A Study of Some of the Factors Affecting the Laboratory Bleach of Soybean Oil

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INTRODUCTION

COOPERATIVE tests on the bleaching of soybean oil by members of the society have resulted in a wide variation of colors being obtained by the participants. Preliminary consideration indicates two possible explanations for this situation. Either the Official Method for bleaching is not sufficiently explicit with respect to soybean oil or there is something inherent in this particular oil which is responsible for differences in bleach results. These two factors might also exert a combined influence upon results obtained.

To determine, if possible, the conditions which must be controlled in order to assure a uniform bleach color and to apply these to the bleach procedure has been the principal objective in this work. Refined expeller oil was used in all the experimental work. This was from several small lots, but all direct comparisons were made on the same lot so that any small variation in bleach color between lots was of no consequence. The standard bleaches for the different lots varied between 2.4 and 2.7 red.

INVESTIGATION OF THE OFFICIAL METHOD FOR BLEACHING

Briefly, the Official Method for bleaching calls for heating the oil to 120°C., adding 6% of Official earth, agitating the mixture for five minutes without allowing the temperature to fall below 105°C. and then filtering. Each step in the method has been carefully examined by varying one condition at a time while keeping the others standard. The additional conditions of rapidly heating the oil to the specified temperature at a uniform rate and adding the earth immediately that temperature was attained were also observed.

TEMPERATURE

The first condition to be varied was bleaching temperature. A number of carefully controlled bleaches was run at sustained temperatures ranging from 90° to 140°C.

TABLE 1

Temp. of Bleaching in C.	*Bleach Color in Lovibond Red*
90	2.8
100	2.4
105	2.4
115	2.4
120	2.4
130	2.5
140	2.6

Table showing bleach colors obtained bleaching throughout at the specified temperature.

All colors obtained between 100° and 120°C. were the same. At 90°C. the color was 0.4 red higher than obtained in the 100°-120°C. range, due very likely to not obtaining the maximum bleaching action at this temperature. Above this same temperature range (100°-120°C.), that is, at 130° and 140°C., the color darkens again — 0.1 and 0.2, respectively.

PER CENT OF EARTH

A study was made of the color variation due to using amounts of earth varying from 4 to 8 per cent.

The rate of change of bleach color with respect to the amount of earth used is still significant at the percentage prescribed by the Official Method. It was found to change 0.4 red between the limits of 5½ and 6½ per cent of earth. (Figure I)

BLEACHING TIME

In studying the effect of bleaching time, the intervals were varied from 2½ to 20 minutes.

