Dec. 13, 1949 C. R. KINCAIDE 2,491,115

METHOD FOR SOLVENT EXTRACTION

Filed Nov. 27, 1943 2 Sheets-Sheet 1

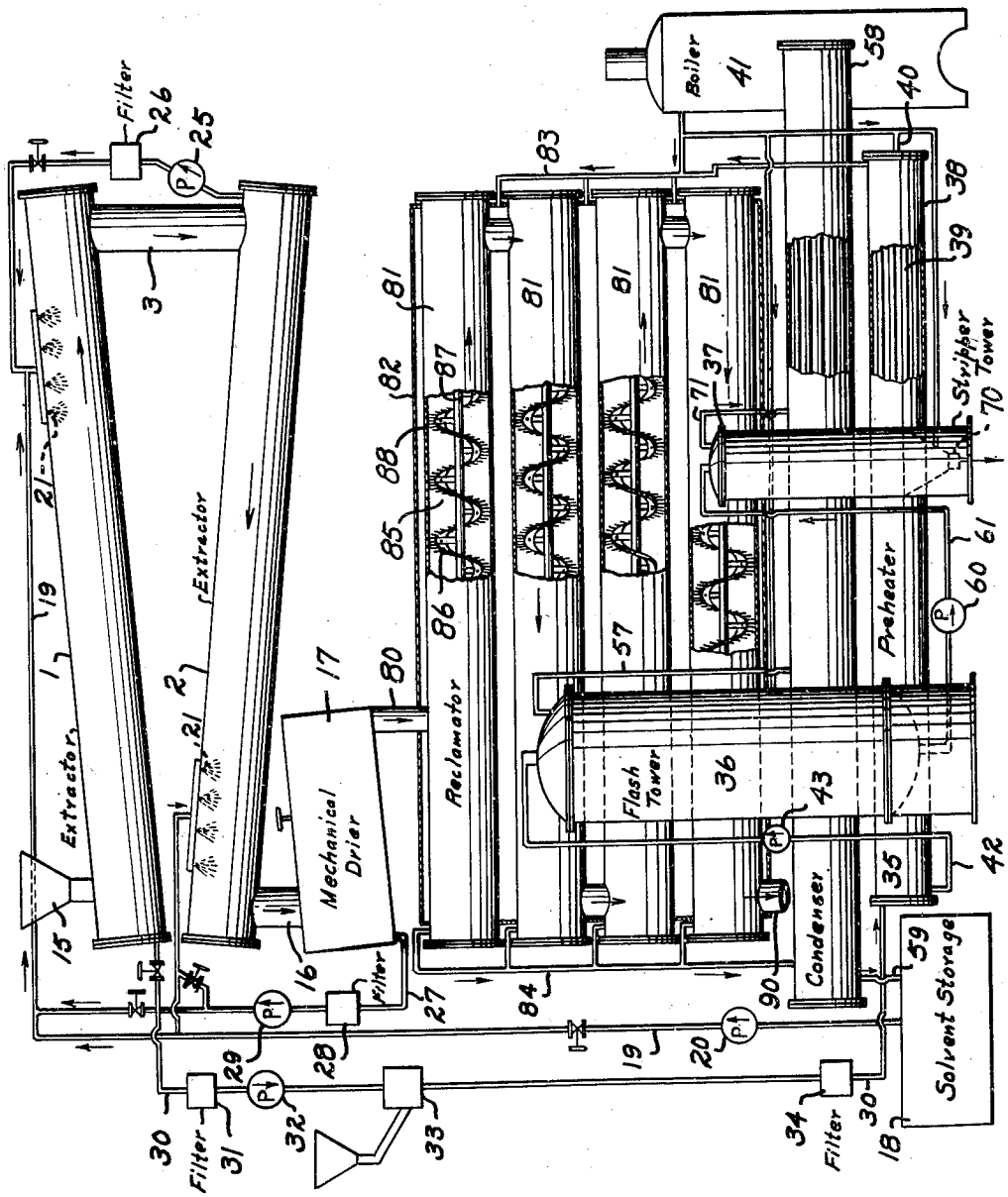


Fig. 1.

