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(54) **Miscella bleaching.**

(57) Miscella is bleached using a bleaching agent which is in the form of granules having an average particle size of ≥ 0.5 mm. An advantage embodiment of the process uses the granules as the stationary phase of a packed column through which the miscella flows. The process lacks a special bleaching agent separation step. The granules can be easily regenerated and the recovered oil is recycled.

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The present invention relates to a process for bleaching a miscella comprising an edible oil by contacting the miscella with a bleaching agent.

A miscella is defined as being a mixture of a crude edible oil or oil mixture obtained by pressing and/or extracting oil containing products and an extracting agent.

In caustic neutralization some of the impurities (free fatty acids, phospholipids, metals, part of peroxides) are removed by neutralization. However, an amount of oxidated triglycerides, soaps and particular colour imparting substances will still be present in the refined oil. Therefore frequently a subsequent bleaching step has to be performed. Bleaching is currently done by stirring of about 0.3 - 15% of activated bleaching earth during 15- 30 minutes, under vacuum at temperatures ranged between 90 - 120 °C. The bleaching earth is further removed by filtration. The solid effluent contains relatively large amounts of oil (about 25 - 50%).

The present invention aims to provide a process in which a miscella of an edible oil can be bleached in a convenient way without high oil losses and without the involvement of high investments in equipment.

To this end the present invention is characterised in that the bleaching agent used is bleaching earth in granular form with an average particle size of at least 0,5 mm.

The miscella elution is greatly facilitated if granulated bleaching earth is used instead of fine powder bleaching earth. As a consequence, bleaching earth packed columns with much lower diameter/length ratios can he used. Furthermore the investment-costs for equipment are considerably reduced and the oil losses appear to be reduced when compared with prior art bleaching processes.

The bleaching agent can be any granular bleaching earth (e.g. aluminium silicate - F-24 Engelhard, Tonsil COG, CO, Granosil) suitable as adsorbent in miscella bleaching. The granules are preferably used as a stationary phase in a packed column through which the miscella to be bleached is flown. By eluting the miscella through a stationary phase in a packed column the miscella can be bleached by the adsorbent without the necessity of complex process steps and complex separation equipment.

The edible oil which is present in the miscella to be bleached is advantageously cottonseed oil.

It appears that by bleaching this oil in the process of the present invention most of the colour bodies of cottonseed oil are removed. Furthermore, an effective removal of the peroxides takes place.

When the bleaching earth is saturated, the oil retained in the bleaching earth is recovered, washing the column with, for example, fresh hexane. The recovered oil dissolved in the hexane can be recylced into the process. This reduces the oil losses in bleaching to very low levels. Pilot plant trials showed that these losses are below 0.02%, which is much lower than in conventional processes (using 0.5% bleaching earth) where the losses are near of 0.3%. Alternatively the bleaching earth can be regenerated by extraction with a suitable extracting agent. To this end, many extracting agents are available.

Advantageously, the used bleaching earth is regenerated by extraction with an alkane/polar solvent mixture, preferably a hexane/anhydrous ethanol mixture.

Regeneration of the used bleaching earth by washing, and thereby removing all retained matter, provides for spent bleaching earth that can be used again for bleaching.

In another embodiment of the present invention the edible oil which is present in the miscella to be bleached is preferably soyabean oil. The bleaching of a neutral miscella of soyabean oil according to the present invention causes a significant reduction of gossypol pigments. Furthermore the amounts of phosphorous and peroxides are also reduced.

Preferably, the bleaching earth used for the bleaching of soyabean oil is regenerated by extraction with a ketone, advantageously being aqueous acetone.

The regenerated bleaching earth, also named spent bleaching earth, can be used again in the bleaching process.

Preferably at least part of the bleaching agent consists of spent bleaching earth which has been regenerated according to one of the above mentioned extraction-methods for used bleaching earth. The use of fresh bleaching earth as well as regenerated spent bleaching earth in the packed column provides for an economical use of the bleaching agent. When the deoiled bleaching earth can not or does not have to be used anymore it can be blended with sand, cement and water for bricks production.

In the process of the present invention there are preferably two or more packed columns present and the miscella is bleached on these columns in a counter-current process.

In the counter-current principle the miscella is first eluted in the first column, having more saturated adsorbent and is then passed through the second column having fresh adsorbent. When the first column is saturated, the adsorbent can be discharged or regenerated. Subsequently, the column is packed with fresh adsorbent and the bleaching cycle starts again, changing the miscella flow direction. In this way maximum efficiency of bleaching can be attained. The counter-current principle can be applied to two or more packed columns.

