Principles and Practices of Process Equipment and Plant Design
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Module - 05
Lecture - 64
Design of a 10 TPD Mono-nitrotoluene plant

Hello, good day to you all. So, today is going to be the last session on our entire course and we are going to conclude with Design of a 10 Tons Per Day Mono-nitrotoluene plant and we will start with that right now.

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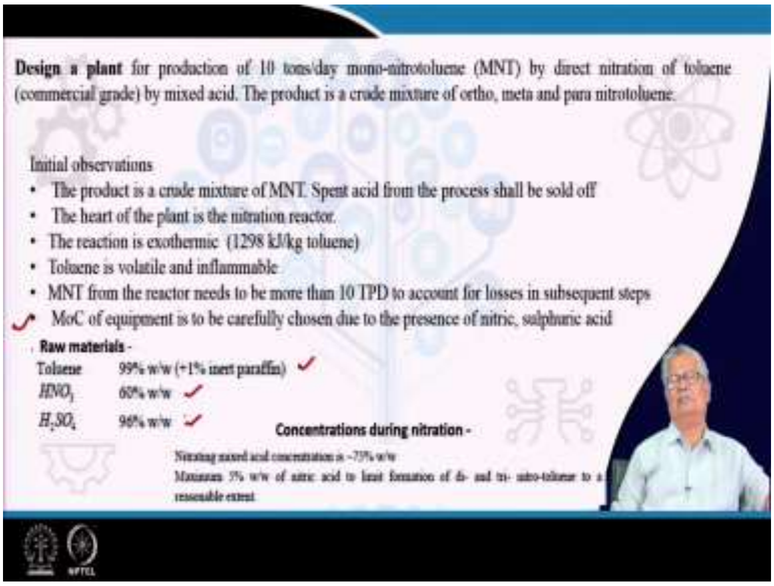

It is like this - let us have a look at a design statement and you already know most of the design statements posed to a consultant like you or even me or anyone for that matter is usually imposed; that means, every information is not available when the proposal has come to design a particular plant.

Here you are supposed to design a plant for production of 10 tons per day of mono nitro toluene. The process is also specified it is by the direct nitration of toluene of commercial grade by mixed acid. Whenever we say mixed acid we really mean it is a mixture of it is a mixture of sulfuric acid and nitric acid which is also called nitration acid. The product is supposed to be a crude mixture of ortho, meta and para nitro toluene depending on where the nitration takes place.

Let us look at the problem and have a few initial observations the first observation is that the product is a crude mixture of different species it is not a single compound. The process will also generate a spent acid and there is a market for that this was told to me not during the design of the plant, but later on. The heart of the plant is definitely the nitration reactor.

Well, it is very important because of few things. The first thing is the reaction is exothermic the heat of reaction is pretty high. It is 1298 kilo joule per kg of toluene. Toluene is a hydrocarbon which is highly volatile and inflammable. It is basically as dangerous as a sample of petrol or gasoline water we may call it.

Mono nitro toluene from the reactor needs to be more than 10 tons per day though the syllable amount has to be 10 tons and this additional amount is to account for the losses in the subsequent steps of processing. Material of construction of this particular plant equipment is to be carefully chosen because of the presence of nitric acid, sulfuric acid; that means, it is a pretty corrosive environment.

Now, let us look at the availability of raw material. Commercial toluene, nitric acid and sulfuric acid; the specifications are 99 percent weight, 60 percent weight and 96 percent weight and toluene can contain up to 1 percent inerts which is paraffin and it will not play any role in the reaction, it will just remain as inert. During nitration, the literature says that there are quite a few conditions.

The nitrating mixed acid concentration is expected to be around 75 percent because that is how under this condition the rate of reaction has the rate equation for this has been generated. Maximum of 5 percent weight by weight of nitric acid is to be the limit of nitric acid concentration in the reactor.

And this would limit the formation of di and tri nitro toluene which are not desirable products. Well, some amount of formation is inevitable but, a practical limit can be set by limiting the nitric acid concentration during nitration to 5 percent.

