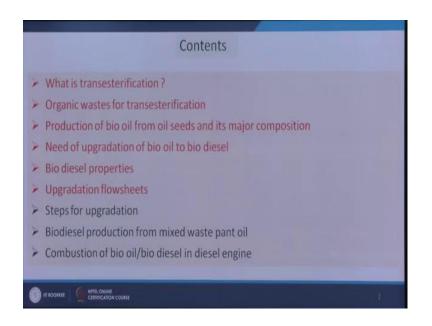
# Waste to energy conversion Dr. Prasenjit Mondal Department of Chemical Engineering Indian Institute of Technology, Roorkee

# Lecture – 35 Energy production from waste through transesterification – 2

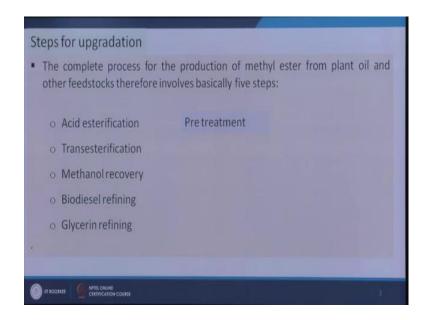
Hi friends, now we will start discussion on the second part of the module energy production from waste through transesterification. In the first part of this module we have discussed on the fundamentals of transesterification process need of upgradations of bio oil and other triglyceride continuing waste.

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And we have also seen the need of upgradation of bio oil and bio diesel properties and in this part of this module we will discuss on steps for upgradation and bio diesel production from mixed waste plant oil and grease and combustion of bio oil or a bio diesel in diesel engine.

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So, in the first part of this module we have already discussed on the general flow sheet for the upgradation of bio oil and other waste containing triglycerides. So, you have seen that there are 5 basic steps which are involved for the whole process. The first step was acid esterification. Second is transesterification, third is methanol recovery, fourth is biodiesel refining. And then glycerin refining out of this steps the acid esterification is also termed as pretreatment step it is under pretreatment operation. And we will be discussing on the importance of these individual steps and of the major role of these steps and then we will discuss how to convert cooking oil to bio diesel and the grease to bio diesel

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# Acid Esterification (AE) Free fatty acids (FFAs) in biodiesel feedstocks react with alkali catalysts like NaOH and KOH and form soaps, which reduce the oil properties of the biodiesel. Triglycerides + FFAs + Alcohol Base catalyst Na/K salt of FAs + Triglycerides Low levels of FFAs (upto 4%) can be managed by using additional catalyst and allowing soap formation. NaOH [%FFA](0.144) + 1 %; KOH: [%FFA](0.197)/0.86 + 1%; Sodium methoxide: [%FFA](0.190) + 0.25 %. Feedstock containing > 4 % FFAs (Animal fats and recycled greases) are first filtered and then pre-processed to remove water and other contaminants such as unwanted solids. The pretreated oil is then fed to the acid esterification process. Most plant oils and some food-grade animal fats do not require AE, (FFA< 4 %).</li> The sulfuric acid, is dissolved in methanol and then mixed with the pretreated oil. The mixture is heated and stirred, and the FAs are converted to biodiesel. Once

the mixture is neated and stirred, and the FAs are converted to biodiesel. Once the reaction is complete, it is dewatered and fed to the transesterification process.

So, now let us on the acid esterifications. As I have just mentioned that acid esterification is also termed as pretreatment step. So, in this step it is basically targeted to remove free fatty acids if available in the feed stocks, so in bio oil or any other organic waste if free fatty acids are available. So, those free fatty acids react with NaOH if base catalyst is used like say NaOH or KOH if base catalyst are used. So, the free fatty acids react with it is alkali and it is converted to sodium or potassium salt of this fatty acids that is also called as soap. So, this soap reduces the quality of the biodiesel. So, as per the reaction takes place the triglyceride plus free fatty acids plus alcohol then under the base catalyst it forms, this sodium potassium salt of free fatty acids and then the triglycerides. So, this is not desirable.