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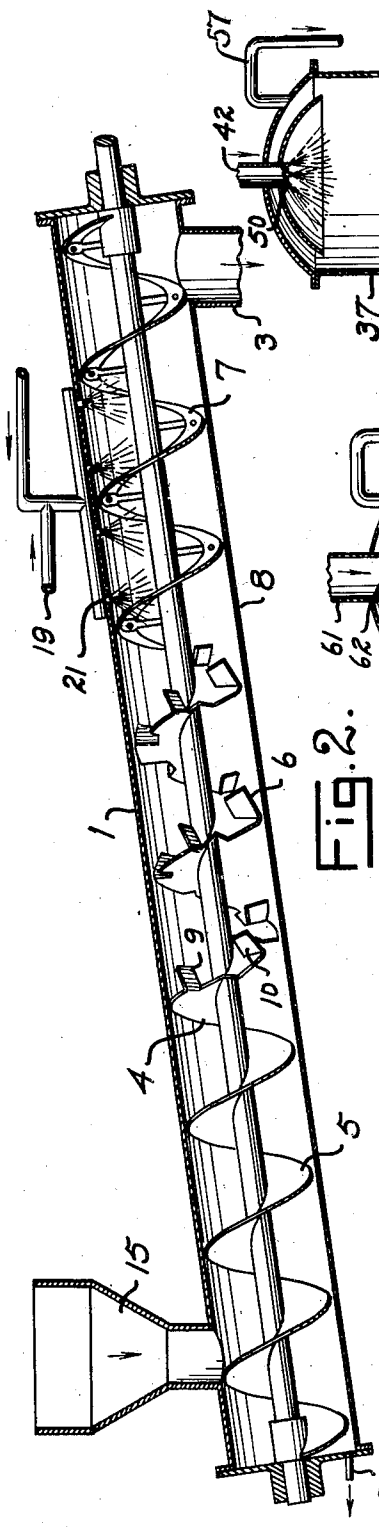


Fig. 2.

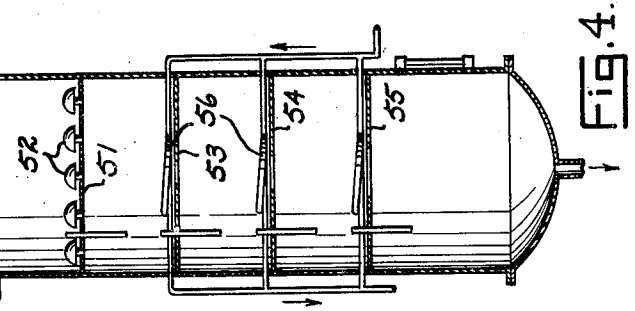


Fig. 4.

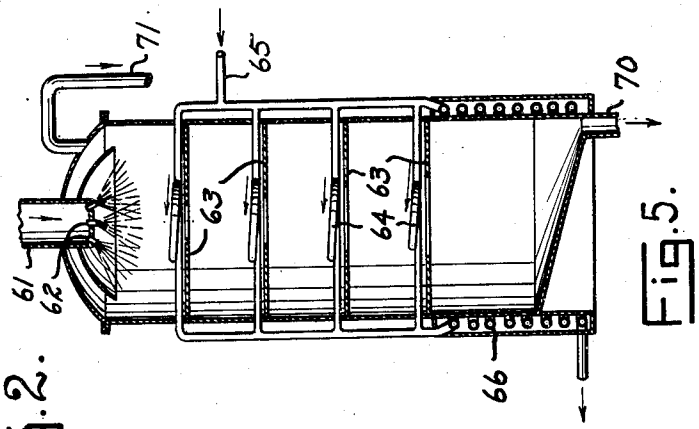


Fig. 5.

