**Food Oils and Fats: Chemistry & Technology**

**Professor H N Mishra**

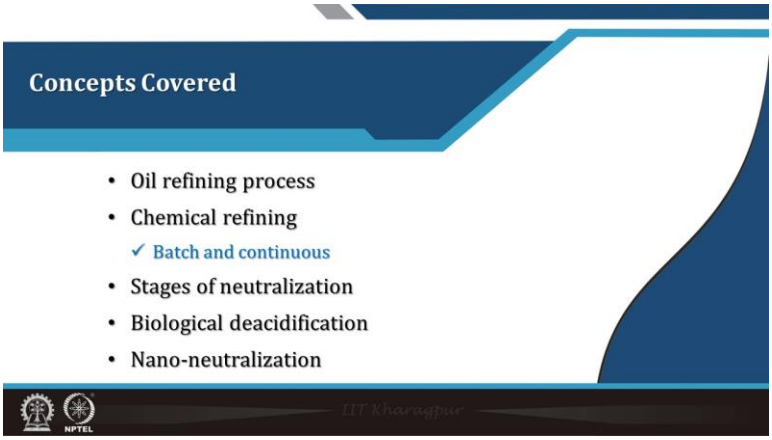
**Agricultural and Food Engineering Department**

**Indian Institute of Technology Kharagpur**

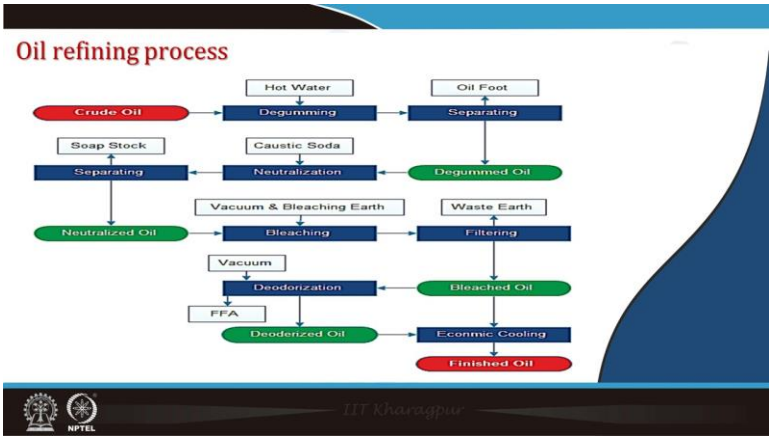
**Module 6: Edible Oils Refining**

**Lecture 28: Chemical Refining and Neutralization**

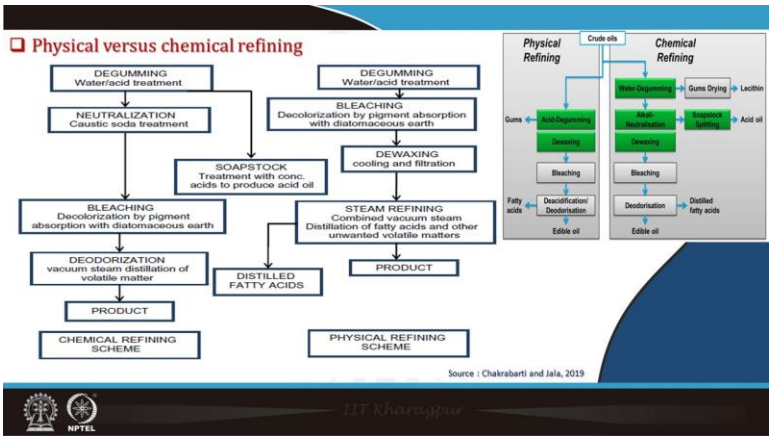

Hello everybody. Namaskar. Now, we are in the lecture 28. In this lecture in the next half an hour or so, we will discuss about Chemical Refining and Neutralization of Food Oil.




The concept that I will cover in this lecture includes oil refining process, then chemical refining both batch and continuous, then stages of neutralization, biological deacidification, and nano-neutralization which is a new concept in the neutralization process.

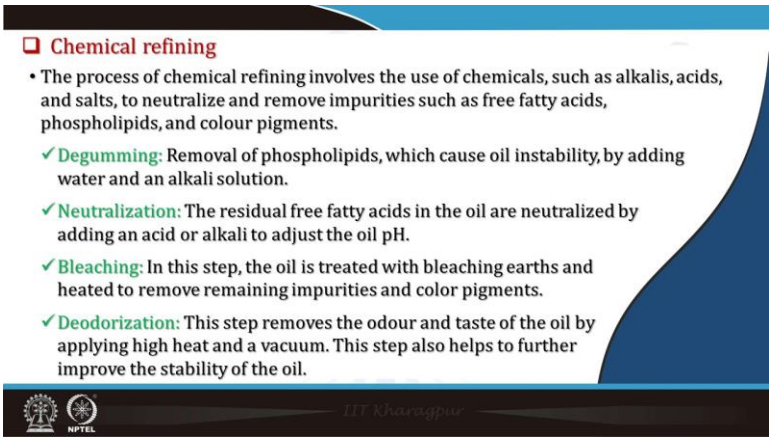



This is the overall refining process earlier also in the two lectures I showed you. So, today we will, in the degumming and filtration we already studied. So, this degummed oil is sent for the neutralization step or that is the where it is treated with the alkali caustic soda. So, this is also known as chemical refining. So, we will concentrate today in this lecture on the neutralization aspect or chemical refining.