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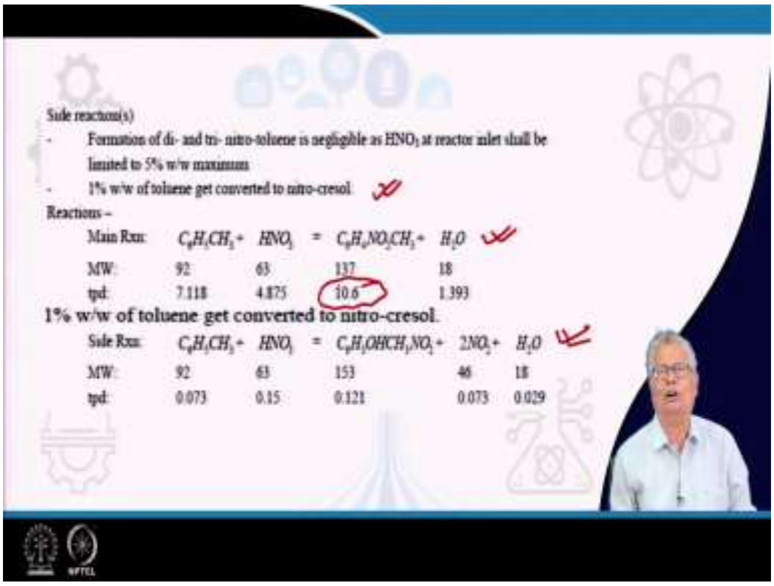

Now, there are few types let us look at the reactions. We are going to consider the formation of dinitro toluene and tri nitro toluene as negligible and however, some amount of formation of nitro cresol is inevitable.

Now, the reactions are the main reaction is here and the nitro cresol formation reaction is here along with the molecular weight of the different species as well as the ton per day flow rates. This is nothing but plane stoichiometric calculations. Now, you will see that instead of producing 10; instead of producing 10 the reactor is expected to produce 10.6 tons of your mono nitro toluene.

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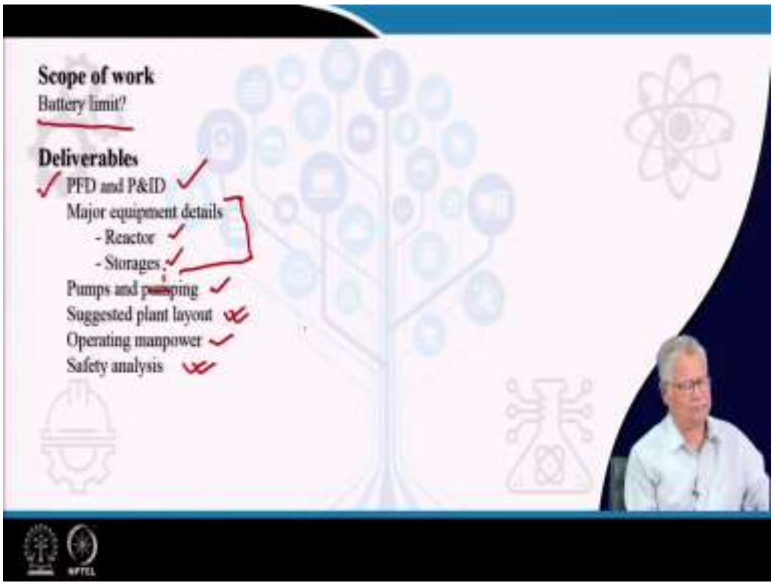

Once we go for the plant design we need to have an idea about the scope of work which is involved. It is a physical system that we got to design; that means, we need to know about the battery limit completely. What all equipment what all facilities will be there within the limits of the design or limits of the plant for that matter we need to be very clear about the deliverables. How will we decide the deliverables in this case? It is a very small plant, very few equipments are expected.

So, we have to have the process flow diagram that is a basic thing. You will definitely be looking forward to I mean to whomever you deliver it, he will be asking for a process and instrumentation diagram because it will include the instrumentation that he has to add to his plant.

The major equipment details have to be supplied because it is basically a process package that we are going to design and that would include the reactor and the storages, pumps and the piping and you are definitely supposed to give out a plant layout.

The operating manpower requirement for your plant you know the plant base. So, how much of manpower is required in order to operate this plant normally has to be specified by the designer. You are also to provide the safety instructions for safely running the plant, dismantling the plant, commissioning the plant, maintaining the plant.

