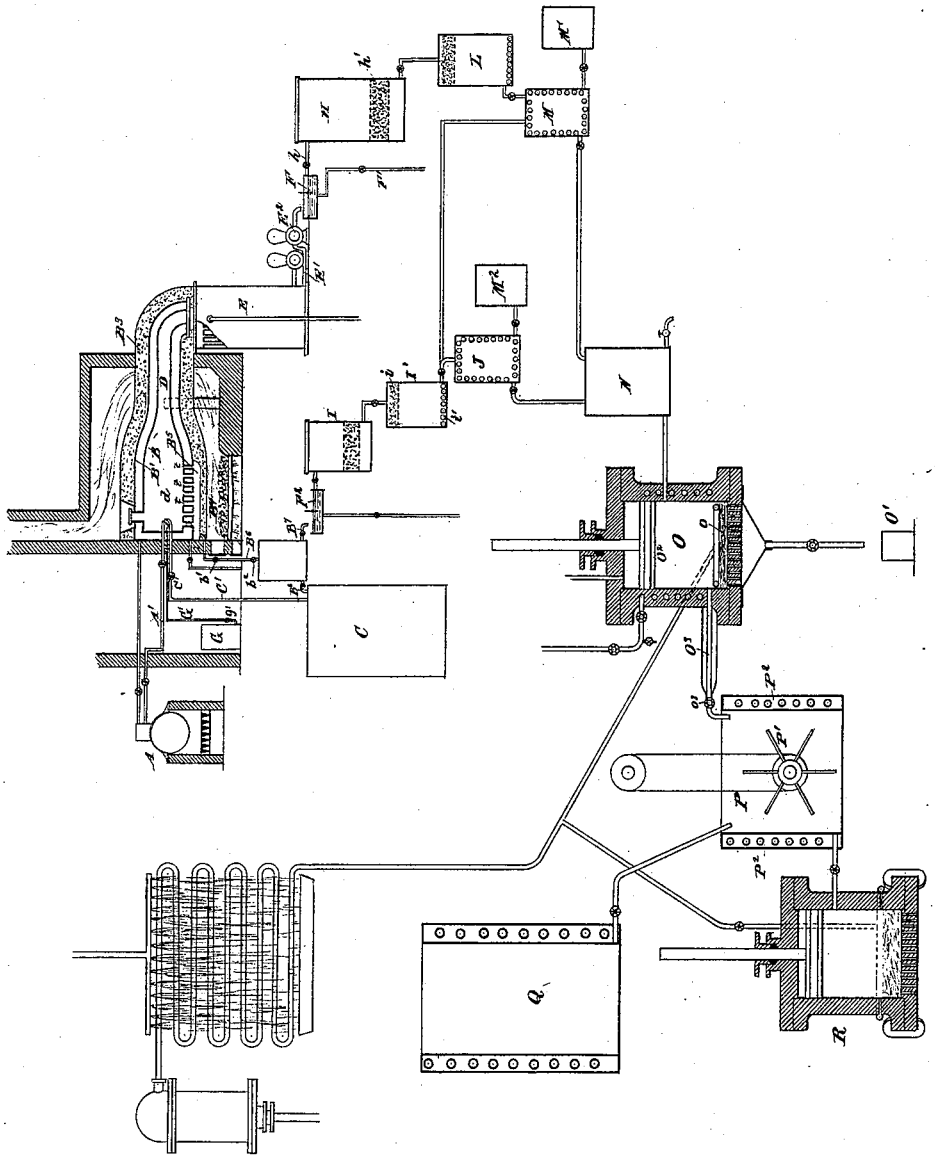
(No Model.)

J. B. GRANT & A. MASON.

METHOD OF TREATING COTTON SEED OIL AND ANALOGOUS OILS.

No. 342,692. Patented May 25, 1886.



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UNITED STATES PATENT OFFICE.

JAMES B. GRANT, OF NEW YORK, AND ALLAN MASON, OF BROOKLYN, N. Y.

METHOD OF TREATING COTTON-SEED OIL AND ANALOGOUS OILS.

SPECIFICATION forming part of Letters Patent No. 342,692, dated May 25, 1886.

Application filed August 1, 1885. Serial No. 173,213. (No model.)

