

UNITED STATES PATENT OFFICE

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SEPARATION OF OIL FROM BLEACHING CLAY

No Drawing. Application filed April 3, 1930, Serial No. 441,422, and in Germany May 16, 1927.

Bleaching clay which has been used for refining mineral oil, contains large quantities of oil which require to be recovered. If the oil containing bleaching clay is extracted with organic solvents, such as petrol, benzol, trichlorethylene, only the oil which was mechanically mixed with the bleaching clay is recovered, and not the part of the oil which was adsorbed. In the known treatment of used bleaching clay by boiling with water also, only the mechanically mixed oil is separated.

In order to remove also the adsorbed oil, the surface of the bleaching clay which has an adsorptive effect must be altered by chemical means. For this purpose the treatment of the oil containing bleaching clay with aqueous lyes is known. The bleaching powder thereby assumes a slimy form which is filtered only with great difficulty, and the working up of large quantities of dilute lyes containing slimy clay has great disadvantages. The mineral oil is also often obtained in strongly emulsified form so that a separation of this emulsion is still necessary, which operation is difficult.

Finally, it has already been proposed to free the used oil-containing bleaching clay from oil by boiling with acidified water. According to the literature of the subject this process produced little satisfactory result.

It has now been found that the adsorbed oil also can be entirely recovered from the used bleaching clay, if it is heated with dilute aqueous mineral acid above its boiling point and under the pressure arising thereby.

In the working up of oil containing bleaching clay which has been used for refining fat, the removal of fat has already been effected by heating with water under pressure, if necessary with the addition of a splitting agent (sulphuric acid). It was however not to be expected that such a process would also be useful for refining bleaching clay in the mineral oil industry. Mineral oil cannot be split. In the known process the acid reacts with the fat, in the present process it does not.

The carrying out of the process is extremely simple. The bleaching clay is to be freed from oil is heated in a closed pressure vessel

with dilute acid to a temperature above the boiling point of the acid. The cheapest is dilute sulphuric acid. The amount of acid is chosen so that after the action is completed the liquid is still acid to litmus. After standing for a short time in a warm atmosphere, the material divides into three well defined layers, at the top, the recovered oil, in the middle a clear aqueous layer still having an acid reaction, and at the bottom the bleaching clay completely freed from oil and saturated with water.

In this method of carrying out the process the oil held mechanically by the bleaching clay is obtained mixed with the adsorbed part. If it is desired to obtain the two quantities of oil separate, the bleaching clay may first be treated with organic solvents in a manner known, and freed from the mass of solvent by settling or filtration, and then subjected to the treatment with mineral acid.

Examples

1. 1 kg. of used bleaching clay containing 30% of oil with 1 litre of 30% sulphuric acid is agitated in a closed vessel by blowing in steam so that the mass is heated and the pressure in the vessel rises to 3 atm. After standing for 3 hours the oil separated above is drawn off by means of the pressure in the vessel through a tube inserted to the appropriate depth. Then the slime free from oil settled below is passed through a filter press under the pressure in the vessel.

2. 1 kg. of used bleaching clay containing 30% of oil is thrice extracted each time with 1 litre of hot petrol, each time being allowed to settle and the clear petrol solution drawn off. By this means about 200 gm. of oil are extracted, which are obtained from the extract as a residue by blowing away the petrol with steam. In quality, this oil corresponds approximately to the original material, which was treated with the bleaching clay, and may be added to the next charge of oil to be treated with bleaching clay. The settled clay, still containing the adsorbed oil and mechanically adhering petrol, is treated according to Example 1. The oil which settles out consists of the mixture of petrol with

the oil which was adsorbed, and is separated by distillation. As a residue, 100 gm. of dark coloured oil are obtained which may be used for inferior purposes.

What we claim is:

1. A process for recovering mineral oil from spent bleaching clay which has been used to refine mineral oil and which contains oil both adsorbed therein and mechanically adherent thereto, which process comprises mixing with the spent clay a dilute aqueous solution of mineral acid of the order of 30% sulfuric acid heating the mixture to a temperature above the boiling point of the mineral acid solution under the vapor pressure of the solution to effect a physical separation of mineral oil from the clay, allowing the material to stand for a time, thereby causing stratification thereof into an upper layer of recovered mineral oil, an intermediate layer of clear aqueous liquid having an acid reaction, and a lower layer of water-saturated bleaching clay completely freed from oil, and finally withdrawing the mineral oil layer by means of the vapor pressure generated during the heating.

2. Process as in claim 1, characterized in that the bleaching clay is treated with an organic solvent for the mineral oil to recover the oil mechanically adhering to the clay, before the clay is heated with the dilute aqueous solution of mineral acid.

In testimony whereof we have signed our names to this specification.

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