

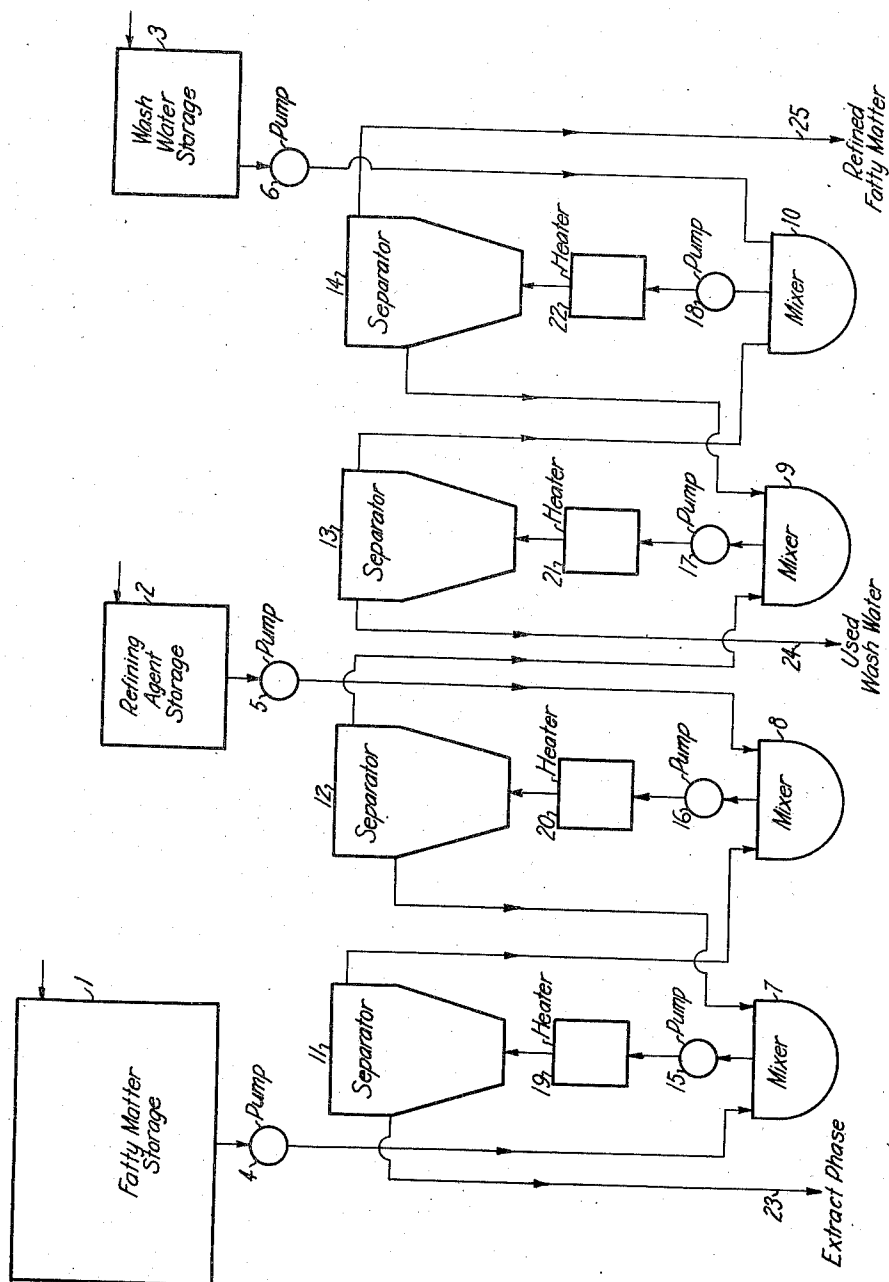
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W. J. HUND ET AL

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FATTY MATTER REFINING PROCESS

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Inventors:

Walter J. Hund

Donald H. Rowe

By their Attorney: *Arthur B. Nichols*

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FATTY MATTER REFINING PROCESS

Walter J. Hund and Donald H. Rowe, Oakland, Calif., assignors to Shell Development Company, San Francisco, Calif., a corporation of Delaware

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This invention relates to a process for removing undesirable constituents such as free fatty acids, certain coloring bodies, mucilaginous material, etc., from fatty matters, and has for its object the provision of a method affording a superior product, a more economical execution and a minimum of loss, and at the same time a method of such flexibility that it may be adapted to the refining of the most diverse crude fatty matters and products.

