

UNITED STATES PATENT OFFICE.

CLEMENS LOSSEN, OF HOBOKEN, NEW JERSEY.

DEODORIZING MINERAL OILS.

SPECIFICATION forming part of Letters Patent No. 537,121, dated April 9, 1895.

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To all whom it may concern:

Be it known that I, CLEMENS LOSSEN, a subject of His Majesty the Emperor of Germany, residing in Hoboken, in the county of Hudson and State of New Jersey, have invented certain new and useful Improvements in Deodorizing Mineral Oils; and I do hereby declare that the following is a full, clear, and exact description of the invention, which will enable others skilled in the art to which it appertains to make and use the same.

This invention relates to the art of deodorizing crude and raw mineral oils, such as Lima oil and Canadian oils, which require a different treatment from the ordinary American oils (generally known as "Pennsylvania oil"), because of their penetrating and disagreeable odor. It is a well-known fact that these kinds of oil are almost unmarketable on account of this disgusting odor and various processes have been invented and patented, having for their object the removal of the disagreeable odor, either from the crude oil prior to or during distillation or from the distillate. As far as I am aware, most of these processes employ metallic oxides in some form or other, because it has been supposed that the disagreeable odor, which characterizes these oils, was due to a chemical combination of sulphur with certain constituents in the oil, the nature of which has never been definitely ascertained. I have discovered, however, that this is incorrect, and that the disagreeable odor is not due to any sulphur combination, but owes its existence to the presence of certain hydrocarbons of a nature wholly different from that of the hydrocarbon which forms the petroleum proper. In other words, I have ascertained, beyond doubt, that the disagreeable odor, which it is the object of my invention to remove, is due to the presence in the crude oil of one of a group of hydrocarbons known technically under the collective name of "acetylenes." Based upon this knowledge, I have discovered a process for removing the odor from the crude oil, which process is in the nature of a preliminary treatment of the oil, prior to its distillation proper. What is chiefly aimed at is to remove the disagreeable odor from the raw product, which can then be placed upon the market and sold to much better advantage than prior to its preliminary

treatment, it being left for the purchaser and refiner to buy the deodorized crude oil and afterward distill the same into its various constituents, as he may see fit; when it will be found that each of the distillates will be entirely free from the disagreeable odor which characterizes this group of oils, the deodorization of which is the object sought to be accomplished.

My invention, therefore, consists in the effectual elimination of the objectionable acetylenes, which results in the purification and deodorization of the oils undergoing treatment by my process.

The principle which underlies my process is to produce an oxidizing action on the acetylenes at a comparatively low temperature, thereby separating them into carbonic acid and water. To effect this, the dissociation of the acetylenes into carbonic acid and water, I employ the second grade of the oxide of copper, or the so-called cuprous oxide (Cu_2O), which is one of the constituents in copper scale, or what is known in German as "*kupfer hammerschlag*." These copper scales are the waste product of copper works, where it accumulates in large quantities so that it is readily obtainable at a small cost. These copper scales, which, as above stated, are the medium employed for the dissociation of the constituents of the acetylenes, are a mixture of the cupric oxide and cuprous oxide; but in carrying out my process, the cupric oxide remains practically inert, while the cuprous oxide, at a certain degree of temperature, accomplishes the dissociation, and thereby the destruction of the objectionable acetylenes, resulting in simultaneous deodorization. Now to carry out my process on this basis, I add from one-half to two per cent. (by weight) of this copper scale, or *kupfer hammerschlag*, to the crude oil, which is gradually heated in a suitable vessel to a temperature of about 120° Celsius, which is approximately the boiling point of the oil; care being taken to stir the mixture of oil and copper scale during the process of heating. When a temperature of 120° Celsius has been reached, and the oil commences to boil, but before vaporization has set in, it is removed from the fire and allowed to cool, and the copper scale is removed by drawing off the oil, by straining it, or in any other suitable manner.

It will be found that as a result of this preliminary treatment, the oil has become deodorized, and is ready for barreling and shipping to the distiller or refiner, who will then
5 treat the deodorized oil precisely in the same manner as he treats the American or Pennsylvania oils; my process being essentially a preparatory step intended to bring Lima oils, and oils of that category, on a par with the
10 "American" or "Pennsylvania" oils, which, owing to the absence of the unpleasant odor which exists in Lima oil, are fit to go to the still of the refiner without such preliminary treatment.