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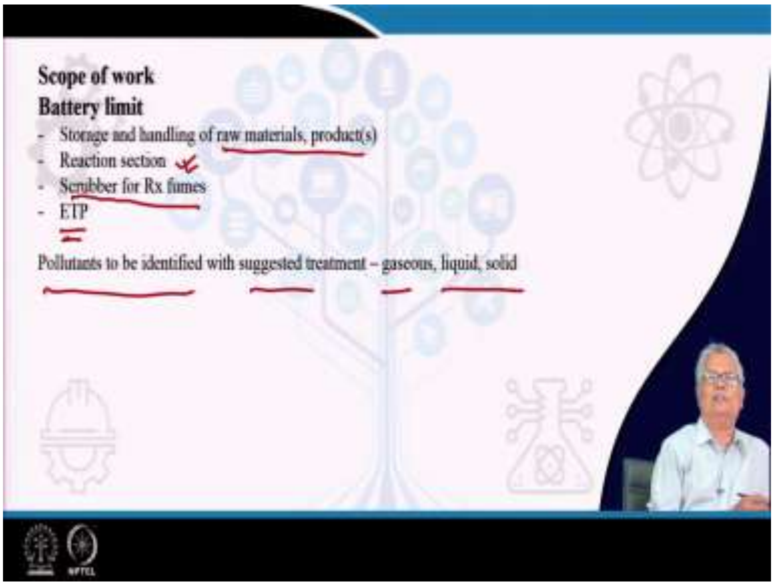

Let us look at the battery limit scope. It must contain the storage and handling of raw materials and products; that means, what it is supposed to contain it must contain the storage tanks, the type of tank, location of the tanks, the facility of the tank and the handling of the raw materials and products will be using pumps and pipelines. You have to have a facility for unloading the raw material from the tank lorries sending it to the tank after some sort of measurement.

So, quite naturally whenever I am talking about the raw materials and products being received there has to be a facility for loading and unloading tank lorries and the quantity amount which is loaded or unloaded normally will be found out by the difference in their weight.

So, you have to have a weigh bridge inside your facility itself. So, you will have tanks, you will have pumps, you will have safety arrangements to handle these very well and naturally you must have a tank lorry facility for loading and unloading.

And, a measurement to be done by using some sort of weigh bridge facility within your premise. The other part is the reactor section this is the heart of your equipment. The reactor the reaction if you have noted the formation of the secondary product is also accompanied by formation of NO 2; that means, there will be fumes from your reactor. This fumes if you cannot let it out to the atmosphere without any treatment.

So, there has to be a scrubber to remove the fumes from the reactor unit. You are going to generate a good amount of effluent also. You are going to you are going to handle hydrocarbon you are going to handle acids you are going to acid handle fumes you are going to scrub the fumes and generate some sort of acidic water. So, all these have to be treated in your effluent treatment plant and only then it can be sent out.

The specifications of what you can send out of your plant we have already understood while discussing the pollution control aspects and the safety aspects earlier. It has to be it is it has to respect the minimum national standards which is MINAS and as well as the state and the CPCB norms. Your scope of work is also expected to identify the pollutants. What are the expected pollutants and what are the suggested treatment for the gaseous, liquid and solid and what is the desired method of disposal there?

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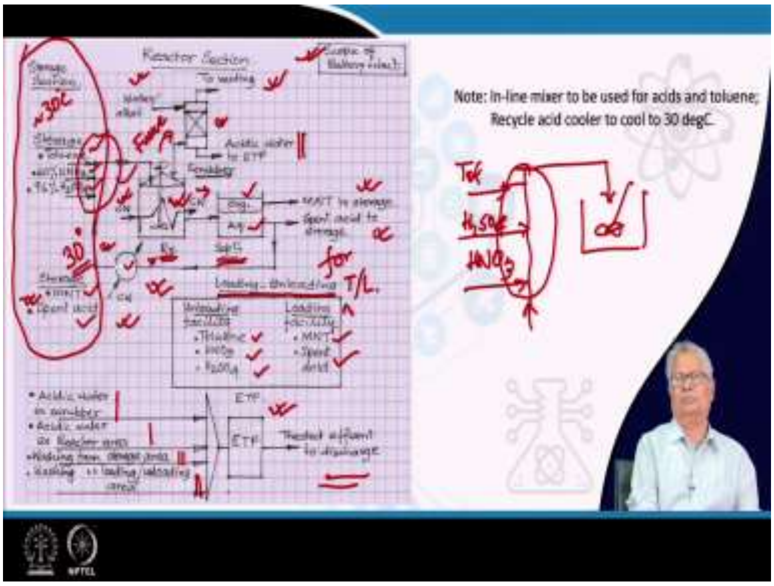

Now, let us see a schematic which represents a plant. To be very frank it is not a proper PNID because it does not show the instrumentation it shows basically the equipment at the different section of your plant primarily to identify the scope of your battery limit. You see here you have a storage section. What does the storage section will contain? It will contain the storages for the raw material toluene 60 percent nitric acid and 96 percent H 2 SO 4. The percentages are percentage weight by weight.