FIGURE I

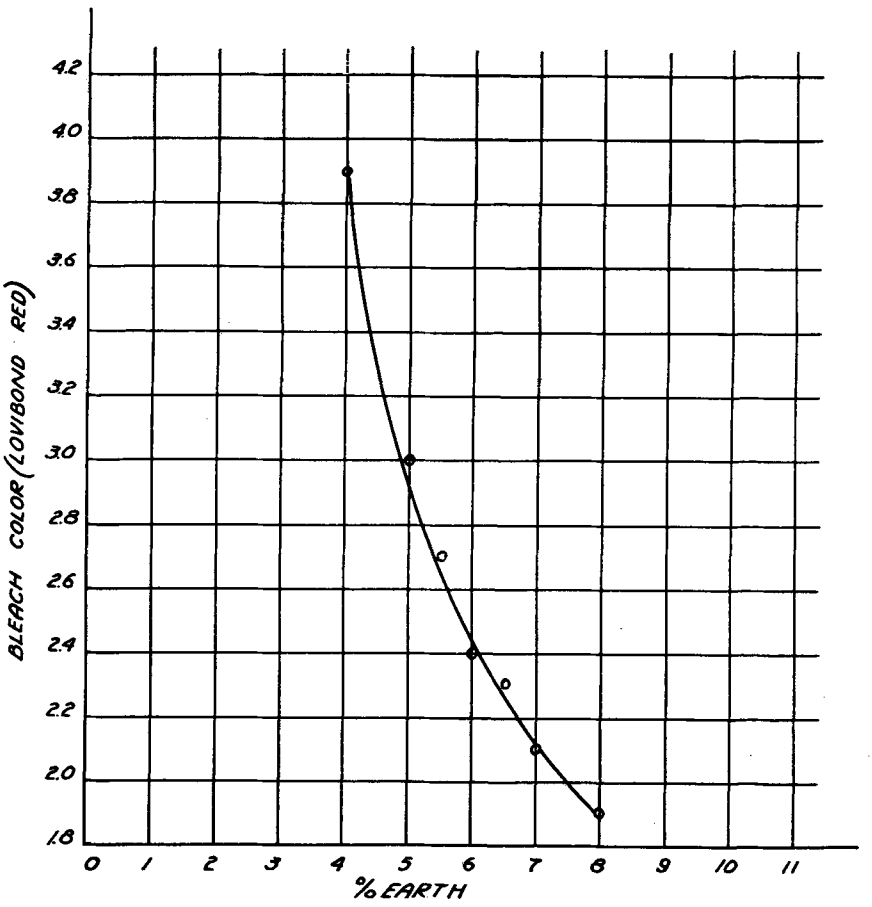
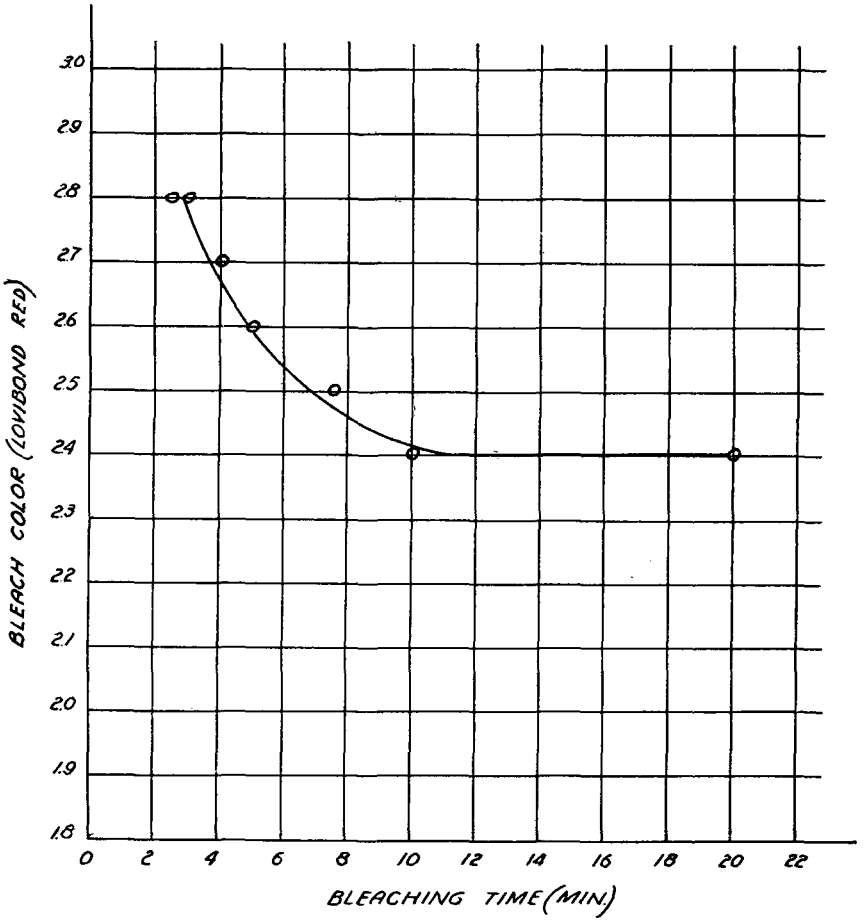


FIGURE II



A slight improvement of color, averaging about 0.04 red per minute between the range of 5 and 10 minutes bleaching time, was found. The differential for the 5-6 minute intervals, however, was slightly more than average; about 0.06 red. Beyond 10 minutes, no measurable change was observed. (Figure II)

LAPSE OF TIME FOR FILTERING

Tests showed that allowing the samples to stand unagitated for as much as 20 minutes after the bleaching period without filtering had no effect on the color obtained.

