

**Novel Technologies for Food Processing and Shelf Life Extension**  
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**Indian Institute of Technology, Kharagpur**

**Lecture – 32**  
**Edible Oil Refining (Part 1) Degumming & Neutralization**

In this lecture, removal of impurities from the edible oil using degumming or neutralization processes will be studied.

**Non-triglyceride (NTG) components present in crude oil**

<ul style="list-style-type: none"><li>• <b>Major</b></li><li>✓ Free fatty acids (FFA)</li><li>✓ Phospholipids</li><li>✓ Diglycerides</li><li>✓ Monoglycerides</li></ul>	<ul style="list-style-type: none"><li>• <b>Minor</b></li><li>✓ Tocopherols</li><li>✓ Sterols and sterol esters</li><li>✓ Tocotrienols in palm and rice bran oil</li><li>✓ Colour compounds</li><li>✓ Oil decomposition products such as polar compound, polymer, aldehyde, ketones and other volatile and non volatile compounds</li></ul>
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Depending upon the method used for extraction of oil and the process parameters, along with the triglycerides some other components also get extracted and they may include free fatty acids, phosphatides, pigments, etc. For example, if the severe pressure or heat conditions etc. are used that may result into some hydrolysis or breakage of the ester linkage of the triglyceride. The phospholipids may also get extracted along with the triglycerides; the free fatty acids development may lead to the amount of diglycerides, monoglycerides etc. The major non-triglyceride compounds present in the oil depending upon the process and process parameters used for oil extraction might include free fatty acids, phospholipids, diglycerides, monoglycerides and so on. Similarly, some of the minor non-triglyceride components which oil might contain include tocopherols, sterols, esters, tocotrienols, colour compounds or even the oil decomposition products such as polar compounds, polymeric aldehydes, ketones and other volatile and non-volatile compounds.

## Refining of crude oil

- Refining is the important processing steps that reduces or removes certain undesirable impurities from the crude oil.
- In refining, physical and chemical processes are combined to remove undesirable components from the crude oil.
- The impurities includes major and minor non-triglycerides.



[mpec.org.my/upload/Understanding-Oil-Fats-Processing-assignment-practice-Kimjong-Gil-POTS-Korea-2015-P1.pdf](https://mpec.org.my/upload/Understanding-Oil-Fats-Processing-assignment-practice-Kimjong-Gil-POTS-Korea-2015-P1.pdf)



## Refining of crude oil

The non-triglyceride components which are present in the oil should be removed before the oil is sent for further use as a crude oil for cooking and frying purposes or for its convergence into various processed products or its use in different food products. So, in order to get good quality oil which contains mainly of triglycerides, the extracted oil or crude oil is subjected to various processing steps for the removal of non-triglyceride components and these are called refining.

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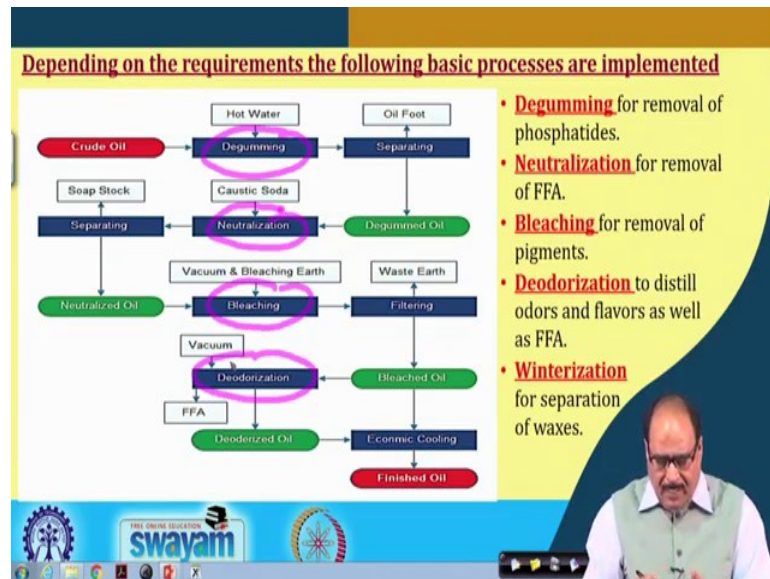
**Objectives of refining process**

