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## Bleaching of Vegetable Oil using Organic Acid Activated Fuller's Earth (Bentonite Clay)

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**Abstract** - Vegetable oil is one of the basic food items which is consumed by almost every human being in this universe. Therefore quality of vegetable oil should be good enough so that it accounts for healthy life. In vegetable oil manufacturing there are four major steps involved which are neutralization, degumming, bleaching and deodorization. Among these steps bleaching is the very important and critical step because it ensures the good color and odor of vegetable oil. The famous method of bleaching in Pakistan is adsorption by inorganic acid activated fuller's earth (bentonite clay). Treatment with inorganic acid (Sulfuric acid and hydrochloric acid) activated bentonite clay is very efficient and shows satisfactory results. But there is a major disadvantage associated with its use. Fumes of hydrochloric acid or sulfuric acid are very dangerous for both the equipment and labor involved in manufacturing of vegetable oil. Therefore the safety of the labor and equipment is compromised. The major purpose of this research work is to give the alternative method for activation procedure of bentonite clay and this method should be the safest method for both the labor and equipment used in vegetable oil industry.

**Keywords:** *bentonite clay; surface activation; organic acids; adsorption; fourier transform infrared spectroscopy (ft-ir).*

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# Bleaching of Vegetable Oil using Organic Acid Activated Fuller's Earth (Bentonite Clay)

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**Keywords:** bentonite clay; surface activation; organic acids; adsorption; fourier transform infrared spectroscopy (ft-ir).

## I. INTRODUCTION

Specific properties of clay minerals and its derivatives made them valuable for their application in various fields. One of the various applications of bentonite clay is its use as a bleaching agent in vegetable oil industry[1]. Vegetable oil production consists of various manufacturing steps and among these steps refining is the most important and critical step. Refining is based on several stages including neutralization, degumming, bleaching and deodorization. Among these four stages bleaching is very complicated step because quality of cooking oil is based on this step. Furthermore appearance, taste and color of vegetable oil are also depending upon this step. On commercial basis there are two types of fuller's earth, natural and activated[2]. Activated fuller's earth is preferred because of its performance due to higher adsorption capacity. Adsorption capacity of fuller's earth

is enhanced by various treatment techniques. Among these techniques acid treatment is the most practiced technique in vegetable oil industries. Other techniques include alkali treatment and organic treatment, but these techniques are not effective enough for enhancing the adsorption capacity of fuller's earth at desired level[3].

In acid treatment normally sulfuric acid and hydrochloric acid is preferred but there are some issues related to it. Fumes of these acids are very dangerous for the labor and equipment involved and moreover some traces of hydrochloric acid remain in vegetable oil after treatment and when this vegetable oil is consumed by human beings it may be carcinogenic if consumed at some extent. It can cause liver and stomach disorders in old ages[4]. So for avoiding these serious issues, treatment with organic acid is introduced in this research paper. Organic acid treatment of fuller's earth is the safest mode of activation because it has no harm to labor, equipment as well as health of human being. Major motivation of this research is the safety of labor and equipment in vegetable oil industry. Average life of equipment is increased which ultimately increase the overall economics of the process[5].

Application of fuller's earth for the bleaching of vegetable oil involves certain issues like filtration, retention of oil and environmental hazards. These problems may arise if the amount of fuller's earth is used in excess amount i.e. more than its requirement[6]. Excessive amount of fuller's earth causes oil losses which ultimately cause oil retention and creates problem during filtration. Moreover the type of clay and their particle size is also an important factor in filtration efficiency. If particles are at large distance from each other than filtration will be easy and if particle size is compact the filtration will be difficult and take more time[7].

There is a lot literature on surface chemistry and modification techniques of fuller's earth. The basic information in the literature is mainly focused on fundamental structural unit and surface characteristics of bentonite clay and their application in process industries. Important parameters which affect the treatment techniques are acid concentration, treatment time, temperature effect, Solid/liquid ratio and moisture content[8].

