A New Method of Refining Oils With Non-Saponifying Alkalies—The Clayton Process

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It is the purpose of this paper to present a brief description of some of the phases of the Clayton method of refining oils and fats by the use of non-saponifying alkalies. I shall confine my discussion to an account of a unit (1) in which crude oils are treated with soda ash to produce neutral refined oil rid of impurities.

The object of refining is the removal of the impurities such as fatty acids, gums, mucilages, phosphatides, etc. The most effective and hitherto almost universally used agent for accomplishing this is sodium hydroxide. But this reagent while effective because of its vigorous action on these impurities also attacks neutral oil. An excess of sodium hydroxide solution must be employed in order to obtain a sufficiently complete removal of the impurities, including an adequate part of the color bodies. This excess is responsible for loss of some neutral oil. In addition to the loss of neutral oil through direct decomposition by the sodium hydroxide, there is a loss of neutral oil occasioned by the occlusion, entrainment, and entrapment of neutral oil in the formation of soapstock, and the subsequent removal of soapstock from the refined oil. We thus have two types of refining losses: (a) unavoidable, i.e., the fatty acids and other impurities, (b) avoidable, i.e., loss of neutral oil. A great measure of success has been achieved during the past decade in reducing the avoidable losses with the introduction of the continuous method (2) of refining oils.

There remained the fact that the sodium hydroxide combined with neutral oil to form soaps, which in turn aggravated the problem of oil entrainment.

The obvious thing to try is a neutralizing agent that does not combine with neutral oil. Soda ash is an alkali answering the purpose and is at the same time the cheapest, most common alkali. But the reagent has many drawbacks. Although sodium carbonate has been advocated as a refining agent its use has been severely limited. A recent appraisal in a book (3) on refining oils is the following: "Sodium carbonate is quite interesting; it presents the big advantage of not attacking neutral oil, but its decomposition by fatty acids causes a release of carbon dioxide, which slows down the settling of the soapstock and increases the volume. Emulsions can form easily in carrying out this process and they are then particularly stable."

Thus while sodium carbonate offered many attractive features to the refiner, he was unable to take advantage of them because of the inherent drawbacks such as excessive foaming, incomplete neutralization, "floaters," slow-settling, voluminous soapstock and insufficient attack on the coloring matters.

One of the outstanding features of the new Clayton process is the use of soda ash to remove the impurities from crude oils to produce neutral refined oil while incurring strikingly low refining losses. In fact the refining losses were so low that we were encouraged to look for a more exacting standard of comparison than the cup test.

At this point I should like to digress. A standard is a measure of progress. The more exacting the standard the greater is the progress. In the days of kettle refining the efficiency of the refining was measured in terms of a laboratory kettle test, or the cup test, under carefully defined conditions. The improvements of the continuous process of refining oils were convincing to the refiner when its performance was compared with the kettle, or cup. With the development of the new method of refining with non-saponifying alkalies, the avoidable losses were reduced to such an extent that a new yardstick, or standard, was sought by which to evaluate the efficiency of the process. Inasmuch as the avoidable losses that plague processes employing caustic soda in excess due to saponification were eliminated and the separation of soapstock from oil was so sharp, a more rigorous standard to gauge the efficiency of the process was sought. This was found in the so-called absolute or Wesson Loss. The method, first devised by Wesson and later thoroughly investigated by Jamieson, yields reliable results when the conditions that Jamieson described for carrying out this determination are rigidly followed (4). A weighed quantity of oil, usually about 10 grams, is dissolved in 50 c.c. of petroleum ether. The solution is then shaken for three minutes with 10 c.c. of a 14 per cent potassium hydroxide solution. Twenty-five c.c. of 50 per cent alcohol are added, and the whole shaken for 15 to 20 seconds and then allowed to stand until the mixture separates into two layers. The lower layer is drawn off and extracted with petroleum ether three times, combining these petroleum ether extracts with the first one made. The combined petroleum ether extracts are washed three times with 15 c.c. portions of 50 per cent alcohol, and the lower layer separated after each extraction. The solvent is evaporated from the petroleum ether extracts and the oil dried to constant weight in vacuo, or in an inert gas atmosphere.

