country than in others. We have been able to get some very good checks by taking fifty-gram samples and first making a moisture determination, then fuming with hydrochloric acid at 130 for one hour, putting them in the ten-mesh sieve and rubbing them with a large rubber stopper. Then make a final polish by putting the seed on a piece of cloth. Then we have determined the moisture content of the lint that is taken off a number of samples and got an average. Using that average, and our original moisture, we have been able to calculate the percentage of lint. We have made some very good checks. We believe following so closely the method used in the analysis of seed will make a better series of determinations than using the acid, which has its dangers and is somewhat more expensive.

SOME NOTES ON THE THEORY AND PRACTICE OF BLEACHING FATTY OILS*

By HENRY ODEEN and H. D. SLOSSON
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THE purification of oils by the removal of many impurities occurring as finely divided suspensions, colloidal bodies and soluble matter has brought into wide use such materials as carbon, clay and silicia in a variety of form and quality. The displacement of colloids and solutes from the oil to the surface of the solid adsorbent particle, to be held there tenaciously, is termed adsorption.

ADSORPTION THEORIES

The removal of such colloidal and soluble impurities from oil as, for example, mucilaginous and coloring matter, is, in the practice of the art, a relatively simple operation, but underlying this simple performance, science has found physico-chemical law and theory to explain the behavior and mathematically to measure the interplay of surface energy at work. One of our earliest references to the scientific study of adsorption is Scheele's research in 1777 on the adsorption of gas on charcoal. Willard Gibbs, eminent American scientist of his time, promulgated the adsorption theorem in 1876 and later H. Freundlich in 1909 published his work on colloidal chemistry giving mathematical expression for the measurement of the amount of adsorption at constant temperature.

For our purpose the theory may be stated that: solutes which lower the surface tension of the solvent will be concentrated at the liquid-solid interface. The theory may be demonstrated as follows:

(1) Assume a pure, colorless oil. Within the body of this oil the molecules have neighbors above, below and on all sides to attract each other. But the molecules in the top or surface layer cannot have this molecular pull upward; they do have an extra attraction downward into the mass of the oil molecules. This inward pull makes the surface act like a stretched membrane or elastic skin, contracting when it can. There is potential energy or ability to do work resting in this surface tension which is trying to expend itself.

(2) Assume the same oil with coloring matter present. The color bodies are larger in size than the oil molecules and while not as numerous, yet they do disturb the lines of attraction which previously held the oil molecules so closely together. If we measure the surface tension now we will find it less than it is in the case with pure oil as in (1) above. We have now come to the meaning of "solutes which decrease the surface tension" for the color bodies are the solutes (dissolved substances).

(3) Assume that adsorbent material has been added to oil containing coloring matter. For conveience let us take only a small portion of this oil wherein we have many oil molecules, a few color bodies and one adsorbent particle.

The main thing to note is that there is another kind of surface aside from the top surface of the oil. Whenever the oil is displaced to make room for the adsorbent particle, including any penetration into its porous structure, there we have other free surface which, in the aggregate, is very large. This surface is called "liquid-solid interface." Probably the first thing which happens on the addition of the adsorbent particle is a momentary change in the surface tension of the surrounding oil—there is a lowering at the particle surface, accompanied by an equivalent increase of intermolecular forces in the adjacent layers. Adjustment of these forces to equilibrium causes a counter-flow back to the adsorbent surface.

(4) Apply heat and agitation to the oil. The color bodies are now presumed to be concentrated at the liquid-solid interface. The agitation has brought the color bodies into intimate contact with the adsorbent particles permitting their concentration at this point.

These interfering color bodies have now been removed so that they can no longer break the intermolecular force of cohesion; accordingly the surface tension again increases. Again the oil molecules, relieved of this interference to some extent, exert their natural, mutual cohesion to each other.

Freundlich's absorption isotherm concerns changes which take place when the amount of adsorbent is progressively increased but the temperature is held constant. The essential formula is:

$$X/M = K\ C^n$$

Where X is amount of solute adsorbed
M is amount of adsorbent used
C is concentration of unadsorbed solute
K and n are constants

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*Presented at the Twenty-sixth Annual Meeting of the American Oil Chemists' Society at Memphis, Tennessee, May 23-24, 1935.

