This invention relates to a process of oxidizing hydrocarbons, more particularly the oxidation of paraffin wax.

An important object of the invention is to oxidize paraffin wax without the formation of gummy oxidation products insoluble in hydrocarbon oils.

The invention broadly contemplates a process of oxidizing hydrocarbons which comprises contacting the hydrocarbons under the influence of heat with an oxidizing gas, the reaction being carefully controlled and stopped just short of the point where the formation of gummy products insoluble in hydrocarbon oil occurs. The invention further contemplates the separation of the oxidized from the unoxidized portion and an additional treatment of the latter portion, preferably in the present of fresh unoxidized hydrocarbons.

The oxidation products of hydrocarbons and particularly of paraffin wax are of importance in the manufacture of soaps. It has also been found that the lubricating value of hydrocarbon oils is greatly increased when a portion of properly oxidized paraffin wax is compounded therewith. In ordinary methods of oxidizing paraffin wax the material is heated while exposed to a current of air until practically complete oxidation takes place. The procedure yields a product which usually has very unsatisfactory properties, rendering it unsuitable for compounding with mineral oils or for use in the manufacture of soaps due to the fact that the high oxidation products are dark colored, gummy materials insoluble in hydrocarbon oil.

I have discovered a process whereby hydrocarbons, particularly paraffin wax, may be completely oxidized to products suitable for compounding with lubricating oils or for soap making, without substantial formation of objectionable gummy products.

In carrying on the oxidation of hydrocarbons in accordance with my invention, I prefer to proceed as follows:

A charge of hydrocarbon, say for example paraffin wax, is placed in a vessel fitted with heating and cooling coils and means for introducing air into the contents. The vessel is preferably constructed of aluminum or at least fitted with an aluminum lining, as it has been determined by experiment that this metal effectually resists any corrosive action which may result from the more volatile acid products of the reaction. The charge of wax is then heated to about 200°-300° F., or preferably to about 325° F., and a current of air is blown through the melted mass. The reaction gives off considerable heat so that no external heat is required to maintain the desired temperature of 325° F. and it is even necessary to effect cooling of the vessel by a proper system of cool water coils or an equivalent device. As the reaction progresses, small samples are periodically removed from the kettle and tested for their acid value by the usual method. When the acid value (expressed as milligrams of KOH required to neutralize one gram of the oxidized wax) reaches about 35, the air is cut off and the batch is ready for further treatment.

My experiments with the oxidation of paraffin wax have shown that when the wax is oxidized under the above temperatures, the reaction can be carried to a fairly definite point, as evidenced by the acid value of the product, before there is any substantial formation of the gum-like products of a more prolonged oxidation. While an acid value of about 40 may be considered the upper limit to which the reaction may be safely carried without the formation of gums, I prefer to only carry the oxidation to an acid value of about 35 which insures the complete absence of gummy constituents.

At this point, however, it will be found that there remains an appreciable portion of the wax which has been only very slightly oxidized or has even remained entirely unchanged. Such unconverted wax is objectionable both in soaps and in lubricating oils and I therefore prefer to treat the oxidized charge in order to separate therefrom the unoxidized portion. This separation may best be accomplished by a "sweating" process similar to that ordinarily applied to the separation of the various fractions of paraffin wax according to their melting points. The process is universally employed in the petroleum industry and further detailed description is not deemed necessary, it being sufficient to state that, due to the appreciably higher melting point of the unoxidized wax as compared to the oxidized portion, separation by sweating is both convenient and effective.

After separation by sweating, the lower
melting oxidized portions of the wax are available for the various uses to which they are adapted. The product is particularly suitable for compounding with lubricating oils to improve the lubricating qualities thereof, for example in the lubrication of brake and transmission linings in automobile power plants and the like, proving an effective preventative for the "chattering" often encountered at these points of frictional resistance. The higher melting portion, separated in the sweating operation, has the property of promoting the oxidation of fresh unoxidized wax and it has been found that the admixture of such partially oxidized wax with a fresh batch materially reduces the time required to carry the reaction to the point indicated by the desired maximum acid value.

For the sake of more clearly illustrating the invention, the following example is given, it being understood that the invention is not limited to the specific conditions and results contained therein. In the example, about 3000 lbs. of paraffin wax having a melting point of 125° to 125° F. is placed in a suitable kettle, heated to a temperature of 325° F. and maintained at this temperature while approximately 6000 cu. ft. of air per hour is passed through the molten mass. Samples are withdrawn at regular intervals and tested for their acid value, the results of which tests given below indicate the progress of the reaction.

<table>
<thead>
<tr>
<th>Time in hours</th>
<th>Acid value of batch</th>
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<tbody>
<tr>
<td>0</td>
<td>0</td>
</tr>
<tr>
<td>2</td>
<td>4</td>
</tr>
<tr>
<td>4</td>
<td>8</td>
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<tr>
<td>6</td>
<td>20</td>
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<tr>
<td>8</td>
<td>28</td>
</tr>
<tr>
<td>10</td>
<td>35</td>
</tr>
</tbody>
</table>

The air blast is discontinued when an acid value of about 35 is reached and the oxidized wax may then be withdrawn from the kettle and may be considered a finished product suitable for soap making or compounding with lubricating oil. However, it is preferable at this point to pump the molten wax to a "sweater" where it is subjected to a "sweating" or fractionating operation yielding approximately 80% oxidized material and 20% wax which is only slightly oxidized. The low melting oxidized portion of the wax is free from gummy constituents insoluble in hydrocarbon oils and may be compounded with lubricating oils of low cold test without producing therein an objectionable paraffin "cloud" or haziness at low temperatures. The very slightly oxidized portion is charged into the reaction kettle with fresh wax and has the effect of catalyzing or promoting the oxidation to such a degree that the approximate time of 10 hours required for the oxidation of the batch given in the specific example may be reduced by one-half or even more.

In practice the invention may be applied in the oxidation of various types of hydrocarbons such as viscous oils, high and low melting point waxes and the like. Pure oxygen may be used although in general air will be found to be the cheapest and most satisfactory oxidizing gas available for large scale operations. In certain cases it may be found to be advantageous to use higher temperatures than the preferred temperatures disclosed but such high temperatures increase the tendency toward formation of the gummy insoluble products, the avoidance of which is one of the most important features of this invention.

Obviously many modifications and variations of the invention, as hereinbefore set forth, may be made without departing from the spirit and scope thereof, and therefore, only such limitations should be imposed as are indicated in the appended claims.

I claim:

1. The process of preparing oxidized paraffin wax substantially free from gummy products insoluble in hydrocarbon oil which comprises exposing the wax to the action of an oxidizing gas while under the influence of heat, discontinuing the oxidation when the product has reached an acid value not exceeding forty.

2. The process of oxidizing paraffin wax without the formation of gummy products insoluble in hydrocarbon oil, which comprises passing air through the wax heated to a temperature not exceeding 330° F. until the wax has acquired an acid value not exceeding forty.

3. The process of oxidizing paraffin wax without the formation of gummy products insoluble in hydrocarbon oil which comprises passing air through molten wax at a substantially constant rate, continuously conducting away the heat of the reaction to maintain the temperature of the wax at 300° F. to 330° F. and continuing the oxidation until the wax has an acid value not exceeding forty.

In witness whereof I have hereunto set my hand this 22nd day of December, 1925.

JOSEPH R. SCANLIN.