The finished product mono nitro toluene also will require a storage tank and you require a spent acid storage as well because from the spent acid tank you have to send out to some customer who will be buying your spent acid. Normally, this type of spent acid is used for neutralizing certain strong alkali, which may be a waste product in some other industry most of the case it is like this.

Of course there may be some other cases where people may be interested in taking out the H 2 SO 4 and HNO 3 it present in your spent acid to the extent possible and if it is economic. The raw material will be fed to your reactor. The reactor is supposed to be a CSTR. It is a continuous starred tank reactor; this is the one.

You will find that all these are mixed; that means, you have your toluene, you have your H 2 SO 4, you have your HNO 3 – all these three are mixed and they are mixed to with a recycled stream as well. It is a recycle stream of the spent acid and it is sent to your reactor.

Now, this is a mixing arrangement that you will be requiring. We have not gone into the detailed design of this, but possibly this is going to be some sort of inline mixing and if required ejectors may be used in this as a mixer.

It is also possible for you to go for some other type of mixing arrangement and mixers, but obviously, it is expected since there will be fume generated when they come in contact and there will be heat generated also. So, it should be some closed system. Inline mixing apparently is a good option in this case.

Now, let us see we already have mentioned that the reaction is highly exothermic. So, your reactor definitely will must will require some sort of cooling. We will see at what temperature we would like to conduct this. But, one thing is true that your storage temperature will be something around 30 degree centigrade; that means, in our design we will be considering something around 30 degree centigrade to be the storage temperature, which is an average storage and a reasonable storage temperature certainly.

The moment there is a unreacted amount of acid or another spent acid coming in, along with the spent acid you will also be having certain amount of water. The water is formed during the secondary reactions it during the reactions in your reactor itself and will be there in the aqueous phase and it gets separated.

Now, you have 96 percent sulfuric acid, 60 percent nitric acid, toluene and the spent acid which also contains water. So, there is some dilution here as well and there will be a heat of dilution which will be evolved in this case.

So, if I just have my recycled acid possibly it is going to add to the temperature of my reactor, instead it is thought that it will be wise to have a separate cooler using some sort of cooling water we will see what cooling water temperature we really require and have it sent here and will if this temperature is 30 degree centigrade, we will assume that this temperature is also 30 degree centigrade; that means, the cooler here will be cooling from whatever temperature here to a temperature of 30 degree centigrade.

So, you can consider isothermal mixing in line often mixing there will be evolution of heat and we will see what it amounts to immediately after this. Now, the reaction here is between liquid phases. There is no catalyst present. It is well mixed because of the presence of the starter that you have in your CSTR. The heat is being removed possibly by some through some heat transfer surface, the other side of which you will have a cooling water to remove the heat possibly some sort of tubes or it could be even jacket.

Possibly in this case since the rate of heat generation is high, just providing an external jacket may not suffice and you may in fact, what we have done while we have checked this we have found that it is you not only you require a single row of tubes, you require several rows of tubes here to remove the heat. We will see the details later on.

Now, as the reaction proceeds there will be product overflow from here. It will be sent to a separator vessel where it will settle into a lighter organic layer which is primarily the mono nitro toluene and an aqueous phase which is the acid phase. The mono nitro toluene will be sent to the storage. The storage is here and the spent acid part of it will be sent to the store I mean the it will be sent to part of it will be sent to the storage and the rest amount will be recycled back through the heat exchanger after cooling.

You will also require in your battery limit a loading unloading facility for the tank lorries and these will be the facility for the toluene, nitric acid, sulfuric acid, mono nitro toluene and spent acid the other two the last two will be sent out; that means, that will be under loading facility the rest will be unloaded there.

If we look at the ETP let us look at the streams which will be going there and before we go there we just see what happens to the fume that gets generated here is the fume what does this fume contain the fume contains NO 2. So, this fume has to be scrapped and the NO 2 has to be removed from there.

This scrubbing can be done with plain water or it could be done with some little bit of alkali, it could be caustic soda, it could be even light dilute lime solution also, but if you are going to use lime solution there is a chance that there may be clogging in the packing that may that you may use in your scrubber.

So, possibly what you will be preferring in this particular case is a very dilute solution of acoustic the fume comes it goes up through the packing. The packing definitely has to be inner to the acid fumes. It meets the neutralizing water or alkali from the top, the acid vapor the acid component gets removed and it is vented out to the atmosphere.