RESULTS

Investigation of the conditions specified by the Official Method for bleaching reveals that:

1. Sustained temperatures within the established temperature range and extending slightly beyond on either side are interchangeable for the purpose of yielding uniform bleach colors.
2. Variation of per cent earth used does have some effect. But the variation caused by even the ordinary means of weighing the oil and the earth in the laboratory is probably not sufficient to make this effect significant.
3. Although the color continues to improve slightly after the specified interval for bleaching, the improvement is of no consequence, providing reasonable adherence to the time interval is maintained.
4. Standing for as long as 20 minutes after bleaching and before filtering does not affect the bleach color.

INVESTIGATION OF OTHER CONDITIONS

In the work just discussed, all bleaches were carried out observing the additional conditions of (1) heating the oil rapidly and uniformly to the bleaching temperature and (2) addng the earth immediately that temperature was reached. No difficulty in reproducing bleach colors was experienced so long as both the Official Method and these latter conditions were observed.

The Official Method does not specify any period of time through which the oil should be heated to 120°C. Neither does this method state whether or not the oil should be agitated during the heating interval nor if it is essential to introduce the earth immediately after the specified temperature is attained. These conditions are left to the judgment and convenience of the operator as well as to the limitations of the bleaching equipment. It seems logical to assume that agitation for the purpose of uniform heat distribution and temperature measurements would be applied but the other two conditions will vary. These variations may not be so much on successive bleaches, run by the same operator on the same equipment, but may be considerable for different operators and different bleaching equipment. Adhering to the specifications for the Official Method while investigating these conditions was consequently undertaken.

TIME OF HEATING AT BLEACHING TEMPERATURE BEFORE EARTH IS ADDED

The first indication that time of heating and agitation prior to the introduction of the earth had an effect on the bleach color was encountered when one sample was allowed to heat to 140°C. and cool down to 115°C. before the addition of the earth. The bleach color of this sample was 0.8 red higher than one run throughout at 140°C. and 1.0 red greater than another made at 115°C. The difference in treatment of this sample and a normal one was 10 minutes' extra hot agitation before the addition of the earth.

This indication, that time of heating and agitating prior to the addition of the earth had an effect on the bleach color, was studied further. A series of samples was bleached in which the oil was agitated 5 and 10 minutes at the specified temperature before adding the earth. Their colors were compared with that of a sample to which the earth had been added as soon as the specified temperatures had been attained. (In this discussion that sample which is heated to the specified temperature within 2½ minutes and to which the earth is added immediately that temperature is attained is referred to as the "blank" or "standard.") It was found 5 minutes extra agitation resulted in an increase of 0.4 red and 10 minutes extra agitation, 0.7 red over the standard. (Figure III)

From the study of bleaching time, we found that 10 minutes agitation at the specified temperature in contact with the earth gave 0.2 red better color than the stand-

ard interval of 5 minutes. The "5-minute" sample, as described above, received 10 minutes agitation at the specified temperature but was in contact with the earth for only the last 5 minutes. It seems a simple deduction that the increase in bleach color is due to the extra agitation at the specified temperature before adding the earth.

TIME OF HEATING TO THE SPECIFIED TEMPERATURE

Having found that additional heating under agitation at the specified temperature prior to the addition of the earth influences the bleach color, the question as to whether the rate of heating to temperature affects the color should also be considered. If it does, the 2½ minute period of heating used for our standard test might represent a point on the "heat-darkening" curve. In this case, the bleach color becomes relative to the heating facilities of the bleaching equipment.

The influence exerted by the rate of heating is demonstrated by our experience with the cooperative samples. With the exception of the last two groups, all of our data were obtained on the same equipment and were on the dark side of the norm. The last two groups, however, were run on other equipment and the values reported were at the norm. The only difference in the bleaching equipment was that it requires 2½ to 3 minutes to heat to the specified temperature with one unit as against 4 to 7 minutes with the other.