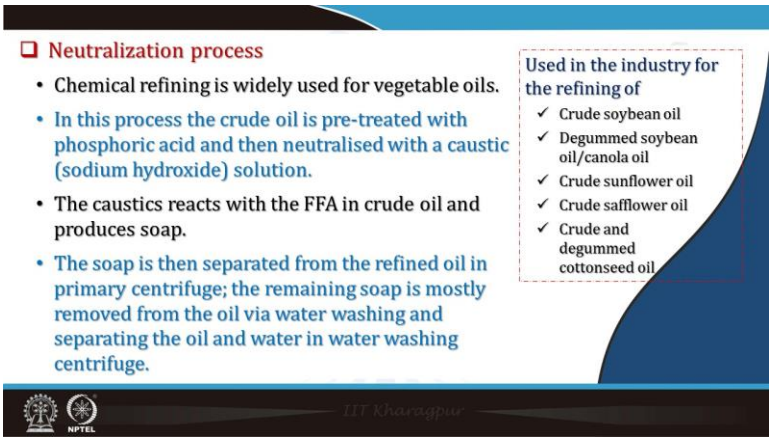



So, basically in the oil refining is a generic term, that is the different stages, that is treatment processes or steps involving the treatment of crude oil starting from the extraction process till it is finally, packaged and sent to the industry for food usage it is oil is passed through various steps. So, this is told as refining process, but this refining further again may be a two process one is the chemical refining and there is a physical refining. The neutralization which are talking about neutralization basically that is the removal of the free fatty acids, but when we talk about chemical refining or physical refining, the oil is treated with the different as you can see here in the last class when we were thing degumming there also I told you, but here now it is specific treatment that in the when this oil, that is it is degummed, water degummed, the gums and lecithins are removed, then it is degummed oil is passed through alkali neutralization, soap stock is removed then it is sent to the dewaxing, bleaching and deodorization. So, all these steps together when it is given to the oil it is known as chemical refining process.

On the other hand, the physical refining you can see here that is although except that that is the acid degumming is done, dewaxing is done then bleaching and then finally, deodorization, this alkali treatment is not done here though no chemical involvement. So, here the free fatty acids are removed by deacidification is done during deodorization process, distillation process. So, that is a physical refining. So, this is the difference between physical refining and chemical refining.

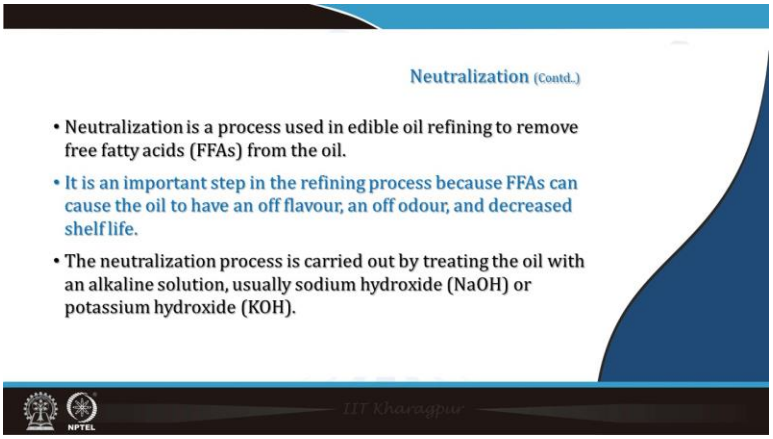



So, when we talk about chemical refining as a whole so, means that is, this process has four major steps in the chemically refined oil means it will be subjected to degumming for the removal of phospholipids then it is neutralized where the free fatty acid, in the neutralization step particularly the free fatty acids are removed and oil becomes neutral. And obviously, the acid free fatty acid so, it is treated with the alkali to neutralize for the removal, then comes the neutralized oil. It bleached in the bleaching step, the oil is treated with the bleaching earth and heated to remove remaining impurities and color pigments. Then deodorization process finally, this step removes the order and taste of the oil by applying high heat and a vacuum. This step also helps to further improve the stability of the oil. So, of course, degumming we have studied earlier in detail bleaching and deodorization also we will study in details in the next classes. In today's class we will concentrate about neutralization aspect itself, but sometime in the industry all these process that is in the continuous operations are all these processes are together. So, where necessary we will tell about briefly about the other aspects as such.



So, the neutralization process that is neutralization means the oil here, that is the it is also called chemical refining and then in this process the crude oil is pretreated with the phosphoric acid and neutralized with caustic soda that is pretreated with phosphoric acid means that is degummed oil. Degummed oil is taken for neutralization and normally there is, in the neutralization obviously, either potassium hydroxide or sodium hydroxide a known amount depending upon the type amount and type of fatty acid that are present in the oil, a particular amount of a known amount of caustic solution is used. This caustic reacts with the free fatty acid in the earlier classes if you remember we discussed the saponification process. So, when this free fatty acid that are there in the oil, they react with the alkali potassium hydroxide or sodium hydroxide and they form sodium or potassium salt of the fatty acid and that is nothing, but soap. So, this soap is formed and then this soap is separated there is a remaining soap is mostly removed from the oil by either water washing or separating the oil and water in a washing centrifugal etc.

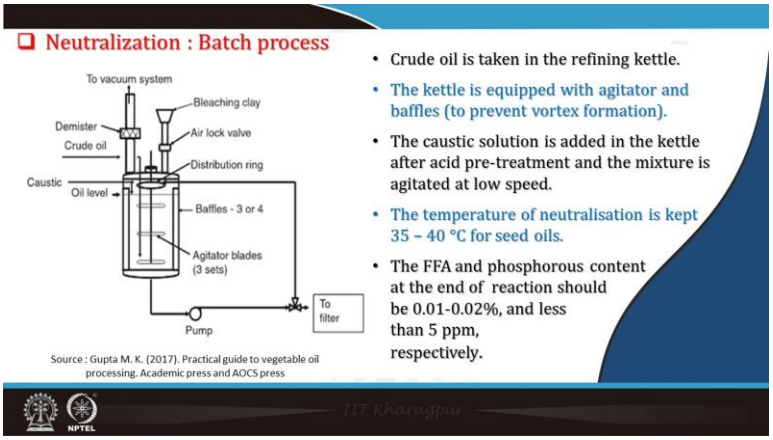
And this is mostly done in the industry for crude soybean oil, degummed soybean oil, degummed canola oil, crude sunflower oil, crude safflower oil, or crude and degummed cottonseed oil etc. All these are that is subjected to neutralization process before sending to the market.