The adsorption curve is a parabola and as such becomes a straight line when plotted on full logarithmic paper.

Gurwitsch[1] disagrees with the Gibbs-Freundlich theory and has his own chemical-mechanical theory. He believes that the molecules of the solvent and the molecules of the dissolved substances both strive to obtain the greatest concentration at the surface of the adsorbent. Therefore, if the attractive power between the solvent molecule and the adsorbent is less than that between the molecules of the dissolved substances and the adsorbent, the dissolved substance will be largely concentrated on the surface of the adsorbent.

Haseman[2] states that free di-silicic acid, through available hydrogen from the OH ions, is the active ingredient in decolorization.

Then, there is the negative and positive charge theory which attempts to explain the action in terms of electrical units. As generally stated, it presupposes that the color body in the oil, for instance, is charged negatively whereas the adsorbent has a positive charge and that the greater this differential between the two, the more certain will the neutralization of charge take place with adsorption occurring simultaneously.

Dr. P. G. Nutting[3] of the Geological Survey, Washington, has made a thorough study of the bleaching clays. He suggests that the activation of a clay consists in the removal of loosely held ions at the surface, leaving open bonds or free valances exposed for the selective adsorption of coloring matter from the liquid or oil in question.

DECOLORIZING CLAYS

Fuller's earth has been used by man since ancient times, particularly for the cleansing of wool. It is mentioned in the Bible and we find that the Chinese were familiar with it at an early date. In the purification of oils its first uses were, perhaps, concerned principally with the clarification of vegetable oils; later development provided an additional purpose—that of color removal. The important clay mineral in the adsorptive earths is generally known as montmorillonite,[4] which is a hydrous silicate of aluminum with accompanying magnesium, calcium and iron.

Fuller's earth is a commercial designation for a natural clay-like mineral substance which has the property of absorbing oils and fats to a high degree and of removing coloring matter therefrom. Industrial uses employ both a coarse granular product for percolation filtration and a finely ground powder in the contact method.

Activated bleaching clay is a designation for acid-treated clays which have appeared on the market in more recent years. They distinguish from fuller's earth in their superior ability to adsorb colloidal impurities and soluble coloring matter.

The Conversion of Raw Clay to an Activated Adsorbent.

Clay-like substances which respond, more or less completely, to chemical activation are considered, generally, as bentonites which in the raw state may have little or no decolorizing property. The purpose of the chemical treatment is to produce, artificially, a high surface activity on the clay particle and this is accomplished by both a physical and chemical transformation of the raw bentonite. Soluble material is leached out whereby minute pores and capillaries are enlarged and created in the original clay particle, giving it greatly increased surface exposure. The chemical composition is altered also, particularly with reference to the silica-alumina ratio.

Briefly, the conversion of raw bentonites into a highly active adsorbent product is accomplished by intimately contacting the ground raw clay with acid solution under certain optimum conditions which are variable for clays differing in origin and physical characteristics. The products of reaction are thoroughly removed by successive washes with large volumes of water. After dewatering the pulp is dried under carefully controlled conditions followed with final grinding to desired mesh specification, then packaged.

The chemical treatment is in itself a comparatively simple operation, but when it is considered that the finished product performs its work in the purification of oils by physical means, it will be obvious that much stress must be given to those processing conditions which influence the physical characteristics of the finished product. Careful routine control by chemical and physical tests at every stage of manufacture is indispensable for the maintenance of uniform quality.

THE EFFECT OF VARIOUS CONDITIONS IN THE BLEACHING OPERATION

Time and Temperature.

Efficient operation requires intimate contact of the adsorbent particle with as much of the oil as will saturate the adsorbent to equilibrium with the impurities to be removed. Therefore, thorough agitation is provided; viscosity of the oil is reduced by application of heat; and stirring is continued for an adequate minimum of time. Each of these factors, however, should be considered with due regard for the destructive effect of atmospheric oxygen.