Now, to tackle this situation if the free fatty acid content is very less, let us say if it is less than 4 percent then no free acid esterification step is applied. In that case additional amount of sodium hydroxide or potassium hydroxide is added. So, as mentioned here you for if n NaOH is added then percentage free fatty acids into 0.144 plus 1 percent and for KOH this is this one and for sodium methoxide, that is this is a catalyst in that case free fatty acids into 0.190 plus 0.25 percent. So, this is an additional amount of alkali used to neutralize these acids, but if it is free fatty acids content is higher relatively more than 4 percent, then we need to separate the free acids first before the transesterification reaction starts. So, in that case acid esterification is important. So, to remove this free fatty acids sulfuric acid is added. So, this sulfuric acid reacts with the free fatty acids and

the free fatty acids are converted to in presence of alcohol to esters that is desirable for the biodiesel.

So, sulfuric acid is dissolved in methanol, and then mixed with the pretreated oil the mixture is heated and stirred and the free fatty acids are converted to biodiesel. So, that is the main role of the acid esterification.

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<ul> <li>200°C, which converts FFAs to monoglycerides and diglycerides.</li> <li>FFA + glycerol ZnCl₂ catalyst monoglyceride and diglycerides + water</li> <li>Water is vented as it is formed. Requires high temperature and slow process.</li> <li>Acid catalysis followed by base catalysis- Acid catalysis for conversion of FFAs methyl esters, until FFA &lt; 0.5% followed by addition of methanol and bacatalyst to finish the reaction</li> <li>Acid such as H₂SO₄ catalyzes both esterification and transesterification reaction</li> </ul>		
<ul> <li>Glycerolysis – Glycerol is added to high FFA feedstock with catalyst (ZnCl<sub>2</sub>) 200°C, which converts FFAs to monoglycerides and diglycerides.</li> <li>FFA + glycerol ZnCl<sub>2</sub> catalyst monoglyceride and diglycerides + water</li> <li>Water is vented as it is formed. Requires high temperature and slow process.</li> <li>Acid catalysis followed by base catalysis- Acid catalysis for conversion of FFAs methyl esters, until FFA &lt; 0.5% followed by addition of methanol and bacatalyst to finish the reaction</li> <li>Acid such as H<sub>2</sub>SO<sub>4</sub> catalyzes both esterification and transesterification reaction Esterification of FFA is fast (1 hour) but transesterification is slow (2 days) at 60</li> </ul>	Alt	ernative method to remove/ manage free fatty acids (FFAs)
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Esterification of FFA is fast (1 hour) but transesterification is slow (2 days) at 60	•	Acid catalysis followed by base catalysis- Acid catalysis for conversion of FFAs to methyl esters, until FFA < $0.5\%$ followed by addition of methanol and base catalyst to finish the reaction
	0	Acid such as $H_2SO_4$ catalyzes both esterification and transesterification reactions. Esterification of FFA is fast (1 hour) but transesterification is slow (2 days) at 60°C.

Now, the main role of acid esterification is to remove the free fatty acids or to convert it. So, there are some other approaches which also do the same job one is enzymatic methods. Some enzymes can do the similar job, but the difficulty is that this is very costly and this is not a very popular option. Then glycerolysis, glycerolysis the glycerol can be added at 2 hundred degrees centigrade in presence of some catalyst like zinc chloride. So, that free fatty acids are converted to monoglyceride and diglyceride plus water is formed when the 200 degree centigrade is applied the water which is produced in the reactor it will be goes off. So, now, acid catalysis followed by base catalysis is another option.

So, in this case initially acid is used, when free fatty acids is released means say 4 percent free fatty acids is there. So, acid will be used to down it below 0.5 percent and then it will be used for sodium hydroxide addition or potassium hydroxide additions. So, that second step that base catalysis reaction will go on and this how it can be managed that 0.5 percent by acid and then by NaOH, this is possible because the acid catalysis

reactions there are 2 types of reaction takes place if we had acid then triglycerides will be converted to esters as well as free fatty acids will also be converted to esters. But preprocessed seed will be very quickly transferred to the esters, but transesterification will be taking more time here it is given say 1 hour is sufficient for free fatty acids to extra formation, but at least 2 days at 60 degree centigrade it is required for the transesterification.