We do not urge this test as a rapid, routine analysis, but in the hands of a careful analyst it yields values that are significant, and certainly within the margin of error that obtains in the determination of refining losses. Now, what is the significance of the Wesson Loss? It affords the only index of the magnitude of the unavoidable losses, the sum total of fatty acids, mucilages, gums, resins, phosphatides, and color bodies present in the crude. These must be removed in order to produce a neutral refined oil. Having suggested the elements of reducing the avoidable loss, through the choice of a non-saponifying alkali, and then proceeding to develop a process whereby the oil was degummed-neutralized and the color considerably reduced with the formation of soapstock that was practically oil-free when separated from the oil, an evaluation of the process in terms of Wesson Loss seemed to us a sound approach.

In fact it is apparent now why this index was all but forgotten as it possessed only a theoretical significance to the refiner because the existing methods of refining were so far away in their efficiency that this index did not have a real objective meaning that could in practice be translated into comparable performance and yield. The refiner, a very practical man withal could be expected to show little interest in a refining loss determination that was remote from the refining losses he actually obtained. This is not intended as a reflection upon the refiner who was as efficient as the limits of his system permitted, but to demonstrate the wide gap between the refining losses produced in the cup or kettle, and the Wesson Loss. In other words, the refiner with a healthy sense of the practical chose a standard that he could approach, and that standard was the cup test. The free fatty acids were determined and upon this with the aid of an empirical table, the utility of which no one will deny, depended the amount and strength of lye to add. It is no wonder then crude oils of the same acidity often gave widely different losses for the reason that the oils had different amounts of gums or minor constituents. The last are the substances that are responsible to a large extent in many instances for the vagaries of refining.

The new refining process vitalizes the meaning and significance of the Wesson Refining Loss, because the results obtained with this process are commensurate with this high standard. Scores of Wesson Losses of crude oils have been determined in our laboratory. It takes a little time and patience for an analyst to master the technique. Analyses are usually run in duplicates and triplicates at the beginning. After an analyst has run several he can check himself to within less than 0.05% of the value of the Wesson Loss.

It is fortunate that the absolute Wesson Refining Loss, an index which, until the advent of this new process had theoretical significance only, and even in this respect was neglected and its utility not fully appreciated, was available. The development of the Wesson Loss technique is as much a tribute to the foresight of the originator as to the painstaking accuracy and reliability of Jamieson who, in corroborating Wesson, put the method on a more secure foundation.

The Wesson Loss supplies us with a quantitative estimate of the gums present in the crude oil. These gums more than any other constituent are the bête noir of the refiner because they are the persistent emulsifiers that retain neutral oil in the soapstock masses. These gums have obscure properties often intermediate between oleophilic and hydrophilic. The properties of the soapstock are not only dependent on the absolute amounts of gums but on the ratio of the gums to free fatty acids. Mixing crude oil and aqueous solutions of alkali results in the formation of emulsions. When the aqueous alkali solution is a solution of sodium hydroxide, the emulsion persists for more or less longer periods depending on the type of process employed. But, in any event, the sodium hydroxide present in excess can attack neutral oil, the gums functioning as emulsifiers that tend to stabilize the emulsions involved: (1) the emulsion of refining agent solution with the crude, (2) the emulsion of soapstock when formed, (3) the emulsion during the interval of contact of the soapstock with the oil, and (4) the emulsion during the period of separation of the oil from the soapstock.

The longer this state of emulsion persists, in scattered local areas, or distributed through the mass of oil, the more likelihood there is for the excess alkali to attack neutral oil, and the greater the mass of soapstock formed, in which larger mass, more neutral oil is more likely to be entrapped. Soap, the product of the action of alkali upon fatty acid, formed practically instantly and spontaneously upon contact of alkali with crude oil, and the major product of the action of alkali with triglycerides, is an emulsifying agent in its own right. This in brief, is the nature of the problem that challenges the refiner: ridding the crude oil of the gums, mucilages, fatty acids, and phosphatides without loss of triglycerides. With the aid of the Wesson absolute refining loss as a criterion, we know in quantitative terms what is the maximum possible yield of neutral triglycerides.