- **Removal of undesired products from crude oils**
  - ✓ Free fatty acids (FFA)
  - ✓ Phospholipids (PL)
  - ✓ Oxidised products
  - ✓ Metal ions
  - ✓ Colour pigments
  - ✓ Other impurities
- **Preservation of valuable components**
  - ✓ Vitamina E or tocopherol-natural antioxidants
  - ✓ Sterols, sterol esters, tocotrienols, and squalene
- **Minimize oil losses**
- **Protection of the oil against degradation**

The slide also features a speaker in the bottom right corner and logos for IIT Bombay and the Swamyam initiative at the bottom.

### **Objectives of refining process**

The objectives of the refining process therefore, includes the removal of undesirable components from the crude oil like free fatty acids, phospholipids, oxidised products, metal ions, colour pigments and other impurities. But at the same time the refining process should be conducted in such a manner that the valuable components like tocopherols, tocotrienols, sterols, sterol esters etc., also get extracted along with the oil. They are the valuable component having good antioxidant properties etc. So, the refining process should not remove these desirable component which the oil contains. Apart from this, oil losses should be minimized and the refined oil should be protected against any deterioration i.e. even the refining process parameter itself should not lead to the decomposition of the oil.



In this slide, the major processes to which oils are subjected are shown. The removal of phosphatides etc. is called degumming. After degumming, the degummed oil (see Figure) is sent to the neutralization step that is again very important. In the neutralization step, free fatty acids are removed. The neutralized oil is sent to the bleaching process where the undesirable colouring pigments are removed. Finally, last step of the refining process is the deodorization which is an important step where the materials or components which are not removed in earlier processes or generated if in case will be retained in this process. So, after this step, the oil should basically be bland in taste, it should be principally only triglyceride; odours, flavours and all other components should be removed. Sometimes even after deodorization, these oils are also winterized. Bleaching earth added during the bleaching process is further separated by the filtration, but it might so happen that a few minor earth particles might remain in the oil. During the storage these earth particles get deposited on the bottom of the tank which should be removed.

**Degumming**

**Aim of degumming operation**

- ✓ The emulsifying action of PL increases oil losses during alkali refining.
- ✓ Gums lead brown discoloration of oil after heating during deodorization.
- ✓ Salts may be formed with copper, magnesium, calcium and iron, accelerating oxidative degradation of oil.
- ✓ Certain PL, such as lecithin, find widespread industrial application.

**Gums : Two types**

- **Hydratable**
  - ✓ Easy to remove by simple water washing
- **Non-hydratable**
  - ✓ Hard to remove from oil.
  - ✓ Requires the use of an acid to convert to hydratable for complete removal.

**Different degumming processes**

- ✓ Water degumming
- ✓ Acid degumming
- ✓ Enzymatic degumming
- ✓ Membrane degumming

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The gums or phospholipids which are present in the oil are of two types either hydratable or non-hydratable. The hydratable gums or phospholipids are easy to remove just by simple water washing whereas, non-hydratable phospholipids are difficult to remove. They require acid treatment for their complete removal. Acid treatments in fact, convert the non-hydratable phospholipids into hydratable phospholipids and they get removed. Different degumming processes include water degumming, acid degumming, enzyme degumming and membrane degumming.

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### Water degumming

Source: <http://lipidlibrary.aocs.org/lipidfacts/content.cfm?itemNumber=40334>

The extracted gums can be processed into lecithin for food, feed or for technical purposes.

- Hydration process; warm water is added to the crude oil at 80-85 °C and the mixture is agitated slowly for approximately 20 min.
- The water dosage used is usually based on the expected amount of PL in the crude oil.
- The hydratable PL agglomerate at the interface of the oil and water, capturing even some non-hydratable PL with them.

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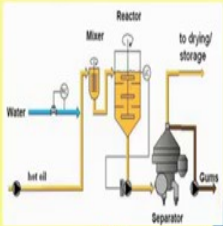
### Water degumming

The process which is followed for degumming of the oil using water can be seen in the flow sheet shown in slide. These steps are normally done at the extraction plant.