According to the present invention, the fatty matter to be refined is extracted with a suitable, substantially immiscible extractant and subsequently washed free of traces of the extractant, said extraction and washing operations being performed in a continuous multi-stage countercurrent manner.

The accompanying drawing illustrates diagrammatically one suitable flow arrangement for a plant wherein the crude fatty matter may be subjected to a two-stage extraction with a refining agent, followed by a two-stage washing, both operations being performed in a continuous countercurrent manner. It is to be understood that this arrangement may be altered considerably without departing from the spirit of the invention. Thus, for example, one of the washing stages may be omitted; either the extraction or the washing or both may be performed in more than two stages; treatment with a third or fourth agent may be inserted in one or in multiple stage; the capacity of the plant may be increased by using several units in parallel, as, for example, four mixer units, three heating units, etc.

Referring to the drawing, the numerals 1, 2 and 3 represent storage tanks for the crude fatty matter, extractant and wash water, respectively. The fatty matter storage tank 1 is preferably equipped with heating means, such as, for instance, a steam coil. Numerals 4, 5 and 6 represent pumps by means of which the above materials are supplied to the system. Said pumps are preferably of such construction as to allow adjustment to a wide range of flow. Numerals 7, 8, 9 and 10 represent mixing devices, and numerals 11, 12, 13 and 14 represent separating means, in this case centrifugal separators. Numerals 15, 16, 17 and 18 represent pumps and numerals 19, 20, 21 and 22 represent heating means, such as, for instance, steam heated coils.

Again referring to the drawing, the crude fatty matter as a liquid at the desired temperature is fed from tank 1 by pump 4 into mixer 7 wherein it is thoroughly contacted with, but not emulsified with, the once used extractant from separator 12.

The mixture is continually delivered by means of pump 15 from mixer 7 to separator 11 wherein the two phases are quickly separated. By means of a heating means 19, the temperature of the mixture is adjusted to give efficient separation. The partially refined fatty matter leaving separator 11 flows to a mixer 8 wherein it is thoroughly contacted with fresh extractant fed continually to the mixer by pump 5 from tank 2. The mixture is delivered by means of pump 16 to separator 12 wherein the phases are again quickly separated. A second heating means 20 may be used to control the temperature and thus aid rapid separation. The twice treated fatty matter from separator 12 then flows to a mixer 9 wherein it is washed with once used water from separator 14. The mixture is then delivered by means of pump 17 to separator 13 wherein the phases are quickly separated. The refined and partly washed fatty matter from separator 13 flows to mixer 10 wherein it is thoroughly contacted with fresh water supplied continually from tank 3 by pump 6. The mixture is delivered by means of pump 18 from mixer 10 to separator 14 wherein the phases are quickly separated. The heating means 21 and 22 allow temperature control during these later steps. The fatty matter issues from separator 14 via pipe 25 in a well refined and substantially dry state.

During the course of refining the extractant and the wash agent flow continually in a countercurrent manner with respect to the fatty matter, i. e., from right to left. Thus the fresh extractant is fed continually by means of pump 5 into mixer 8 wherein it meets the partially refined fatty matter from separator 11. After being thoroughly contacted in mixer 8 and the phases separated in separator 12, the once used extractant flows to mixer 7 wherein it is thoroughly contacted with the crude fatty matter continually fed from tank 1 by pump 4. The twice used extractant then issues from separator 11 through pipe 23.

The wash water travels a countercurrent path similar to that of the extractant being fed continually from tank 3 by pump 6, passing consecutively through 10, 18, 22, 14, 9, 17, 21, 13 and issuing via pipe 24.

