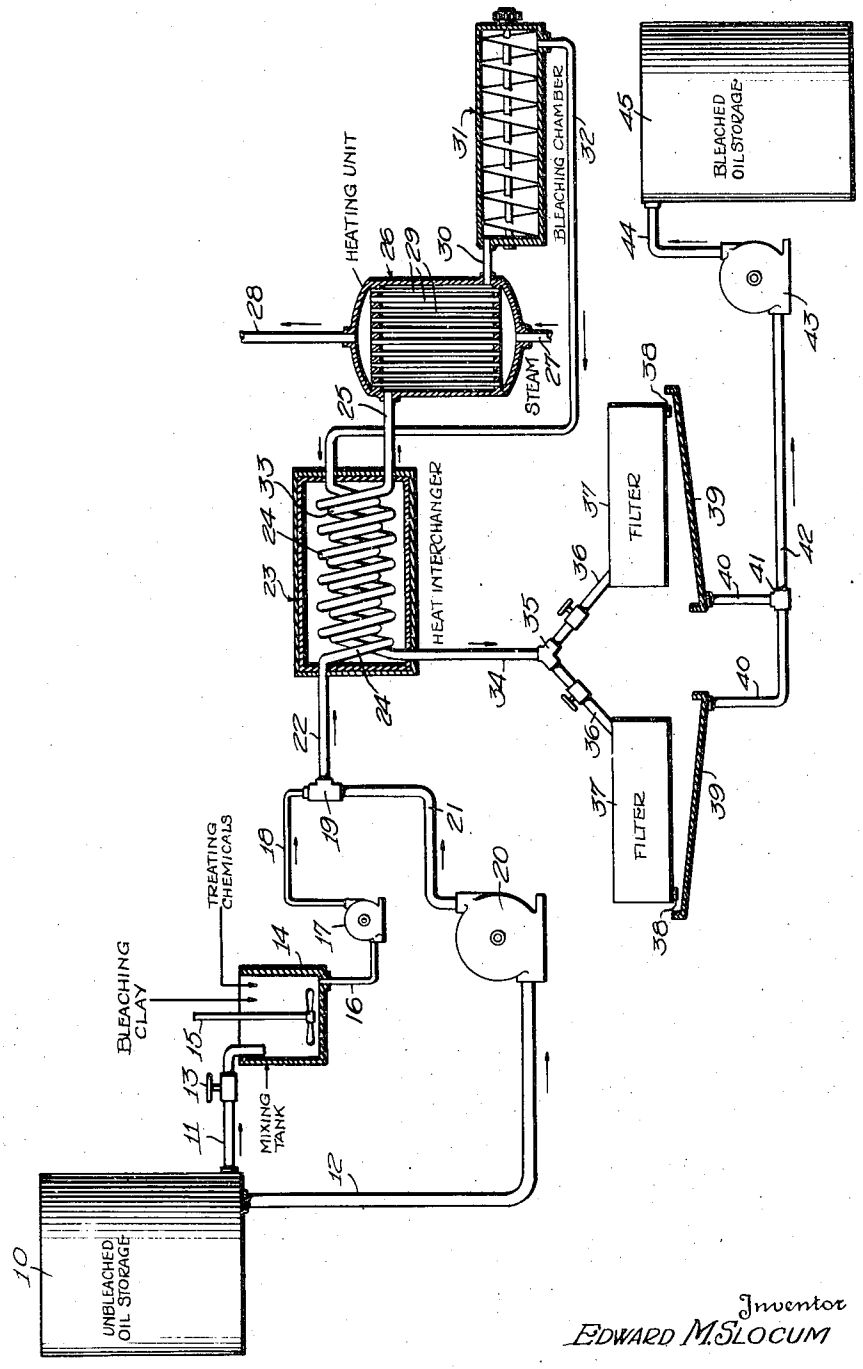
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PROCESS OF PREPARING ADSORBENT CLAYS

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PROCESS OF PREPARING ADSORBENT CLAYS

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This invention relates to the bleaching of animal and vegetable oils and fats. It also relates to the activation of bleaching clays and to the recovery of residual oils or fats remaining in spent bleaching clay after the use of such clay in the bleaching of animal and vegetable oils and fats.

This is a division of my copending application Serial No. 726,823, filed May 21, 1934.

The principal object of this invention is to provide an improved process of bleaching animal and vegetable oils and fats wherein numerous of the disadvantages attending similar processes heretofore employed are avoided.

An important object of the present invention is to provide a continuous process of bleaching animal and vegetable oils and fats.