The acidic water generated from the bottom of this has to be neutralized at least neutralized and before it is sent out from the plant itself it has to conform to this specifications which is permitted. For that you definitely will be requiring the effluent treatment plant facility or ETP within your premise.

So, the streams coming to the ETP will be the acidic water which I have just mentioned from the scrubber. The acidic water see you have an reactor which handles a good amount of acid. So, there will be some amount of leakage, spillage and the water, which is used for washing in that area will possibly be acidic as well.

So, you are going to receive some amount of acidic water from the reactor area and you are also going to receive a good amount of washing wash water from the storage areas also and there will be wash water from your loading unloading facility as well. It is expected that your wash water will contain hydrocarbon small amount, but it will still be there and it is also expected to be acidic.

The acid has to be neutralized that should be required step before it is treated further and sent out to the effluent discharge from your plant. So, that is basically the scope of another plant, which is going to be an affluent treatment plant and the treatment facilities will be removing primarily the acids and your small quantity of hydrocarbon.

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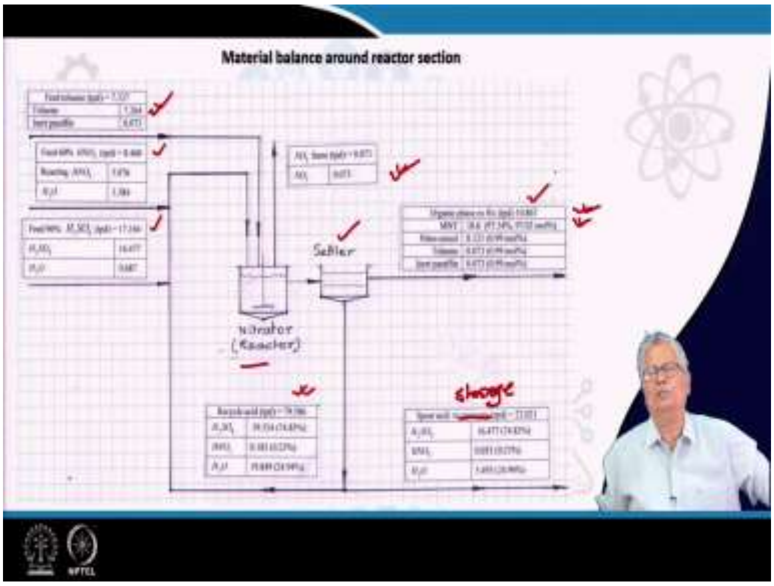

Now, let us move forward let us see. We have a look at the material balance around the reactor section. So, what you have there is a nitrogen reactor and a settler. You notice here there is a large amount of recycle; the recycle is close to about 80 tons per day the reason is the ratio of the acid to your toluene has to be high.

Because if you are and it has to be maintained at a specific rate, which is arrived at through a set of calculations. You will also note that a good amount of fume has to be disposed of every day; it amounts to about 73 kgs a day. Your organic flow rate will be about 10.867 which is approximately your 10 point instead of 10.6 it is 10.7. In fact, it has got roughly around 10.6 of mono nitro toluene. The others are nitrocresol unreacted toluene and small quantity of inert paraffin which remains there.

The entire thing is arrived at by calculations based on the conversion and the stoichiometric calculations. You will note here a spent acid to recovery section basically it is not exactly recovery you can call it will be going to the storage section for sailing later on will be approximately 22 tons a day.

So, you are going to send out roughly about 22 tons a day of spent acid, which will amount to about 25 about say 20 meter cube. So, you will be sending out roughly one tank truck load of spent acid out of your factory. If you look at the capacity of the toluene which is going to come in it is roughly about 7.26 which is this amount.

So, you will have every day at least one tank lorry of 10 meter cube coming in. If you look at the nitric acid and the sulfuric acid which is totally amount to about 25, 26 tons so, this will require about 3 tank lorries to be decanted every day.

Your product, which is going to be about 10 point sorry, about 10.9 tons so, you will be requiring roughly about one tank lorry of about 15 kilo liters leaving with your product mono nitro toluene which is in the crude form. So, what I would like to say is if you look at this you will be having your idea that what all should be your in modes of input, what all should be your modes of dispatch from your plant as well.