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## II. MATERIAL AND METHODS

### a) Sample preparation

Fig.(1) shows the steps for methodology involved in this research. The fuller's earth for activation and analysis was taken from the "Good Earth" company in Sheikhpura (Pakistan) which manufactures the activated fuller's earth or bleaching earth for different vegetable oil processing industries[9]. 100 grams of weighed fuller's earth are taken separately for sample preparation. Each of these samples was ground to powder form. Then these samples were treated with four organic acids which are most suitable for activation according to the literature[10]. These four organic acids were oxalic acid, phosphoric acid, citric acid, acetic acid. These acids with different concentrations are used but 1N acid solution is optimum for treatment and showed satisfactory results so 1N acid solution is used for treatment[11].

### b) Phosphoric acid treatment

Phosphoric acid is best recommended for the removal of aluminum ions. It reacts with aluminum and aluminum phosphate is formed, which settles down and easily removed. Literature shows that treatment of clay in Algeria with 70% acid dosage and 24 hours residence time, 66 % conversion of bleaching earth is achieved. Concentration of acid and residence time both highly depends upon the nature of the clay. When maximum conversion is achieved then bleaching earth is washed with water so that acid is completely removed. Then in next step it is dried in 200°C for almost complete removal of moisture. Then its adsorption capacity is tested by treating with edible oil[12].

### c) Oxalic acid treatment

Second sample of fuller earth is then treated with oxalic acid. According to literature survey oxalic acid is good for removal of both aluminum and iron. Research on Brazilian clay reveals that 81% oxalic acid dosage and 24 hours residence time gives 74% conversion. In this research different dosages of oxalic acid are selected to obtain maximum conversion. After acid treatment it is washed with water and dried at 200°C for maximum removal of moisture. Finally adsorption capacity is tested by treating it with edible or vegetable oil[13].

### d) Citric Acid Treatment

Citric acid bleaching is mostly recommended when soybean oil refining is required. Literature does not reveal conversion of bleaching earth using citric acid. Therefore different dosages of acid will be checked for maximum conversion of bleaching earth. When maximum conversion is achieved then same procedure of washing and drying is adopted as mentioned above and then finally its adsorption capacity is tested by oil treatment[14].

### e) Acetic Acid treatment

Acetic acid is recommended when palm oil refining is required. According to literature 1N acetic acid with 0.5 hours' time give 66% conversion. Acetic acid is recommended for removal of magnesium. By varying its concentration and residence time conversion rate also changes. So that concentration will be selected at which maximum conversion occurs. After that same procedure of washing, drying and oil treatment is adopted for testing adsorption capacity[15].