A further object of the invention is to provide a process of bleaching animal and vegetable oils and fats wherein the apparatus employed is of more simple and inexpensive character than the apparatus commonly employed in similar methods heretofore used.

A further object of the invention is to provide a process of bleaching animal or vegetable oils and fats which is productive of a product of substantially better color and stability than prior processes of bleaching oils and fats.

A further object of the present invention is to provide an improved process of activating bleaching clay.

A further object of the invention is to provide a bleaching clay which is substantially free from water soluble electrolytes.

Another object of the invention is to provide an improved process of reclaiming oil from spent bleaching clay.

Another object of the invention is to provide a process of reclaiming oils and fats from spent bleaching clay without utilizing the conventional step of blowing the spent clay with air.

Other objects and advantages of the invention will become apparent during the course of the following description.

The method in general use for bleaching animal and vegetable fats and oils is to introduce a large quantity, say from 10,000 to 100,000 pounds, of the oil or fat to be bleached into a large iron tank equipped with a steam heating coil and agitators. The oil is heated to a temperature of around 212° F. Powdered bleaching clay is dumped in and stirred for about fifteen or twenty minutes. The mixture of oil and clay is then pumped in through a leaf filter press provided with canvas filtering cloths. The oil is discharged through brass faucets into a trough. The oil in this trough is pumped back into the bleaching tank until turbidity disappears and a satisfactory color is obtained. The oil running from the filter press is then pumped into a storage tank, agitation of the oil and clay being continued until all the oil has been filtered.

After all of the clay has been pumped into the filter press, it is then blown with air and thereafter blown with steam for the purpose of removing the residual oil from the spent clay. The oil so removed from the spent clay is very dark and poor in quality so that in most cases it can be used only for making so-called "soap powder".

The conventional procedure as referred to above is often very detrimental to the stability of the oil so processed. Its resistance to becoming rancid is in many cases cut in half. Another disadvantage is that the oil is heated for a considerable period in the presence of air and in contact with iron, both of which are injurious to the oil. Moreover, the oil forms a hardened film of oxidized oil around the fibers of the canvas filter cloths, reducing speed of filtration and leading to the breaking of the cloths. There are several other recognized disadvantages in the conventional bleaching procedure.

One of the most important disadvantages from a commercial standpoint is the fact that the oil lost in the spent clay runs from 18 to 45% of the dried spent clay. This is by far the heaviest expense in bleaching oil. The value of the oil lost in the spent clay may be several times that of the clay used in the process.

As a result of extensive research directed to improving the disadvantageous conventional processes of bleaching animal or vegetable oils and fats, I have devised a method which avoids most of the objectionable features of the present method in common use.

In a broad way, my process comprises mixing the required amount of bleaching clay for treating the volume of animal or vegetable fat or oil with a portion of the material to be bleached to provide a dispersion of the clay therein, to which dispersion may be added treating chemicals of the character hereinafter referred to. This dispersion is then continuously mixed in required amount with the unbleached oil or fat, the resulting mixture thereafter being heated to a temperature favorable to the bleaching of the oil or fat and agitated in a bleaching chamber, after which the mixture is passed to a filter where the bleached material is separated from the spent bleaching clay. It is an important part of my process that the material to be bleached is con-

stantly maintained out of contact with the air or oxidizing gases. This is accomplished by employing a closed system which at all times is substantially completely filled with the material under treatment, thereby excluding air without necessity of the creation or maintenance of a vacuum in the system. The commercial importance of my process is also dependent in part on the procedures which I employ for activating the bleaching clay prior to the use thereof in the bleaching process and for recovering oil or fat of good quality from the spent bleaching clay, as hereinafter described in detail.

In the accompanying drawing I have shown in diagrammatic form an assembly of apparatus suitable for use in the practice of my process.