We have found that the present method of refining is superior to the other known methods, especially as regards quality of product, losses incurred during refining, adaptability to the refining of various fatty matters, as also the preservation of valuable vitamine forming constituents, amount of extractant required and throughput

capacity. The various advantages realizable through the use of our invention are best appreciated in the light of the problems encountered in the art of fatty matter refining.

5 The refining agents generally used in fatty matter refining, such as, for example, aqueous alkalis, alkylolamines, dilute mineral acids, etc., besides functioning as refining agents, all react with the fatty matter to a greater or less extent
10 to effect a certain amount saponification, etc., of the fatty matter, which, in many cases, represents an appreciable loss. The loss resulting from this cause is by no means restricted to the actual amount of fatty matter reacted, but is
15 considerably greater. Since the products of saponification, etc., are usually active emulsifying agents, they considerably lengthen the time required for phase separation, thereby causing
20 reaction, and by preventing a sharp interface, cause the loss of unsaponified oil by removal with the emulsified layer.

Since the refining agents function to a large extent as true extractants, the degree of refining
25 and the percentage losses are, other things being equal, dependent upon the effective time of contact, and, being so, are dependent one upon the other. In such cases, therefore, where a highly refined product is desired, the refiner is usually
30 confronted with a substantial loss, and this loss frequently determines the degree of refining economically practical.

We have found that the present continuous multi-stage countercurrent method is superior to
35 other methods in this respect, since, other things being equal, the present method produces a more highly refined product.

Notwithstanding the fact that in executing the present invention, the fatty matter is actually
40 subjected two or more times to the action of the refining agent, we have found that, other things being equal, the present method incurs less loss than the known methods. This is a decided advantage, especially when treating costly materials, such as, for example, the refining of cod
45 liver oil or carrot oil. This advantage is achieved by several features inherent in the continuous multi-stage countercurrent process.

Although a certain amount of loss due to reaction
50 is incurred during the mixing operation and is unavoidable, a substantial proportion of such loss occurs during the separation of the phases, during transfer, in waiting tanks, etc. Thus, fatty matters, after being treated with a refining agent and
55 separated therefrom, always contain small quantities of the refining agent which may react with the fatty matter at an appreciable rate. In order to reduce the losses to a practical minimum, the present method is so adapted as to afford the
60 least contact between the reactants except that required during the mixing operation.

Immediately upon issuing from the mixing operation, the phases are separated, as, for instance, in a centrifugal separator, and the separated
65 phases are immediately subjected to the next following step, etc., until the product has been completely treated.

Thus, the fatty matter from the first refining stage is piped directly to the second mixer
70 (mixer 8). The product from the last stage of refining is subjected immediately and continually to a washing treatment, followed by a separation of the phases. We prefer to subject the raffinate to a continuous two-stage washing treatment, although this second stage is not usually necessary.

The once washed raffinate contains only minute quantities of refining agent, and therefore, does not necessitate further promptness in handling. The once washed raffinate may be batch washed,
5 stored, filtered, bleached, dried or otherwise treated as the case may require.

Since it is desirable to eliminate all unnecessary contact of the refining agent and the fatty matter, and since the time required for phase separation represents such undesirable contact, it
10 is advantageous to execute the phase separation promptly and as efficiently as practically feasible. We have found that centrifugal separator machines are eminently suited for such separation. If the fatty matter in question contains appreciable quantities of sludge forming materials, a centrifugal separator provided with a sludge type bowl may be advantageously used for the first
15 separator, (separator 11). However, any means whatsoever which will effect a prompt separation may be used. In some cases where the fatty matter responds to treatment with a very dilute or weak agent, such as a 1% solution of NaOH, ordinary gravity separation may be applicable.

The present method has the further advantage, other things being equal, of requiring less
25 refining agent. This advantage can in some cases result in a substantial lowering of the refining costs.