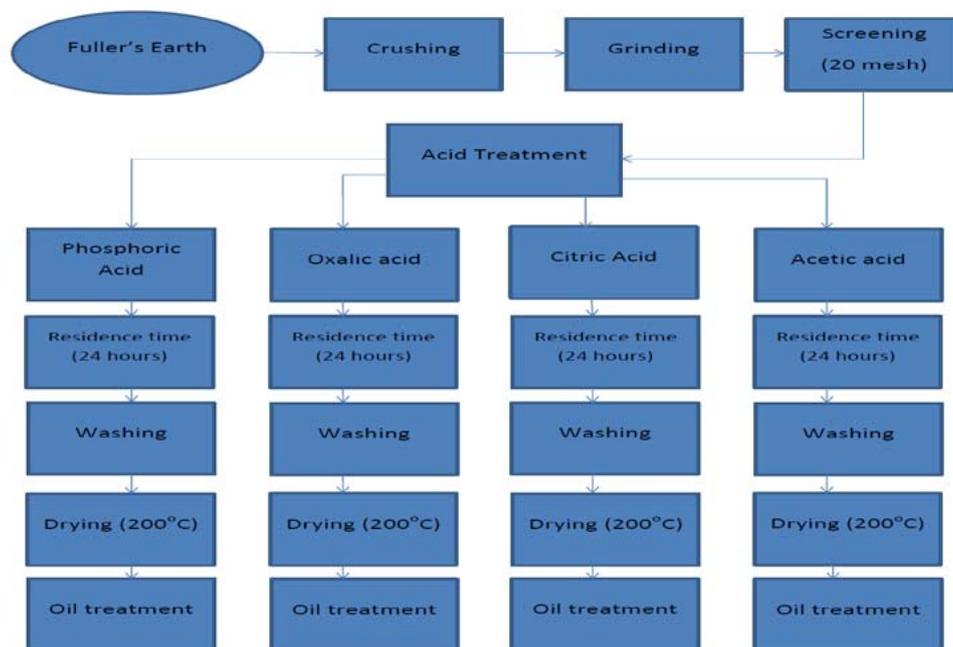


Fig.1 : Research methodology steps

### f) Method of Characterization

The analysis technique which is used for the characterization of both the untreated/treated clay is the Fourier's Transform Infrared Spectroscopy (FT-IR). The quality features of infrared spectroscopy are one of the most effective tools of this vast and advanced method for characterization[14]. For so many years, large research has been done in terms of the basic frequencies for absorption (also known as group frequencies) which is very important tool for understanding of the structure and spectral co-relation of the associated molecular vibrations. Application of this precious information at serves to be a combination of both art and science[16].

## III. RESULTS AND DISCUSSIONS

After making four samples of organo-clays, bleaching capability of these clays have been tested by

their application on used vegetable oil. Four samples of 50 ml of used vegetable oil havemade and 2 gram of each organo-clay has been poured in it. After heating it at 200°C and 24 hours residence time the oil has been filtered and its results have been analyzed through FT-IR technique. Results of this oil have been compared with the un-used or new vegetable oil. For this purpose used and unused oil samples of SUFI vegetable oil has been taken. SUFI vegetable oil is a very popular and most consumed brand in Pakistan.