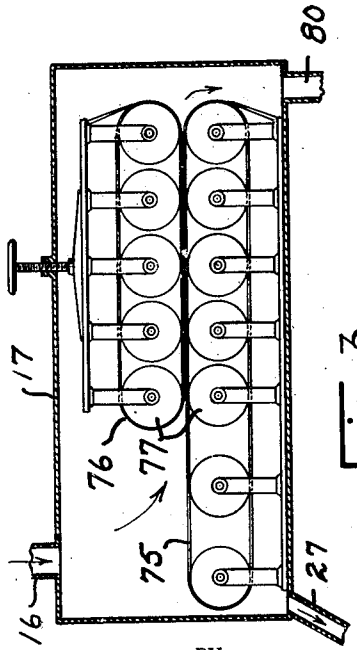


Fig. 3.

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UNITED STATES PATENT OFFICE

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METHOD FOR SOLVENT EXTRACTION

Charles R. Kincaide, Houston, Tex.

Application November 27, 1943, Serial No. 511,969

1 Claim. (Cl. 260—412.8)

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The invention relates to a means and method of solvent extraction of oils, fats, waxes, gums, resins, etcetera, from solids such as seeds, beans, and vegetable fibre as well as those of animal and mineral origin.

It has been found that the success of a solvent extraction process generally depends upon conducting the process in a minimum of time at a temperature somewhat below that at which decomposition sets in so as to avoid initiating such decomposition on the one hand and the discoloration of both the extractive and the meal on the other hand.

The term "raw solids" is used to designate vegetable, animal and minerals from which oils, fats, waxes, gums, and resins may be removed by a proper solvent; "extractive" will be used as descriptive of the large class of materials which may be recovered by solvent extraction; "meal" or clean solids will be used for the fibre or solid matter remaining; and "raffinate" designates the homogeneous liquid solution of extractive in solvent.

The present application is a continuation-in-part of prior copending application Serial No. 344,770, filed July 10, 1940, for a Method and apparatus for solvent extraction and also relates to the subject matter of copending application Serial No. 512,053, filed November 27, 1943, for Method of producing chlorinated hydrocarbons, both now abandoned.

It is one of the objects of the present invention to provide a solvent extraction process wherein a solvent is agitated in intimate contact with raw solids in a finely divided state so as to remove the desired extractive and to thereafter treat the resulting raffinate to reclaim the solvent for reuse and recirculation.

Another object of the invention is to provide a closed system vapor tight solvent extraction apparatus where the raw solids are agitated in the presence of a counterflow of solvent to produce a rich raffinate.

Another object of the invention is to provide a plurality of chambers for contact and agitation of the solids and solvent wherein the solids are exposed to a plurality of separate applications of the solvent to each chamber to remove the extractive.

Still another object of the invention is to provide an extraction or contact chamber where the agitation is applied by three distinct types of flights so as to obtain the desired rate of uniform movement while first immersing the material in the solvent and thereafter washing the material with solvent to obtain the maximum efficiency of solvent on the one hand and the maximum removal of the extractive on the other hand.

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Another object of the invention is to filter the rich raffinate at atmospheric temperature to effect the removal of solids and collodial matter in combination with a neutralization of the extractive so as to polish the extractive and avoid any decolorizing treatment.

Another object is to provide a flight conveyor for intimate association of the raw material and solvent to produce a rich raffinate wherein the conveyor is brush tipped to insure a wiping action against the chamber wall.

Another object is to conduct a simple and economical process of solvent extraction where a minimum of heat is applied to the extractive so as to avoid decomposition and discoloration and filtering of the meal is eliminated.

Another object is to neutralize and clarify the rich raffinate prior to the application of any heat.

Other and further objects of the invention will be readily apparent from the following description wherein—

Fig. 1 is a diagrammatic view partly in section illustrating an embodiment of the apparatus ready for operation.

Fig. 2 is a sectional view through one of the extractors showing the novel agitating multiple flight conveyor construction utilized in the invention.