15 The copper scale, after it has been removed from the deodorized oil, may be regenerated by roasting to a dark cherry red heat, after which it is cooled off, when it is ready for use over again; and in this manner a great quantity of copper scale may be used over and over
20 again for deodorizing purposes with only a nominal waste.

In order to satisfy myself that my theory regarding the cause of the unpleasant odor
25 in the category of Lima oils is due, not to the existence of sulphur in chemical combination with some peculiar constituent of the oil, but is due to the presence of the acetylenes in the oil, I have tested the oil after it has undergone my preliminary treatment, and have in-
30 variably found, as a result of such tests, that the percentage of sulphur in the deodorized oil has not been lowered. In other words, the batch of oil, after undergoing my preliminary
35 process of deodorization, will be found to contain the same percentage of sulphur as the crude oil contained prior to my preliminary treatment;—a fact which proves conclusively that the unpleasant odor cannot be due to the
40 presence of sulphur compounds, because if that were so, the test of the deodorized oil would show the entire absence of sulphur, or, at least a greatly reduced percentage thereof. Moreover, if the odor was due to sulphur com-
45 pounds, the cupric oxide contained in the copper scales would be sufficient to oxidize and neutralize the sulphur, whereas by my improved process, which operates on the acetylenes, the cupric oxide is practically inert,
50 the effect being wholly due to the chemical action of the cuprous oxide upon the constituent parts, (carbonic acid and water,) of the acetylenes.

That my theory is correct is further proved
55 by a test of the residue in the heating vessel, which shows no trace of sulphur, and also shows that the cupric oxide has not been affected by the process, the protoxide and the acetylenes being the only elements which
60 have undergone change by the said process.

One of the most important results of my process, from a commercial standpoint, is the fact that by my process the oil will become permanently deodorized; that is to say, by the
65 destruction of the acetylenes the odor is permanently removed and cannot possibly return, whereas, in deodorizing processes which

are based upon the destruction of the sulphur compounds, it is well-known that the objectionable odor is liable to return to the oil after a time. This is another proof of the correctness of my theory, because, if the objectionable odor was due, as heretofore erroneously supposed, to the presence of sulphur, the effectual removal of sulphur or its compounds would, of course, result in the effectual and permanent destruction of the disagreeable odor, the return of which can only be accounted for on the ground that the acetylenes, to which this odor is really due, have
70 not been destroyed, such old processes being directed toward the destruction of the sulphur compounds in the oil and having no reference whatever to the destruction of the acetylenes.
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In view of the fact that the deodorization by my process is wholly due to the action of the cuprous oxide (Cu_2O), upon the acetylenes, it will be obvious that I can use the cuprous oxide alone, and that the oxide of copper is added accidentally, simply because
80 it forms one of the constituent parts of the copper scale, which is the cheapest form in which the cuprous oxide can be obtained at the present time.
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It is absolutely necessary to the successful carrying out of my process, that the mixture of oil and copper scale should be heated to (approximately) 120° Celsius in order to produce a reaction of the cuprous oxide. The
90 so-called "cold processes" which aim at the deodorization of the oil simply by mixing it with certain metallic oxides, without heating the moisture or by heating the moisture up to the point at which distillation begins, are not
95 applicable to my purpose, because unless the mixture is heated to the boiling point, the reaction of the cuprous oxide will not take place, that is to say, the cuprous oxide will not give off its oxygen, by the combination of which with the acetylenes, the constituent
100 parts of the latter, viz., carbonic acid and hydrogen are dissociated.
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Having thus described my invention, I claim and desire to secure by Letters Patent
115 of the United States—

1. The process of deodorizing mineral oils, which consists in mixing the oils with cuprous oxide and then subjecting the mixture to heat until the acetylenes contained therein
120 are destroyed, substantially as set forth.

2. The process herein described of deodorizing mineral oils, which consists in dissociating the acetylenes contained in said oils by mixing them with cuprous oxide and heating
125 the mixture to a temperature of the boiling point of the oils, substantially as set forth.

In testimony that I claim the foregoing as my own I have hereunto affixed my signature in presence of two witnesses.

CLEMENS LOSSEN.

Witnesses:

H. BAUR,

GEO. O. GLAVIS.