To all whom it may concern:

Be it known that we, JAMES B. GRANT, of the city and county of New York, in the State of New York, and ALLAN MASON, of Brooklyn, Kings county, in the State of New York, have invented certain new and useful Improvements in the Method of Treating Cotton-Seed Oil and Analogous Oils, of which the following is a specification.

We have discovered that it is a great advantage under certain circumstances to refine cotton-seed and other analogous oils in the presence of heat so regulated as to be uniform and even, and yet not of such a high degree as to produce destructive distillation or to resolve the product being refined into any of its elements.

It has heretofore generally been considered necessary to use more or less of acids and alkalies in the refining of oil. They are usually applied by means of tanks and subsequent decantation. Our process is intended to obviate so much handling and to economize time, at the same time accomplishing the desired results more effectually and thoroughly. It is an improvement on our process for treating cotton-seed oil and analogous oils described in our application filed April 27, 1885, Serial No. 163,672, allowed June 1, 1885, and greatly expedites the same.

The oil obtained in large quantities from cotton-seeds and analogous sources contains more or less albuminous not readily vaporized matter. We have discovered that such oil may be purified and refined by being brought in a diffused condition into violent contact with acid or alkali in a vacuum-retort in the presence of the vapor of water, and by the mechanical force of a strong current of such vapor, in combination with efficient means for maintaining a vacuum and preventing any back action from the force of the steam, and instantaneously transferring it to a vacuum-condenser, and after it leaves the condenser passing it through an alkali or acid. It is then marketable refined oil. It is then further treated by saturation with carbonic-acid gas, which removes the tendency to rancidity. It is afterward frozen, pressed, mixed with lard-oil or other animal-oil, and again frozen, all this part of the process being as described in our application for patent filed April 27, 1885, Serial No. 163,672, allowed June 1, 1885, and which gives it additional superior qualities and adapts it to more varied uses. We receive the oil in a vacuum-retort with provisions for maintaining a uniform heat, and meet it upon its reception therein with a stream of sulphuric acid or alkali, and also a stream of live steam under sufficient pressure to thoroughly mix the acid or alkali and the vapor of water with the oil and instantaneously force the whole mass into the vacuum-condenser arranged to receive it, from whence it is pumped or allowed to flow through alkali or acid into a tank, in the top of which is placed another filter to insure perfect neutralization of acid or alkali. In this tank any moisture that may remain in the oil because of having been in the presence of the vapor of water or from any other cause is removed. It is then decanted into a tank, where it is saturated with carbonic-acid gas. We present the steam to the oil and acid in the form of a jet, having such force and being so presented to properly-shaped nozzles that it will draw up the oil and acid or alkali from reservoirs located a little lower. Such devices have been long known for atomizing perfumery. Such in a limited form have been applied at various stages in the treatment of oils for producing vapor, but always for the purpose of destructive distillation. We provide for making and maintaining a nearly perfect vacuum in the retort or evaporating-chamber. This greatly facilitates the vaporization and atomization without requiring so high a temperature as would produce destructive distillation. Receiving the oil into a vacuum-retort and striking it with such force with high pressure, saturated steam atomizes and diffuses the entire mass without separating it into any of its elements, while the acid or alkali being sprayed through the oil while in this diffused condition cuts and loosens the undesirable portions, which are caught and retained, and the acid itself neutralized by the alkali so that no trace of the acid remains. It is condensed simply to get the vapors in a liquid form; but no decomposition of the oil or separation into its elements occurs at any stage. We have made provision for accidents