Immediately after extraction, they are treated with a definite amount of water depending upon the quality of the crude oil and this process is called hydration process.

- Hydration process; warm water is added to the crude oil at 80-85 °C and the mixture is agitated slowly for approximately 20 min or passed to the reactor for about half an hour.
- The water dosage used is usually based on the expected amount of PL in the crude oil.
- In the reaction vessel the hydratable phospholipid agglomerate at the interface of the oil and water, capturing some of the non-hydratable phospholipids with them. They precipitate and in the next step by using appropriate separation method normally centrifuge etc. they are removed. So, these gums which are removed from here they are sent for purification. The oil is sent to the drying and storage so that whatever water has been added here can be removed before sending it to the next step.

- Oil is also trapped by the PL, forming an emulsion, referred to as “gums” or “wet gums”.
- After a certain reaction period the hydrated PL can be separated either by decantation (settling) or continuously by means of centrifuges.



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### Acid degumming

#### Dry acid degumming

- Dry acid degumming is particularly suitable for processing oils with low gum contents such as palm oil, coconut oil, or animal fats.
- Intensive mixing is implemented following addition of acid to the pre-heated crude oil.
- The conditioned gums are absorbed into the bleaching earth and are separated by filtration.
- The benefits of the dry acid degumming process are
  - ✓ Efficiency as a result of
    - ❑ Low energy consumption,
    - ❑ Low operation and maintenance costs
  - ✓ Long service life (the components are acid proof),
  - ✓ Low investment costs,
  - ✓ Environmental-friendly as no waste water or soap stock occur.



### Acid degumming

Then, after water degumming done at the extraction plant, the oil is pumped through appropriate transportation or conveying means to the refining plant. And it is said that the refining of the oil starts from the storage tank itself, because during the storage also depending upon the environmental conditions and pumping practices followed, some of the insoluble oil get settled in the storage tank and separated. So, from this oil storage tank the oil is passed through the acid degumming unit.

## **Dry acid degumming**

Acid degumming unit may be of two types: dry acid degumming and wet acid degumming.

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**Wet acid degumming**

- Initially oils with higher gum contents (e.g. corn oil) are similarly processed as in dry acid degumming.
- However, to achieve gum hydration, water is added following acid apportioning.
- The gums are removed by a separator prior to bleaching.
- This process is beneficial as centrifuges enable easy separation of gums in oil with higher non-hydratable gums (e.g. rapeseed oil and soybean oil).
- The consumption of bleaching earth is reduced because the oil has already been extensively degummed.

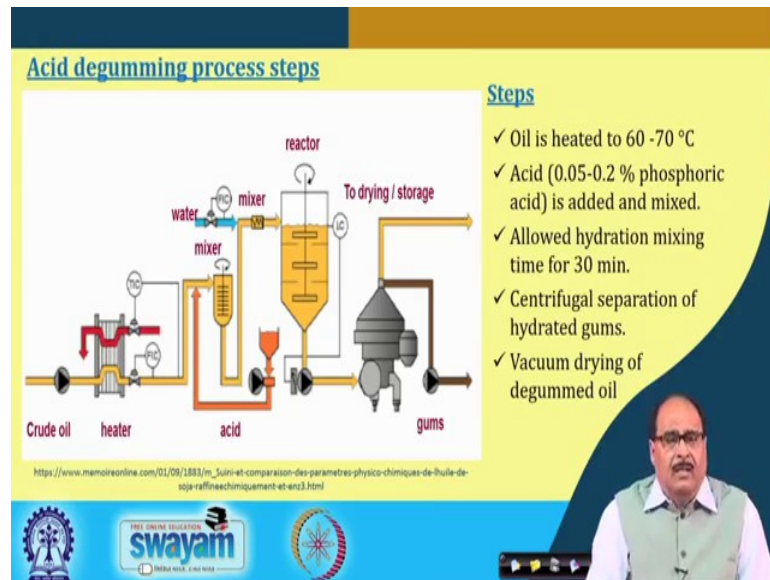
The slide features a blue header, a yellow main content area, and a blue footer. The footer contains logos for 'THE OPEN UNIVERSITY', 'swayam', and 'INDIA'S SKILL DEVELOPMENT' along with a small video inset of a man in a green vest.

