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User's Guides of Activated Clay

TYPES OF PRODUCTS AND THEIR PROPERTIES

Activated Clay has been produced as two types applicable for different processes. First is the granule type for the percolation process and the second is the powder type for the batch operation.

1. GRANULAR TYPE

1.1 Shacnite 136

SHACNITE 136 is applicable in petroleum processing such as catalysts for acid-catalyzed reactions and as absorbents for the decolorization of the petroleum/petrochemical products. These catalyzed reactions are heavy oil cracking, condensation, polymerization, alkylation, dehydration and isomerization in a fixed bed system. The amounts as well as the strength distribution of acidity were finely controlled according to the utilization-purposes of the reactions. This is very effective for the removal reaction of olefins in the BTX stream by dimerization reaction.

1.2 Shacnite 136A

SHACNITE 136A has more acid amount than SHACNITE 136 for long-term use. This product is recommended for the use of BT refining process, in which the feed stream has lower Bromine Index.

1.3 Shacnite 136N

In the process for the naphtha or heavy oil cracking, when BTX were extracted from the product, it is required to remove residual sulfolane (400 ~ 1200 ppm) from the distillation stream. SHACNITE 136N was prepared to remove this residual sulfolane up to the non-detectable level by Gas Chromatography equipped with FID.

1.4 Fuller's Earth Clay

At the end treatment of light hydrocarbon such as kerosene and jet fuel, FULLER'S CLAY has been used for the removal of the colored impurities and the moisture content.

2. POWDER TYPE

Powder types are produced for the batch process for the uses of oil/fat and petroleum products.

2.1 Oil and Fat Refining

Oil and fat contain impurities like free fatty acid, phosphatides, coloring matter and others, which could be refined by activated clay through their bleaching process. The activated clay has the higher specific surface area and also higher selectivity of the colored matter and others to be absorbed. For the refining of the oil and fat, the application procedure depends strongly on the feed properties, such as nature of oil/fat.

2.1.1 DA-P1

This is the versatile Grade for the edible and technical oil/fat.

2.1.2 DA-P2

This was mildly activated for the use of easy-to-bleach from coconut, palm kernel, cottonseed and soybean.

2.1.3 DA-P10

This was activated for the use of wastewater treatment as flocculating agent and adsorbent for the removal of the colored matters in wastewater.

2.1.4 DA-P100

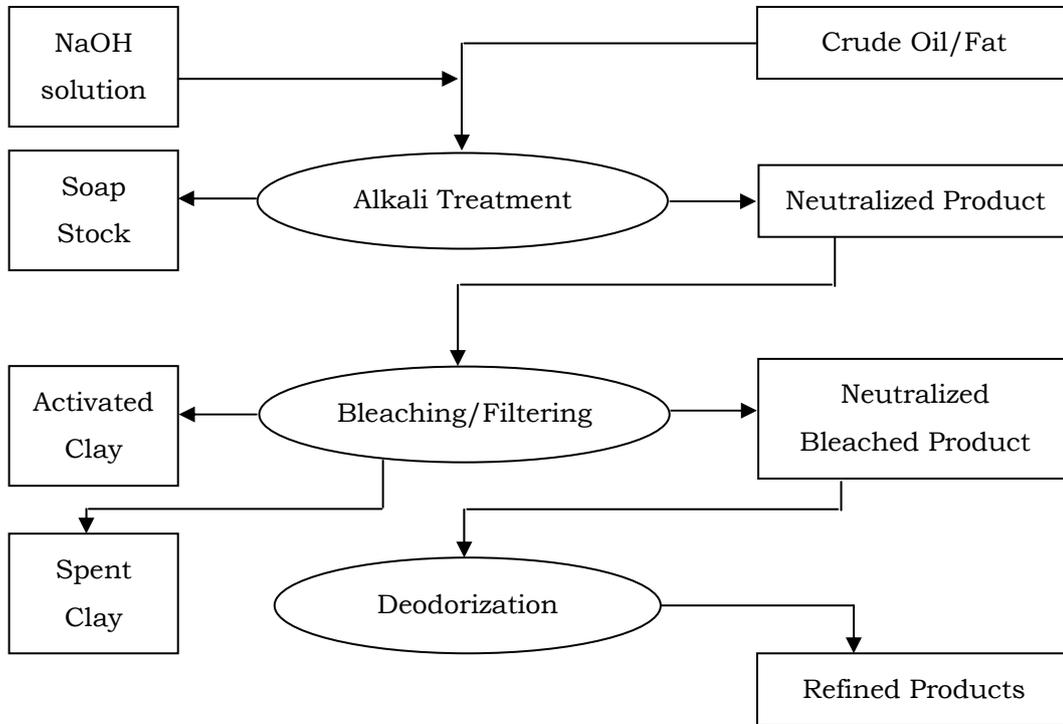
This clay was activated for the use in petroleum products such as electrical insulating oil, lubricating oil, recycled oil and others to improve the quality. (60 ~ 200°C).