Referring to the drawing, the numeral 10 designates a storage tank for the oil or fat to be bleached, which tank is provided with outlet pipes 11 and 12. The outlet pipe 11, which is provided with a valve 13, is adapted to deliver oil or fat into the mixing container 14 which is preferably provided with a suitable mixing or agitating device 15. The mixing container 14 is provided for preparing the oil or fat dispersion of bleaching clay, to which dispersion other treating chemicals may be added. The pipe 16 is adapted to deliver the dispersion from the mixing container 14 to a pump 17 which forces the material through the pipe 18 to a union 19 where it is mixed with the material to be bleached which is delivered from the storage tank 10 through the pipe 12 to a pump 20 which forces the material through the pipe 21 to the union 19. From the union 19 the mixture of the dispersion from the pipe 18 and the unbleached material from the pipe 21 is passed through the pipe 22 to a heat insulated heat interchanger generally designated by the reference numeral 23, the mixture passing through a coil 24 arranged within the heat interchanger 23 and being discharged therefrom through the pipe 25 which connects with a heating unit 26 having steam inlet and outlet pipes 27 and 28 and heating tubes 29 which may be arranged in the manner of the usual tube boiler construction. The material which passes through the heating unit 26 is delivered by the pipe 30 into a bleaching chamber 31 which is provided with a stirring device preferably in the form of a screw conveyor. The material discharged from the bleaching chamber 31 is passed through the pipe 32 to a coil 33 arranged in juxtaposition to the coil 24 in the heat interchanger 23. The material under treatment is discharged from the coil 33 through the pipe 34 which connects with a union 35 from which the material may be directed through either of the valved pipes 36 to a preselected one of a series of filters 37. The filters 37 may be of conventional type such as a leaf filter press, a conventional self-emptying filter press, a centrifugal filter, or the like. While in the accompanying drawing only two filters are designated, it will be apparent that additional filters may be used where required. Each of the filters 37 is provided with an outlet 38 which is adapted to discharge the filtered animal or vegetable oil or fat into the conventional troughs 39 from which the filtered material is carried by pipes 40 which connect at the union 41 to a pipe 42 connected to a pump 43 which is adapted to pump the bleached oil or fat through a pipe 44 into a storage tank 45.

In the preferred practice of my process, specially activated bleaching clay is employed. This is recommended in all cases and is particularly desirable where a leaf filter press is employed in the process and where the clay used would, in the absence of the special activation referred to, be high in water soluble electrolytes and particularly salts of calcium, magnesium and the like, which would form insoluble soaps in a subsequent treatment for removing residual oil from spent bleaching clay.

In order to provide the activated clay which I preferably employ, the clay in the undried solid gel state in which it comes from the ground is first subdivided to form granules approximately the size of small pebbles. The subdivided clay is then introduced into a tank provided with a mixing device and is covered with an acid solution which is thereafter heated to a boiling temperature under ordinary pressure. The boiling is continued until laboratory test shows that the clay has been sufficiently activated. This ordinarily requires from 6 to 10 hours. In treating the clay with an acid solution, practically any acid may be employed, either organic or inorganic, hydrochloric acid, sulfuric acid, and oxalic acid being but three of the many acids which can be employed. I have found that all of the acids are of about equal value and, therefore, it is preferable to use the cheapest, i. e. sulfuric acid. The acid solution which is employed is preferably of about 60% strength although it will be obvious that the strength of acid is subject to great variation. The amount of the acid solution employed is also subject to great variation but it is recommended that sufficient acid solution be used to completely cover the subdivided particles of clay in the treating container.

After the boiling treatment has been carried out for the required period of time, the excess of acid solution is drawn off and water is sprayed on to the mass and permitted to trickle through. This spraying treatment thoroughly washes out all water soluble electrolytes present in the clay.

It is of course recognized that activation of bleaching clay by acid treatment is an old procedure. However, in the processes heretofore employed it has been customary to treat dried crude bleaching clay with water to reduce the clay to a pulp or mudded condition, after which the clay is treated with an acid. It has been found that it has been substantially impossible satisfactorily to remove water soluble electrolytes from the clay thus treated and in no instance has it been possible to thoroughly wash such clays by spraying. As pointed out above, I have discovered that if the clay is treated in the undried solid gel state in which it occurs in the ground instead of in the pulp or mud form in which it is usually treated, the water soluble electrolytes may be conveniently and easily removed by merely spraying water over the particles of clay in a container. Tests have demonstrated that bleaching clay activated in the manner described above is substantially free from water soluble electrolytes, the amount of such electrolytes being reduced below about one-hundredth of one per cent. These clay particles after washing are suitable not only for drying and grinding to produce a commercial bleaching clay but, without drying or grinding, are suitable for other purposes, such as in water purification systems.

In preferred practice, the clay particles activated in the manner described above are dried and ground in conventional manner. The dried clay is then ready for use in the bleaching of animal or vegetable oils and fats.