## **Wet acid degumming**

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## Acid degumming process steps

### Steps

- ✓ Oil is heated to 60 -70 °C.
- ✓ Acid (0.05-0.2 % phosphoric acid) is added and mixed.
- ✓ Allowed hydration mixing time for 30 min.
- ✓ Centrifugal separation of hydrated gums.
- ✓ Vacuum drying of degummed oil.

### Enzymatic degumming

- The enzyme solution (aqueous solution of citric acid, caustic soda and enzymes) is dispersed into filtered oil at mild temperature.
- A high speed rotating mixer is used for effective mixing of enzyme solution and oil.
- The conversion of non-hydratable PL into hydratable PL is attained by the effect of enzyme.

<https://www.slideshare.net/jazzyy/fat-processing>

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### Enzymatic degumming

Enzymatic degumming is another potential process for removal of phosphatides or gums etc. from the oil (see Figure). In fact, the processing steps are same only difference here is that, in earlier case water or acid solution is used, here enzyme solution is used.

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### Membrane degumming

- MF and UF have been explored for membrane based degumming.
- Membrane degumming of the oil can be done in two ways
  - ✓ Degumming of crude solvent free vegetable oil
  - ✓ Degumming of oil-solvent micella
- The major problem in membrane degumming of the crude oil was the drastic flux drop at the initial experiment stage which was attributed to the pore blocking, concentration polarisation, and cake formation by the rejected solids.

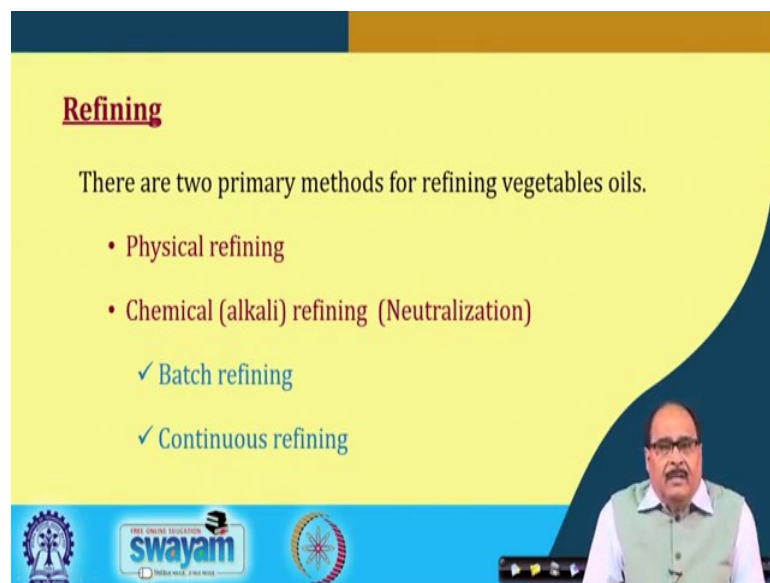
Source : de Moura et al. (2005)

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## Membrane degumming

The principle of membrane degumming is same as membrane separation process. Micro filtration or ultrafiltration offer promising potential in oil industry for the removal of phosphatides and other components which are present.

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- Membrane degumming of the oil can be done in two ways:
  - ✓ **Degumming of crude solvent free vegetable oil**
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- The major problem in membrane degumming of the crude oil was the drastic flux drop at the initial experiment stage which was attributed to the pore blocking, concentration polarisation, and cake formation by the rejected solids. So, operation becomes little difficult or costly to manage.