and irregularities, which will be described further on. The steam is employed in its natural state, by which we mean not superheated. The entire mass of oil and acid and water in the form of vapor of low tension is instantaneously forced out of the retort into a surface-condenser, where it comes in contact with other surfaces kept at a low temperature by a liberal flow of water between the pipes or other elements of which the condenser is composed. We have no deposit of oil in the retort. There will sometimes be found in the retort the result of premature condensation of particles of the oil which are intended to be forced over, but which from said irregularity collect on the side of the retort and drip into a pocket prepared to receive them, or from the acid not being properly regulated and cutting and separating too rapidly, or from the steam not being admitted under sufficient pressure to carry over all the particles, which would also cause a greater or less quantity to drip into the pocket prepared to receive them. These would be some of the irregularities referred to; but in the case of cotton-seed oil and analogous oils, or animal-oils of any kind, the operation would differ from the treatment of hydrocarbon oils in this way: That the material coming from the pocket, never having been vaporized, would be in liquid form, but having been by means of a triple atomizer introduced into the retort in very diffused condition, and the particles having been impinged upon the acid or alkali with great force, the product contained in the pocket would have the undesirable portions loosened and in a more or less separated condition, and would simply have to be passed through the alkali and saturated with the carbonic-acid gas to be a marketable, useful, and superior product, and nearly the same product and in the same condition as the product which has been passed into the condenser and passed through the alkali and subsequently saturated with carbonic-acid gas, except that the product of the pocket, not having been in such a rarefied state or so long in the presence of acid or alkali, might require to be further treated with alkali or acid before final saturation with carbonic-acid gas. For this we provide. In the case of hydrocarbon oils, which are so much more volatile, the contents of the pocket are returned to the vessel from which the oil is received and subjected to the same process, together with the crude material in the tank; but this we do not always, and, in fact, very seldom, find necessary in the treatment of cotton-seed oil and analogous oils or animal oils of any kind. The presence of saturated steam, by which we mean not superheated, together with the vacuum maintained in the retort, does not allow the temperature in the retort to reach a very high degree. The heat in the retort is kept nearly uniform by suitable appliances. It may range from 212° to 300° Fahrenheit, according to the work required to be done.

The apparatus and appliances, as shown in the drawings, will form the subject-matter of a separate application for patent.

We do not in this process invention confine ourselves to any particular kind of apparatus, the object being to present the oil to the sulphuric acid in a diffused condition, thus causing a more thorough and prompt action of the acid or alkali and a great saving in quantity and subsequent or previous thorough saturation with carbonic-acid gas. Acid and alkali have been before used, but the striking of the oil in a highly diffused condition by the acid or alkali, or impinging the acid or alkali on the globules of oil, has not been practiced before to our knowledge or belief; nor has saturation with carbonic-acid gas to preserve the oil in a pure state, and to remove and prevent rancidity, ever been, to our knowledge, discovered or practiced before our invention. We prefer sulphuric acid as the acid referred to, and lime or caustic soda as the alkali, but do not confine ourselves to these. For purposes where the fullest use of our invention is desired this oil is afterward further treated by straining with pressure at a low temperature, induced, preferably, by the direct contact of expanded carbonic-acid gas. This separates it into an oil which is fluid at all temperatures, and a hard fat. The hard fat is then mixed by strong mechanical agitation with beef-fat or mutton, or other suitable animal-fats, and immediately frozen. This so unites the fats that they ever after remain practically combined.

Our treatment produces oil and hard fat possessing peculiarly valuable properties.

The accompanying drawing forms a part of this specification, and represents what we consider the best means of carrying out the invention. It is a central vertical section, partly in elevation, and shows the novel parts with so much of the ordinary parts as is necessary to show their relation thereto.

Referring to the drawing, A is a steam-boiler, from the dome of which steam is led at high pressure into a jacket or space between two shells, B B′, of an evaporating and atomizing chamber or retort, *d*, which is surrounded by a body of sand, B^3, serving to preserve and equally distribute the temperature. Heat produced by fire in a furnace or chamber, B^4, below aids to prepare the temperature in the retort and to maintain the temperature of the steam in the jacket, notwithstanding the great consumption thereof due to the rapid evaporation of the material going on in the interior of the retort. A steam-pipe, A′, conducts ordinary saturated steam at a high pressure from the boiler A to what we term a "triple injector," a device located within the evaporating chamber or retort *d*. The construction and arrangement of the triple injector is such that by means of a pipe, C′, leading upward from a tank, C, containing the oil to be refined, and a pipe, G′, also leading upward from the tank G, containing acid, (pure, or more or less diluted,) it will, by the action