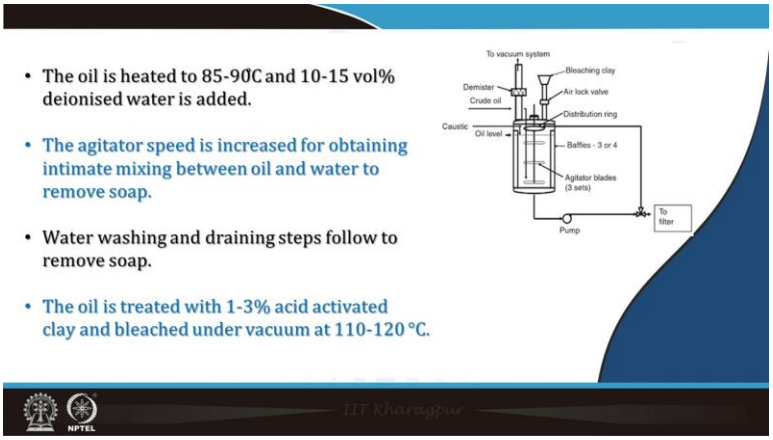

So, I hope it is clear by now to you that neutralization is a process which is used in edible oil refining to remove mainly the free fatty acids. It is an important step in the refining process because the free fatty acids can cause the oil to have an off flavor, an off order and decreased shelf life because if they are free fatty acids or the unsaturated fatty acids then it may cause auto oxidation and other problems and even the more amount of free fatty acid in the oil seriously affects the properties and characteristic frying properties, cooking properties, other conditions of the oil. So, the neutralization process is very important it is carried out by treating the oil with alkali solution as I told you usually sodium hydroxide or potassium hydroxide.

So, the process if you see that what is the neutralization process it may be batch or continuous. First, we will discuss a batch process. In the batch process, what is done there is a kettle which is provided with all accessories, it is basically a vessel which is called refining kettle or neutralization tank, and this kettle is equipped with an agitators and

biofuels to provide vortex formation etc. and other control and measuring instrument and accessories like temperature measurement, pressure measurement etc. they are provided with this.

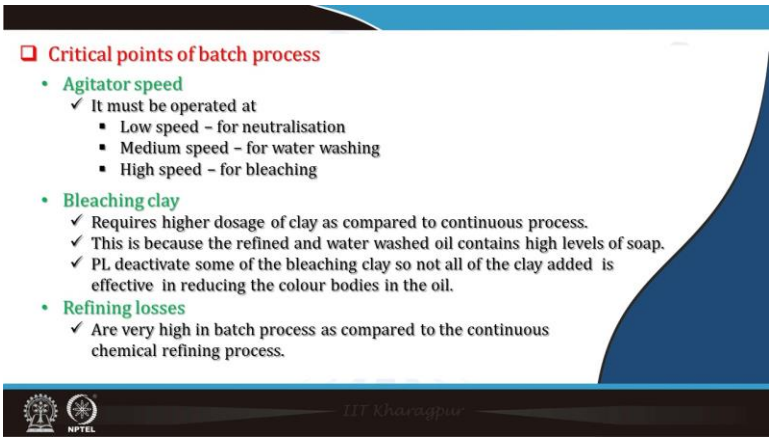



So, the caustic solution is added in the kettle that is after acid pretreatment and the mixture is agitated at a slow speed. The degummed oil is taken and, in this oil, the caustic solution is added. The temperature of the neutralization first for the seed oils. The oil enters at a temperature of around 35 to 40 degree Celsius in this and the free fatty acid and phosphorous content are at the end of the reaction should be as low as 0.01 to 0.02 percent and this free fatty acid and phosphorous at the end should be less than 5 ppm, respectively. So, accordingly depending upon how much is the free fatty acids initially present that is this amount of sodium or potassium hydroxide required can be calculated and then can be used.



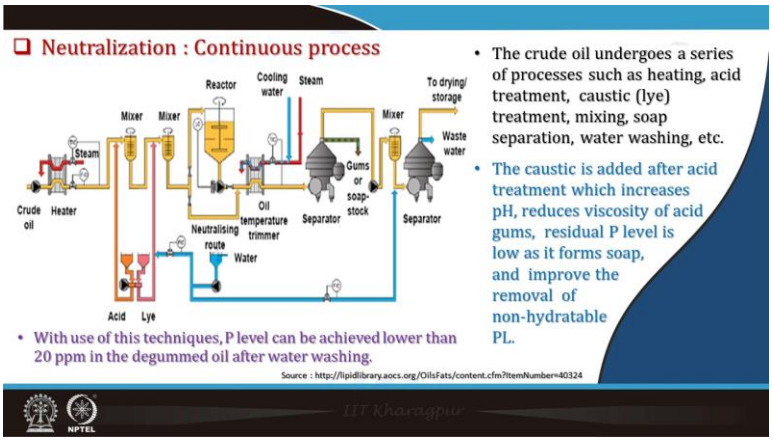

So, when the oil is sent to the reaction vessel, it is heated that is the reaction vessel is provided with the steam inlet and other assembly for heating the oil and measuring and indicating its temperature etc. So, oil is heated to 85 to 90 degree Celsius and once at that time 10 to 15 volume percent of the deionized water is added in the hot oil. The agitator speed is increased for obtaining intimate mixing between the oil and water to remove soap. After this is a washing step, that is once the soap is formed earlier in the neutralization process then its temperature is increased and then water is added and water

washing and draining steps are followed to remove the soap and even sometime 2 or 3 or 4 washings are done to completely remove all the soaps. The oil is treated with alkali solution 1 to 3 acid percent acid activated clay, the oil is bleached under vacuum at around 110 to 120 degree Celsius that is the after the neutralization, the activated clay is used. The details of this we will discuss in the next class.