When refined fatty oil is bleached in open kettle, 15 to 20 minutes of contacting at the maximum temperature will ordinarily be sufficient, provided moisture has been completely removed in the meantime. The optimum temperature will, as a rule, range between 220° and 240° F. Aside from the question of moisture removal, the temperature will vary somewhat according to the optimum for a given adsorbent. For example, some clays will exert their maximum effects slightly below 200° F. where others will function best 20 or 30 degrees higher. In the case of a plant bleaching experiment on two different batches of refined, washed and vacuum dried corn oil, a given adsorbent clay yielded a color of 35 Y-4.5 R at 170° F. and 35 Y-4.1 R at 220°.

As an illustration of the time-temperature relation in the bleaching of a refined sardine oil with 3 per cent of activated clay the following data are given:

TIME-TEMPERATURE RELATION BLEACHING REFINED SARDINE OIL WITH 3% ACTIVATED CLAY

5 minute contact at 220° F.	Color 15 Y — 1.2 R
30 minute contact at 220° F.	Color 20 Y — 1.4 R
60 minute contact at 220° F.	Color 23 Y — 1.7 R
60 minute contact at Room temp.	Color 35 Y — 1.5 R
15 minute contact at 186° F.	Color 20 Y — 1.3 R
30 minute contact at 186° F.	Color 20 Y — 1.4 R
60 minute contact at 186° F.	Color 25 Y — 1.5 R
120 minute contact at 186° F.	Color 25 Y — 2.0 R

In the above tests, the 5 minute contact at 220° was decidedly the best; with longer contacts, apparently, oxidation or polymerization, or both, gave effect to a progressive decrease in decolorization, and less at 186° than at 220°.

Effect of Moisture

A common belief exists that the oil should be thoroughly dried before the adsorbent is to be added, but in the writer's experience this has not been found to hold either in the case of open kettle or vacuum bleaching. As a matter of fact, there appears to be good reason for the introduction of the adsorbent before drying and allowing dehydration and contacting to proceed simultaneously. It would seem to be preferred from a theoretical standpoint that the adsorbent be present during the period of raising the temperatures above, say, 160° F. to its final temperature, to adsorb coloring matter which may have a tendency to "set" at the higher temperatures. And from a practical consideration the time of contact is thus somewhat longer without increasing the time cycle. If, perchance, the operator is unduly rushing his batches through, this will serve as a margin of safety. Where vacuum equipment is employed the adsorbent and oil may be charged to the drier at the beginning of the operation and save the subsequent interruption to "suck in" the bleaching material and with it atmospheric oxygen.

In demonstration of this point, two 48,000-lb. batches of refined and washed corn oil were bleached under vaccum, all conditions being identical, except that in the one case the batch was vacuum dried first and then contacted; in the other case oil and adsorbent were contacted and dried simultaneously:

Contacting the dried oil 35 Y—4.5 R Color.

Drying and contacting simultaneously gave 35 Y—4.4 R Color.

Aside from these considerations it is contended by some investigators that a small percentage of moisture present during adsorption is an actual aid to bleaching, especially when the adsorbent material is very dry.

Effect of Soap.

As a rule, refined oil ready for bleaching will contain small amounts of soapstock in suspension and solution unavoidably carried over from the previous caustic treatment. The presence of soap in quantities within the usual limits of refinery practice does not noticeably diminish the bleaching action of clay but beyond slight traces does give effect to a rise in free fatty acids—more in the case of some fatty oils, for example, coconut than in cottonseed—when contacted with decolorizing clay. And the increase will vary with the amount of soap present. Possibly the explanation lies in the fact that as soap is decomposed under these conditions the liberated sodium ion is adsorbed by the clay and the free fatty acid remains in solution in the oil. Moreover, the action of bleaching earths accelerates decomposition of soap.

A study of the following plant data from different refineries using different bleaching earths will demonstrate the relation of soap to free fatty acids in the case of well refined and settled oil, versus refined oil containing excessive soap:

CASE 1. F.F.A. DETERMINATION BEFORE AND AFTER BLEACHING ON A WELL REFINED AND SETTLED OIL

	F.F.A. Before Contacting	F.F.A. After Contacting
Cottonseed oil	.10%	.10%
Cottonseed oil	.10%	.09%
Cottonseed oil	.14%	.12%
Cottonseed oil	.03%	.02%
Cottonseed oil	.03%	.03%
Cottonseed oil	.02%	.02%
Coconut oil	.04%	.05%
Coconut oil	.04%	.06%
Coconut oil	.04%	.07%

Case 2. Refined cotton oil having excessive soap in suspension, some of which was skimmed off in the bleacher before adding the adsorbent material.