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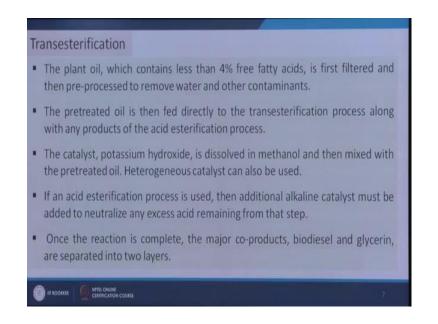
Conversion of FFA			
The conversion of FF	As can be determined fro	om the following Equation	
$\eta = \frac{S_0 - S_i}{S_0}$	Where S <sub>o</sub> is initial acid reaction time	value and $S_i$ is the acid value at	t some
neutralized ethanol a	nd is fully dissolved by he	of sample is added to some am eating. Phenolphthalein (0.5 mL h standard KOH solution.	
The acid value is cal		$\frac{\cdot V \cdot c}{m}$	
	is the volume of KOH e	; c is the concentration of the K mployed for titration (mL); m is Ding et al., 2012, Energies, 5,	the mass
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Now, one thing is that that 4 percent free fatty acids or somewhere it is mentioned that if that is 2 percent free fatty acid is the upper limit. So, whatever may be the amount 2 or more percent, but we have to know the free acid concentration, now what is the free acid concentration. So, how can you measure it or how can you measure the conversion of this free fatty acids that is equal to S o minus S i by S o where S o is the free acid content at the initial stage and then S i is the acid content at any time t and then this is the formula through which we can get the conversion of free fatty acids. So, S that is your acid value we have to know at initial stage and after certain time. So, how can you get the acid value? Acid for the determination of acid value some amount of oil is mixed with some amount of neutralized ethanol.

So, neutralized ethanol is prepared as per the standard procedure mentioned in the protocol. So, then neutralized ethanol and the oil sample id added with phenolphthalein indicator and titrated it against KOH solution there is known concentration known

strength of KOH solution normally n by 10. So, then titer point end point is achieved then the acid value is calculated by using this formula the acid value is equal to 56 point one into V into c divided by m and V is the volume of KOH solution how much we are using it. So, that is in m l, and c is the concentration of the KOH solution that is in mol per liter, and m is the mass of the sample taken to be analyzed that is in gram. So, that way we can get the S values and we can get the fatty acid conversion.

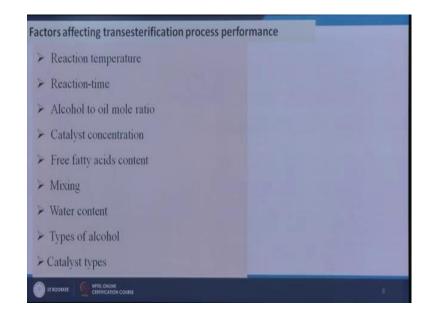
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Now, will start transesterification and we will try to understand what is the importance of transesterification. So, this is the reaction through which triglyceride is converted to glycerol and fatty acids esters alkyl esters of the fatty acids. So, this is our main reaction for the conversion of bio oil or other organic triglyceride contain organic waste to biodiesel. So, here the catalyst is required as you have discussed in the previous part of this module, that the catalyst may be acid catalyst base catalyst that is those are homogenous catalyst or heterogeneous catalyst may be.

So, homogenous catalyst particularly the base catalyst it has more performance, but it has major drawback that it produces soap. So, it is not desirable. So, heterogeneous catalysts are getting more interest, nowadays and developments are going on to produce heterogeneous catalyst to make the process more efficient. So, if we use acid esterification process then there will be chance of some extra acid present in the output of this step which is entering in the transesterification reactor. So, we have to add additional amount of NaOH, if we use the alkali catalysis process for transesterification.

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Now, we will see what are the factors that can influence the performance of the transesterification process. So, here reaction temperature reaction time alcohol to mole ratio of the oil and then catalyst concentration and then free fatty acid content mixing water content types of alcohol and finally, the catalyst type. So, all those factors or the parameters influence the performance of the transesterification reactions. So, what solvent we are using; that means, what alcohol we are using that will help what will be the temperature and more the time more conversion will be taking place and most important is the catalyst as you have discussed the heterogeneous catalysts are getting more interest because the homogenous catalyst like base catalyst are having some typical problem.