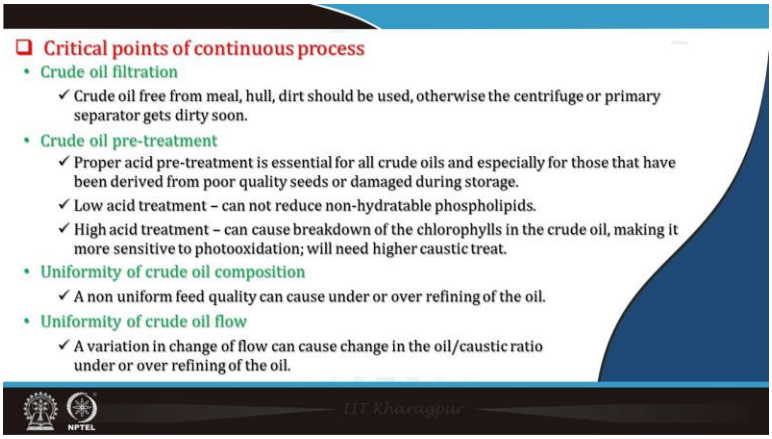



So, the critical points that need to be controlled and taken into consideration while this in the batch process of neutralization that is number 1, agitator speed in the reaction medium you can see in the figure that these are agitators etc. are provided. So, its speed this agitator must be operated at a low speed for neutralization at medium speed for water washing and high speed for bleaching. So, in all the 3 stages neutralization washing and the speed of the agitator must be properly adjusted so that because in the neutralization step it should be low that so that the soap is formed, it should not break it should not form emulsion. If you use a high speed, it will break.

Next is the bleaching clay, that is bleaching clay requires higher dosages of the clay as compared to the continuous process. This is because the refined and water washed oil contains high levels of soap. PL deactivate some of the bleaching clay so not all of the clay is that is added is effective in reducing the color bodies in the oil. Then refining losses are very high, comparatively high refining loss is there in the batch process as compared to the continuous processes.

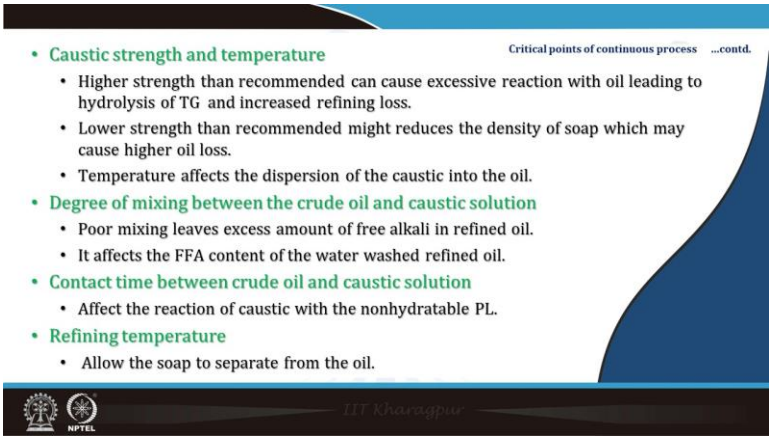


Now, the continuous process you can see here in the schematic. Here, that is the there are different mixers are provided, reaction vessels, cooling tower, steam, they are generating and introducing things. Oil temperature timers are there, then acid and alkali solution assembly for tanks, and assembly for the and all the systems required for proper mixing of the acid and alkali solution in the oil then a system for heating the oil. So, you this you see yellow line is that oil how it moves, these red lines, that is pink lines, acid and alkali solutions and the other is this water solution/ So, this crude oil undergoes, in this case in the continuous process continuously that is such as heating, acid treatment, caustic treatment, mixing, soap separation, and water washing because these are the steps. So, these are all continuous operations and in the system all the equipment and system are provided for all these processes. The caustic is added after acid treatment as you can see here in the schematic process. And it increases the pH, reduces viscosity of acid gums, residual phosphorus level is low as it forms soap and it improves the removal of the non-hydratable phosphor lipids. With the use of this technique, even phosphorus level can be achieved lower than 20 ppm if the degummed oil is water washed properly.