Because the decoloring powder of the activated clay depends strongly on the feeds, it is recommended to ask and consult the manufacturer.

The following two processes can be carried out for the refining.

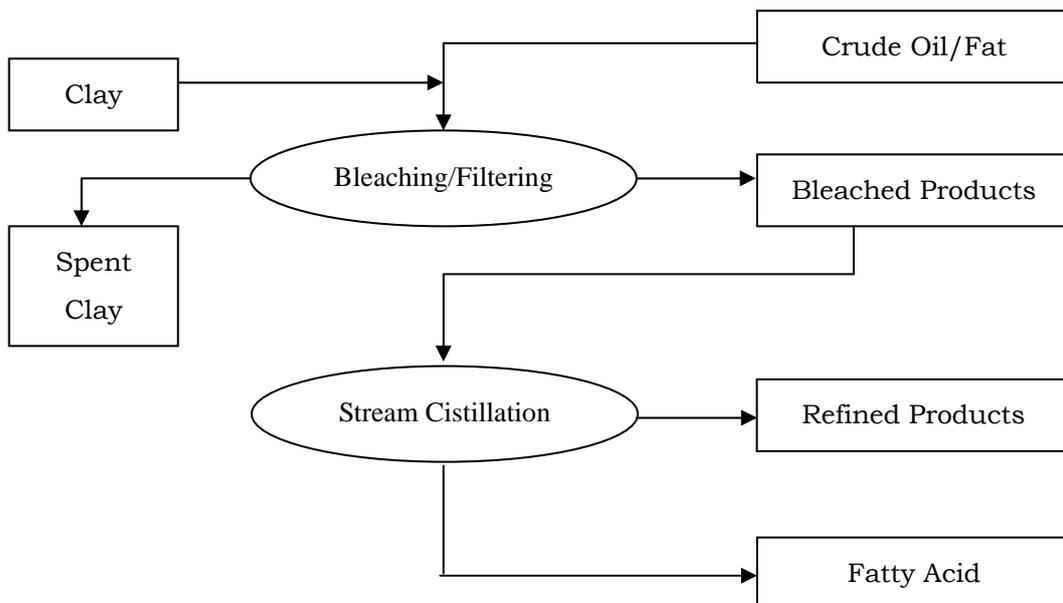
1. ALKALI REFINING

Bleaching after Chemical Treatment (Neutralization of oil/fat)



2. PHYSICAL REFINING

Bleaching without Chemical Treatment (Neutralization of oil/fat)



The **bleaching efficiency** depends strongly on the history of crude feed, from which the applying temperature of the bleaching and the amount of the clay are sometimes determined. For example,

1. bad-harvested / unfavorably stored raw,
 2. long-stored, overheated and/or
 3. extracted one
- require the different conditions of the treatment.

The **pretreatment** ahead of the bleaching gives better results. Settling, filtration and centrifuge for the raw materials remove suspended large particles, water and brine. For the removal of the finely dispersed particles, **acid-treatment** is required for some kind of oil For example,

1. Palm oil by phosphoric acid or citric acid
2. Linseed oil by sulfuric acid

Alkali solution of caustic soda is good for the removal of the colored matter through forming soap from the fatty acid.

The **bleaching process** is actually performed under the vacuum or in an open vessel. The vacuum provides advantages, like low temperature drying of oil, moisture removal from the clay and the avoidance of contact with the oxygen in the air.

Activated clay is fed to oil heated up near bleaching temperature in the vessel.

In a continuous process, the activated clay is premixed with a portion of oil, which is continuously introduced to the bleaching vessel. The bleaching temperature ranges between 80°C and 150°C. Bleaching for the feed that is difficult to be bleached requires higher temperatures.

Bleaching at a higher temperature, however, causes increase of free fatty acid of the bleached oil. Absorption of coloring matters on activated clay can be equilibrated within 30 minutes under sufficient agitation. The grade and dosage of activated clay have to be selected economically and technically through laboratory test. After bleaching, spent clay is separated from bleached oil by filtering with a filter press. Particle size distribution of 's Activated Clay was prepared for giving fast filtration and low oil content in spent clay cake for every filtering equipment.

2.2 Refining of Petroleum Products

The oil stocks to be treated by clay are electrical insulating oil, turbine oil, heavy duty lubricating oil and recycled oil in rolling mill, which can be improved for their color, oxidation stability, emulsion characteristics, copper corrosion, etc. by using activated clay.

Liquid paraffin and paraffin wax are refined by activated clay after sulfuric acid treatment for improvement of color, acid wash color and ultraviolet absorbance.

Light distillate of petroleum, jet fuel, kerosene and light oil are finished by percolator of Fuller earth clay expecting improvement of color and moisture content with 10-60 of hourly space velocity at room temperature.