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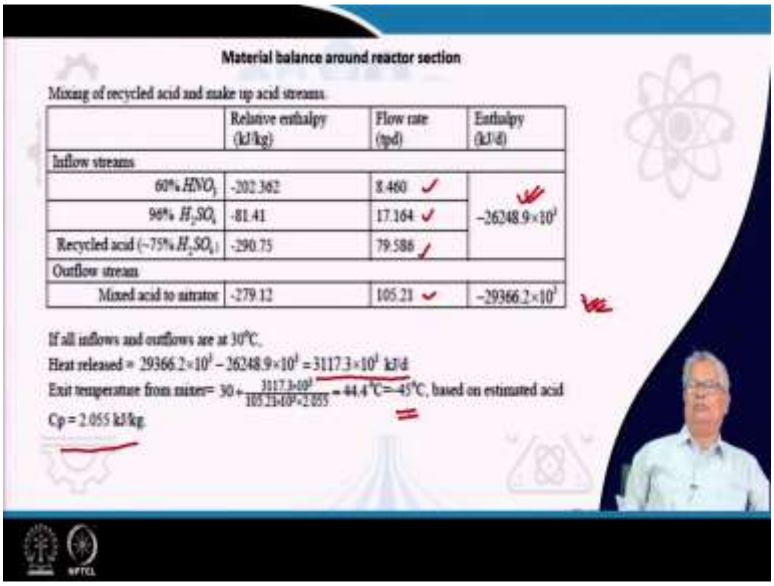

Now, let us go into more details of this reactor section. The quantities are known now. In tons per day and the flow rates are written here. These three are mixed and the total acid to the nitrator is this. Now, what happens is the enthalpy coming in to the nitrate is this amount and you will see; that means, the heat release quantity is going to be the difference between these two; that means, you have to have arrangement to re take out this much quantity of release.

Based on a specific heat of around 2.055 kilo joule per kg for your reaction mixture, the exit temperature from the mixture assuming everything enters at 30 degree centigrade comes to around 45 degree centigrade; that means, you will it means one thing; that means, your nitrator will be operating at a temperature of around 45 degree centigrade provided you are removing this much quantity of heat.

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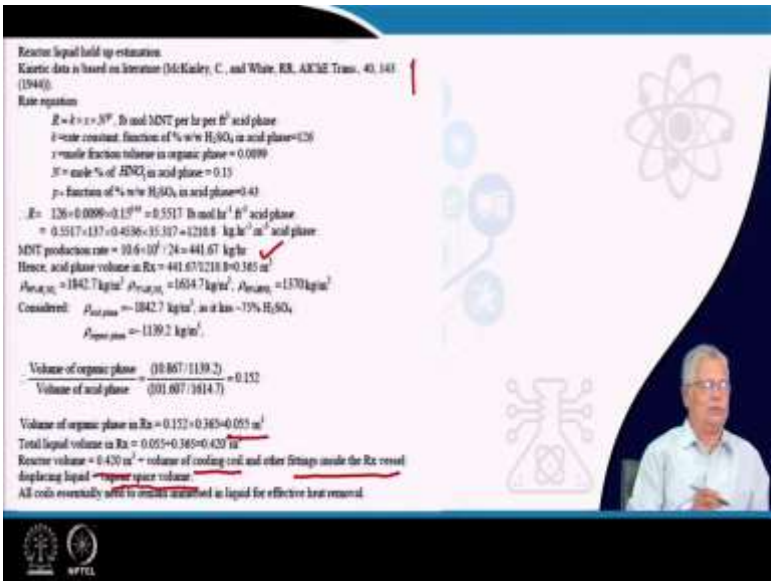

Now, let us look at the reactor hold up estimation; that means, the volume of the reactor. It is a liquid-liquid homogeneous reaction. The kinetic data is well established and the reference is available here. The rate equations are given and we know the production rate also is amount amounting to this much. Based on this if you calculate you will find that the volume of the organic phase required is roughly about 0.055 meter cube.

Now, there is something. The reactor volume will be how much? The reacted volume will be 0.055 plus the inorganic volume which is basically the aqueous volume of the acids, which is making a large part of it. The proportion of the aqueous phase present in your reactor is much large as compared to your volume of the organic phase. This is primarily required to limit the concentrations so that you get your desired distribution of products.

The total reactor volume thus amounts to about 0.42 plus the volume of the cooling coil if you are having cooling coils inside and other fittings which is inside your reactor vessel, plus definitely you must have a vapor space volume to added to this also you will notice one thing all coils must essentially remain immersed in liquid for effective heat removal.