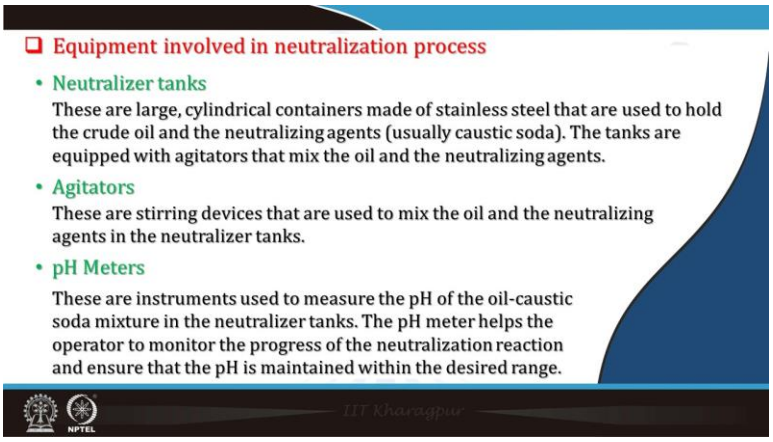



So, here some critical points which needs consideration that is the crude oil filtration is very important in the continuous processing operation. The crude oil should be free from any meal, hull, dirt, etc. Otherwise, the centrifuge or primary separators get dirty soon and it will require more and more refining losses that and there were more and more frequent cleaning and maintenance. Then crude oil pretreatment is another a very important step here. Proper acid pretreatment is essential for all crude oil and especially for those that have been derived from poor quality seeds or damaged during storage. So, the proper acid treatment so that these impurities etc. are properly removed. If you have a low acid treatment, cannot reduce the non-hydratable phospholipids, and a high acid treatment can cause the breakdown of the chlorophylls in the crude oil making it more sensitive to photooxidation and will need higher caustic treatment etc. So, that is again here that is the proper acid treatment pretreatment of the oil is very essential step, necessary step to get the good quality neutralized oil. Then uniformity of the crude oil composition, a non-uniform feed quality can cause under or over refining of the oil and it may be difficult that to know that what is the exact amount of potassium or sodium hydroxide, its concentration, how much it is used and process operation. So, uniform

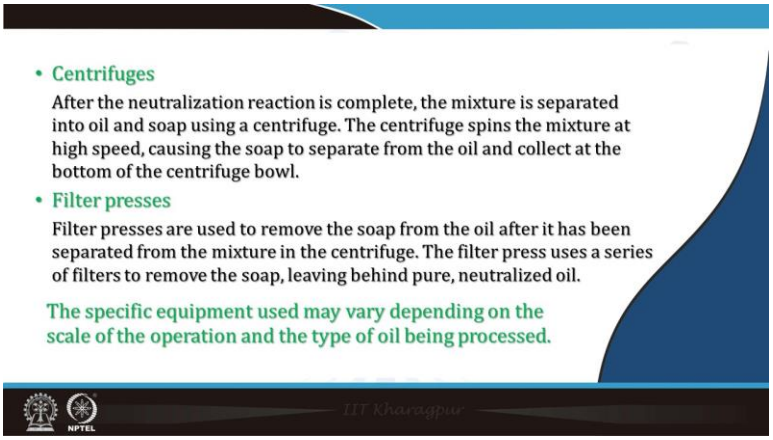
crude oil composition is important. Similarly, uniformity of the crude oil flow, a variation in change of the flow can cause change in the oil or caustic ratio under or over refining of the oil.




Then caustic strength and temperature, that is very again the even the caustic solution sodium hydroxide solution. Normally it is recommended that between 1 to 3 normal solution should be used and how much it is there, that is depending upon the fatty acid present, but the normality is important because if you use normality higher than the optimum recommended, it may cause hydrolysis of the triglyceride and generate rather more and more fatty acids. If you use a very weak solution less than 1 normal, then it may form the soap, proper soap formation may not be there, it may form rather weak emulsion and its separation from the oil may be a problem. So, this is very important that the proper strength, proper amount, quantity as well as strength of the solution of sodium hydroxide etc. should be followed. Temperature affects the dispersion of the caustic into the oil, so proper temperature also. Then degree of mixing between the crude oil and the caustic solution. Poor mixing leaves excess amount of free alkali in the refined oil, it affects the free fatty acid content of the water washed refined oil. Then another important aspect is the contact time between crude oil and caustic solution. It affects the reaction of the caustic with the non-hydratable phospholipids. Then refining temperature, it allows the soap to separate from the oil, so proper. So, it is very important these are the factors should be properly and optimum conditions must be used.

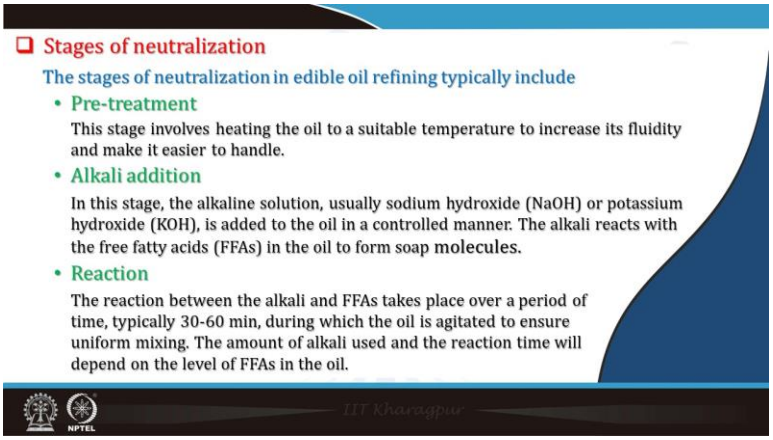


Then the equipment which are involved in the neutralization process as you could see in the schematic diagram in the earlier we showed. So, there are various equipment it is needed that is one is the neutralizer tank. These are the large cylindrical containers made of stainless steel that are used to hold the crude oil and the neutralizing agents like caustic soda. These tanks are equipped with agitators that mix the oil and the neutralizing agent properly. Agitators are stirring devices that are used to mix the oil and the neutralizing agents in the neutralizer tanks. Then pH meter, these are the instrument which are used to measure the pH of the oil caustic soda mixture in the neutralizer tank. The pH meter helps the operator to monitor the process or progress of the neutralization reaction and ensure that the pH is maintained within the desired range that is very important.