of the strong blast of steam escaping from the pipe A′, draw up the oil and sulphuric acid together, and mingle them with the steam. At the same time the oil and acid are struck with such force by the steam escaping through the pipe A′ that they are thoroughly commingled, and the acid so impinged upon the globules of oil as to do its work effectually and promptly. The intimate mingling with the saturated steam also has a good effect. It washes and cleanses the oil. The force with which the oil and acid are struck by the steam, and the continual escape of the steam from the pipe A′ under such high pressure, together with the effect of maintaining a vacuum in the retort *d′*, blows and carries the oil, acid, and steam immediately into the neck D of the retort and into the condenser E, with the exception of any particles which may through any irregularities or from the action of the acid or alkali, or density of the oil, or the presence of foreign non-volatile matter, drip down the sides of the retort through the small orifices *t t* into the pocket B^5, where it is allowed to run at intervals through the pipe B^6, if necessary to subject it to the same process again; or, if this be not necessary, through the pipe B^7 into the trough F^2 into the filter I, containing alkali or acid, and, when necessary, through the additional filter *i* into the tank I′, where all moisture is removed by operation of the steam-coil *i* into the tank J, where it is thoroughly saturated with carbonic-acid gas, by means described in our description of tank M, further on, or the tank I′ can be connected by a pipe or trough with the tank M, and when the oil is in proper condition can be treated with carbonic-acid gas in the tank M. When this operation is performed, the cock *b′*, which has been opened to allow the material to run down the pipe B^6 as far as the cock b^2, is closed, and the cock b^2 opened, which allows the contents of the pocket B^5 to pass off with very slight disturbance of the vacuum, which is instantly overcome and removed when the cock *b′* is closed and the cock b^2 opened and the pump E^2 has been worked for a few moments. All the remainder of the mass, consisting of oil, sulphuric acid, and vapor of water, is, by the force of the steam, combined with the action of the pump E^2, and, aided by a nearly complete vacuum in the retort, carried instantly over into the neck D and condenser E.

The condenser E can be of any suitable style or form adapted to the ends in view. We at present use "Lighthall's," which has vertical tubes, the material being conducted through the interior of the tubes by the vacuum and the constant suction, a liberal flow of cold water being circulated in the intermediate spaces.

In the condenser E the vaporized portion of the mass is condensed, and the entire liquid mass, consisting of oil, acid, and water flows from the bottom of the several pipes, and is drawn through a pipe, E′, by an air-pump, E^2, operated by a steam-engine or other suitable power. The liquid being thus forced from the vacuum-retort and condenser is discharged into a trough or long tank, F, in which the water is allowed to separate. The water remaining on the bottom is drawn out through a pipe, F′, and need not be further referred to. The character of the oil is here observed, and the amount of filtration determined upon. It is then, together with what acid and impurities it may contain, allowed to flow through the pipe *h* into the tank H, which is provided with a filter, *h′*, consisting of alkali or acid, according as the material in the tank H is acid or alkali, through which it is allowed to filter, and decanted into the tank L, provided with a coil of pipe in the bottom, or other suitable apparatus for thoroughly removing any remaining moisture. It is then decanted into the tank M, provided with perforated pipes or other suitable apparatus for saturating with carbonic-acid gas. Here the carbonic-acid gas at a natural temperature is applied. Should the operator decide, when the oil is passing through the long tank or trough F, that additional care is necessary, the product of the tank H would be passed through an additional filter placed between the tank H and the tank L, or in the top of the tank L, as represented; otherwise the filter could be removed. When the saturation with carbonic-acid gas in the tank M or other suitable tank is completed, the product is then ready for market. No trace of impurity or acid is left, and it can be placed in suitable vessels or receptacles for transportation or use.

The carbonic-acid gas may be made practically pure in large quantities at a moderate price. It can be produced in such a vessel as M′ M^2 by treating marble-dust or limestone with sulphuric acid.

The oil treated as described and saturated with carbonic-acid gas is pure and free from rancidity. It can be stored or sold for use, and kept a long time, perhaps indefinitely, without becoming rancid.

In order to realize fully the benefits of this valuable product and the varied uses to which it may be applied, we operate in the following manner: The oil is allowed to flow from the tank M, where it had been saturated with carbonic-acid gas, into the storage-tank N, from which it is drawn, as required, into a chamber, O, where it is frozen by suitable means for producing a low temperature, but maintaining a dry atmosphere in the chamber O. The frozen product is then compressed by suitable means and the uncongealed portion expressed through the strainer *o*, placed in the bottom of the chamber to prevent clogging into the receptacle O′. The product in the receptacle O′ will not congeal, except at a temperature lower than that maintained in the chamber O during the freezing process.