Aromatic hydrocarbons extract, so-called BTX, can be improved its acid wash color and bromine index by using SHACNITE 136 series catalysts percolator with liquid phase under elevated pressure at 150 - 200°C of temperature by 1 - 3 of hourly space velocity.

There are two general processes for refining of petroleum products by activated clay.

1. Contact Process

Oil stock is contacted with activated powder clay in a vessel like bleaching of oil and fat. In case of refining of recovered lube oil, liquid paraffin and paraffin wax, oil stock is treated by sulfuric acid (0.5% to 5% of concentrated sulfuric acid by weight under slow agitation at room temperature to 50°C). The oil is left to settle after acid tar floks become larger during slow agitation and separated from precipitated acid tar sludge prior to activated clay treatment.

Activated clay treatment is carried out for 30 - 45 minutes at a temperature between 60 and 225°C. Treating temperature by activated clay is recommended as follows.

Electrical Insulating Oil	:	60 - 80°C
Lubricating Oil	:	100 - 250°C
Liquid Paraffin	:	70 - 100°C
Paraffin Wax	:	90 - 120°C

In case of high temperature treatment (more than 150°C), oil should be processed in an inert gas or superheated steam and cooled down to approximately 100°C before filtration to avoid oxidation of the oil.

2. Percolation Process

Oil stock is treated with passing through a fixed bed of granular activated clay. The operation factors on this process are a granular size of clay to be used, phase of fluid, temperature and pressure of bed, fluid velocity through bed or retention time in bed and life of bed. Outlines of factor have been given as aforesaid. Granular size of clay has to be selected to minimize pressure loss of fluid and to maximize the effect coming from the viscosity and velocity of fluid. Velocity of fluid and life of clay bed should be evaluated by laboratory test to decide an optimal size of bed and a frequency for replacement of bed by comparing with the standard activated clay.

2.3 Quality Control of the Clay

The contacting of clay with mineral acid is the beginning for the activation of raw clay. The activation results in the removal of aluminum, magnesium, iron and other cation from clay mineral structure, which depend on the treatment conditions and the raw clay properties. The treated clay gives the increased surface area, the improved porosity and the chemical/catalytic activity.

Washing process after activation is to remove acid soluble matters and excess acid from acid-treated product. Maximum content of free acid is controlled to be 5 mg of KOH value (the consumed quantity of KOH to neutralize a hot-water-extract from 1 g of product).

Washed semi-product is filtered by filter press to provide a filter cake with low and uniform moisture for minimization of fuel consumption and of a variation of moisture in clay.

Semi-product is operated to dry the filter cake in the range of 10% to 12% of moisture for a regular product and 3% to 5% for a low moisture product under strict temperature control to prevent our product from over-heating.

Dried product is milled with grinding machine combined with air-separator

and prepared for fine powder that is through 200 mesh sieve by minimum 85% and has a particle size distribution for minimizing of filtering rate of oil after application of 's Activated Clay for oil bleaching process.

In other processes, granular activated clay product, SHACNITE 136 series and 's FULLER'S EARTH are produced as 20 to 50 mesh and 30 to 60 mesh in the range of particle size.

The product is stored in tank for the final examinations in quality and packed in bag (25kgs) and other flexible bags.

Evaluation of bleaching activity of clay or performance test of product is most important in quality control. For the general evaluation of bleaching activity, neutralized **soybean oil is bleached at 110°C for 15 minutes** with the lot sample of the product and standard type sample corresponding to the grade to be produced. The color of the bleached oils is determined with a Lovibond Tintometer and the bleaching activity of lot sample is evaluated in comparison with standard type sample by their respective color of bleached oil. To meet practical demand, other oils and fats may be used under the various bleaching conditions, which follow the actual plant conditions closely.

A standardized lubricating oil stock is treated by the same way oil and fat is treated for evaluation of 's Activated Clay to be applied for refining of mineral oil. As occasion demands, oxidation stability, copper corrosion, ultraviolet absorbance and emulsion characteristics of bleached oil are determined besides color evaluation.

Column percolation techniques are applied under various conditions such as temperature, pressure, and space velocity for evaluation of refining and/or dehydration activity of SHACNITE 136 grade. Life of the clay bed is evaluated by determination of characteristics (Bromine Index and the level of the soluble coke fractions) of percolated oil and also through laboratory test.

The evaluation of product is to follow the actual plan conditions as closely as possible and then to obtain more accurate results in practical application. The followings are in subsidiary items to be evaluated for quality control.

1. Elemental analysis
2. Determination of specific surface area, porosity and average pore diameter by nitrogen absorption at liquid nitrogen temperature.
3. Acidity determinations (amount and strength distribution, hydrogen exchange capacity, free acidity)
4. X-ray analysis (deformation of the structure)
5. Determination of chemical characteristics (catalytic activity for the reaction and absorption capacity for the absorbate)
6. Others