Another advantage of the present method is its flexibility, i. e., its adaptability to the refining of
30 the most varied fatty matters with the usual refining agents. According to the quality of the crude fatty matter and the ultimate use to which the refined product is to be employed, a wide variety of problems are encountered in the refining of fatty matters. Thus for example,
35 oils that have been stored for some time behave differently than fresh oils, and, therefore, sometimes require radical changes in the refining process. Likewise, cold pressed oils are usually much more difficult to refine than hot-pressed oils. Oils obtained from old or bruised seed contain certain other impurities which require special
40 methods for their removal. All oils are known to vary with the season of harvesting and the locality of production. Certain oils often contain impurities which act as emulsifying agents, and such oils usually require a substantial departure from the usual refining methods. Such
45 variations as are required for each individual case, such as giving a preliminary treatment with water, dilute alkali, dilute acid, or variation of the strength, kind or amount of refining agent, alteration of the temperature, or time of mixing,
50 etc., are easily accommodated by the present method. Any of the usual refining agents, such as various strengths of HCl, H₂SO₄, H₃PO₄, KOH, NaOH, Na₂CO₃, NaHCO₃, Na₃PO₄, alkylolamine, dialkylolamine, etc., may be employed in the
55 present invention. In the usual refining practice these agents function both as reactants and as extractants, and in such cases they are used in considerable excess. In a few cases, such as when it is merely desired to remove the fatty
60 acids, the refining agent may be used in approximately the stoichiometric amount. When desirable, as when using an alkylolamine as a refining agent, the used agent may be recovered by any of the known methods and reused.

The use to which an oil is to be employed requires further variation in the refining methods. Thus, for example, a sardine oil destined for poultry feed must be refined without serious loss of
65 vitamins, while if the same oil is to be used as a

paint oil, the preservation of the vitamins is of no consequence and the refining process must be executed so as to substantially remove all the coloring bodies, and at the same time improve the drying qualities of the oil. In the refining of edible oils, little attention has been given in the past to the preservation of such valuable components as carotin. This substance, which is a valuable source of vitamine, is exceedingly susceptible to oxidation, absorbing about 34% of its weight of oxygen to revert to a colorless valueless constituent. According to the present method, the fatty matter subjected to treatment is allowed little opportunity to oxidize. Since under the preferred mode of operation the mixer units 7, 8, 9 and 10 are completely filled with liquid, oxidation of the carotin during mixing is substantially obviated.

The present invention is applicable to the refining of fatty matter of animal, vegetable or synthetic origin. Thus, for example, hot pressed, cold pressed and unclarified hot pressed California cottonseed oil, soya bean oil, palm oil, linseed oil, sardine oil, perilla oil, corn oil, peanut oil, cohune nut oil, and sundried and smoke dried copra oils have been successfully refined by the present method.

While, in order to efficiently refine a given fatty matter, it is quite often necessary to alter the refining method materially, such as by using an entirely different refining agent, by using a plurality of refining agents, by giving accessory treatments, etc., it often happens that fatty matters differ mainly in the relative amounts of the impurities present, and in such cases adjustment of the effective contact is usually sufficient to insure maximum efficiency of refining. Since the effective contact is dependent upon the time of contact, degree or intimacy of contact, and strength and quantity of reagent, this value may be fixed by adjustment of any of said variables. An optional means of altering the time of mixing may be afforded through the use of a subdivided mixing unit. Instead of using one mixing device, the mixing in one or more cases may be effected in a plurality of smaller mixing means, preferably connected in such a manner as to allow their use in series or in parallel. With such an arrangement, it is possible to alter the effective volume and thereby the effective time of mixing without changing the throughput rate of the plant, as, for instance, by shunting out one unit of the mixing battery. In some cases, where a single mixing device is used, it is possible to control the time of mixing through variation of the quantity of liquid therein. However, while this later method is applicable to the refining of varnish oils, etc., it necessitates the providing of an oxygen free atmosphere above the liquid when the vitamine content of the fatty matter is of import.

The capacity of a satisfactory mixing device or the total capacity of the units, if subdivided, may vary considerably. We have found that a capacity equal to about one-seventh of the capacity of the plant per hour is one suitable size.