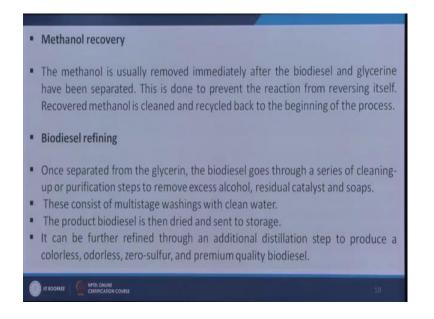
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	Solvent used	Catalyst	Operating conditions				
Feed stocks			Temp (°C)	Alcohol oil ratio	Catalyst loading wt%	Reaction time (h)	Yield (%)
Sunflower oil	Methanol	SrO	65	12:1	3	0.5	90
Soybean oil	Methanol	K <sub>2</sub> CO <sub>3</sub> /TiO <sub>2</sub> (Ti:K = 0.65:1)	60	12:1	6	2	25.15
Babassu coconut oil	Methanol	Amberlyst-15: Sulfonated ion exchange resin	60	100:1		8	80
Waste cooking oil	Methanol	Sulfonated earbon based catalyst	80	30:1	10	4	92

So, I will you some example of the use of heterogeneous catalyst and different types of feed stocks that is different types of bio oil. So, waste cooking oil. So, from this we can get some idea about the range of different parameters which have been investigated. So, the temperature range is basically 60 to 65 is normal case, but in some cases here we are getting 80 degrees centigrade.

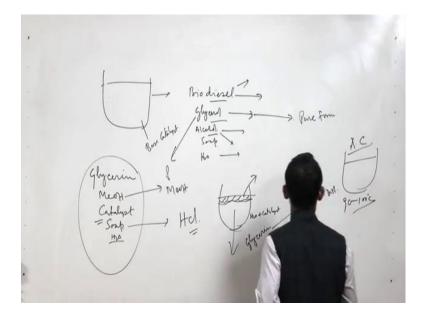
So, alcohol to oil ratio if you see 12 is to 1 in reaction we have seen that 1 mole of triglyceride reacts with 3 mole of alcohol, but in actual case the ratio is not 3 is to 1. It is more than that that is 12 is to 1 somewhere it is 30 somewhere it is 100. So, normally in most of the cases 10 to 9 12 these ratios are used and then catalyst loading catalyst loading is we can see here the various amount of catalyst has been used ranging from 3 percent to 10 percent. So, 4 5 6 percent are normally used and reaction time is also varying. So, maximum 8 hour. So, 15 hours to 8 hours in this table we are seeing yield we are also getting different than 20 5 percent to around 92 percent.

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Now, we will discuss on methanol recovery. So, in transesterification process we will get the products the bio oil or triglyceride is converted to diesel products that is the fatty acid alkyl esters. And then also some glyceride is formed and unreacted methanol that will be present in the product, and that has to be separated because that that can be used again for this process then biodiesel refining biodiesel will be; obviously, containing say.

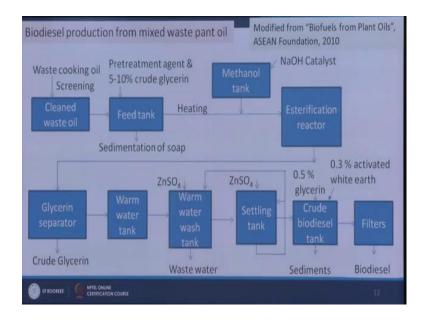
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The alcohol say we have the transesterification reactor. So, we are getting here biodiesel, we are also getting glycerol we are also getting alcohol. So, those are not used

some soap, formation will also be there if we use here base catalyst. If we use base catalyst, then there will be soap. So, we need pure biodiesel. We need pure glycerin we need pure alcohol. So, these 3 pure components we need.

So, biodiesel refining is necessary because these impurities has to be removed moisture. There will be water will also be there. So, that water has small amount of water may be present. So, that has to be removed. Now what that glycerin refining, what is the role of this step the rit is role of this step is to get purer glycerol from this, we will be getting purer form. So, we need to develop some methods through which we can get it. So, there is series of operations are there and we can get 99 percent or higher purity of glycerol.



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Now, we will discuss biodiesel production from mixed waste plant oil. So, here we see waste cooking oil, we have as a feed stock we want to produce biodiesel from waste cooking oil. So, our first step is screening. So, we will be screening it to remove any floating material on it. Then the cleaned waste oil will be having and we will store it in a vessel. So, this cleaned waste oil will be pumped to the feed tank. This feed tank may be filled up to say 80 percent of it is volume then it is maintained at say 50 to 60 degree centigrade temperature and pretreatment agents are added here.

It also includes 5 to 10 percent of crude glycerin in some cases. So, 5 to 10 percent crude glycerin if it is added that helps to prevent the solidifications of the feed stock and pretreatment agents helps to convert free fatty acids to soap and then ultimately we will

get the soap here. So, that soap will be sediment here. So, separation is required. So, for this we need initial stirring first. So, some stirring will be there in the feed tank and then we have to allow certain time for the settling of the particles or the sludge will be produced here.