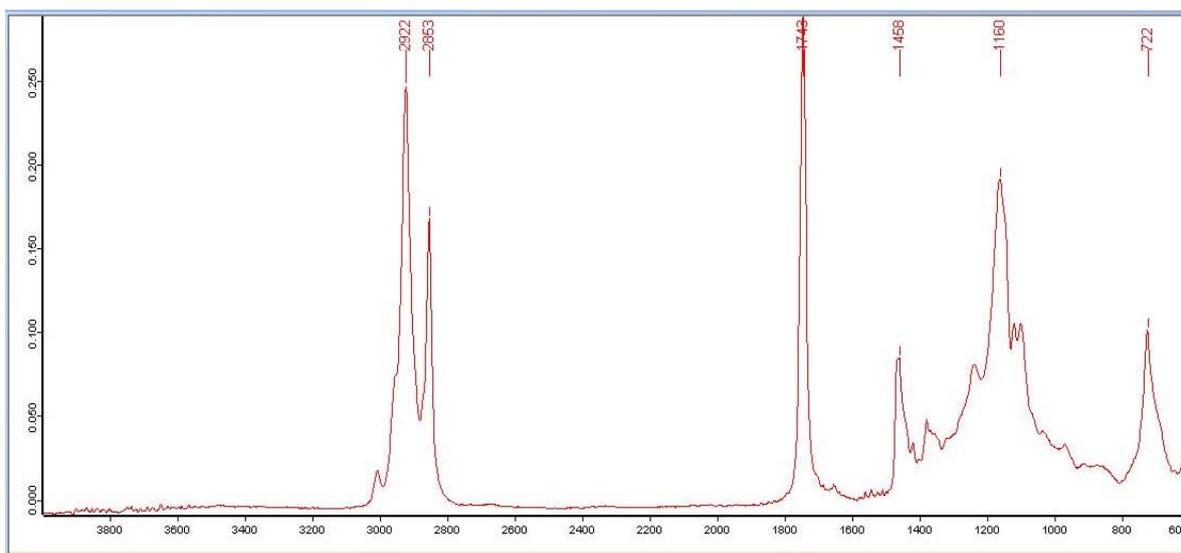


Fig. 2 : Analysis of used vegetable oil

Analyses of used vegetable oil (fig.2) before treatment shows that greater number of saturated hydrocarbons is present i.e. straight chain alkane, alkene and their respective derivatives. If we go from right to left sharper peaks mostly formed between the ranges (1000-2000  $\text{cm}^{-1}$ ), between 2000-2500  $\text{cm}^{-1}$  no peaks are formed and after 2500  $\text{cm}^{-1}$  some shaper peaks are formed showing straight chain methane-oxygen functional group and from 3000-3500  $\text{cm}^{-1}$  no larger and sharper peaks are formed. If the analysis of used vegetable oil is compared with analysis of unused vegetable oil (fig.3) than it can be observed that peak pattern in the range up to 3000 $\text{cm}^{-1}$  is almost the same but after 3000  $\text{cm}^{-1}$  although the sharper peaks are not formed in both cases but the intensity of spectrum is greater in used vegetable oil. In unused vegetable oil spectrum is lying approximately on x-axis line showing

approximately zero intensity while the spectrum analysis of used vegetable oil shows little bit higher intensity of spectrum after 3000  $\text{cm}^{-1}$ .

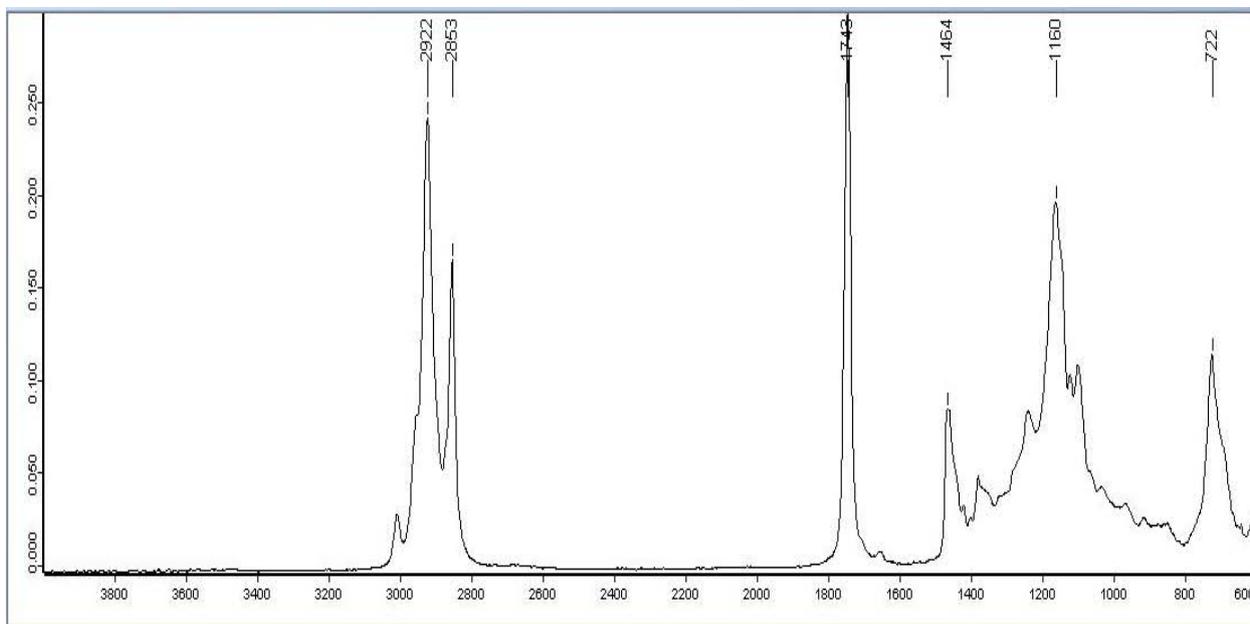


Fig. 3 : Analysis of pure vegetable oil

Analysis of acetic acid activated clay treatment of vegetable oil (fig.4) shows variation in peak pattern if compared with the result of pure vegetable oil analysis. Peak pattern obtained in the range up to 1500 cm<sup>-1</sup> is almost the same as in pure vegetable oil. In the range between 1500 to 2000 cm<sup>-1</sup> intensity is higher and then

from 2000 to 3000 cm<sup>-1</sup> spectrum pattern is almost the same as in pure vegetable oil. After 3000 cm<sup>-1</sup> again intensity rises up to 3500cm<sup>-1</sup> and starts declining afterwards, showing variation in adsorption spectrum in comparison with pure vegetable oil.