Referring to the accompanying drawing, the necessary amount of bleaching clay to treat the

amount of oil to be bleached is weighed into the mixing tank 14. The amount of clay employed is determined in conventional manner by laboratory test and, in general, ranges between approximately 0.5% and 1.25% on the weight of the oil or fat to be bleached. From the storage tank 10, sufficient of the oil or fat therein is delivered through the pipe 11 under the control of the valve 13 into the mixing tank 14. This oil or fat should be at as low a temperature as the solidifying point of the oil or fat will allow and still have the material thoroughly liquid. This temperature is, roughly, between 60° and 120° F. The amount of material introduced into the mixing tank 14 from the storage tank 10 is subject to considerable variation but I have found it advantageous to mix with the bleaching clay about nine times its weight of oil or fat from the tank 10. The agitator 15 is then started so as to mix the clay into a smooth dispersion with the oil or fat. As will be apparent, instead of using but one mixing tank 14, several such tanks may be employed.

In preferred practice, I also introduce into the mixing tank 14 certain treating chemicals to be incorporated with the dispersion therein. However, these chemicals may be added separately or together later in the process by introducing them directly into the system or by the use of separate mixing tanks. However, in order to avoid unnecessary handling of materials, it is preferable to mix the treating chemicals with the dispersion in the tank 14.

One of the treating chemicals which I recommend be incorporated with the material to be bleached is an inhibitor of enzyme and bacterial action. I have discovered that water soluble halogen compounds are particularly suitable for this purpose, such as ammonium and alkali metal bromides, fluorides, iodides and chlorides and their complex ions. In preferred practice I employ sodium fluoride. Of the soluble halides, sodium chloride is the least effective inhibitor but even it can be used with some measure of success. The amounts of the soluble halides employed will vary greatly as will be apparent to those skilled in the art. The amount required is determined by subjecting the oil or fat to be treated to the conventional "stability test". Generally, the amount used will range from one-fiftieth of one per cent. to one-tenth of one per cent. on the weight of the material to be bleached.

I have also found it advantageous to incorporate an anti-oxidant with the dispersion in the mixing tank 14, preferably one which does not tend to discolor animal and vegetable fats and oils. Various suitable anti-oxidants are known, such as the various derivatives of pyrogallol and other strongly oxygen-absorbing organic chemicals, such as diphenols. The anti-oxidant is generally employed only in traces, say one-fiftieth of one per cent. on the weight of the material being bleached.

If desired, carbon black may also be incorporated in the dispersion prepared in the mixing tank 14. As is well known, carbon black is frequently employed in the bleaching of animal or vegetable fats and oils for the purpose of delaying rancidity. However, I have found that it is not necessary to employ carbon black in the present process and since its use may prove troublesome, I prefer not to employ this material. However, it can be employed if desired and, if used, it is introduced in the approximate amount of four pounds of carbon black to one hundred thousand pounds of the material to be bleached.

As set forth above, the treating chemicals need not be added in the mixing tank 14 but may be subsequently introduced into the system. These are preferably introduced in solution or suspension in a portion of the oil or fat being treated.

From the tank 14 the dispersion therein is mixed by any suitable means with a definite volume of the oil or fat to be processed. In preferred practice, I accomplish this by the use of pumps 17 and 20 which convey the dispersion from the tank 14 and the oil or fat to be processed from the tank 10 to the union 19 where the mixing takes place. The pumps are set so as to mix the correct volumes of the oil or fat from the storage tank 10, which ordinarily has been subjected to the usual preliminary refining treatment, with the dispersion from the tank 14. For example, if preliminary tests indicated that 1% of the bleaching clay on the weight of the oil or fat should be employed, and a 10% clay dispersion had been prepared in the tank 14, approximately nine parts of the oil or fat to be bleached would be mixed with one part of the 10% dispersion.

After the mixing of the unbleached material and the clay dispersion, the mixture is passed through a heating device and the hot mixture is then sent through a closed container, equipped with a stirring device, of such dimensions that the oil or fat leaving it has been bleached and purified to the desired degree. Ordinarily, the apparatus should be designed so that, where an activated quick-bleach clay is employed, a period of about eight to fifteen minutes will be required for the passage of the material from its point of mixture, say at the union 19, to its point of discharge from the bleaching chamber. The bleaching temperature to be employed will, of course, vary considerably according to the material under treatment, the purpose for which the bleached material is to be employed, and other factors well known in the art. As a general rule, the bleaching temperature employed varies roughly from 145° to 300° F. Since the present process safely allows the use of high bleaching temperatures without injury to the oil, I prefer to heat the oil to a high bleaching temperature, since the higher the temperature, the better the bleach.