CASE 2. F.F.A. DETERMINATION BEFORE AND AFTER BLEACHING ON A REFINED OIL CONTAINING EXCESSIVE SOAP

	F.F.A. Before Contacting	F.F.A. After Contacting
Cottonseed oil	.025%	.045%
Cottonseed oil	.03%	.08%
Cottonseed oil	.02%	.045%
Cottonseed oil	.04%	.07%

If a soapy batch is heated to around 220° F. with a minimum of stirring during the application of heat, and then allowed to stand quiescent for an hour or two, much of the soap will form as a compact surface layer. Skimming off this soap before contacting with the adsorbent will materially check the rise in F. F. A. For example, two batches of very soapy oil were drawn into two bleachers and given the same bleaching treatment except that in the one, the surface soap layer was removed before adding the decolorizing material and in the other it was not removed:

F.F.A. DETERMINATIONS BEFORE AND AFTER BLEACHING OIL WITH EXCESSIVE SOAP PRESENT AND WITH SOME OF IT REMOVED

	F.F.A. Before Contacting	F.F.A. After Contacting
With some soap removed	.03%	.05%
With soap left in....	.03%	.08%

The free fatty acids in normally *well* refined cotton oil will be reduced slightly by the action of most good bleaching earths; the reduction being of the order of .01 to .02 per cent, as shown by the following plant bleaches:

REDUCTION OF F.F.A. IN BLEACHING A WELL REFINED COTTONSEED OIL WITH GOOD ADSORBENT CLAYS

	F.F.A. Before Contacting	F.F.A. After Contacting
Cottonseed oil	.10%	.09%
Cottonseed oil	.08%	.06%
Cottonseed oil	.03%	.02%
Cottonseed oil	.10%	.08%

The case is not one of chemical neutralization as will be evident in the following instance where the one clay is acid to phenolphthalein by .007 per cent and the other neutral, as gauged by their water extracts. The comparison in each case was made under identical operating conditions on the same oil. Two cases are given, each for a different refinery:

COMPARISON OF THE REDUCTION OF F.F.A. IN WELL REFINED COTTONSEED OIL BETWEEN TWO DIFFERENT TYPES OF ADSORBENT CLAY—ONE CONTAINING A TRACE OF ACIDITY AND THE OTHER NEUTRAL

	F.F.A. Before Contacting	F.F.A. After Contacting
Case A:		
Slightly acid clay..	.10%	.08%
Neutral clay	.10%	.09%
Case B:		
Slightly acid clay..	.05%	.03%
Neutral clay	.05%	.03%

The reduction may perhaps be explained by the preferential adsorption of free fatty acid by the clay.

A bleached oil is not properly finished merely because it shows the desired color; it must be entirely free of soap lest its contamination persist in the finished deodorized product. Moreover, maximum decolorization is dependent upon soap-free condition, for most of the original color in the crude oil is dissolved in the soapstock formed during the caustic refining. Appreciable amounts of soap present during the bleaching step demand excessive heating time to insure their complete removal and this slows down operations with attendant increase of f.f.a. and color reversion.

Applying the Decolorizing Materials.

The decolorizing materials should be added to the oil in a single dose. When applied in increments less decolorization takes place; time is wasted; and the oil is unnecessarily exposed to oxidation while at a comparatively high temperature. The writers have not investigated the effect of vacuum in this connection. The effect on decolorization was studied in a series of laboratory bleaches on refined cotton oil, taking a 2,000 gram charge of oil and contacting at 220° F. for 5 minutes with one-half of one per cent of activated clay, then withdrawing a 200 gram sample of the charge with a pipette, filtering and reading its color. The main charge was then given another increment of clay and contacted and sampled for a color reading as before. This procedure was continued until 5 successive increments had been applied in a total dosage of 2 per cent. For comparison with single dose application, a second series of bleaches was made with 5 minute contacts at 220° F., adding the clay charge in a single application in corresponding amounts. The results were as follows:

BLEACHING REFINED COTTONSEED OIL BY APPLYING THE ADSORBENT IN TWO DIFFERENT WAYS

Series No. 1: (Applying the Dose by Increments)