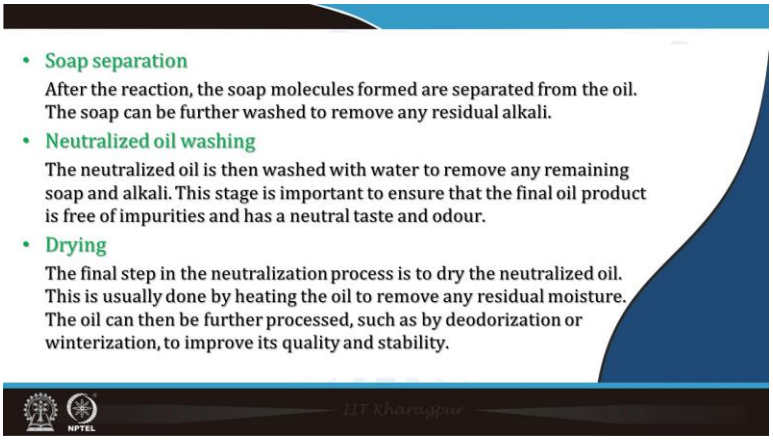



Then centrifuges of proper design and proper capacity because after the neutralization reaction is complete the mixture is soap stock particularly is separated from the oil and the soap that is used in a centrifuge. So, the centrifuge spins the mixture at high speed causing the soap to separate from the oil and it is collected at the bottom of the centrifugal bow.

Then filter presses are provided, these are used to remove the soap from the oil and it has been separated from the mixture in the centrifuge. The filter press uses a series of filters to remove the soap leaving behind the pure neutralized oil. So, the specific equipment used however, may vary depending upon the scale of operation and the type of oil being processed.

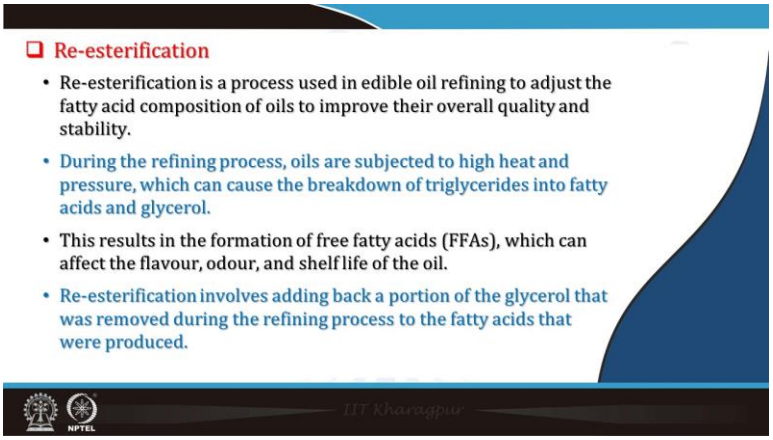



The stages in the neutralization process include pretreatment, alkali addition and then finally, reaction. In the pretreatment what is done is this stage involved the heating of the oil to a situation or to a suitable temperature to increase the fluidity of the oil and to make it easier to handle. So, after the oil comes to a proper temperature then the second stage is the alkali addition. In this stage, the alkaline solution usually sodium hydroxide or potassium hydroxide is added to the oil in a controlled manner as I told you proper amount and proper concentration, the alkali reacts with the free fatty acids in the oil to form soap molecules. It is given proper reaction time that is the reaction between the alkali and free fatty acid take place over a period of time, typically, it may be 30 to 60 minutes depending upon the quantity of free fatty acid present and quantity of the alkali solution used. During this period the oil is agitated continuously or intermittently to ensure uniform mixing that is giving proper contact between ensuring a proper contact between the acid molecule, FFA molecule, and the alkali. The amount of alkali used in the reaction time will depend upon the level of FFA in the oil.

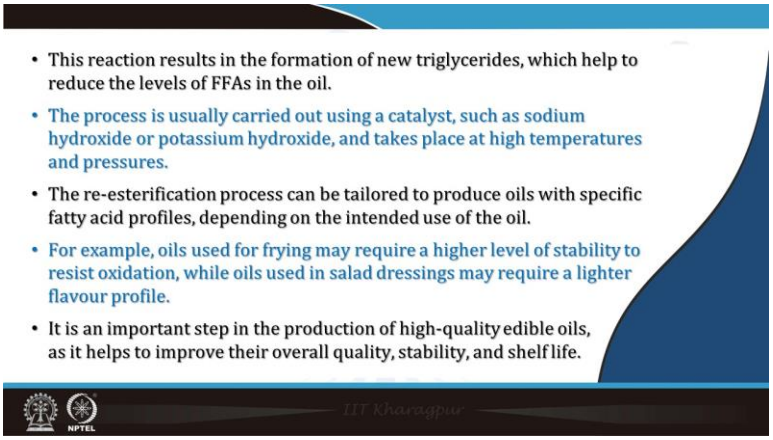



Soap separation in the next stage. After the reaction, the soap molecules formed are separated from the oil. The soap can be further washed to remove any residual alkali. Then neutralized oil washing in the next stage. In this stage, oil is washed with water to remove any remaining soap and alkali. This stage is important to ensure that the final oil product is free of impurities and has a neutral taste and odour. Finally, drying, the final step in the neutralization process and this is usually done by heating the oil to remove any

residual moisture from it. The oil can then be further processed such as sent to the deodorization or winterization to improve the quality and stability or it can be sent to the further product manufacturing or bottling for market distribution.