Fig. 3 is a sectional view through the continuous press for removing excess liquid from the extracted solids.

Fig. 4 is a sectional view through the flash tower or still for separating the raffinate into solvent and solute.

Fig. 5 is a sectional view through the stripper tower which constitutes an element of the extraction system.

Referring to the drawings the numerals 1 and 2 refer to successive extractors which are oppositely inclined and which have their spaced

ends interconnected by means of a pipe 3. Each of these extractors is provided with a special agitator conveyor 4 (Fig. 2) driven from a suitable source of power (not shown) to agitate and carry any solids from the lower ends of the extractors for discharge at their respective upper ends.

The agitator conveyor 4 is of special construction to effect an intimate association of the solids and solvent and comprises three distinct conveyor flight sections 5, 6 and 7 which are so designed as to provide agitation and conveyance at a desirable rate of travel of solids within the cylinder 8. The lowermost section 5 of the agitator conveyor 4 is a section of solid flight which is designed to move and fold over the solids in the solvent in the lower end of the extractor chamber thereby preventing packing or clogging of the solids at this point.

The section 6 of the agitator conveyor is a section of a cut-and-folded flight in which the sections 9 and 10 are severed and flanged outwardly so as to provide surfaces which agitate and lift the solids to effect more vigorous and intimate mixing contact with the solvent while at the same time conveying the solids upwardly toward the ribbon type of flight 7 which, due to its open or skeleton construction, permits the incoming atomized solvent at 21 to move against or countercurrent to the movement of the solids.

The solids to be treated have been prepared by crushing, flaking, grinding or otherwise, so that the surface of the particles will present the maximum exposed area for contact with the solvent. These solids will be deposited in the hopper 15 and fed into the extractor 1 so as to be moved through the extractor cylinder by the conveyor 4.

The solvent from the storage tank 18 is conveyed by the pump 20 through the pipe 19 to be atomized or sprayed upon the solid matter by the nozzles 21. The solvent will be sprayed into the extractor at such a rate that there will be an accumulation thereof in the lower portion of the extractor and preferably the horizontal level of the liquid may be about half way up the inclined extractor.

When the solvent is mixed with the solid matter and removes the extractive therefrom it is thereafter known as raffinate and there will be a bath of this raffinate in the lower part of the extractor so that the first operation is the immersion of the particles in the raffinate bath. They will be agitated and conveyed by the solid flight 5 and gradually moved upwardly along the cylinder until they arrive at the cut and folded sections 9 and 10 where the agitation is substantially increased. At about this level the solid matter moves out of the bath and the additional agitation tends to allow the raffinate to drain from the particles. As the particles move along in the cylinder they arrive at the ribbon type conveyor 7 which additionally agitates the particles and they are subjected to a washing action by the atomized solvent distributed by the sprays 31. It seems obvious therefore that the solids have been subjected to at least two contacts with first the raffinate and then with the solvent so that a substanial part of the extractive borne by the particles is removed therefrom. The solid matter after having been washed passes through the pipe 3 into the second extractor where the operation is repeated. The solvent gradually being converted to raffinate as it contacts the solid, moves down the extractor cylinders by gravity in a counter current flow.

The rich raffinate which has absorbed a substantial portion of the extractive is drawn off through the pipe 30 at the lower end of the extractor 1 while the weak raffinate is picked up by the pump 25 and conducted through a pipe and the filter 26 which is connected to the pipe 19 so that the weak raffinate from the second extractor is recirculated in the desired proportion with the incoming solvent in the extractor 1.

In a similar manner the liquids removed from the mechanical dryer 17, to be more fully described, are conducted by means of conduit 27, filter 28 and the pump 29 to the nozzles at the upper ends of the extractor 1 to be recirculated.