Grams Oil	Grams Clay	Percent Clay (Cumulative)	Lovibond Color
2000	10	.5	35 Y — 5.63 R
1800	9	1.0	35 Y — 4.31 R
1600	8	1.5	35 Y — 3.75 R
1400	3.5	1.75	35 Y — 3.69 R
1200	3	2.0	35 Y — 3.5 R
Series No. 2: (Applying in a Single Dose)			
1800	9	.5	35 Y — 5.63 R
1800	18	1.0	35 Y — 4.19 R
1800	27	1.5	35 Y — 3.45 R
1800	31.5	1.75	35 Y — 3.07 R
1800	36	2.0	35 Y — 2.76 R

In the first series it will be noted that little decolorization was gained beyond the third increment. By comparison with the second series, the total dose of 2 per cent actually accomplished approximately only 75 per cent of its capacity; i. e., 2 per cent gave about the same bleach as 1.5 per cent when added all at one time.

In plant operation this behavior is at least of equal order. As a practical hint from these observations it is suggested, for the sake of economy, that if the initial amount of adsorbent material does not produce a sufficiently low color on a particular batch of oil, that it be disposed of by blending with lower colored oil or even rebleached after filtration.

Loss of efficiency when the adsorbent is added by increments, invites theoretical speculation and from the standpoint of adsorption theory, it is offered that successive increments find the remaining portion of coloring matter in the oil more resistant to adsorption than some of the loosely bound color bodies already attached to other adsorbent particles. And these color bodies may act as connecting links between the older, more or less "saturated," adsorbent particles and the newer ones of later increments. The net effect would thus be an agglomeration of adsorbent particles with an attendant reduction of the effective adsorptive surface. If, on the other hand, all of the adsorbent is contacted at the same time, each particle has equal opportunity with its neighbors for an even share of the various types of solutes which differ in their susceptibility to adsorption; each particle, therefore, will bear an equal share of the burden.

If we apply the negative and positive charge theory of adsorption, the explanation may proceed from the hypothesis that the positively charged adsorbent particle has received an excess of negatively charged solute and when new increments of adsorbent come in contact, union takes place through neutralization of charges and a consequent agglomeration results, thereby reducing the exposure of adsorptive surface.

MULTIPLE BLEACHING

The adsorption curve, which is a parabola, predicts that multiple bleaches on the same oil will produce greater decolorization than when the same amount of adsorbent is applied in a single dose. Theoretically this is true, but in practice it does not seem to hold for some fatty oils when they are bleached in contact with air. This may be true because of oxidation and polymerization, either or both, owing to the long exposure of the oil to air and heat which are the subject conditions of treatment. A comparative study was made in the laboratory on some of the same oil and adsorbent clay which were used in Series No. 1 and No. 2 above. The procedure that was followed consisted of adding a total dose of 2 per cent of adsorbent in five separate bleaches with filtering of the oil between each application of the adsorbent; contacting at 220° F. for 5 minutes. The results are shown in the following table:

APPLYING ADSORBENT ON THE SAME CHARGE OF OIL IN MULTIPLE BLEACHES WITH INTERMEDIATE FILTRATION

Grams Oil	Grams Clay	Percent Clay (Cumulative)	Lovibond Color
1600	8	.5	35 Y — 5.63 R
1500	7.5	1.0	35 Y — 4.4 R
1400	7	1.5	35 Y — 3.84 R
1300	3.25	1.75	35 Y — 3.8 R
1200	3.0	2.0	35 Y — 3.8 R

As in Series No. 1 above, no gain in decolorization was made beyond the third operation. Bleaching under vacuo is here suggested for further research to help determine the contributing causes.

However, in the case of a crude perilla oil, air was excluded by a continuous saturation of the oil with carbon dioxide and the temperature was maintained at not over 120° F. during a series of 9 multiple bleaches. A parallel series bleached in contact air at the same temperature gave uniformly better colors. Had nitrogen, or some other inert gas, been used instead of carbon dioxide, the results may have been different. The multiple bleaches in both of these series were somewhat poorer for corresponding amounts of adsorbent used in a single dose. Perilla oil was used in this experiment because it decolorizes readily at room temperature.