The Process

Sodium carbonate has two well-known desirable features that have intrigued the refiner: (1) the property of not attacking neutral oil, that is triglycerides, and (2) relatively low cost. But, when used in place of sodium hydroxide to refine crude oils, the drawbacks encountered have heretofore proved too great. These are, to repeat: incomplete and insufficient neutralization, foaming, occlusion of carbon dioxide in the foam to prevent reaction from proceeding, danger of foam rising and going over sides of kettle, no marked reduction in color, slow settling, and porous soapstock of large volume with a consequent high refining loss.

The new refining process employing sodium carbonate, has overcome these difficulties. I shall now attempt to describe a continuous stream-line variant of this process, having in mind a commercial installation where this process has been in operation for some time. The process may be divided into the following functional zones:

(1) Refining; removing gums and neutralizing with soda ash.
(2) Dehydration in vacuo.
(3) Hydration of mixture of dried oil and soapstock.
(4) Caustic wash to reduce color when necessary or desirable.
(5) Water washing of refined oil.
(6) Drying of refined oil in vacuo.

Crude oil pumped from a supply tank is preheated in flow and proportioned quantities of soda ash solution, usually 15° to 20° Bé., in excess, preferably usually in amount at least double the quantity to neutralize the free fatty acids, are added and mixed with the crude. The mixture then passes through a coil, or zone, which may be further heated, in which the soda ash solution and oil are in contact before the mixture is sent to a dehydrator kept under a vacuum of 27-29½″, where the water and any carbon dioxide from the reaction of neutralization are removed. To insure the homogeneity of the mixture of dried soapstock and oil in the dehydrator, there is a stirrer near the bottom. The mixture of dried soapstock and oil is then pumped at a constant regulated pressure to a coil before which a hydrating liquid, usually a solution of soda ash of a concentration of 6° to 20° Bé., is injected into the mixture of dried soapstock and oil. The resulting mixture is then led through a coil before being separated centrifugally. The number of centrifugal machines employed is dependent on the volume of throughput and the capacity of each machine.

As the oil at this point is neutral and rid of the gums, mucilages, phosphatides, and the color considerably reduced in the case of cottonseed oil, and adequately reduced in many instances in the case of corn and peanut oils it would be well to pause to examine in greater detail the various stages of the process up to this point. Before proceeding with other matters, I cannot stress too much the importance of maintaining a positive flow of materials at a constant rate in any given stage of the process. Whenever a reagent is added, it is advisable to add the same by means of some proportioning device. Further, when the process is an over-all continuous one, such as the one described here, synchronization of the mechanical components is an important element, not only because of the desirability of maintaining conditions constant in a given zone or area from instant to instant, but because of the interdependence of the states of operation. Once the system is started up, there should be an unvarying, uninterrupted supply of materials. Provision for this has been amply provided in units already installed.

The purpose of adding at least double the quantity of soda ash to the crude oil is to help the reaction of the soda ash with the fatty acids and other acidic components proceed without violent evolution of carbon dioxide. The possibility of some of the contents of the dehydrator kicking over is entirely eliminated because of "chemical" reasons when at least this excess is employed. When the mixture is led into the dehydrator the water is flashed off leaving behind dry oil soap and gum. In other words, whatever emulsions there were present have been destroyed, now that the water has been evaporated. The gums have undergone a marked change in physical characteristics. They have lost a great deal of their oleophilic characteristics, but still retain enough of their hydrophile nature to go with or be wetted by an aqueous phase. Similarly the dehydrated soap becomes inert, and upon contact with an aqueous phase hydrates, the hydrated soap being insoluble in the oil medium. The hydrating medium most commonly employed is the same as that for neutralization, a concentrated solution of sodium carbonate. This simplifies the practical application to some extent, as the neutralization reagent and hydrating medium are the same, and can be stored in and pumped from a single tank.

The refining of crude oils with aqueous solutions of alkalies can be looked upon as a study of emulsions. The formation of an emulsion when crude oil contacts or is mixed with aqueous alkali solution, is spontaneous and in this sense cannot be prevented. The persistence of the emulsion however depends on many factors, some of which are controllable. Thus the emulsion forms a very useful purpose, maintaining effective contact of the two media, during which time, neutralization and other refining reactions are initiated. Consequently the emulsion-on-contact, whether it persists for a fraction of a second or for longer periods, may be regarded as unavoidable, where aqueous solutions of alkalies are the refining agents. But a series of reactions has been set in motion, some of which are desirable, and others detrimental. Sodium hydroxide solution, present in excess, is likely to form emulsion producing substances, that is, soap, in proportion to the amount of sodium hydroxide, with the excess capable of forming soap through attack on neutral oil. There are at least two emulsions: (1) the major emulsion, the one that the crude oil and lye form upon contact, and (2) minor emulsion, the one that persists to a greater or less extent in the soapstock phase, from the time of the initial formation of soapstock in the "break," during the period of contact of the "break" with the refined oil and during the separation of the soapstock from the refined oil.