In the preferred practice of my process, the mixture of material to be bleached and the clay dispersion is passed from its point of mixture at the union 19, through the pipe 22 into the coil 24 arranged in the insulated heat interchanger 23 where there is imparted to the unbleached material a portion of the heat from bleached material passing in a countercurrent direction through the juxtaposed coil 33. The material is passed from the coil 24 through the heating unit 26 where it is heated up to an elevated temperature, say up to 300° F. Thereafter the material is passed through the bleaching chamber 31 where it is thoroughly stirred by the screw conveyor therein. From the chamber 31 the bleached material and spent clay dispersion is passed through the coil 33 in the heat interchanger 23 and out of the heat interchanger through the pipe 34. In the preferred practice of the process, the material passing through the pipe 34 has been cooled to a temperature in the neighborhood of 180° F.

The material at this point is then conducted to a suitable filter to remove the bleached oil or fat from the associated spent bleaching clay. In preferred practice, I employ a series of filters of sufficient number so that there will be at least one filter available while the spent clay in the other filters is being removed therefrom or being treated

to reclaim residual oil or fat therefrom. In the accompanying drawing I have shown two filters 37 to which material from the pipe 34 may be directed through the pipes 36. As shown, each of the pipes is provided with a valve in order that the material from the pipe 34 may be directed to the proper filter.

I may use any suitable conventional filter such as a leaf filter press, a so-called self-emptying filter press, a centrifugal filter or the like. The bleached oil or fat passing through the filter is then subjected to such additional refining treatment as may be desired, such as a conventional deodorizing treatment, after which it is introduced into a storage tank.

When the bleached oil or fat is to be left in storage tanks for any substantial period, it is preferable to store the material in contact with a non-oxidizing gas such as methane, which may be derived from natural gas, nitrogen, carbon dioxide or the like. When a fat or oil is to have a gas beaten into it to provide an emulsion of the fat or oil with such gas, as is the practice with much of the compound shortening now manufactured, a non-oxidizing gas such as methane, nitrogen, carbon dioxide or the like should be used in place of the air now commonly used. An oil or fat thus treated will be found to be more stable than a similar oil or fat which has had air beaten into it.

As stated above, in the ordinary process of bleaching animal or vegetable fats and oils there is ordinarily lost about 18 to 45% of the oil on the weight of the clay due to its retention by the spent bleaching clay and where a portion of this oil has been recovered, the quality of the recovered oil was so poor that it could not be marketed as high grade oil. In the present process, I have provided a method of reducing the oil loss to about 7% in one embodiment and to about 15% in another embodiment of the process.

Where the oil is filtered with conventional leaf filter presses, the first unit employed is closed after it has been used for sufficient time to render its further use impractical and the material under treatment is then directed to another similar press which is ready for use. The spent clay in the first unit is then washed with a cold or luke-warm soap solution. Any convenient method of applying the soap to the spent clay may be employed. In preferred practice, there is sent through the first filter press unit containing spent clay and bleached oil in its press leaves an oil dispersion of a water soluble soap. The soap is preferably suspended in a portion of the refined material being treated. For example, if cottonseed oil is being bleached, the soap is suspended in refined cottonseed oil and this dispersion is forced through the press under treatment. This is continued until sufficient soap has been deposited in the spent clay to permit it to be washed thoroughly. The amount of soap to be deposited will vary according to the particular conditions in a particular plant but, in general, the soap would approximate between one-tenth of one per cent. and one per cent. on the weight of the spent clay to be processed.

When a sufficient amount of soap has been deposited the supply of the oil dispersion of soap is cut off and cold or lukewarm water is then sent through the spent clay in the press. If too hot water be sent through the spent clay it will not hydrate the clay sufficiently to reduce the surface attraction of the clay for the oil.

The water passing through the spent clay washes the liberated oil through the filter cloths and the resulting mixture of oil and water is then introduced into a container where it is permitted to stand so that the oil may be separated from the surface of the water.

The treatment of the spent clay described ordinarily sets up channels through the spent clay structure so that the water does not come into contact with the entire mass of the spent clay. When the amount of oil being carried off by the water is reduced substantially, it is desirable to turn off the supply of water and thereafter force an elastic non-oxidizing gas, such as steam, through the filter leaves. By the elastic pressure of the gas, the water is forced into new channels. Thereafter, water is again introduced into the press and this may again be followed by a second treatment with an elastic gas such as steam and a third treatment with water.

As a result of this treatment the oil lost in the spent clay may be reduced to approximately 15% of the dry spent clay.

There are several other advantages attending this method of treating spent clay in leaf filter presses. The bleached oil is removed from the filter press substantially completely without blowing the press with air as is now the usual method. This use of air injures the oil removed by it, as well as the oil still remaining with the spent clay in the press.

