

# UNITED STATES PATENT OFFICE.

SIGMUND GEORGJEVITCH ROSENBLUM, OF LONDON, ENGLAND.

## PROCESS OF REFINING OILS.

SPECIFICATION forming part of Letters Patent No. 632,148, dated August 29, 1899.

Application filed November 21, 1898. Serial No. 697,092. (No specimens.)

*To all whom it may concern:*

Be it known that I, SIGMUND GEORGJEVITCH ROSENBLUM, a subject of the Czar of Russia, residing at London, England, have invented  
5 certain new and useful Improvements in or Relating to the Refining and Decolorizing of Cotton-Seed Oil and other Analogous Oils, (for which application for patent has been made in Great Britain, No. 9,824, dated April  
10 28, 1898,) of which the following is a specification.

This invention relates to a new process by means of which certain vegetable oils can be effectively freed from the various impurities  
15 and coloring principles contained in them.

As is well known, cotton-seed oil as expressed from the seed contains a certain coloring-matter which imparts to the oil a cherry-red or black color and prevents its utilization  
20 for most of the industrial uses it can be put to. At present the oil is very effectively deprived of this coloring-matter by a treatment with caustic alkalies, and a well-refined oil of a pale straw color is thereby obtained. This  
25 treatment is, however, very costly on account of the great loss (up to eighteen per cent.) which arises from the use of alkali through the partial saponification of the oil. It is with a view to obviate this loss and to produce, if possible, a superior product that the process which forms the subject of this invention has been devised.

Though the peculiar coloring-matter of crude cotton-oil has often been the subject of  
35 chemical research its real nature has up to the present remained unknown. My investigation of this subject has led to the discovery that there are two coloring principles contained in the crude cotton-seed oil, and at  
40 least one of these I was able to identify beyond any doubt and upon it to base my process of refining and decolorizing the oil. The greater part of the coloring-matter belongs to the class of coloring principles known as "tannins" and gives their characteristic black-color reaction with iron. After this coloring-matter, to which the red or dark-brown color of the oil is due, has been removed by suitable chemical means, as specified hereinafter,  
45 there still remains another coloring-matter which imparts to the oil a yellow color, and this coloring-matter most probably belongs to

the class of xanthophyls. Its identity has, however, up to the present not been completely established. As is well known, the tannins have  
55 the property of combining with and being precipitated by many metallic compounds—such, for instance, as those of iron, aluminium, copper, &c.—but so far I have found that certain salts of iron or aluminium are the  
60 most suitable for my purpose. In order to bring about a perfect combination of the tannin coloring-matter which is held in solution by the oil with iron or aluminium, it is obviously necessary to employ such compounds of  
65 these metals as would be entirely soluble in the oil. Such compounds are, for example, all the iron or aluminium salts of the fatty acids, like ferric or aluminium oleate, stearate, palmitate, &c., also the iron or aluminium salts of other acids similar in nature  
70 to the fatty ones, like iron or aluminium resinate and others. Though any iron or aluminium soap may be used with the same effect in my process I for reasons of economy  
75 prefer to use the mixture of iron or aluminium salts obtained by the double decomposition of an alkaline cotton-seed-oil soap with a suitable iron or aluminium compound. The resulting compound practically consists of a  
80 mixture in varying proportions of iron or aluminium oleate, stearate, palmitate, and linoleate; but if soaps from other oils be used the resulting compound will vary in proportions and composition. When, for example,  
85 the compound iron oleate alone is added to crude cotton-seed oil, the iron compound combines with the tannin coloring-matter, and a deep brownish-black color is imparted to the oil. On examining a thin layer of the so-  
90 treated oil on a transparent surface it will be found that the oil which before addition was perfectly uniform in appearance now contains a very finely-divided black precipitate, which imparts to it the dark color, while the  
95 oil itself is of a light-yellow color. This precipitate is, however, so finely divided that as far as I am able to ascertain it cannot be separated from the oil by filtering media of the closest texture. In order to separate the  
100 now light-colored oil, it is necessary to bring this very finely divided precipitate into such a form as to enable it to be retained by filtering media which are in ordinary technical use.