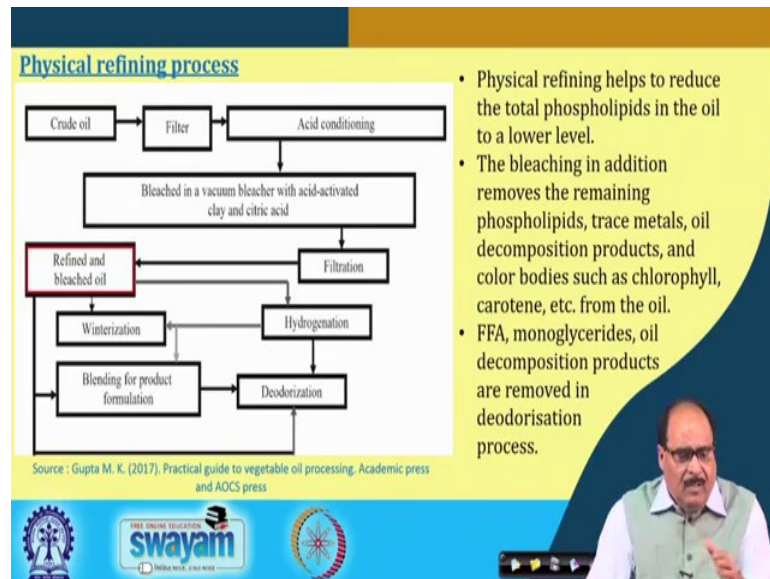
**Refining**

There are two primary methods for refining vegetables oils.

- Physical refining
- Chemical (alkali) refining (Neutralization)
  - ✓ Batch refining
  - ✓ Continuous refining

The slide features a yellow background with a dark blue curved shape on the right side. At the bottom, there is a blue banner with logos for 'swayam' and 'MOE, GOVT OF INDIA'. A small video inset in the bottom right corner shows a man in a white shirt and green vest speaking.

After degumming, the degummed oil with no water content is sent to the refining. The refining is basically removal of free fatty acids or such other components from the oil. There are two methods used for the refining process, it may be physical refining process or chemical generally called alkali refining process or neutralisation, which may be accomplished in batch or continuous process.



### Physical refining process

The crude oil is sent to filter and acid conditioning, after removing the gum etc. it is bleached in a bleacher with acid activated clay and citric acid. And then filtration to remove the suspended particles or even free fatty acids etc.

- It helps to reduce the total phospholipids in the oil to a lower level.
- The bleaching in addition removes the remaining phospholipids, trace metals, oil decomposition products, and color bodies such as chlorophyll, carotene, etc. from the oil.
- FFA, monoglycerides, oil decomposition products are removed in deodorisation process.

### Chemical refining (neutralization) process

- Chemical refining is widely used for vegetable oils.
- In this process the crude oil is pre-treated with phosphoric acid and then neutralised with a caustic (sodium hydroxide) solution.
- The caustics reacts with the FFA in crude oil and produces soap.
- The soap is then separated from the refined oil in primary centrifuge; the remaining soap is mostly removed from the oil via water washing and separating the oil and water in water washing centrifuge.

#### Used in the industry for the refining of

- ✓ Crude soybean oil
- ✓ Degummed soybean oil/canola oil
- ✓ Crude sunflower oil
- ✓ Crude safflower oil
- ✓ Crude and degummed cottonseed oil



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#### Neutralization : batch process

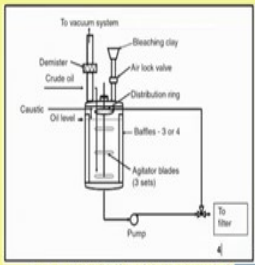
Source : Gupta M. K. (2017). Practical guide to vegetable oil processing. Academic press and AOCs press

- Crude oil is taken in the refining kettle.
- The kettle is equipped with agitator and baffles (to prevent vortex formation).
- The caustic solution is added in the kettle after acid pre-treatment and the mixture is agitated at low speed.
- The temperature of neutralisation is kept 35 – 40 °C for seed oils.
- The FFA and phosphorous content at the end of reaction should be 0.01-0.02%, and less than 5 ppm, respectively.


## Neutralization : batch process

Crude oil is taken into the refining kettle. The kettle is equipped with the agitator as well as baffles to prevent vortex formation. The caustic solution is added into the kettle after acid pre-treatment and the mixture is agitated at low speed. The temperature of the neutralization is kept 35 - 40 °C for the seed oils. The free fatty acids should be maximum 0.01 - 0.02 % and the phosphorous should be less than 5 ppm after the neutralization treatment.