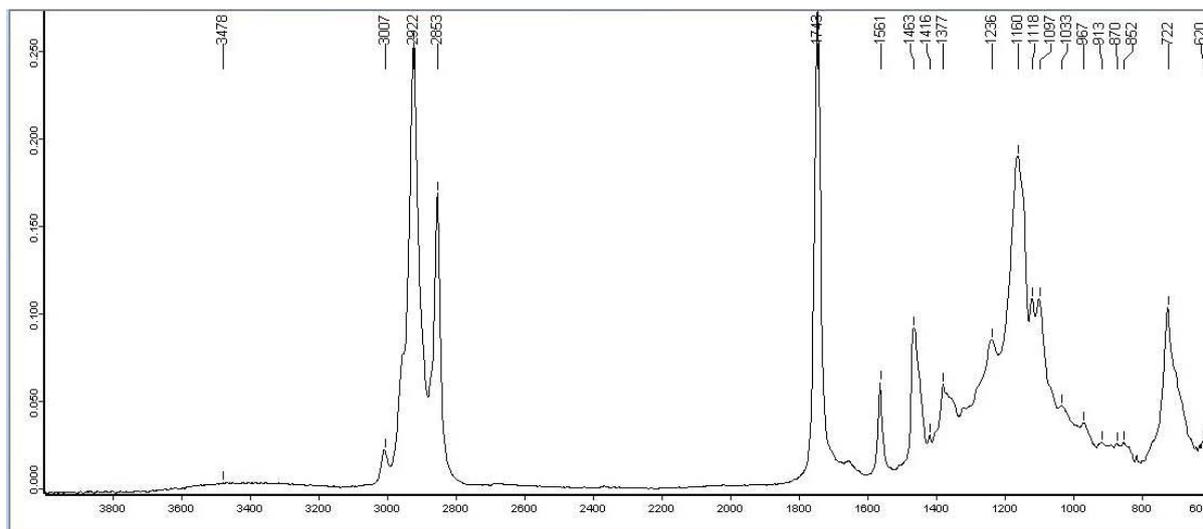


Fig. 4 : Analysis of Acetic acid clay treatment of used vegetable oil

Analysis of phosphoric acid activated clay treatment of used vegetable oil (fig.5) shows that intensity variation throughout the spectrum. At some regions intensity is same as in between 1500-2000 cm<sup>-1</sup>. However in the beginning of the spectrum more peaks are formed showing greater number of straight chain hydrocarbon functional groups. Sharper peaks are formed mostly in the range 600-750 cm<sup>-1</sup> and 1000 to 1100 cm<sup>-1</sup>. In range 2600-2900 cm<sup>-1</sup> intensity degree of

sharpness of peaks is reduced if compared with pure vegetable oil sample and after 3000cm<sup>-1</sup> spectrum intensity is little higher than pure vegetable oil sampled.