This object can be attained by a very great variety of means; but for the purposes of my invention I have so far found it most suitable to proceed as follows: I add to the oil treated as  
 5 above a small amount of strong ammonia (ammonium hydrate) and incorporate it by suitable mechanical means very intimately with the oil. The effect of the ammonia is to precipitate the ferric tannate (mixed with ferric hydroxid) in the form of a black flocculent precipitate, which is then easily removed from the now light-colored oil by a simple filtration. As alluded to above, a great variety of compounds will have a similar or the same effect  
 15 upon the ferric tannate as ammonia. Such compounds are, for example, alkaline carbonates, hydroxids, phosphates, ammonia-gas, and many others; but so far I have found that ammonium hydrate is most suited for my  
 20 purpose.

Having given an outline of the main reactions upon which my process is based, I will now shortly describe how I propose to carry it out in practice. I dissolve in the crude  
 25 cotton-seed oil at a moderate temperature a small amount of the fatty ferric compound—say ferric oleate—(0.1 to 0.2 per cent. of the weight of the oil has been found to be quite sufficient,) and I then leave the oil to stand  
 30 for some time at a moderately warm temperature—say about 130° to 150° Fahrenheit—mixing it from time to time in order to bring about a complete combination of the tannin coloring-matter with the iron. I then  
 35 pour into the oil a suitable amount of ammonium hydrate (one per cent. of the weight of the oil has been found to be sufficient) and emulsify the mixture by any suitable mechanical means. The mixture at first assumes a  
 40 rather thick consistency, but after the emulsifying has proceeded for a short time the mixture reverts to the normal consistency of the oil, and this stage of the process is then completed. The oil is then passed through any  
 45 suitable filtering material which is capable of retaining the black precipitate, and an oil of a golden-yellow color is thus obtained. The water of the ammonium hydrate is entirely absorbed by the black precipitate, and thus  
 50 the oil after having been separated from the precipitate is clear and practically free from water. The oil, however, contains a trace of gaseous ammonia which imparts to it a slight though characteristic smell of ammonia. This  
 55 smell is easily removed from the oil by passing a rapid current of air through the oil for a few minutes or by heating it for a very short

time at a moderate temperature. Both these methods will also remove any traces of water which may be still in the oil, and thus dry it  
 60 effectively. The oil is now of a golden color. This yellow color is, as far as I have been able to ascertain, due to another coloring principle belonging to the class of xanthophyls and can  
 65 be easily removed from the oil by heating it briskly to a temperature of about 150° Fahrenheit. This done, the oil will be of a very pale straw color.

Oil treated by the above process is not only freed from the coloring-matters contained  
 70 therein, but also from the so-called "mucilage," which is carried down by the black precipitate during the process and filtered out together with it. In fact, as ascertained by  
 75 chemical analysis oil treated by my process is of a much higher degree of refinement than the best refined cotton-seed oils at present on the market. The process if carried out properly does not involve any appreciable loss  
 80 of oil and does not introduce any impurities which could be prejudicial to the use of the oil.

Oil prepared by this process is absolutely free from iron. The amount of free fatty acids which of necessity is introduced into  
 85 the oil by the use and subsequent decomposition of the fatty iron salt or salts is too small to be of any practical disadvantage.

In the above process although iron oleate was added alone to the cotton-seed oil equally  
 90 good results are obtained by the use of any iron, aluminium, or other similar metallic soap.

I claim—

1. The process of refining cotton-seed and  
 95 other analogous oils by treating the crude oil with a metallic soap, consolidating the precipitate thus formed by adding an alkali, separating the precipitate and subsequently heating the oil to remove the yellow coloring-  
 100 matter.

2. The process of refining cotton-seed and other analogous oils by treating the crude oil with a metallic soap and consolidating the precipitate thus formed by adding an alkali—  
 105 such as ammonia.

In testimony whereof I have hereunto set my hand in the presence of the two subscribing witnesses.

SIGMUND GEORGJEVITCH ROSENBLUM.

Witnesses:

SELMA WENSTENFELD,  
 WILMER M. HARRIS.