Following the rich raffinate as it leaves extractor 1 through the pipe 30, it will be passed through a suitable filter 31 which is utilized for the purpose of removing any solid or colloidal matter carried by the raffinate. One of the essential features of the present process is to minimize the time in which the solvent is in contact with the solids and it is desirable to remove all of the solid matter from the raffinate prior to the application of any heat thereto so as to avoid discoloration or decomposition. The pump 32 carries the raffinate from the filter 31 into the chamber 33 which may be designated as a neutralizer and clarifier, there being an attachment on this clarifier 33 whereby suitable material may be added to neutralize, clarify and dehydrate the raffinate. An illustration of such a material is pulverized quick lime and calcined diatomaceous earth mixed together in substantially equal parts. This chemical is added and mixes with the raffinate as it continues its course through the pipe 30. The filter 34 then removes the added material so that the raffinate passes on to a preheater 35. With this procedure the raffinate is prepared for heat treatment and it should be particularly noted that all of the operations so far in the removal of the extractive and treatment of the raffinate, that the process is carried on at atmospheric temperature and pressure so that no decomposition or decoloration of the extractive has occurred.

The raffinate could be conducted directly into the flash tower 36 and thence to the stripper tower 37, but it has been found to be desirable to preheat the raffinate and the preheater 35 therefore comprises a cylinder 38 having the heater tubes 39 therein which are supplied with steam through the connection 40 from the boiler 41. The raffinate coming in contact with the heated tubes 39 is raised to the desired temperature and moved by the pump 43 through the pipe 42 to the nozzles 50 in the top of the flash tower as seen in Fig. 4.

This flash tower has a plurality of partitions or trays therein such as 51 provided with the bubble caps 52. The raffinate moves downwardly in succession over the trays 53, 54 and 55, each of which is provided with a heating coil 56. The temperature in the flash tower approximates 212° F. The solvent used preferably has an initial boiling point of approximately 100° F. and an end boiling point of approximately 175° F. so that the temperature in the flash tower is substantially in excess of the end boiling point whereby the solvent will be instantly vaporized from the rich raffinate entering the flash tower. Such a solvent and the production thereof comprise the subject matter of copending application Serial No. 512,053, to which reference has above been made. In this manner there will be no tendency for the various constituents making up

the solvent to vaporize independently at their own various boiling points between 100 and 175° F. The residual extractive passes passes through the bottom of the flash tower so that the period during which the extractive is subjected to heat is a minimum so as to prevent decolorization or even the initiation of decomposition.

The vaporized solvent rises instantly in the flash tower and escapes through the outlet 57 into the condenser 58 which is suitably cooled by the provision of the pipes therein so as to effect condensation of the solvent and its return through the pipe 39 to the solvent storage tank 18.

The extractive moving down through the flash tower is conducted through the pipe 61 by the pump 60 into the stripper tower 37 which is seen in section in Fig. 5. This stripper tower has the trays 63 and the coil 64 whereby steam from the lines 65 may heat the coils and vaporize any residual solvent from the solid material. The lower portion of the stripper tank is jacketed with the coils 66 and provided with an inclined bottom so that the extractive will be directed through the outlet 70. The vaporized solvent will pass through the pipe 71 into the condenser 58. The extractive will be free of solvent, it has been discolored and initial decomposition has not set in so that the additional steps of treating the extractive for the purpose of clarification, decolorizing and dehydrating are unnecessary. It might be stated that with other solvent processes where the rich raffinate is not filtered, clarified, neutralized nor dehydrated prior to vaporization that some of the solvent and colloidal therein tends to cause decoloration or decomposition of the extractive when it is heated.

One of the essential features of the present process is that in the first place the solid matter is maintained in contact with the solvent at atmospheric temperature for a minimum period. The rich raffinate thus formed is then immediately filtered to remove any solid or colloidal matter. It is then neutralized, clarified and dehydrated, all prior to the application of any heat. The pecularities of the solvent in that it has an end boiling point below the temperature at which decoloration and decomposition of the extractive sets in permits the operation of the process in such a manner that the extractive is not discolored nor is decomposition initiated. The vaporization of the solvent occurs instantly so that the extractive is benefited thereby because it has been found that too long a contact of the extractive with the usual type of solvents and a prolonged period of heat tend to destroy and discolor the extractive.