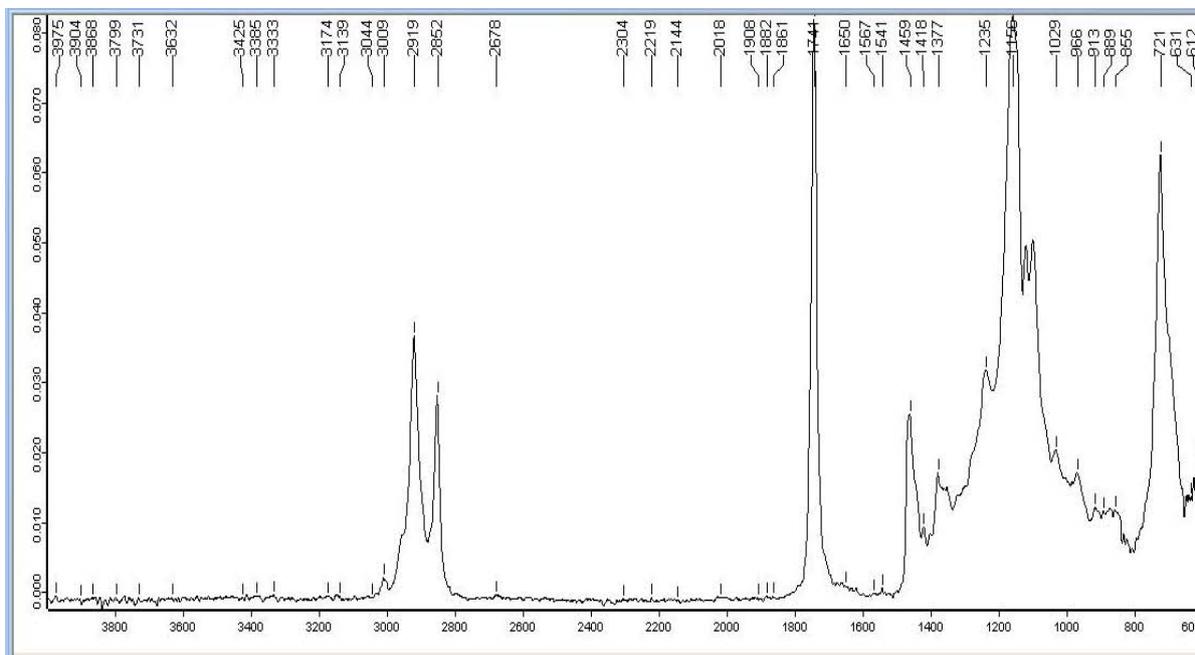


Fig. 5 : Analysis of phosphoric acid activated clay treatment of used vegetable oil

Analysis of citric acid activated clay treatment of used vegetable oil (fig.6) shows variation in spectrum intensity if compared with pure vegetable oil sample. Citric acid activated clay shows almost same results as in fig.5 except at some regions. At the beginning same peak pattern and intensity has been observed clearly as in phosphoric acid treatment but 1800  $\text{cm}^{-1}$  intensity is higher up to 2400  $\text{cm}^{-1}$  and after 2400  $\text{cm}^{-1}$  it starts declining and after 2800  $\text{cm}^{-1}$  same spectrum intensity is

observed. In the range between 3000-3200  $\text{cm}^{-1}$  intensity is approximately at zero level and almost same as in pure vegetable oil but after 3200  $\text{cm}^{-1}$  spectrum intensity again starts increasing and till end intensity increases to much higher level as compared to previous samples showing large variation in spectrum intensity in this region. Large variation in this regions shows that power of bleaching of citric acid is more intensive than its requirement.