- The oil is heated to 85-90°C and 10-15% deionised water is added.
- The agitator speed is increased for obtaining intimate mixing between oil and water to remove soap.
- Water washing and draining steps follow to remove soap.
- The oil is treated with 1-3% acid activated clay and bleached under vacuum at 110-120 °C.




Source: Gupta M. K. (2017). Practical guide to vegetable oil processing. Academic press and AOCs press



The oil is next heated to 85 - 90 °C and 10 - 15 % deionised water is added, agitator speed is increased for obtaining intimate mixing between oil and water to remove soap. Water washing and draining step are followed to remove soap. Oil is treated with 1 - 3 % acid activated clay and bleached under vacuum at 110 - 120 °C.

**Critical points of batch process**

- **Agitator speed**
  - ✓ It must be operated at
    - Low speed – for neutralisation
    - Medium speed – for water washing
    - High speed – for bleaching
- **Bleaching clay**
  - ✓ Requires higher dosage of clay as compared to 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 added is effective in reducing the colour bodies in the oil.
- **Refining losses**
  - ✓ Are very high in batch process as compared to the continuous chemical refining process.



### **Critical points of batch process**

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- Refining losses
  - ✓ Are very high in batch process as compared to the continuous chemical refining process.

So, the process parameter etc. should be accordingly optimized and standardized.

**Neutralization : Continuous process**

Source : <http://lipidlibrary.aocs.org/OilsFats/content.cfm?itemNumber=40324>

- The crude oil undergoes a series of processes such as heating, acid treatment, caustic (lye) treatment, mixing, soap separation, water washing, etc.
- The caustic is added after acid treatment which increases pH, reduces viscosity of acid gums, residual P level is low as it forms soap, and improve the removal of non-hydratable PL.

□ With use of this techniques, P level can be achieved lower than 20 ppm in the degummed oil after water washing.

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## Neutralization : Continuous process

The principle in the continuous process is same as that of the batch process. Here also it is intimate mixing of the oil with the suitable concentration of alkali, and giving proper reaction time, then settling and removal of the soap. The crude oil undergoes a series of the process like heating, acid treatment, caustic treatment, mixing, soap separation, water washing etc. continuously (see Figure).

**Critical points of continuous process**

- **Crude oil filtration**
  - ✓ Crude oil free from meal, hull, dirt should be used, otherwise the centrifuge or primary separator gets dirty soon.
- **Crude oil pre-treatment**
  - ✓ Proper acid pre-treatment is essential for all crude oils and especially for those that have been derived from poor quality seeds or damaged during storage.
  - ✓ Low acid treatment – can not reduce non-hydratable phospholipids.
  - ✓ High acid treatment – can cause breakdown of the chlorophylls in the crude oil, making it more sensitive to photooxidation; will need higher caustic treat.
- **Uniformity of crude oil composition**
  - ✓ A non uniform feed quality can cause under or over refining of the oil.
- **Uniformity of crude oil flow**
  - ✓ A variation in change of flow can cause change in the oil/caustic ratio under or over refining of the oil.

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Critical points of continuous process ...contd.

- **Caustic strength and temperature**
  - Higher strength than recommended can cause excessive reaction with oil leading to hydrolysis of TG and increased refining loss.
  - Lower strength than recommended might reduce the density of soap which may cause higher oil loss.
  - Temperature affects the dispersion of the caustic into the oil.
- **Degree of mixing between the crude oil and caustic solution**
  - Poor mixing leaves excess amount of free alkali in refined oil.
  - It affects the FFA content of the water washed refined oil.
- **Contact time between crude oil and caustic solution**
  - Affect the reaction of caustic with the nonhydratable PL.
- **Refining temperature**
  - Allow the soap to separate from the oil.

The slide also features logos for IIT Bombay, Swayam, and other educational institutions, along with a small video inset of a speaker in the bottom right corner.

- **Caustic strength and temperature**
  - Higher strength than recommended can cause excessive reaction with oil leading to hydrolysis of TG and increased refining loss.
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  - Poor mixing leaves excess amount of free alkali in refined oil.
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