Dehydration functions among other things to eliminate the problem of emulsions during the period of neutralization and dehydration. As the neutralization and dehydration occur in the presence of a non-saponifying alkali in the new process, there is no loss of neutral oil from this source. Having brought the system to an "inert" state, the problem is to maintain this state of inertness, so to speak, as much as possible, while separating the oil from the soapstock, with the production of a neutral refined oil on the one hand, and a relatively oil-free soapstock on the other.

I shall discuss one expedient that has proved successful, and that is, adding an aqueous medium containing sodium carbonate to the mass of soapstock and separating the hydrated soapstock from the oil in a centrifugal separator. When the property of concentrated sodium carbonate solutions of not saponifying neutral oil and not forming emulsions with the oil is known, plus the fact that sodium carbonate is a comparatively cheap reagent the advantages of using it become apparent. These circumstances are responsible for the extreme flexibility of this reagent. A wide range of concentrations and amounts of soda ash solutions may be employed without fear of attack on neutral oil, graining and fractionation of the soapstock, emulsifying neutral oil or running into emulsion troubles. Soda ash solutions of sufficient concentration act as emulsion de-stabilizers without at the same time graining out soap. Where a continuous centrifugal is used to separate soapstock from the oil, and this method is now very widely employed, a reagent in which these properties reside is of considerable practical importance because the centrifugals can still perform their function of separating the components. In fact the mixture lends itself to a sharp separation in the centrifuge.

Inasmuch as I am discussing one particular application of the Clayton non-saponifying alkali refining process, namely, one that involves the centrifugal separation of soapstock from oil, I shall confine myself to conditions for maintaining efficient prolonged centrifugal separation of soapstock from oil. Let us now go back to the stage of the process where the oil and soapstock are in the dehydrator. The water and carbon dioxide have been removed from the mixture. The gums have been rendered inert. They are no longer as soluble in oil, nor do they tend to form emulsions as readily. But on the other hand, their immediate re-dispersion in water is also slightly impaired.

This is of no consequence as long as their emulsion and oleophilic propensities have been reduced. Concentrated sodium carbonate solutions hydrate the soap and gums and together form a much heavier phase than the oil. Separation in the centrifuge is thus facilitated and the efficiency of the separation is greatly increased. Further, concentrated sodium carbonate solutions de-stabilize the emulsion between the oil and the soapstock phase, making the soapstock phase oil repellent, thus squeezing out the entrained oil globules. In the centrifuge, the action of centrifugal force completes the squeezing out of the oil from the soapstock phase in which the former is held only very superficially because of the oil-repellent nature of the sodium carbonate solution.

All other factors being equal and adjusted, the operation of a continuous type centrifugal is more efficient over prolonged periods when the soapstock phase is discharged in a constant uninterrupted flow. The more liquid, or the lower the viscosity, within certain operating limits, is the soapstock, the more efficient is the separation, and the greater is the likelihood of the centrifugal continuing to discharge oil-poor soapstock and clear oil for extended periods. One of the functions of the soda ash solution is to dilute the soapstock sufficiently to create a phase possessing flow properties under the conditions of separation and discharge from the centrifugal. The components of the soapstock phase slide along the walls of the centrifugal bowl, the "inert" sodium carbonate solution serving as a lubricant. Two important aspects of the dehydration-hydration sequence, with respect to emulsions, are that in the dehydration stage, emulsions are eliminated and the oil neutralized, and in the hydration stage, starting with an inert system through the choice of the proper hydrating liquid that functions at the same time as a destabilizer, the emulsion is never stable, so that the oil can be separated cleanly from the soapstock. It is the first instance of the separation of soapstock from oil, where the emulsions are synthetic and are under the control of the operator. The other existing commercial methods of refining oils all have "natural" emulsions, the persistence of which depends on the method of refining and the skill of the operator. When the oil-soapstock mixture is dehydrated, as in the new process, the emulsion is destroyed. From this point, the type of emulsion with the use of the proper hydrating solution here described is under the control of the operator.