So, the temperature already I have mentioned that is 50 to 60 degree centigrade and around 20 minutes stirring is there is required, and then around one hour time is given for the sedimentation. Then the output from this feed tank that is hearted to around the 70 degree centigrade and then it mixed with sodium hydroxide and methanol solution. So, methanol tank in the methanol tank sodium hydroxide catalyst is used, and the ratio is around say 4 percent of sodium hydroxide is added in the methanol tank with respect to it is the volume of methanol and then it is mixed with the heating oil which is coming here.

Now, this temperature is around say 50 degrees and that NaOH addition produces some heat here. And that heat makes it suitable to raise the temperature to around 50 degrees centigrade. So, 50 and 70 degrees centigrade these materials are mixed and then it is send to the transesterification reaction reactor. So, in the transesterification reactor it be may be of different types the tubular if it is a tubular type of reactors. So, a typical time is around half an hour and the temperature is say 65 degree centigrade 60 to 65 degree centigrade in the transesterification reactor. So, here the basic reactions take place for the transesterification. So, triglyceride is converted to glycerol and the fatty acid esters alkyl esters. And then some unconverted alcohol is there catalyst are there.

So, we need to purify this product. So, the product which we are getting here the product is coming out it will be having the full of impurities. So, we need to remove the impurities from our desirable product our desirable product is your first is biodiesel. Second is the glycerol and third one is also methanol. If you use methanol for this esterification reaction then the product it is going to glycerin separator. So, glycerin separator how this can be if we think about the different 3 major components, so a biodiesel glycerol and alcohol out of this it has maximum density rho is maximum then this and then this alcohol. So, on the basis of density, it we will get different layers.

So, the crude glycerin separator the glycerin separator can work on the basis of separation in the density. And the crude glycerin will come from the bottom part and

from the top we will be getting the biodiesel. And methanol will be present in both the cases, as catalyst will be present in the both the streams. And your additional catalyst will be present in soap if some soap is formed in the reaction that will also be present in both the streams. So, we need further purification of both the streams, that is biodiesel stream and there is glycerin stream. So, for this purpose the biodiesel is first force passed through the warm water tank.

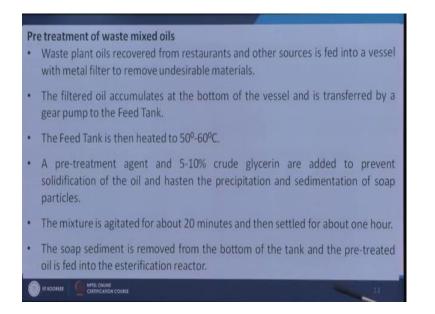
So, warm water tank helps the vaporizations of the alcohol or methanol particularly which is present here from the biodiesel and then it is going to warm water wash tank. So, we use warm water to wash it. So, that the soap is there catalyst is there. So, all those materials will come into water phase. So, catalyst and soap will come into water phase and we will be getting the biodiesel in different phase and sin there and it is biodiesel is coming to settling tanks. So, if we allow some time here. So, then if there is some other materials impurities present in this biodiesel that can also get some chance to settle and in both the cases you are using zinc sulfate and zinc sulfate helps to break the emulsion.

So, water alcohol and biodiesel emulsion that is broken down by this the presence of zinc sulfate, and more waste water is generated and this is the output of the settling tank that is purified and again sent to this warm water wash tank which helps for the purification then from the setting tank, we get apparently pure biodiesel, but this is not very pure it is also require some further treatment and it is sent to crude biodiesel tank.

So, in crude biodiesel tank we add 0.5 percent glycerin and 0.3 percent activated white earth. So, these 2 are used the main purpose is to get the sediments separated from this mixture and if we need further purification we can send it through some filters in the paper filters like this and we can get the biodiesel. So, here we have we need here say around say certain time we need say 15 minutes of stirring and overnight it is settling time for the process of the separation of sediments from the crude biodiesel tank. So, this is the overall flow sheet for the production of the biodiesel from mixed plant oil.

So, these are the in this slide the pretreatment of waste mixed oils how the pretreatment is done it is given in details, already we have discussed now esterification process we have also discussed it is given some detail.