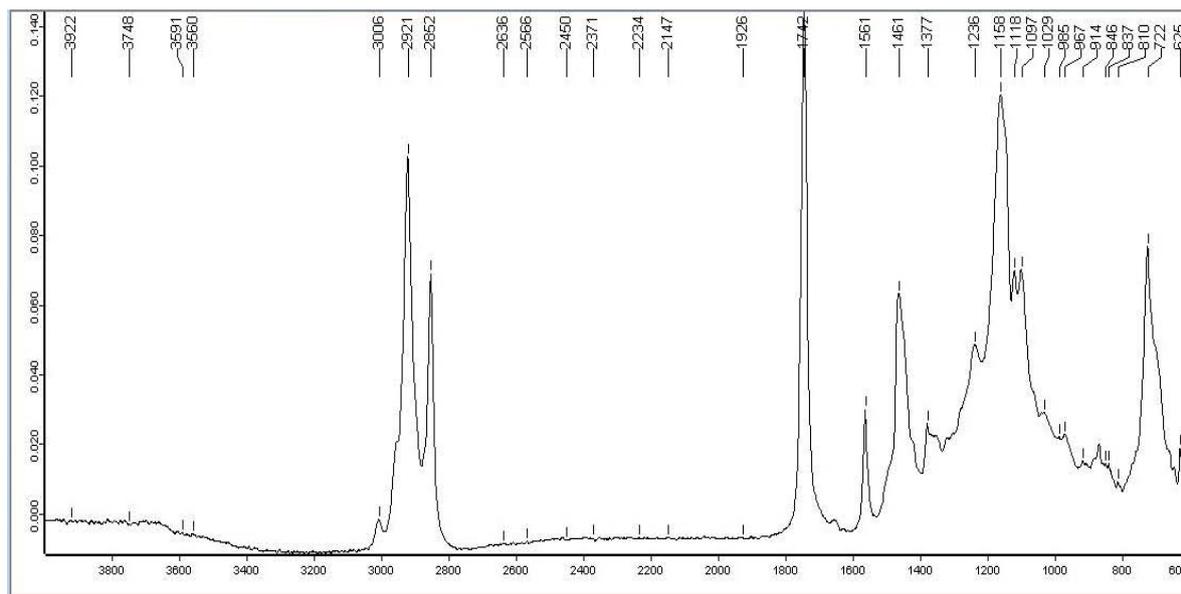


Fig. 6 : Analysis of citric acid activated clay treatment of used vegetable oil

If the oxalic acid activated clay treatment of used vegetable oil (fig.7) is compared with pure vegetable oil sample the it is clearly observed that the spectrum intensity from beginning to end and peaks

formation and their intensity throughout the same. Hence oxalic acid activated purifies the used vegetable oil more effectively as compared to other three organo-clays.

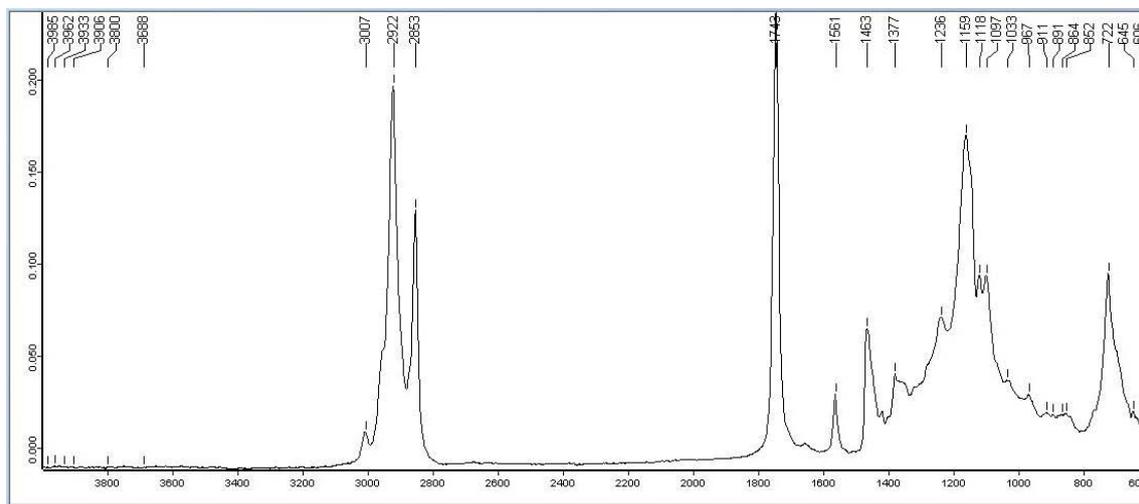


Fig. 7 : Analysis of oxalic acid activated clay treatment of used vegetable oil

#### IV. CONCLUSION AND RECOMMENDATIONS

From the above discussion if four of these organo-clays are considered the best organo-clay which is suitable for the treatment of bleaching process in vegetable oil industry in Pakistan is Oxalic acid. Oxalic acid treated clay analysis shows that bleaching power of oxalic acid is effective enough that it can purify the used vegetable oil and convert it into approximately new vegetable oil. So if this clay is used in bleaching portion in vegetable oil manufacturing industry, it will show more satisfactory results in comparison with inorganic acid treated clay. If some further research work is carried out, these organo-clays can be used as an adsorbing agent in purification of used lube oils as well. Therefore more research is required in this regard.

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