In practice this fact is strikingly illustrated in the levelling or standardizing action of dehydration. Oils of different types often respond without significant variation of the concentration and per cent of soda ash solution to be used for hydration. The amount of gums in the crude oil determines to a large extent how much hydration solution to add. The character of the discharging soapstock is the quickest index of the amount of hydrating solution needed, more being added when the soapstock discharges too sluggishly. The operator has two things to look for as far as the reagent is concerned, after he has determined the F.F.A. of the crude, (1) add two times the amount of soda ash solution, usually 15° to 20° Bé., required to neutralize the F.F.A., and (2) then inject enough soda ash solution to cause the soapstock to discharge freely from the centrifugals. He will quickly learn what the approximate setting for the hydration solution should be for a given crude. The changes are made quickly, literally by a turn of a valve. From an operating control standpoint the process is surprisingly simple.

Another instance of the regimentation or simplification of the process, is the equal facility with which degummed and undegummed crude oils are refined. The refining of low-gum and completely degummed crude oils with sodium hydroxide is at times a ticklish job, because the separation into clear oil and oil-poor soapstock is difficult to maintain for extended intervals. The system under such circumstances is very sensitive and is easily thrown out of balance with the consequent fractionation of the soapstock resulting in dirty oil. But, with the dehydration-hydration system, no such difficulties are encountered. Degummed crude oils and ordinary crude oils are refined with equal facility. Inasmuch as the gums determine perhaps more than any other factor the amount of hydration solution to use, degummed oils require less than undegummed crude oils. Another characteristic that affects the amount of solution is the nature of the oil, that is, whether the oil contains more unsaturated fatty acids and so forms more soluble soaps than an oil with more saturated acids. For this reason an extracted crude soyabean oil, which has relatively small amounts of gums, required but 2.5% of hydrating solution. Water alone without dissolved electrolyte, proved an adequate hydrating medium in this instance. The soapstock discharged as a thin flowing liquid. This crude oil, F.F.A. 0.3%, was processed through the unit with a refining loss of 1.15% as against a laboratory cup loss of 3.0%. I cite these results as much to illustrate the extreme flexibility of the process as to show the extent of the savings even in low cup loss oils.

I have dwelled at length on the process up to the point of separation of the sodium carbonate soapstock from the neutral color-reduced oil. The latter is further treated in a commercial unit. When the oil is cottonseed oil, further reduction in color is obtained through subjection to a caustic wash. The oil, cooled in flow to about 70-90° F., is pumped through a coil at the same temperature before which sodium hydroxide solution, about 1 to 2% of a 10° or 12° Bé., is proportioned and mixed with the oil. The amount and strength of sodium hydroxide solution are governed by the nature of the oil. A highly colored oil, or one that resists reduction in color may require stronger lyes. The mixture is continued through a coil heated to 130-160° F., and then led to a centrifugal, or a battery of centrifugals, where the oil is separated from the soapstock which discharges as a thin trickle of dark liquor. The loss sustained in an operation of caustic washing just described is about 0.1%. The oil is then sent through an all-closed washing and drying system. The caustic washed oil, which may be heated in flow through a coil, is pumped through a coil heated to about 160-190° F., before which water is mixed into the flow, then sent to a high capacity centrifugal. The centrifuged oil is then pumped into a vacuum kettle where the water is evaporated to produce a dry oil. The refining losses reported here for this process were determined on single and several tank car lots from the weight of crude oil used and that of the dried, washed, refined oil recovered.

It is revealing to compare the analyses of the soapstock discharged from the centrifugals that separate the sodium carbonate soapstock from the neutralized oil, with soapstocks of other processes. Kettle soapstock usually had 45-50% T.F.A. and the ratio of free, or neutral oil, to fatty acids was about 1 to 1; soapstocks from the new process have T.F.A. ranging from 12 to 45%, depending largely on the amount of liquid used to rehydrate. The ratio of neutral oil to fatty acids varies from about 1:4 to 1:12, with some of the common ones around 1:6. In view of the absence of loss due to saponification of neutral oil, the avoidable loss is virtually the free oil in the soapstock, plus the small losses incurred in the caustic wash and water wash steps.