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And then these are things that I have already we have discussed now we are going to glycerin recovery. So, in the flow sheet we have seen that here we are getting glycerin and then this stream is for biodiesel and it is purification and here we are not shown any steps for crude glycerin separation, but you see what is the composition in crude glycerin. So, glycerin methanol, catalyst soap basically and to some extent moisture any in the feed stock that is the composition of this crude glycerin.

So, we have to separate. So, again the boil if we heat it if we heat this one the whole mixture then methanol will go off, methanol will go off. Then we need to separate catalyst and soap and H 2 O.

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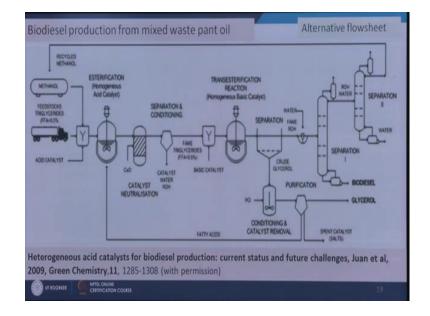
Glycerine recovery
The crude glycerine from the <b>glycerin separator</b> is stored in a <b>neutralization tank</b> , which is glass-lined and equipped with a closed-type agitator.
It is heated under normal pressure to distil out the methanol, which is recovered from the condenser located at the top of the <b>neutralization tank</b> .
The remaining crude glycerine is neutralized by the addition of hydrochloric acid and the residual soap is converted into oil, which is removed from the upper layer.
Crude glycerine is taken from the bottom of the <b>neutralization tank</b> and pumped first to the <b>crude glycerin tank</b> and then to the <b>crude glycerin concentrator</b> for further treatment.
The crude oil that is skimmed off from the <b>neutralization tank</b> is burned as fuel for heating the <b>vacuum distillation unit</b> .
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So, first step of this glycerin purification is that it is stored in neutralization tank and neutralization tanks and then it is it is glass lined and equipped with a closed type agitator, and then it is heated under normal pressure to out of to out the methanol. After that when methanol is being out we are having these things and soap is also there if we add some acid say HCL. So, soap will be converted to ester and again it will form one oil and it will goes to the top of this mixture if we have glycerin mixture. So, HCL is added.

So, some oil will form here because the soap will be converted to esters and then we will get some oil and that oil is separated and used for other applications. Then the next part which we are getting that is basically having H 2 O and catalyst. So, when we will neutralize the catalyst sodium hydroxide part is also removed. So, then crude glycerin is taken from the bottom of the neutralization tank and pumped first to the crude glycerin tank and then to the crude glycerin concentrator for further treatment. So, after that after this HCL treatment and removing of this upper part of that oil that oil we will be getting relatively pure glycerin, but still if we need more purification of this then we have to go for further steps.

So, what are those further steps, then we can go for vacuum distillation. We can go for vacuum distillation, if we need further purification the vacuum them we can go for addition of activated carbon. Activated carbon if we use add if we allow certain time here. So, the activated carbon will capture all the impurities present in the glycerin

particularly the colors and it requires a 15 minutes' time, at 90 to 100 degree centigrade temperature, and the requirement of activated carbon is around 2 to 3 percent of the total glycerin which is present. So, that way we can get the 99.5 percent pure glycerin.

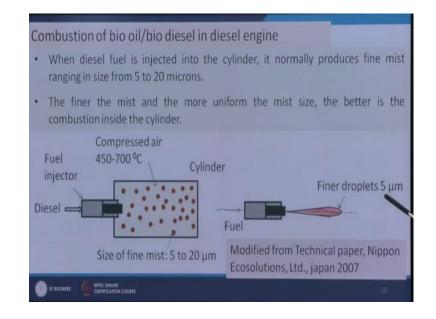


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Now, we will see one flow sheet for the conversion of grease or where the fatty acid content is higher to biodiesel. At first the feed stocks are coming into the esterification that is acid esterification step acid catalysis is used and then the free fatty acid is converted to esters. And then it is coming it is neutralization acid is neutralized then it is coming for separation, and then we are adding some base catalyst and then transesterification reaction is going on than we are, getting the products the product separation is going from the bottom we are getting the glycerin part from the top we are getting biodiesel and alcohol then this is separated here by distillations and biodiesel and methanol are going from the top parts.