O is the cylinder, and O^2 the piston of a hydraulic press. After the piston O^2 is raised by any suitable means (not shown) and the

chamber below is filled or nearly filled with oil and frozen, the pumps (not shown) are operated to depress the piston and induce a severe pressure on the oil. The thin portion exudes through the strainer *o*, as described. Next, admitting steam from a boiler (not shown) into a jacket surrounding the chamber O, and also surrounding a discharge-pipe, O^2, the hard fat is melted, and on opening the valve o^3 it is delivered, as required, into a mixing-vessel, P, where it is mixed with any desired proportion of animal oil or fat from a tank, Q, and agitated by a revolving wheel, P′, or other suitable means for agitating. This fat thus mixed with the cotton seed fat may be melted beef suet, mutton-tallow, lard, or other animal fat adapted to impart any desired qualities which the purified and practically odorless cotton-seed oil described does not possess. The mixing tank P is surrounded with a jacket, P^2, into which steam is admitted to keep the oils and fats being mixed therein in a liquid condition and to aid the thorough admixture of the products. After being thoroughly combined, the mixed product is discharged into a tank, R, where it is again frozen by means for producing a low temperature, but not allowing the product to come in contact with moisture. The fat mixed and instantly frozen under these conditions will remain permanently united and may be put up in proper packages for transportation and use. It is valuable for cooking and all culinary purposes.

In the treatment of hydrocarbon oils by a somewhat similar process, which is the subject of an application for patent by the parties hereto, filed June 30, 1885, Serial No. 170,252, more care is necessary with the retort *d*, and a finer regulation of the heat is required than for vegetable and animal oils. In the case of the hydrocarbons amalgamation with the vapor of water in the retort *d* is absolutely necessary. In the case of vegetable and animal oils such is not necessary. The process and machinery differ in other respects, although the retort may be made practically the same for convenience; but the impinging of the acid or alkali on the diffused petroleum and other hydrocarbons, and subsequently passing the condensed vapors through a filter of alkali or acid after being taken from the condenser E, is a valuable improvement upon the process as described in our application for patent, filed June 30, 1885, Serial No. 170,252.

By our method of refining *in vacuo* and rapidly transferring to the condenser we prevent oxidation. The great rapidity with which the vapor of the water is condensed prevents the free oxygen in the water from being absorbed.

The inclosing of the retort *d* and the jacket B B′, together with the neck D of the retort, in sand, as shown in the drawings, is for the purpose of maintaining uniformly the temperature desired, and to prevent premature condensation. A glass gage (not shown) on the end or side of the pocket B^5 indicates when the oil which has not passed over should be removed from the pocket B^5, as above described.

In the practical operation of our invention we first heat the sand surrounding the steam-jacket B B′ and retort *d*, operate the air-pump E^2, and allow steam to pass into the retort from the boiler A through the pipe A′, test the vacuum and uniformity of heat maintained throughout the retort *d*, neck D, pocket B^5, and pipe B^6, and the efficiency of the condenser E, and when all is conditioned satisfactorily we open the cocks *c′* and *g′* and allow the oil and sulphuric acid to be drawn into the retort *d* from the tanks C G. As soon as the oil enters the retort *d* from the pipe C′ it is impinged upon by the acid and steam, which continuously enter through the pipes A′, and by this combined action is thoroughly cleansed, and the undesirable portions cut out. It is accomplished instantaneously, and by the further combined action of the air-pump E^2 and the force of the steam is instantaneously conveyed to the neck D of the retort and into the condenser E.

The intermingling with the saturated steam in the retort *d* prevents the temperature in the retort from reaching such a degree as to cause decomposition of the oil or separation into its elements.

The fires in the furnace or chamber B^4 may be extinguished as soon as the sand is properly heated, and when the operation commences, unless it should be found necessary at times to combine it, because of the rapid evaporation going on in the interior of the retort *d*, or the inefficiency of the steam-jacket B B′ to maintain the uniform heat desired. This rarely occurs, as the sand-bath after being once heated is a sufficient adjunct to the steam in the jacket B B′, which may be, if necessary, superheated, but in no case must superheated steam be admitted into the retort *d*.