For the purpose of more accurate time measurement of the bleach period, it would be natural for the operator to allow the faster heating samples to mark time at the specified temperature while the slower ones caught up before adding the earth. In some cases, this represented additional agitation out of contact with earth to the extent of 2 or 3 minutes at 120°C. Such samples, we now feel, would have a bleach color 0.2 to 0.3 red above standard (Figure III). It was suspected the slower heating samples also had a color darker than standard. This was confirmed by tests showing that as the heating period varied from 1½ to 6 minutes, the color increased 0.3 red. (Figure IV)

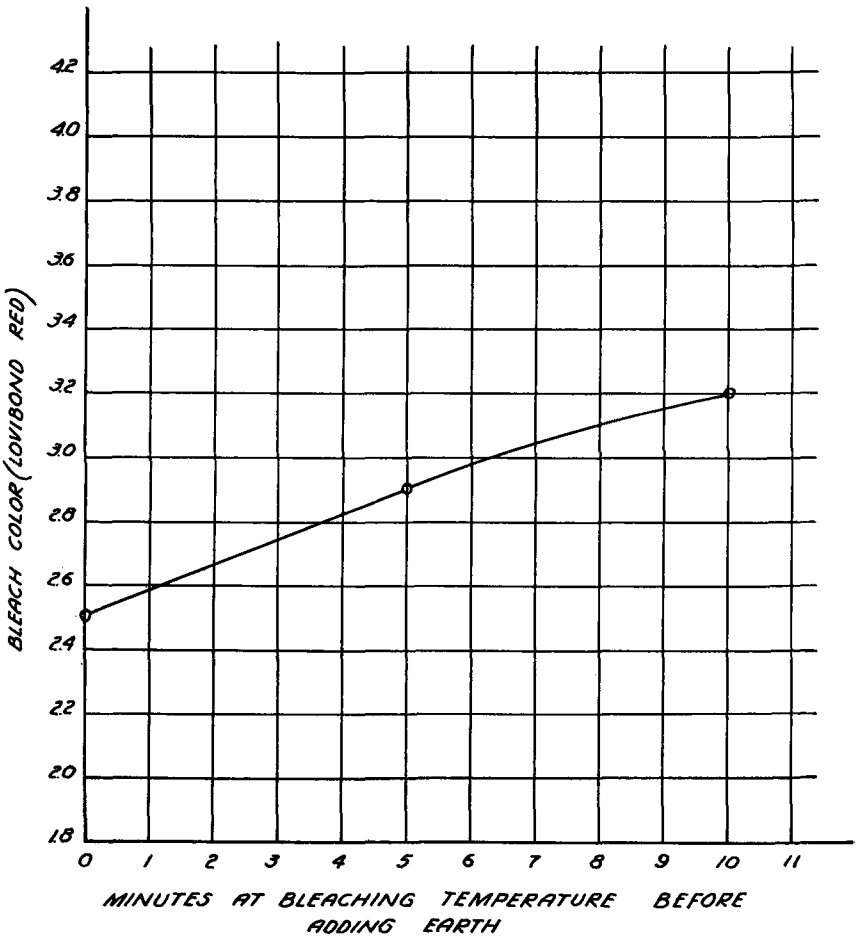
The bleach color obtained then depends not only on the time of agitation at the specified temperature before addition of the earth but also on the rate of heating to the specified temperature. As a corollary to this, it follows that the 2½ minute period taken as a standard in our work does represent a point on the "heat-darkening" curve. Hence, it is apparent bleaching equipment with variable facilities for heating will yield different bleach colors.

HEATING OIL IN CONTACT WITH EARTH

Referring back to Figure IV, where the color increased 0.3 red as the time of heating varied from 1½ to 6½ minutes, we find that the slope of the curve increases as the heating period decreases. Extrapolation to a zero heating period shows a 0.3 to 0.4 red decrease in color from the standard. This point should represent the color where the so-called "heat-darkening effect" is nil, a color which may more nearly approach the intrinsic bleach color of a soybean oil. In order to eliminate this effect and at the same time conform to the Official Method, the oil would have to be heated instantaneously to 120°C. and the earth added simultaneously. This, of course, is impracticable. As an alternate course, a deviation from the Official Method was made and the effect determined of introducing the earth prior to the heating period.