As best seen in Fig. 3 the solids passing through the pipe 16 are deposited upon an endless foraminated conveyor 75 which cooperates with a shorter conveyor 76 and a plurality of rollers 77 to adjustably apply pressure to such solids and thereby squeeze a major portion of the residual solvent from the solids. This solvent, as already explained, is conducted from the dryer through the conduit 27.

The solids discharged from the dryer 17 pass through a steam jacketed pipe 80 to the first of a series of reclamators 81 which are surrounded by a steam jacket 82 supplied with steam from the boiler 41 through the steam line 83. Any residual solvent remaining in the solids passing through the reclamators 81 is vaporized and is conducted from the respective units through the vapor line 84 and to the condenser 58 so that such vapor will be condensed and returned to the solvent storage tank 18.

In order that the clean solids shall be uniform and free from traces of the solvent used in the process, each of the reclamator chambers 81 is provided with a special conveyor 85 driven from a suitable source of power (not shown). Each of these conveyors comprises a ribbon flight 86 mounted upon a shaft 87, the flight being maintained in spaced relation with each the shaft and the inner walls of the surrounding chamber 81. In order to agitate and convey the solids within the chambers and to avoid caking of the solids to the inside walls of the chambers at any point therein the flight 86 are provided with fringed outer edges which are normally of slightly greater diameter than the interior walls of the chambers and which possesses such resiliency that the fringe 88 maintains constant brushing or scraping action on the inside walls of the reclamator chambers. In this manner any tendency of solids to stick to the chamber is overcome as the solids are constantly moved toward the outlet 90 whence the clean solids are conveyed in any suitable manner to storage for use in commerce.

The solvent per se is being described and claimed in my copending application Serial No. 512,053 heretofore mentioned, but generally for the purposes of the present disclosure it may be stated that this solvent has no color, is water white, is non-explosive, non-inflammable, it has an initial boiling point approximating 100° F., an end boiling point of approximately 175° F., a weight approximating 11.288 lbs. per gallon, the maximum vapor pressure at recovery is 10 lbs. and a latent heat of evaporation approximates 192 B. t. u. per pound. It is impossible to determine the molecular of weight because it is a miscible solution of a large number of chlorinated hydrocarbons formed from the isomers of the hydrocarbons with chlorine which produces many different compounds having individual boiling points which overlap each other, but which it is impossible to separate into the different constituents thereof by any form of distillation.

As just indicated no heat is applied to the soluble constituents until all solids have been removed therefrom. In a similar manner no heat is applied to the solids until such solids have passed through the mechanical dryer 17 and into the reclamators 81. The treatment of the solids in the reclamators removes all of the solvent in a manner and at such temperature that the clean solids withdrawn from the outlet 90 are likewise of higher quality.

Broadly the invention comprehends a continuous system and method for solvent extraction of the soluble constituents of raw solids so that liquid and clean solid products of higher quality are obtained from the raw solids.

What is claimed is:

A solvent extraction process including the steps of effecting an intimate mixing of finely divided particles of oil bearing material by immersing the particles in the solvent, agitating the mix while gradually moving the solids out of the raffinate thus formed, washing the solids by spraying solvent thereover while agitating the solids, withdrawing the rich raffinate thus formed from the area of immersion, drying the solids and vaporizing the solvent therefrom to discharge dry extractive free solids, polishing the rich raffinate by filtering to remove any solids or colloidal matter therefrom, treating the raffinate for effecting

neutralization, clarification and decolorization all prior to the application of other than atmospheric pressure or temperature thereto, refiltering the raffinate, subjecting the raffinate to flash heating for a minimum period so as to avoid any substantial setting in of initial decomposition and above the end boiling point of the solvent to instantly vaporize the solvent without discoloring the extractive, condensing the solvent for recirculation and stripping the extractive of any remaining solvent.

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