A proper evaluation of all the factors involved will undoubtedly reconcile theory with practice in the bleaching of a fatty oil.

THE AMOUNT OF ADSORBENT TO USE

Colloidal impurities and soluble coloring matter are removed by treatment with caustic soda and adsorptive agents and each treatment should contribute a certain relative share, more or less, of the work to be done in order to effect an economically balanced refining treatment. While reduction in color and colloidal impurities is also effected, very markedly, by efficient hydrogenation and deodorization, it may not be economy in the final analysis to depend upon these operations for decolorization beyond certain limits.

If an oil is to be hydrogenated, the optimum dosage of adsorbent in the previous bleaching operation will be such that all traces of soap and catalyzer poisons are removed in order to economize on hydrogen gas, catalyzer and conversion time.

Deodorization under high heat and vacuum causes precipitation of mucilaginous and coloring matter and within certain limits the action is progressive. Undoubtedly there is danger of causing incipient or complete decomposition of delicate fat molecules with a resultant premature rancidity in the finished fat if heat treatment is depended upon for removal of undue amounts of color. Moreover, the accumulation of this precipitated matter necessitates frequent shut-down and caustic cleaning of the plant, which, in turn, requires a subsequent prewash of the system with refined oil before proceeding with operations in order to absorb caustic residue and oxygen unavoidably left in the system. The precipitated matter will be a continual source of contamination to the deodorized oil. In many plants the oil is clarified and brightened by filtering through paper lined presses which, by the way, is an excellent precaution under the usual circumstances. However, the presence of undue amount of precipitated matter causes rapid blinding of the finished oil press with consequent extra labor for redressing and the possibility of autocontamination of the oil itself.

Because of these considerations, it will be obvious that below certain minima of adsorbent dosage, economy is defeated and finished oil quality is likely to be impaired.

The destearinization or winterizing of animal and vegetable oils and fats is materially accelerated when inhibiting impurities are first removed with adequate adsorbent treatment. In addition the stearine precipitates in much better crystalline form, thus making its separation from the liquid oil with greater speed and completeness.

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DISCUSSION

Pres. Hutchins: Thank you, Mr. Odeen. Are there any questions that any of you would like to ask Mr. Odeen? I feel sure he would be glad to answer them.

Mr. Kilgore: I was very much interested in this theoretical discussion on adsorption, but some of the actual results it seemed, disagreed with his very complete discussion of adsorption. I would like to point out that a common fallacy is made by writers on adsorption in trying to apply a formula worked out by the famous Willard Gibbs, as referred to in this paper, to the interface instead of the surface. If you will examine Gibbs' original papers carefully, you will find that the adsorption isotherms are applicable only to surfaces, not to interfaces. That fallacy has persisted down through the literature, until recent work has shown on a very good mathematical basis that the isotherm does not even approximately hold in the case of interface adsorption. This was particularly interesting to me, because the results seem to actually verify the recent work on that. I would suggest that in going over this theoretical work, as has been done so thoroughly, this recent work be taken into account, because it certainly has a heavy bearing on the problem which has just been discussed.

President Hutchins: We appreciate the paper very much, and it will be published along with all the others, so that you can dwell on it and think over the many points more than you can this morning.

REPORT OF THE REFEREE BOARD

For the Annual Meeting, May 1935

AT A JOINT meeting of the Governing Committee and the Referee Board in Chicago on October 10, 1934, the consensus of opinion regarding the future policy of our Society on cooperative samples was as follows:

1. The A.O.C.S. should regularly sponsor a set of cooperative samples of broader scope than the check meal samples of the Smalley Foundation Committee. Cottonseed samples and crude oil samples are believed to be appropriate additions to the cooperative samples regularly sponsored by the Society.
2. Participation in this work should be required of referee chemists and made available optionally to others.
3. In the tabulation of the results, there should be a grading system and a standard set for what is considered satisfactory work.
4. It should not be the policy of the Society to issue certificates generally to those performing the work and meeting the standard prescribed.
5. Referee certificates should still be issued, substantially as in the past, and the results of the cooperative work should be taken into account as determining in part the qualifications of referee chemists. However, all present at the meeting agreed that the results of cooperative work should not be emphasized to the point of detracting from