The invention will now be further explained with reference to the examples and the attached drawings, in which:

- figure 1 shows a schematic survey of the miscella bleaching principle according to the present invention; and
- figure 2 shows a flow sheet of the counter-current miscella bleaching principle according to another embodiment of the present invention.

Figure 1 shows a packed column which can be used for miscella bleaching according to the present invention. The neutral miscella enters the column at the top and passes in downward direction through the column which is packed with granular bleaching earth. By passing through the column most of the colour bodies as well as an amount of phosphorous and peroxides, present in the oil phase of the miscella, are adsorbed and filtered by the granular bleaching earth. When the miscella leaves the column the amount of above mentioned components is greatly reduced.

Miscella can be bleached on the same column until saturation of the packed column is reached. In that case the column is filled with fresh granular bleaching earth or the used granular bleaching earth is regenerated.

Figure 2 shows two adjacent columns A and B packed with granular bleaching earth, which can be used for counter-current miscella bleaching. To this end the miscella is initially fed to column A at the top of the column. The miscella flows through column A in downward direction, leaves the column at the bottom and enters the adjacent column B at the top of this column. It flows in downward direction through column B and leaves the column at the bottom as bleached miscella. During this process column A will be saturated before column B. When saturation of column A is reached, the column will be filled with fresh adsorbent, whether or not in the form of regenerated bleaching earth. Subsequently, the bleaching cycle starts again, but this time in the opposite direction, as shown with dotted line in figure 2. In this second bleaching cycle column B will be saturated before column A. When saturation of B is reached, the column will be refilled and the bleaching cycle starts again in the original direction. This process can infinitely be repeated.

As mentioned before, it is also possible to employ more than two subsequent columns; in that case the subsequent columns have increasing fresh bleaching earth in the flow direction of the miscella.

With some oils which are difficult to bleach, e.g. rice bran oil, an additional column filled with granulated activated carbon can be used in order to improve the colour fo the oil.

EXAMPLE 1

Cottonseed oil as well as soyabean oil were bleached according to the present invention in a column according to figure 1. Tabel 1 below shows the analytical values of neutralized miscella and bleached miscella on packed columns for cottonseed oil. Tabel 2 shows the analytical values for soyabean oil.

TABLE 1

Analytical values of neutralized and bleached cottonseed oil.		
Analysis	Neutralized cottonseed oil	Bleached cottonseed oil
FFA (%)	0,03	0,03
phosphorous (ppm)	3,7	0,7
peroxides (meq/kg)	1,4	0,3
colour*		
red	11,5	5,3
yellow	40,0	40,0
blue	2,0	0,0
1. 8,5 lts of neutral miscella was eluted in a column packed with 20 g of Tonsil Optimum FF (mean particle size 40-100 microns).		

* Lovibon colour $5\frac{1}{4}$".

TABLE 2

Analytical values of neutralized and bleached soyabean oil.		
Analysis	Neutralized soyabean oil	Bleached soyabean oil
phosphorous (ppm)	1,4	0,7
peroxides (meq/kg)	2,3	0,3
colour*		
red	8,2	4,7
yellow	50,0	40,0
blue	0,8	0,0
1. neutral miscella was eluted in a column packed with granulated bleaching earth (F-24 Engelhard).		

* Lovibond colour $5\frac{1}{4}$".

Claims

1. A process for bleaching a miscella comprising a vegetable oil by contacting the miscella with a bleaching agent, **characterised in that** the bleaching agent used is bleaching earth in granular form with an average particle size of at least 0,5 mm.

2. The process according to claim 1, **characterised in that** the granules are used as a stationary phase in a packed column through which the miscella to be bleached is flown.

3. The process according to claim 1 or 2, **characterised in that** the vegetable oil to be bleached is cottonseed oil.

4. The process according to claim 1 or 3, **characterised in that** the used bleaching earth is regenerated by extraction with an alkane/polar solvent mixture.

5. The process according to claims 1, 3 or 4, **characterised in that** the used bleaching earth is regenerated by extraction with hexane/anhydrous ethanol mixture.

6. The process according to claim 1 or 2, **characterised in that** the vegetable oil to be bleached is soyabean oil.

7. The process according to claims 1 or 6, **characterised in that** the used bleaching earth is regenerated by extraction with a ketone.

8. The process according to claims 1, 6 or 7, **characterised in that** the used bleaching earth is regenerated by extraction with aqueous acetone.

9. The process according to any of the preceding claims, **characterised in that** at least part of the bleaching agent consists of spent bleaching earth which has been regenerated according to claims 4, 5, 7 or 8.