Samples bleached according to this procedure are designated as E-1. A great number of E-1 and "blank" color comparisons were made, all of which disclosed a difference of 0.3 to 0.4 red in favor of the E-1 samples. It is true that the E-1 sample is subjected to a bit more bleaching action than the "blank." This amounts to 2½ minutes at a uniformly progressive temperature increase of from 25° to 120°C. Investigation of the time-effect revealed that 2½ minutes increased bleaching time at a sustained temperature of 120°C. affects the color only 0.1 red. Also, studying the temperature-effect showed bleaching action was less for temperatures below 100°C. Thus, it seems the improvement of color in the E-1 sample due to increased bleaching action is probably not more than 0.1 red. So

FIGURE III



the effect of increased bleaching time alone cannot account for this difference.

The observation that an E-1 sample agitated 5 minutes over-all, that is, 2½ minutes to temperature and 2½ minutes thereafter, gave 0.3 or 0.4 red lighter color than a blank sample adds support to this indication. This seems particularly the case when we consider the blank sample is subjected to a greater total bleaching action than the E-1 sample agitated 5 minutes over-all. Even an E-1 sample filtered at the point where the specified temperature is reached gave a slightly lighter color than a blank sample though it had undoubtedly received less bleaching action at 120°C.

Finally, a comparative E-1 and blank time-color study (Figure V) shows that subjecting the blank to bleaching action for an extended interval does not result in the minimum color attained by the E-1 sample in 5 minutes bleaching time at 120°C.

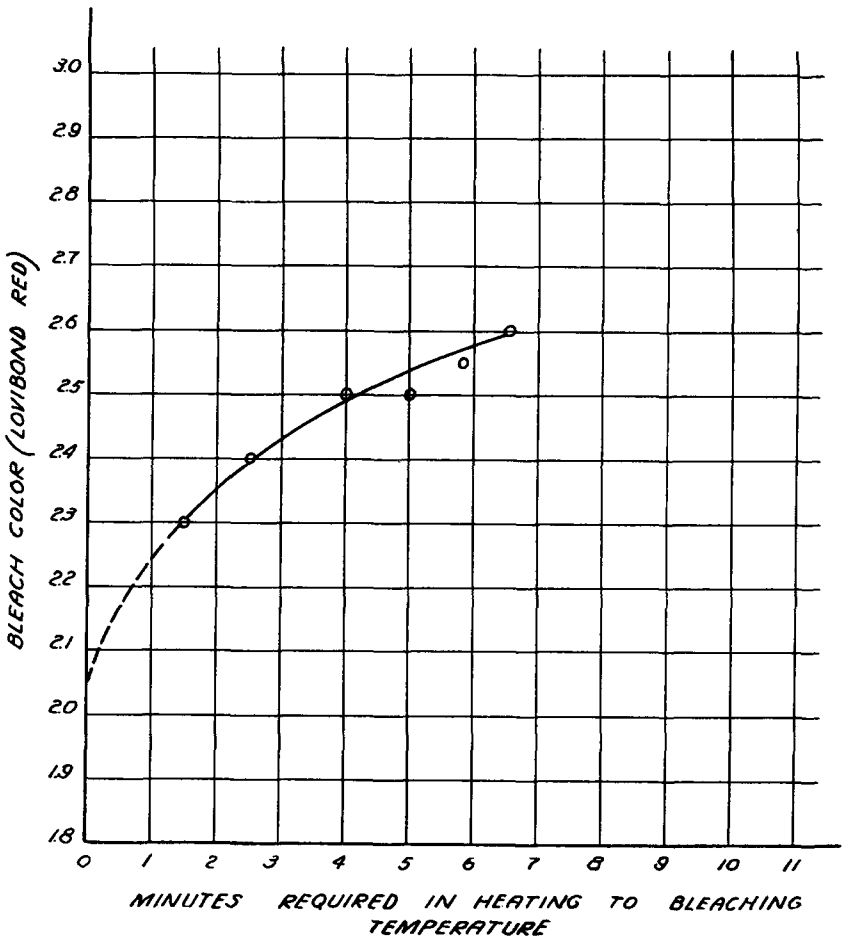
The presence of earth, then, during the heating period acts to improve the color at least 0.2 or 0.3 red over the standard; or more precisely, to inhibit the "heat-darkening effect" at least 0.2 or 0.3 red. This difference corresponds rather well with the color improvement procured when the curve in Figure IV was extrapolated to the zero heating period.