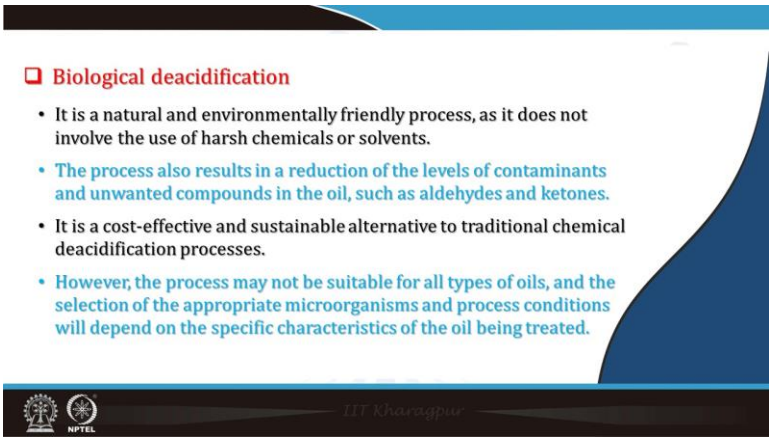



Another one is the concept is re-esterification. Re-esterification is a process used in edible oil refining to adjust the fatty acid composition of oil to improve the overall quality and stability of the oil. During the refining process, the oil is subjected to high heat and pressure which can cause the breakdown of triglyceride into fatty acids and glycerol. So, the triglyceride quantity may be reduced. This formation of free fatty acids can affect the flavor, odour, shelf life etc. of the oil. So, re-esterification involved the adding back a portion of glycerol that was removed during the refining process to the fatty acids that were produced. So, this glycerol and fatty acid and proper reaction time and reaction parameters are given so that it re-esterifies fresh oil and oil quantity is increased, oil yield will increase free fatty acids are further esterified.

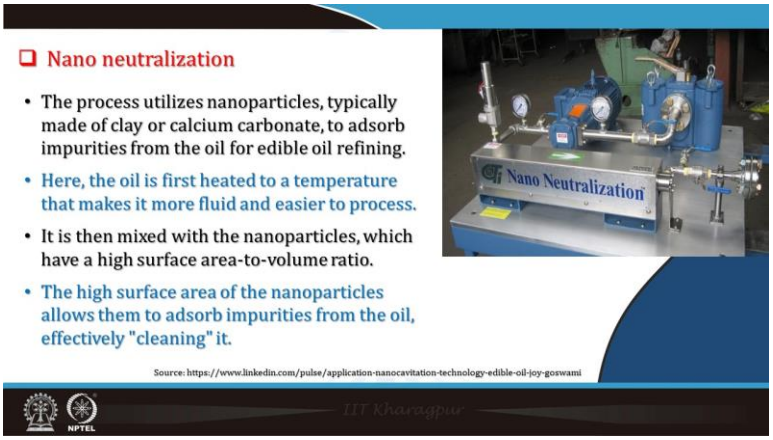



So, this reaction results in the formation of new triglyceride which help to reduce the level of FFA in the oil. The process is usually carried out using a catalyst such as sodium hydroxide or potassium hydroxide and takes place at a high temperature and pressure. The re-esterification process can be tailored to produce oils with a specific fatty acid profile depending upon the intended use of the oil. For example, oils used for frying may require a higher level of stability to resist oxidation while oils used for salad dressing

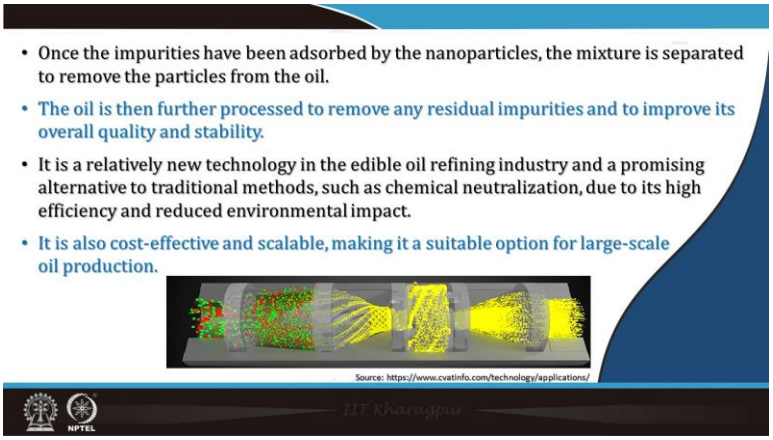
may require a higher flavor profile. So, accordingly the re-esterification process can be used to adjust the free fatty acid composition etc. of the oil and there is also one inter-esterification that we will take up separately in some class. So, it is an important step in the production of high-quality edible oils as it helps to improve the overall quality, stability and shelf life of the oil.



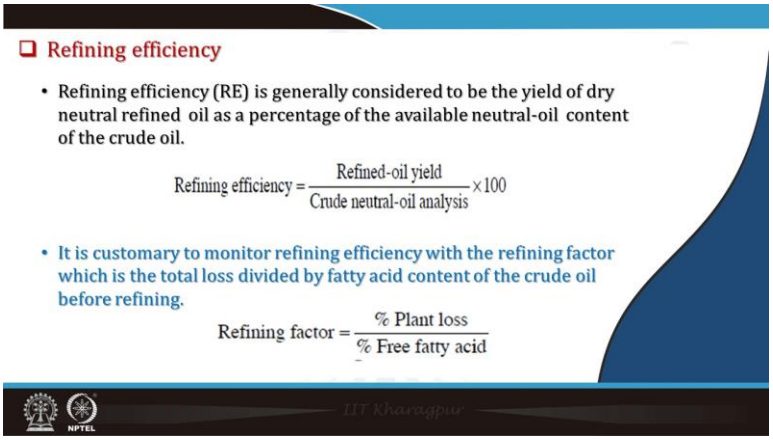

Then under the biological deacidification, it is a natural and environmentally friendly process as it does not involve the use of harsh chemicals or solvents. The process also results in a reduction of the levels of contaminants and unwanted compounds in the oil such as aldehydes and ketones. It is a cost effective and sustainable alternative to traditional chemical deacidification process. However, the process may not be suitable for all types of oil and the selection of the appropriate microorganisms and process conditions will depend on a specific characteristic of the oil being treated.