After the treatment described, the press cloths are washed with soapy water following each use of the filter press. The amount of oil left on the fibers of the press cloths is definitely lessened. The press cloths are thus cleaner, they filter more freely, and last longer.

Where in the above referred to treatment cold or lukewarm water is used, the alkaline soap solution reduces the free fatty acids in the recovered oil. The soap also dissolves some of the color from the recovered oil. What color is developed is very readily bleached out so that the recovered oil without loss of quality in the finished product can be added to the next batch of oil to be bleached. This is not true of the oil recovered by the air-steam blow method.

The above described treatment of the spent clay with soap is successful only with bleaching clay whose content of soluble lime and magnesia, and other injurious metallic salts, is low enough not to form insoluble metallic soaps on the surface of the clay particles and so prevent the passage of liquid through the spent clay. Therefore, as pointed out above, when filtering is done with a leaf filter press and the raw bleaching clay contains a substantial amount of these injurious compounds, the clay should be activated in the manner described above in order that the clay may be substantially freed from water soluble electrolytes.

Instead of using a leaf filter press for separating the bleached oil or fat from the spent bleaching clay, I may use any convenient known type of self-emptying filter press or any convenient known type of centrifugal filter. If a self-emptying press is employed, metallic filter cloths can be used. If a centrifugal filter is employed I prefer to use a type which can be rapidly freed of the solid phase and also a type in which an inert gas atmosphere can be maintained in contact with the filtering oil or fat.

Where a self-emptying press or a centrifugal filter is used, it is preferable to treat the oily spent clay apart from the filter rather than in the filter as where a leaf filter press is employed.

Accordingly before treating the spent clay to recover therefrom the oil or fat retained thereby, the spent clay should first be removed from either the self-emptying filter or the centrifugal filter.

The spent clay which has been removed from the filter is preferably run into a tank which preferably is provided with a stirring or mixing device, such as a paddle. An aqueous solution of a chemical which reduces the surface adhesion of the clay towards the oil or fat is added and mixed in thoroughly, preferably until a relatively smooth mix has been obtained. Water is then run into the tank in an amount sufficient to cover to a substantial depth the clay after it has settled. When the water has thus been run in, the oil or fat which has been liberated by the treating chemical rises to the top while the clay settles to the bottom as a thin mud. The oil is then drawn off from the surface of the water and the clay mud run into the sewer. Since the spent clay remains in a light condition which will not form heavy gummy deposits it can be safety emptied into a sewer. By the above treatment the oil or fat present in the clay is reduced to about 7% on the weight of the dry spent clay.

I have found that there are three different classes of reagents which may be employed to reduce the surface adhesion of the spent clay toward the oil or fat in the treatment above described. For example, soluble fluorides may be satisfactorily employed, and I prefer to use an aqueous solution of sodium fluoride for mixing with the spent clay to be processed. The amount of sodium fluoride to be employed in preparing the aqueous solution will, of course, vary substantially but the exact amount can readily be determined by previous laboratory trial. Ordinarily sodium fluoride in the amount of from ¼ of 1% to ½ of 1% on the weight of the clay is sufficient.

A second class of reagents capable of reducing the surface attraction of the spent bleaching clay for certain oils and fats are coagulating polyvalent kations, specifically, soluble salts of iron, manganese, chromium and aluminum. When one of these metallic salts is employed it may be present, for example, to the extent of from 1 to 10% on the weight of the spent clay being treated.

A third class of reagents capable of reducing the surface attraction of the spent clay for certain oils and fats are coagulating polyvalent anions, specifically, soluble salts of amphoteric metal acids, such as sodium aluminate, sodium stannate, sodium tungstate and the like. When one of these salts is employed it may be present, for example, to the extent of say ½ of 1% to 5% on the weight of the spent clay being treated.

After the oil or fat which has been separated from the spent clay has been recovered, it is ordinarily advisable to add the recovered material to another batch of oil or fat to be bleached, although in some instances this will not be found desirable since the material without further treatment may be put to other uses than the main body of oil or fat which has been bleached.