Where the per cent of total fatty acids in the soapstock is low, acidulation becomes a preferred procedure in disposing of the soapstock. The excess soda ash in the soapstock consumes additional sulphuric acid, but the value of the acid is but a fraction of the increased value of the neutral oil saved. Acidulation proceeds readily when proper care is taken to add the acid slowly in increments, allowing it to decompose the excess sodium carbonate and permitting the evolution of carbon dioxide to be complete before adding more acid.

Performance

The new Clayton process has given consistent, uniform low refining losses close to the Wesson Loss. The closeness of approach to the Wesson Loss is primarily roughly proportional to the amount of gums in the crude and to the color of the crude. With low-gum, light colored crudes we have come within several tenths of one per cent of the Wesson Loss. With high-gum crudes the difference between the Clayton Refining loss and the Wesson Loss is higher. Crude cottonseed oil from the Valley, characterized by low cup losses, low free fatty acids, but high ratio of gums to F.F.A., have responded particularly well to the treatment of the new process. Crude corn oil has been refined to give low refining loss. In one instance recently, a low-gum, dry-milled, crude corn oil, refined through a new process unit, gave a loss of 2.5% as against a Wesson Loss of 2.34% of which 1.1% was gums. The cup loss of the crude was 4.9%. Peanut oil, tallow and other oils have been processed through a new process unit with encouraging results. Peanut oil, in particular, has yielded losses very close to the theoretical.

The efficiency of the process is illustrated in the table below in which the results of the refining of several different crude oils are given. The plant losses were determined from the weights of crude oil pumped into the system and the washed dried refined oil recovered. From 1 to 4 tank cars of crude oil were used in each instance.

TABLE I

Performance of the Clayton Soda Ash Oil Refining Process

Crude oil	F.F.A.	Wesson loss	Plant loss Clayton soda ash process	Official cup loss
Crude Cottonseed Oil—Mississippi Valley	0.6%	2.33%	2.72%	4.25%
Crude Cottonseed Oil—Southeastern	1.1%	3.17%	3.58%	6.45%
Southeastern	1.8%	3.80%	4.41%	7.35%
Crude Corn Oil—dry milled	1.24%	2.34%	2.5%	4.9%
Crude Peanut Oil	0.7%	1.80%	2.17%	4.75%

The soda ash soapstock discharged from the primary centrifuge possesses unique properties. The oil-soluble nature of some of the gums becomes manifest when the "free oil" is determined in the accepted manner of extracting a dispersion of the soapstock in 50% aqueous alcohol with petroleum ether. A considerable portion of the gums goes into the petroleum ether layer. A simple method of extracting oil, relatively free from gums, was devised. The soapstock was first dried in an oven at 100°C. The dried residue was then extracted with acetone. The extracts, filtered, were collected in a weighed flask and the solvent evaporated. The solvent-free residue was an oil. When the soapstock, dispersed in 50% aqueous alcohol, was extracted with petroleum ether, the extract, rid of the solvent, was a semi-solid mass.

I do not wish to give the impression that the new process gives theoretical, absolute losses, but what I do want to stress is that the performance is so good that the existing standards become obsolete, and some more exacting standard such as the Wesson Loss is within reach.

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The Determination of Small Amounts of Free Sulphur in Bleaching Earth

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Sulphur is one of the impurities found in bleaching earths which are used in treating edible oils. In bleaching earth this element exists in the combined and free states. The combined sulphur usually exists as a complex iron sulphide, a sulphate, and sometimes, although rather infrequently, as free sulphuric acid.

This paper is primarily concerned with the detection and determination of free sulphur in bleaching earths since it is in this form that sulphur easily unites with cottonseed, corn, soya bean oils, and the like, especially at the temperatures normally used in bleaching operations. Since the bleaching operation is carried out after the refining operation, any sulphur that finds its way into the refined oil usually remains in it during storage.

When an oil containing sulphur is hydrogenated the sulphur interferes with the activity and selectivity of the catalyst, causing longer hydrogen absorption time which in turn lowers plant output and also requires the use of more catalyst. As a result hydrogenation costs are increased. Since the amount of free sulphur in a bleaching earth is quite small, being well under 0.05 per cent, it often escapes notice. We