This coincidence lends support to the idea that the pre-addition of the earth, strictly speaking, does not improve the color but rather inhibits the "heat-darkening effect." This idea gained further support from the result of tests which showed that E-1 samples bleached for 5 minutes, once they were at temperature, yielded the same bleach color regardless of the length of heating period.

RESULTS

From this it is logical to conclude when the oil is heated to the specified temperature without the presence of earth, there is a positive effect which tends to give a darker bleach color. This is observed even through comparatively short heating periods. With our present knowledge of the causes for this effect, we have simply referred to it as the "heat-darkening" effect. The presence of earth during the heating period apparently nullifies this action.

FIGURE IV



OTHER TESTS

The "heat-darkening" effect has been described as resulting from a combination of heating and agitating the oil without the earth. Other tests have shown that omission of the agitation during the heating period has a mitigating influence but to no greater extent than 0.1 red. Comparative bleaches run in iron, porcelain, and glass vessels showed that these three types of surface were interchangeable insofar as uniform bleach color is concerned.

Cottonseed oil was investigated with reference to its behavior toward this "heat-darkening effect." Only one set out of three showed any difference between the bleach color of an E-1 sample and a sample agitated for 5 minutes at the specified temperature before introducing the earth. This difference was only about 0.1 red. Similar treatment of soybean oil consistently yielded a difference by these two methods of from 0.8 to 1.0 red. This is a rather clear indication that soybean oil under the conditions of laboratory bleaching behaves quite differently from cottonseed oil.

The scope of our investigation has been limited to establishing and describing the "heat-darkening effect." Consideration of the possible reasons for its manifestation in soybean oil suggests a separate investigation along the lines of identifying the presence of some compound not found in otherwise similar oils.

SUMMARY AND CONCLUSION

This investigation has shown that the bleach color of soybean oil can be reproduced but not without observing certain conditions which are not specified in the Official Method for bleaching. These are the time required to heat the oil to the specified temperature and the interval of sustained temperature before adding the earth. It has been shown that any period of heating the oil without earth, either while approaching or at the specified temperature, acts to darken the bleach color in proportion to the length of this period. This has been described as the "heat-darkening effect."

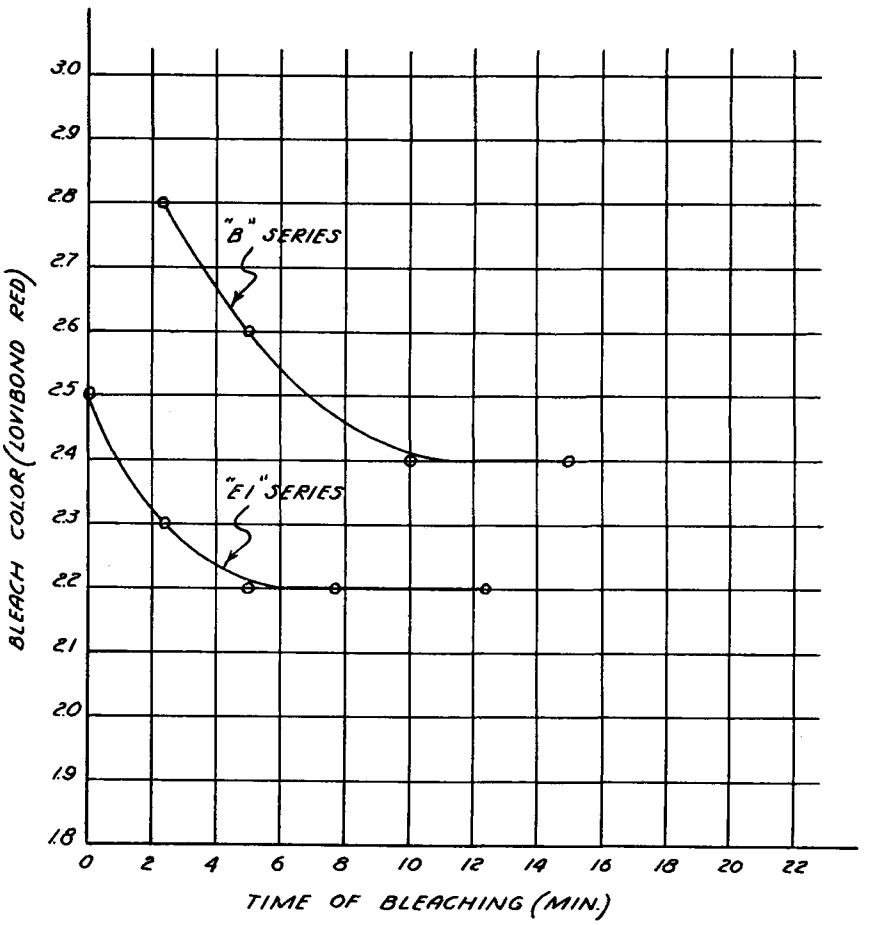
Bleach colors thus obtained become relative to the judgment of the operator and the heating facilities of the bleaching equipment. From the influence these variables exert with soybean oil, it can be seen that wide color variations may be obtained between bleaches and especially between operators with no infraction of the Official Method.