As will be apparent from the foregoing, my process possesses numerous advantages over the processes heretofore employed for the bleaching of animal and vegetable oils and fats. In the first place, the process is a continuous one which is obviously a very important advantage over the prior art processes customarily employed. Moreover, the process maintains the oil or fat out of contact with any great quantity of air or oxidizing gas throughout the process and yet avoids the necessity of creating or maintaining a vacuum in the system. Where a quick bleaching clay is employed, as in the preferred practice of the process, a small apparatus can be employed. Such a small apparatus may, without running into prohibitive expense, be made of metals or alloys more suitable than the iron or brass now in common use in bleaching kettles and filter processes. Another important advantage of the process is that there is no necessity to blow the filter medium with air to decrease the amount of oil entrained thereby. The oil or fat recovered in the process is of lower acid content and in better condition than the oil recovered by the present air and steam treatment. The oil lost in the spent clay is substantially smaller in amount than that lost after air-steam blowing. There is no solvent lost nor solvent to be removed from the recovered oil or fat as in the organic solvent methods of recovery from spent clay. Where leaf presses are used for filtering, the press cloths are left cleaner, filter more rapidly and last longer than in the case of prior processes using leaf presses. Self-emptying presses can be used for filtering in the process, if desired. The oily spent clay from self-emptying presses can be easily treated to give low oil retention. The spent clay after treatment to remove entrained oil or fat is a light pulp which can be run into a sewer without difficulties resulting from gummy muds settling out. Centrifugals can be used and the spent clay obtained by their use successfully treated to give low oil retention of the spent clay discharged at the end of the process. The retention of bleaching clay in the undried solid gel state instead of in a pulp or mudded state allows of thorough washing of chemically treated clays. Heat losses can be minimized through heat interchangers between bleached and unbleached oils. The optimum bleaching temperature can be maintained in the bleaching chamber while maintaining the oil or fat out of contact with the air without the necessity of creating or maintaining a vacuum in the bleaching chamber. If self-emptying presses or inert gas types of centrifugals are employed, the oil can be filtered at temperatures most convenient for rapid filtration and the oil can yet be kept out of contact with the air. The use of an inhibitor of enzyme and bacterial action not only inhibits the lipase-splitting of vegetable and animal fats and oils but these inhibitors also serve as disinfecting agents against moulds, yeast and bacteria. Since the water soluble halides employed as inhibitors are insoluble in oils and fats, they do not affect the oils or fats themselves. When the dispersed crystals come in contact with wet colloidal seed pulp dispersed in oil, the lipase in it is rendered ineffective and the pulp material itself is no longer food for moulds, yeast and bacteria. Oils and fats stored in contact with an inert gas or in which an inert gas has been dispersed possess increased protection against oxidation. Finally, it has been demonstrated that the animal and vegetable oils and fats processed in accordance with the present invention are stable against rancidity for a substantially longer period than the oils and fats treated in accordance with the usual prior practice.

The term "bleaching clay" as employed herein is not intended to be restrictive to any one class of clays, but is to be understood as including all argillaceous material, such as bentonite, semi-

bentonite, fuller's earth or the like which either with or without prior treatment with chemicals is adapted to remove color from vegetable and animal oils and fats.

In the practice of the above described process, the water which is employed in washing the activated bleaching clay after it has been subjected to an activating agent should be low in concentration of injurious ions. For example, a water approaching zero hardness should be employed.

Obviously, materials other than acids may be used in activating bleaching clays and it is an important feature of my invention that where the bleaching clay is activated either with an acid or other activating agents, the water soluble materials remaining in the clay can be readily washed out if the clay is treated in the form of clay granules in solid gel state. While I have referred above to the washing of such clay granules by spraying water thereon and permitting the water to trickle through the mass, it will be apparent that washing may be accomplished by any other suitable means which involves the relative movement of the washing water with respect to the clay granules.

As pointed out above, I prefer to employ in the practice of the present process an inhibitor of enzyme and bacterial action which comprises a soluble halide. While, as stated, this material is preferably added to the oil dispersion of clay in the mixing tank 14, and passes through the system in the presence of the clay, to be substantially filtered out when the bleached material is passed through the filter, I have discovered that it is also advantageous in treating edible fats and oils which are not bleached or which may not be bleached by a process of the character above described to add to such edible fat or oils a minor proportion of a water soluble but oil insoluble halide and, thereafter, to substantially remove the crystals of the halide as by filtering. While the filtering will remove the major proportion of the crystals of the halide, there will still be left traces of the halide which will be sufficient to exhibit enzyme and bacterial action without injuring the digestibility of the treated oil or fat.