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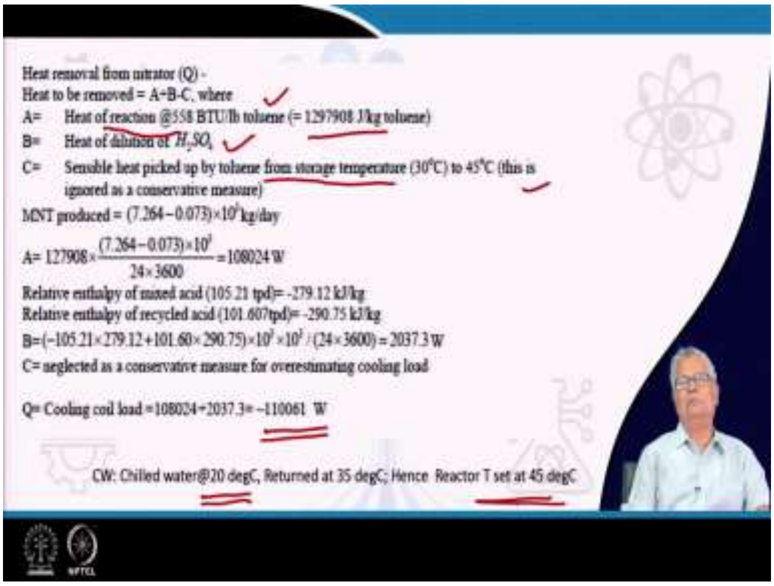

The heat removal from the nitrator is equal to the heat of reaction which is 558 BTU per pound of toulene which amounts to this value which we have talked about earlier plus the heat generated by the heat of dilution minus the sensible heat picked up by the toluene from the storage etcetera. This is in fact, ignored based on this the cooling coil heat load is estimated to be 110 kilowatt approximately.

You will notice one more thing also here. What you require is a cooling to a temperature of close to 30 degree centigrade. The conventional cooling tower which gives you cold cooling water at a temperature around 35 degree centigrade will not work well here. So, what you require is a chiller.

And the chiller is expected to have a temperature of around 20 degree centigrade chilled water for cooling available to you. If you wish you can use the circulating system of chilled brine also. This is also quite common. Based on this the reactor temperature is set at 45 degree centigrade, which matches pretty well with the literature reported kinetic data generation temperature.

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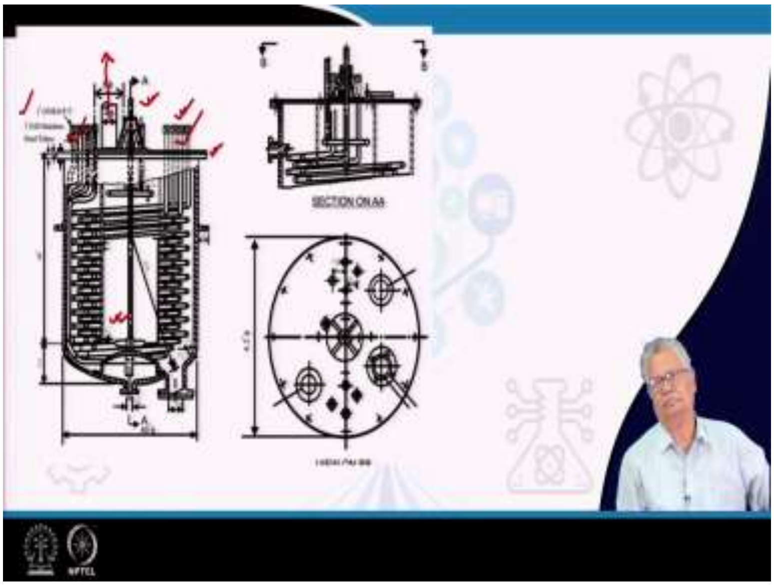

Now, here is the details of the reactor. In finding out the details of the reactor what you are expected to do is you are expected to find out the amount of heat transfer surface area that you are supposed to provide with your coils. For that what you require? You require the heat transfer coefficient at the cooling water side which is inside your tubes and the effect of starting.

Once you have found out the amount of hold up in your reactor and the amount of and the dimensions of your reactor so that you have all your tubes submerged it will be possible for you to refer to the basic design of the impellers for studying in one of your earlier classes and design your starter and its power requirement.

If you know your heat transfer coefficient inside your tube which you know basically because you are choosing here 1 inch OD stainless steel tubes and that you are using for your coils. And, if you are designing your coil diameters accordingly you will definitely be in a position to estimate the total heat transfer area required for the coils and move on.