So, this alcohol or methanol again used for this reactions, and from the bottom we are getting the glycerin it is being purified and catalyst are being resonated and we are getting the catalyst spend catalyst. So, this is the flow sheet with respect to the conversion of high fatty acid containing oil to biodiesel, this is the biodiesel.

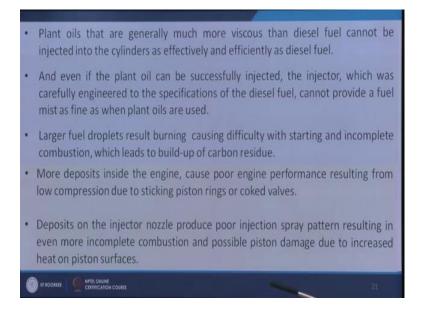
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Now, we will discuss how this biodiesel can be combusted or can be used in engine particularly the diesel engine. So, in diesel engine what happens when the fuel diesel fuel is put through the nozzles in the cylinder? So, droplet us are formed. So, these droplet us particularly 5 to 20 microns range as shown here. So, if it is a nozzle the liquid fuel is passing through it. So, here we will be having the 5 micro meter particles and if we move towards this nozzle, the diameter is also increased. So, 20 to 5 micrometers this is the normal range of the droplet us which is generated in the cylinder.

Now if we use the biodiesel. So, due to it is high viscosity the droplet size will be higher at first due to high viscosity the nozzle will not be that much efficient. So, it will not be able to spray it and to make the droplet us and. Secondly, if it is possible then the diameter will also be higher.

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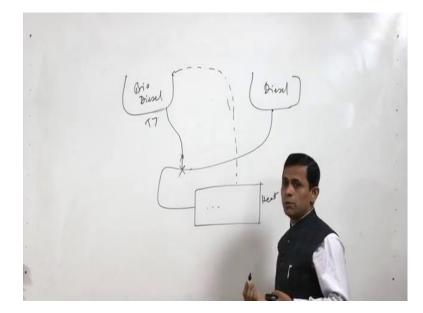
So, higher the diameter it will restrict the full combustion. So, there will be some deposition of coke and due to the deposition of coke there will be many problems in the engine that is why the blains are used the 20 b 20 like say 20 percent biodiesel and 80 percent diesel. So, more deposit is inside the engine poor engine performance resulting from low compression due to sticking piston rings or coked valves, and deposit is on the injector nozzle if injector nozzle. There is some coke deposition then that will not be able to inject the materials or the produce the droplet us efficiently. And there will be some damage on the engine and lower the performance of the engine.

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Modification in engine technology for handling viscous oil like bio oil
Twin Tank or Dual Fuel System
The engine is started on the conventional diesel fuel and the waste heat generated by the engine is used to heat the plant oil.
This avoids running viscous plant oil through a cold engine at start up.
A temperature gauge in the fuel line determines when the plant oil is hot enough for the fuel supply to be switched to 100% plant oil. The fuel supply is switched by a three-way valve.
Before shutting down for extended periods, the fuel supply is switched back to conventional diesel fuel and the engine is allowed to run for about 3 to 5 minutes so that any residual plant oil is flushed from the fuel system.
The vehicle or engine is then ready to be restarted on diesel fuel.

Then what is the remedy of this the research is going on to improve on engine side as well as on oil side and one of such efforts that is twin tank or duel fuel system. So, duel fuel system, and twin tank means in this case the conventional diesel and biodiesel both are used in separate tank. So, there are 2 separate tanks one is for conventional diesel and other is for biodiesel.

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So, from the conventional biodiesel and this is say diesel petro diesel. If it is coming and is going to the combustion chamber, so this combustion chamber at the initial stage or the starting of the engine will be using this fuel it will be combusted here heat will release, waste heat will used for the waste heat will be used to raise the temperature here.

So, temperature will increase once the temperature is increased to sufficient extent then it is viscosity will get down, and it will get the flow ability and at that time it is flow will start it will go through this. And there will be 1 valve 3 a valve. So, that 3 a valve will manage the flow from this tank or this tank and when we will stop the engine again we will stop this line then this line will open and 4 5 minutes will be this diesel will be used. So, that there will be no chocking inside the lines it will be clear. So, for further applications there will be no difficulty. So, this is one of the important development on the engine that is twin tank or dual fuel system. So, that way to some extent the difficulty or the disadvantage of the biodiesel is tackled. So, up to in this module.

Thank you for your patience.