Since fatty matters vary considerably in their tendency to form emulsions, mechanical mixing devices, such as paddles, propellers, etc., capable of variable speed are advantageously used. Through adjustment of the speed, the degree of mixing may thus be regulated to give the maximum effective contact for any particular fatty matter without unduly emulsifying the same. Other means of agitation, such as baffles, orifices,

etc., if properly constructed, should be applicable.

The present continuous, multi-stage, counter-current process, since it is inherently more efficient than any of the known processes, is effectively executed with less violent contacting of the fatty matter and extractant and consequently allows a quicker and easier separation of the phases. This not only reduces the losses, but increases the throughput capacity of the plant. Since the operations are rapid and not interrupted by waiting tanks, etc., the present process affords a plant requiring a relatively small space and having a high throughput capacity. The compact nature, thus afforded, permits of ease of cleaning, small loss of heat, and a minimum tendency for high melting fats, etc., to freeze in the pipes.

As illustrative of the use, conditions and results of one embodiment of our invention, we offer the following:

Example I

A particularly refractive cold pressed cottonseed oil having an appreciable content of sludge forming material, when refined under optimum conditions by the best of the known methods, produced an oil having a color of 35 yellow and 12.6 red (Lovibond Tintometer scale). The color of this oil upon bleaching reduced to 20 yellow and 4.6 red. 804 lbs. of this same oil, treated in a continuous two-stage countercurrent manner, using the same amount and kind of reagent, namely, 3.1% by volume of 24° Bé. caustic soda solution, yielded 768.5 lbs. of treated product having a color of 35 yellow and 8.1 red. Upon bleaching, the product had a color of 20 yellow and 2.2 red.

In this experiment, the separations were achieved in centrifugal separators running at about 7200 R. P. M. The first separator was not equipped with a sludge type bowl and this necessitated running the separation at a higher temperature than customary. The mixers consisted of two one-gallon mixing devices provided with motor driven submerged paddles, and connected in series. The total time of residence was 40 minutes which was divided into 20 minutes for washing and 20 minutes for extracting, this latter being in turn subdivided into 16 minutes for mixing and 4 minutes for separating, heating and transportation from one step to the next.

Upon consideration of the advantages offered by the present continuous multi-stage, counter-current method, it is seen that this method has distinct advantages not possessed by counter current tower extraction, single phase extraction, batch methods, etc., and that it is not equivalent to simply running a product through a refining plant two or more times. Thus, the present process offers economy of space and adaptability to the most varied fatty matters. Depending upon the feature most desired, the present method can be used to advantage to produce a more highly refined product, reduce the losses or effect a saving in the amount of refining agent used.

It is to be noted that the advantages realizable through the use of the present invention spring from the continuous, multi-stage counter-current manner in which the process is executed. While in the preferred embodiment, the process is arranged and executed in such a manner as to be distinctly advantageous for the refining of the most varied fatty matters, it is to be remembered that for a particular case it may not be necessary to utilize all the advantages simul-

taneously, and that for this reason considerable variation in the details of the execution may some times be possible without departing from the spirit of the invention.

5 We claim as our invention:

1. A process for the refining of fatty matters in a continuous, multi-stage countercurrent manner, which comprises contacting a fatty matter in the first stage with a substantially immiscible
10 basic acting extractant in the liquid state which is reactive with said fatty matter, said extractant having been previously contacted with and separated from the partially refined fatty matter in the second stage, separating the bulk of the extractant from the fatty matter, contacting the
15 once extracted oleaginous phase in the second stage with a purer portion of extractant, separating the bulk of the extract phase from the fatty matter, and contacting the extract phase from the second stage with the crude fatty matter in the first stage.

2. A process for the refining of fatty matters in a continuous, multi-stage countercurrent manner, which comprises contacting a fatty matter in the first stage with a substantially immiscible
25 basic acting extractant in the liquid state which is reactive with said fatty matter, said extractant having been previously contacted with and separated from the partially refined fatty matter in the second stage, heating the mixture, separating the bulk of the extractant from the fatty matter, contacting the once extracted oleaginous phase in the second stage with a purer portion of extractant, heating the mixture, separating the
30 bulk of the extract phase from the fatty matter and contacting the extract phase from the second stage with the crude fatty matter in the first stage.