Now, there is something which I would like to which I would like to draw your attention. If you look at the plan or even the side view you will notice a few things. The first thing you have here a vent from the flat top. This is for sending the vapor from this the fumes from this which are primarily nitrogen dioxide to the scrubber.

The head the head itself is removable. It is removable along with the entirety of your piping’s for the cooling water as well as your starter mechanism itself. So, the entire thing has to be fixed in such a fashion, so that it is fitted with some lifting arrangement possibly with a chain pulley which could be mechanized or it could be manual even so that the entire thing comes out and you can attend to the inside of your reactor.

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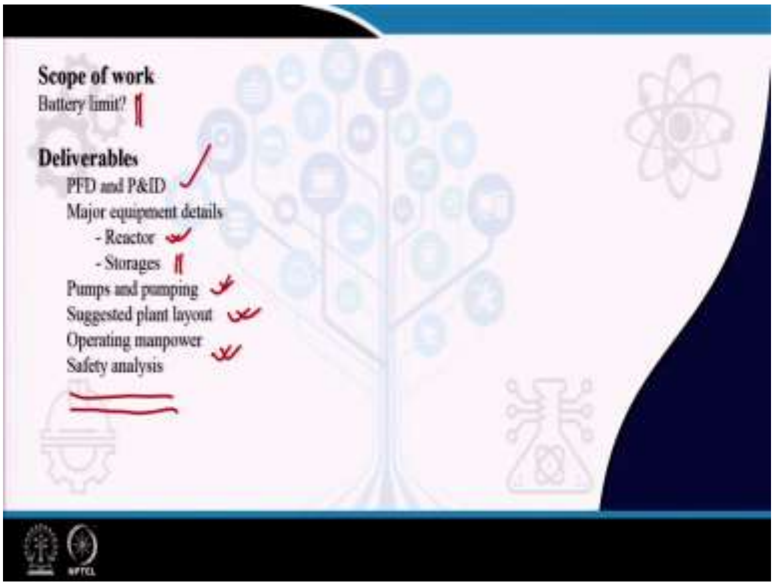

Now, what we do is we have more or less talked about the details of this plant, but we have concentrated more on the reactor end and the facilities that will be reacted in your battery. So, I will say we have fairly understood what the battery limit facilities are required, what battery limit facilities will be essential for this particular plant. It is possible for you to finally, develop the PFD and P and ID from the information which is given to you.

The major equipment details like the reactor is given to you; the storages are the storage vessels which normally you can follow your other process design class or you can refer to any standard book by HK and Ralston or any other book and find out what all will be the details for your storages typically the storages are considered for a storage time of approximately 2 to 3 days maximum 7 days in most of the cases.

The pumps and pumping details you can finalize by considering possibly in this particular case about one to one and a half inch pump and piping size in most of the cases and you can have a suggested plant layout based on the common sense that you will have to have your effluent treatment plant and your loading unloading in one side of your plant where you are having less risk and a high risk site should be segregated from the rest of the plant possibly by a wall or some such barrier.

The operating manpower normally you decide by different sections. Normally in during operation you will be requiring one operator for your who is going to attend for your tankage one for your in fact, two persons for your reactor and one person for your loading unloading facility.

This is apart from the maintenance crew that you will be requiring for regular maintenance which keeps on happening in every plant. Regarding safety analysis it is very important to note here that you have to do a HAZOP analysis for your plant and the basics of HAZOP you have been introduced to, but we are not keeping it within the scope of today's thing.

And, I suggest that you attend and have some idea of the hazard analysis by HAZOP particularly for this type of operating plants and try to do it for yourself first and that is going to give you a fairly completely generated process design which you can document and send to your client. I think with this I will conclude the course and I will just draw your attention to a few features that is whenever you are to design something decide on the battery limit. What is the scope of work, define it very very clearly.

Split your battery into different sections look at the exchange of energy and the material the streams particularly and the relationship between the different sections. Design these individual sections with full concentration keeping in mind the constraints of connectivity; that means, there has to be the same standard to be followed for every section.

The safety analysis has to be done individual section wise and also for the entirety of the plant because safety in one section can always get jeopardized if the safety in the other section is not closed properly followed. I think with this I will conclude the course and on my behalf and on behalf of professor Gargi Das we thank you for being attentive and hope you get benefited from whatever is being delivered to you.

Thank you all, bye.