Nano neutralization, you can see here in the figure that is a set up shown. This process utilizes the nanoparticles, typically made up of clay or calcium carbonate to absorb impurities from the oil for edible oil refining. Here, the oil is first heated to a temperature that makes it more fluid and easier to process. It is then mixed with the nanoparticles, which have a high surface area to volume ratio. The high surface area of the nanoparticles allows them to absorb impurities from the oil and effectively cleaning it.




You can see here that, how it is done in this figure it is shown. Once the impurities have been absorbed by the nanoparticles, the mixture is separated to remove the particles from the oil. The oil is then further processed to remove any residual impurity and to improve its overall quality and stability. So, it is a relatively new technology in the edible oil refining industry and a promising alternative to traditional methods, such as chemical neutralization, due to its high efficiency and reduced environmental impact. It is also cost effective and scalable technology making a suitable option for large scale oil production. However, there are much more things a suitable equipment, suitable system needs to be developed and more R&D work is required before this process is made commercially viable process.



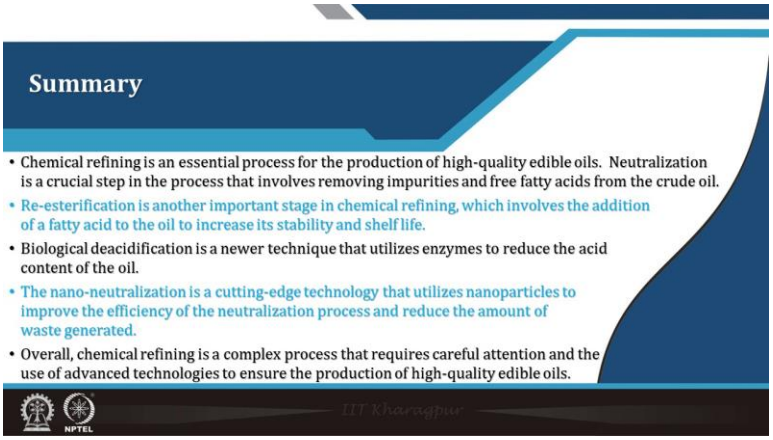

So, the refining efficiency finally, that is it is the generally considered to be the yield of dry neutral refined oil as a percentage of the volatile neutral oil content of the crude oil. That is what was the initial free fatty acids and what percentage of free fatty acids that has been removed the refining. So, the refining efficiency and refining factors are these two indicators which are used to judge the refining process.

$$\text{Refining efficiency} = \frac{\text{Refined} - \text{oil yield}}{\text{Crude neutral} - \text{oil analysis}} \times 100$$

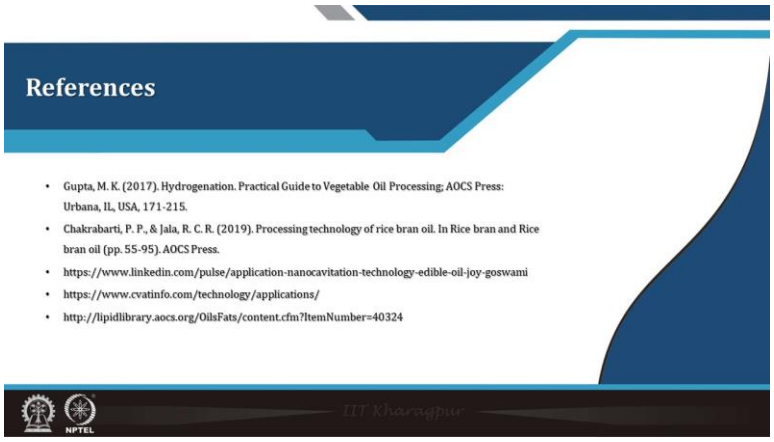
$$\text{Refining factor} = \frac{\%\ \text{Plant loss}}{\%\ \text{Free fatty acid}}$$

So, from that you can one can calculate the refining efficiency and then refining factor is a percent plant loss divided by percent free fatty acids that is what are the total losses that has occurred during the refining process and what is that percent divided by the percent of the initial free fatty acids. So, that is the called refining factor. So, refining efficiency refining factor sometime even fatty acid factor, acid oxidation factors. These are some indicators, factors which are used to judge the refining process.


Then refining byproducts obviously, the crude oils you see contains a number of material that are removed to produce neutral light coloured oil and these impurities have been considered waste products constituting a disposal problems. However, they can also be valuable byproducts when effectively recovered and processed and maybe toward the end of this course we will take up a few lectures on the oil in the processing industry byproducts and their utilization there we will discuss various aspects. So, two major byproducts from the refining processes are soap stock from the chemical refining and then other hydrated gums from the degumming process prior to caustic refining or the physical refining pretreatment stages. So, both these soap stock as well as gums they can be their ways to use it properly, used for the food, feed, or other that is industrial purposes even like soap stock can be used further for the breaking the soap and taking the fatty acid distillation etc. for making fatty acid all those things.



So, finally, I would like to summarize this lecture by saying that the chemical refining is an essential process in the production of high-quality edible oils. Neutralization is a crucial step that involves removing free fatty acids and impurities from the crude oil. Re-esterification is another important stage in the chemical refining, which involves the addition of a fatty acid to the oil to increase its stability and shelf life. Biological deacidification is a new concept that utilizes enzymes to reduce the acid content of the oil. Nano neutralization is a cutting-edge technology and utilizes nanoparticles to improve the efficiency of the neutralized neutralization process and it reduces the amount of waste generated. Overall, chemical refining is a complex process that requires careful attention and the use of advanced technologies to ensure the production of high-quality edible oils.




These are the references that were used in this lecture.


This thank you all very much. Thank you for your patience here. Thank you.