3. A process for the refining of fatty matters in a continuous, multi-stage countercurrent manner, which comprises contacting a fatty matter in the first stage with a substantially immiscible basic acting extractant in the liquid state which is reactive with said fatty matter, said extractant having been previously contacted with and separated from the partially refined fatty matter in the second stage, promptly separating the bulk of the extractant from the fatty matter, promptly contacting the once extracted
40 oleaginous phase in the second stage with a purer portion of extractant, promptly separating the bulk of the extract phase from the fatty matter, and promptly contacting the extract phase from the second stage with the crude fatty matter in the first stage.

4. A process for the refining of fatty matters in a continuous, multi-stage countercurrent manner, which comprises contacting a fatty matter in the first stage with a substantially immiscible
60 basic acting extractant in the liquid state which is reactive with said fatty matter, said extractant having been previously contacted with and separated from the partially refined fatty matter in the second stage, centrifugally separating the bulk of the extractant from the fatty matter, contacting the once extracted oleaginous phase in the second stage with a purer portion of extractant, separating centrifugally the bulk of the extract phase from the fatty matter, and contacting the extract phase from the second stage with the crude fatty matter in the first stage.

5. A process for the refining of fatty matters in a continuous, multi-stage countercurrent manner, which comprises contacting a fatty matter
75 in the first stage with a substantially immiscible

basic acting extractant in the liquid state which is reactive with said fatty matter, said extractant having been previously contacted with and separated from the partially refined fatty matter in the second stage, separating the bulk of the extractant from the fatty matter, contacting the once extracted oleaginous phase in the second stage with a purer portion of extractant, separating the bulk of the extract phase from the fatty matter, contacting the extract phase from the second stage with the crude fatty matter in the first stage, and finally subjecting the extracted fatty matter from the last stage to a washing treatment.

6. A process for the refining of fatty matters in a continuous, multi-stage countercurrent manner, which comprises contacting a fatty matter in the first stage with a substantially immiscible basic acting extractant in the liquid state which is reactive with said fatty matter, said extractant having been previously contacted with and separated from the partially refined fatty matter in the second stage, heating the mixture, promptly separating the bulk of the extractant from the fatty matter, promptly contacting the once extracted oleaginous phase in the second stage with a purer portion of extractant, heating the mixture, promptly separating the bulk of the extract phase from the fatty matter, and promptly contacting the extract phase from the second stage with the crude fatty matter in the first stage.

7. A process for the refining of fatty matters in a continuous, multi-stage countercurrent manner, which comprises contacting a fatty matter in the first stage with a substantially immiscible basic acting extractant in the liquid state which is reactive with said fatty matter, said extractant having been previously contacted with and separated from the partially refined fatty matter in the second stage, heating the mixture, centrifugally separating the bulk of the extractant from the fatty matter, contacting the once extracted oleaginous phase in the second stage with a purer portion of extractant, heating the mixture, separating centrifugally the bulk of the extract phase from the fatty matter, and contacting the extract phase from the second stage with the crude fatty matter in the first stage.

8. A process for the refining of fatty matters in a continuous, multi-stage countercurrent manner, which comprises contacting a fatty matter in the first stage with a substantially immiscible basic acting extractant in the liquid state which is reactive with said fatty matter, said extractant having been previously contacted with and separated from the partially refined fatty matter in the second stage, heating the mixture, separating the bulk of the extractant from the fatty matter, contacting the once extracted oleaginous phase in the second stage with a purer portion of extractant, heating the mixture, separating the bulk of the extract phase from the fatty matter, contacting the extract phase from the second stage with the crude fatty matter in the first stage, and finally subjecting the extracted fatty matter from the last stage to a washing treatment.

9. A process for the refining of fatty matters in a continuous, multi-stage countercurrent manner, which comprises contacting a fatty matter in the first stage with a substantially immiscible basic acting extractant in the liquid state which is reactive with said fatty matter, said extractant having been previously contacted with and separated from the partially refined fatty matter in the second stage, promptly separating centrifugally the