As is well known, in expressing edible oils from nuts or seeds, the nuts or seeds are cut into small pieces and subjected to great pressure whereby the oil is pressed out. This oil always contains a considerable proportion of pectinous material from the cells of the nuts and seeds. While a substantial proportion of this pectinous material can be removed by filtration and other treatment, a portion of this organic material which is present in colloidal condition cannot be removed. It is very finely dispersed in the body of the oil so that it will pass through an ordinary filter. It is this filterable dispersion of colloidal organic material which is considered to be responsible for the spoiling of the oil through rancidity. By adding a water-soluble, oil-insoluble inhibitor of enzyme and bacterial action, as described above, the dispersion of pectinous material present in the body of the oil will take up some of the inhibitor so as to prevent enzyme and bacterial action. As pointed out above, the inhibitor preferably consists of a water-soluble, oil-insoluble halide, preferably a water-soluble, oil-insoluble fluorine compound, and this material is preferably added in the form of crystals of small particle size. When the treated oil is filtered, the crystals of the fluorine compound will be removed from the oil itself but a small portion of the inhibitor will be dispersed in the filterable pectinous material which is dispersed through the oil. Of course, the particles of the fluorine compound having been filtered out of the oil itself and the amount of fluorine compound dispersed in the pectinous material being small, the final product will not be injured with respect to taste or digestibility, although stabilized against enzyme and bacterial action.

Where reference is made herein to activating bleaching clay it is to be understood that the term "activating" is used in its common accepted technical sense, i. e. as applying to the subjecting of the bleaching clay to the action of an acid solution to remove some but not all of the basic constituents of the silicate complex, whereby the resulting product remains as a hydrated silicate of alumina and not a silicic acid.

The term "bleaching clay" as employed herein is used in its commonly accepted technical sense, i. e. as applying to an argillaceous hydrated silicate of alumina possessing marked adsorptive powers and having the characteristic of "readily sliming", by which is meant that when dried and placed in water or a conventional activating acid solution it will readily disintegrate into a slime or mud or pulp. The term does not include, and is not intended to cover herein, shale-like substances containing notable proportions of calcium and magnesium carbonate, such as certain fuller's earths which are essentially rock-like. Moreover, the term does not include, and is not intended to cover herein, kaolins which do not possess marked adsorptive powers either before or after treatment with an acid activating agent, or stable silicate aggregates, such as the glauconites or green sands, which are essentially rock sands.

While I have described in detail the preferred practice of my process and several modifications thereof, it is to be understood that the details of procedure, the arrangement of steps, the proportions of ingredients and the like, may be variously modified without departing from the spirit of the invention or the scope of the subjoined claims.

I claim:

1. In a process of activating readily sliming bleaching clay wherein the clay is subjected to the action of an acid solution and the acid-activated clay is thereafter washed, the improvement which comprises treating with the acid solution bleaching clay which is in its original gel-like form and which contains sufficient of its original water content to prevent substantial disintegration of the clay into a pulp or mud during the treatment with the acid solution.

2. In a process of activating readily sliming bleaching clay wherein the clay is subjected to the action of an acid solution to activate the same, the improvement which comprises treating with the acid solution bleaching clay which is in its original gel-like form and which has retained up to the point of such treatment with acid solution sufficient of its original water content to prevent substantial disintegration of the clay into a pulp or mud during such treatment with acid solution, and washing the substantially non-disintegrated clay substantially completely free from water-soluble electrolytes.

3. In a process of activating readily sliming bleaching clay wherein the clay is treated with an acid solution to activate the same and the acid-activated clay is thereafter washed, the improvement for preventing substantial disintegration of the clay into a pulp or mud upon treat-

ment with acid solution which comprises subjecting to the acid treatment bleaching clay which is in solid gel-like state and which has substantially the same water content as said clay had when in the ground.

4. In a process of activating readily sliming bleaching clay wherein the clay in the form of subdivided lumps is treated with an acid solution to activate said clay, the improvement for preventing substantial formation of a pulp or mud and for retaining the clay in said lump form upon said acid treatment which comprises subjecting to the acid treatment subdivided lumps of bleaching clay which is in solid gel-like state and which has substantially the same water content as said clay had when in the ground, and washing the resulting lumps of activated clay substantially completely free from water-soluble electrolytes.

5. A product of the character described comprising lumps of acid-activated clay in solid gel-like state, the form of said lumps being substantially the same as the form imparted to the original clay prior to acid-activation thereof, said original clay being of readily sliming type, the lump form of said acid-activated clay permitting rapid washing thereof substantially completely to remove water-soluble electrolytes therefrom.

6. A product of the character described comprising an adsorbent, acid-activated clay in the form of lumps corresponding substantially to the form of the original clay when subjected to acid-activation, said original clay being of readily sliming type, the clay in said lumps being in undried solid gel-like state, said product being substantially free from water-soluble electrolytes.

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