The addition of earth before the heating period protects the oil from this "heat-darkening effect." It also eliminates the necessity for observing a uniform rate of heating to the specified temperature; for regardless of this interval, within reasonable variation, a uniform and minimum bleach color is obtained after the usual 5-minute bleaching period at the specified temperature.

RECOMMENDATIONS

We should like to submit for consideration the recommendation that the earth be added to soybean oil before heating to the specified temperature in order to place laboratory bleach colors on a more absolute and reproducible basis.

FIGURE V



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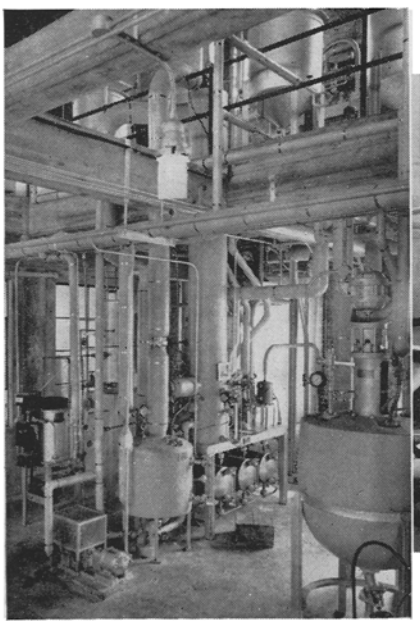
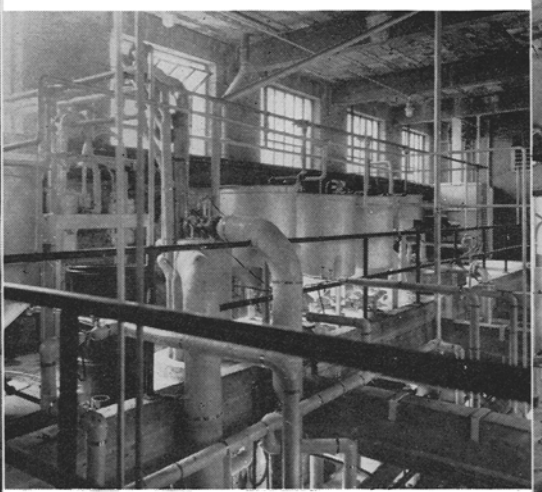
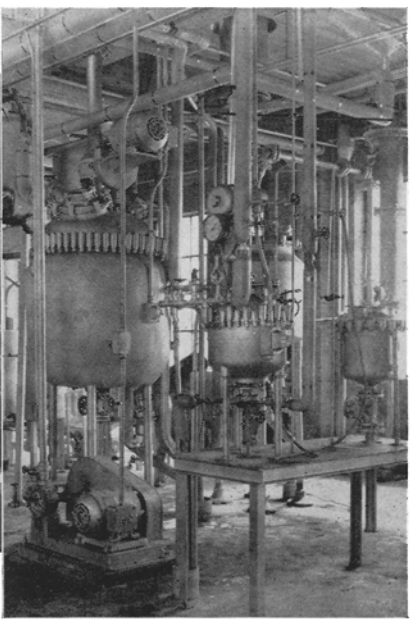


1) Fractionating Still Chemical Pilot Plant.



2) Pilot Plant for Edible Oils.



3) Glass-lined High Pressure Reduction Kettles.