Observe, we do not decompose the oil, nor resolve it into any of its elements. The oil brought from the condenser E, and also that brought from the pocket B^5, is passed through the alkali bath, as described in the specification, to remove the impurities and undesirable portions cut out by the sulphuric acid, and also to neutralize the acid. It is then pure virgin oil. It is next saturated with the carbonic-acid gas, as described, to prevent rancidity and any tendency thereto. It also produces other good results, which we need not explain. It greatly increases the uses to which the oil can be applied.

Modifications may be made in the forms and proportions of the apparatus.

We can in many cases dispense with the sand bath in the case of vegetable or animal oils, and in such cases we can dispense with the fire for heating the sand bath and use superheated steam in the jacket.

In the case of some vegatable and animal oils we can dispense with the sand bath, the

jacket, and the condenser, and allow the vapor of water to pass off through the neck of the retort and through the pumps provided for maintaining the vacuum, and draw off the product from the bottom of the retort by proper means to prevent disturbing the vacuum.

We can use any other shape or style of retort that will admit of the oil being impinged upon by acid or alkali in the retort or receiving-vessel provided with means for preventing so high a temperature as to cause any decomposition of the oil.

Part of the invention may be used without the whole.

We can freeze by other means than the expansion of carbonic-acid gas and preserve the dry temperature; but we prefer the carbonic-acid gas because it can be introduced into the immediate presence of the product and with greatly beneficial results.

We can use a single air-pump or a series of air-pumps to maintain the vacuum and aid the steam in bringing over the particles passing through the retort.

We can use hot air for the jacket B B', if deemed expedient.

We can use a series of tanks and a series of filters, if desired.

We can have the acid meet the oil in opposite directions at right angles or in any direction that may be deemed most expedient.

We can force the oil and acid or alkali in a diffused condition into any retort or receiving-vessel by other means than the suction of steam, as described herein—as for example, force-pumps.

We can drop the oil on the acid and provide such additional provisions as would be required to remove the undesirable portions.

We propose to galvanize or tin the lining of the retort *d*, neck D, and condenser E.

We can have the retort *d* and neck D at any desirable angle.

We claim as our invention—

1. The process of impinging acid or alkali on highly diffused oil or impinging highly diffused oil on the acid or alkali, subsequently filtering through an alkali or acid and afterward saturating the oil with carbonic-acid gas, substantially as herein specified.

2. The process of introducing oil in a continuous stream into a vacuum-retort in the presence of acid or alkali, and subsequently filtering through an alkali or acid, substantially as herein specified.

3. The process of treating oil or fat with acid and alkali, saturating with carbonic-acid gas, and freezing and separating the frozen from the parts remaining liquid, substantially as herein specified.

4. The process of treating oil or fats with acid and alkali, saturating with carbonic-acid gas, freezing and separating the frozen from the parts remaining liquid, melting the frozen portion, mixing it with other oil or fat, and freezing again, substantially as herein specified.

5. The process of treating oil from cotton-seed or analogous sources in a highly-diffused condition by contact with acid or alkali in the presence of the vapor of water, subsequently filtering through alkali or acid, and afterward saturating with carbonic-acid gas, substantially as herein specified.

6. In the treatment of oil or fat from cotton-seed or other sources, the preservation of the oil or fat by saturation with carbonic-acid gas, substantially as herein specified.

7. The product described, being oil treated with acid and alkali and saturated with carbonic-acid gas, substantially as herein specified.

8. The product described, being oil treated with acid and alkali, saturated with carbonic-acid gas, and frozen and pressed, substantially as herein specified.

9. The product described, being oil treated with acid and alkali, saturated with carbonic-acid gas, frozen, pressed, mixed with other oil or fat, and frozen again, substantially as herein specified.

In testimony whereof we have hereunto set our hands, at New York city, this 27th day of July, 1885, in the presence of two subscribing witnesses.

JAS. B. GRANT.
ALLAN MASON.

Witnesses:
E. BROOKES,
MANIERRE ELLISON.