10. The process according to any of the preceding claims, **characterised in that** two or more packed columns are used and that the miscella is bleached on these columns in a counter-current process.

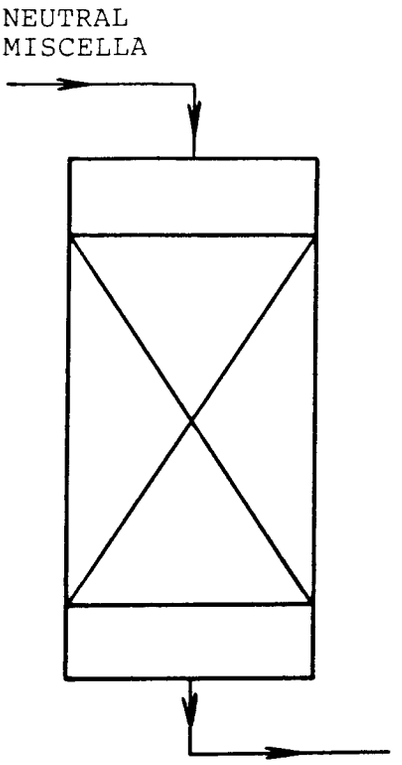
NEUTRAL
MISCELLA

Fig. 1.

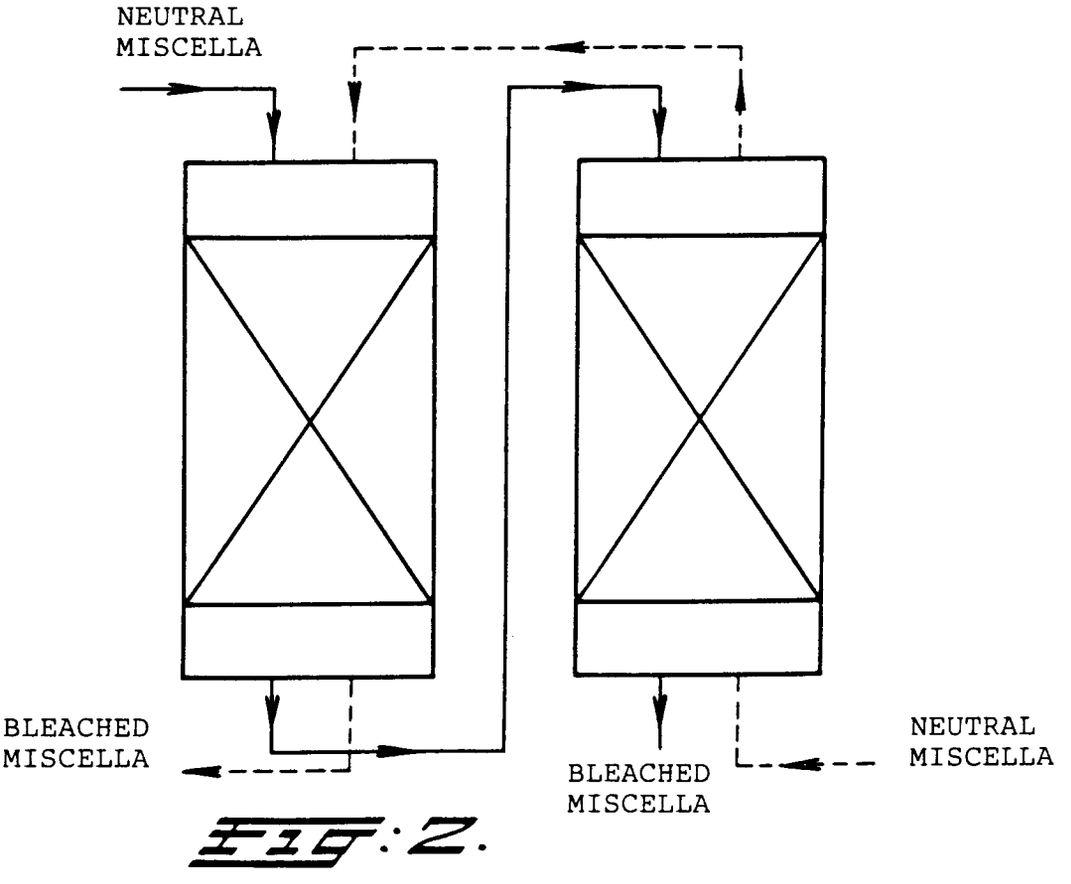
NEUTRAL
MISCELLA
BLEACHED
MISCELLA
BLEACHED
MISCELLA
NEUTRAL
MISCELLA

Fig. 2.