

Electrochemical Technology in Pollution Control
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Lecture – 26
Process waste handling 2

(Refer Slide Time: 00:40)

METAL FINISHING INDUSTRIES INCLUDE:

- i. Electroplating
- ii. PCB Manufacture
- iii. Phosphating, Anodising , Coating
- iv. Painting

Hello students greetings to you, we were discussing the Metal Finishing Industries that included Electroplating, PCB Manufacturer, Phosphating, Anodizing, Coating and Painting etcetera.

(Refer Slide Time: 00:54)

AFTER METAL FABRICATION THEY ARE FINISHED TO FINAL PRODUCT REQUIREMENTS THIS INVOLVES:

- **STRIPPING**
- **REMOVAL OF UNDESIRABLE OXIDES**
- **CLEANING**
- **PROCESSING – PLATING, PHOSPHATING , ANODIZING ETC.**
- **RINSING**
- **DRYING**

And we were also covering what type of operations they do that is stripping, removal of undesirable oxides, cleaning, processing plating, phosphating, anodizing, rinsing, drying and all these things.

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The total liquid wastes in electroplating are not voluminous but are extremely dangerous and /or toxic.

Toxic contaminants are acids, alkalis, metals such as As, Cr, Zn, Cu, Ni, Sn, Ag, Rh, Pd, cyanides, alkaline cleaners , grease, oil, polymers, ammonia, Complexing agents, polyelectrolytes etc.

WASTES ORIGINATE FROM :

- a) BATCH OPERATIONS
- b) RINSE OPERATIONS

Followed by we had classified the waste that is total liquid waste, toxic contaminants etcetera and then I had shown you this slide followed by batch operation and rinse operations.

(Refer Slide Time: 01:19)

MAJOR TYPES OF WASTES FROM ELECTROPLATING

1. Proprietary solutions – process accelerators, surface finish
2. Cyanide concentrates – plating solutions and dips
3. Chromates – plating and rinsing
4. Cyanide rinse waters
5. Conc. Acid and pickling – stripping and cleaning of metal
6. Strong acid rinse waters – Acid dips, process solutions
7. Conc. alkalis – spent alkaline cleaning solution
8. Other wastes – metal compounds, oils, soaps, etc.
9. Cooling water, steam condensates – do not require treatment



(Refer Slide Time: 01:26)

The Acids – HCl , HNO_3 , H_2SO_4 } Present upto 10% in
Alkaline baths – Na_2S , CN , NaOH } saturated solutions

Cleaning is accomplished by organic solvents, pickling, emulsifiers, NaOH , H_3PO_4 and other phosphates, silicates, carbonates, wetting agents etc.

Cyanide salts are extensively used in plating because they are good oxide solvents and give brighter and less porous plating.

The character and strength of plating waste varies considerably depending on plating and rinsing operations. Cyanide baths are alkaline and chromate baths are acidic.



And then major types of wastes we had discussed and we had discussed about the salts cleaning etcetera.

(Refer Slide Time: 01:33)



TREATMENT OF METAL PLATING WASTE

1. MINIMIZATION OR ELIMINATION OF WASTE AND RINSE WASTES
2. PHYSICO-CHEMICAL TREATMENT

And I we were somewhere here when I finished my last class, that is minimization or elimination of waste and rinse wastes, followed by physico-chemical treatment.

(Refer Slide Time: 01:46)

IN THE FORMER SEVERAL STEPS ARE TAKEN DURING PROCESS

- ✓ Gravity feed, non-over flowing, emergency holding tanks for toxic metals and salts.
- ✓ Elimination of breakable components.
- ✓ Designing special drip pans, spray traps, shakers etc.
- ✓ Reduction of spillage, dragout losses and other losses.
- ✓ High pressure fog rinses in place of high volume water washes.
- ✓ Reclaiming valuable metals
- ✓ Returning the reclaimed metal to plating baths.
- ✓ Recirculating wet washer wastes from fume scrubber.

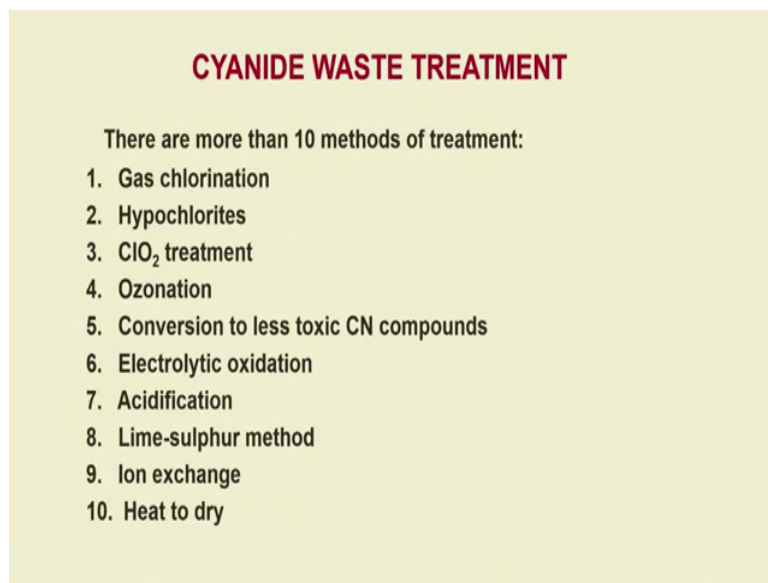
And in the former we had discussed gravity feed non over floating over flowing emergency holding tanks for toxic metals and salts. Here I had explained to you that we need the dikes for taking care of the overflow and then we also discussed about the elimination of breakable components, this we did not discuss actually but we were almost somewhere here when I closed the class. And this is of course, a very important component, because of the waste electro chemical waste and for to removes to stop all such things we will have to design special drip pans, spray traps, shakers etcetera.

And there are other ways for the reduction of spillage drag out losses and other losses, this is for the reduction of the effluents. So, high pressure fog rinses in place of high volume water washes helps quite a lot in reducing the drag out losses and the another aspect of electroplating is reclaiming the valuable metals that is very important. And especially if you are plating costly metals like gold and silver you would not know how much gold and silver you

will be losing during the process and post process, it makes sense to recover all the metals. Not only for environmental protection reasons, but also for recovery of the loss resources.

So, quite often we return the reclaimed metal to the plating baths, thereby reducing the operational costs and also recirculate the wet washer waste from the fume scrubber this is another process. Only big industries can afford to spend time and energy and money on such operations, especially recirculating wet washer wastes from fume scrubber etcetera. For small garage type industries such things would be unthinkable.

(Refer Slide Time: 04:17)



CYANIDE WASTE TREATMENT

There are more than 10 methods of treatment:

1. Gas chlorination
2. Hypochlorites
3. ClO_2 treatment
4. Ozonation
5. Conversion to less toxic CN compounds
6. Electrolytic oxidation
7. Acidification
8. Lime-sulphur method
9. Ion exchange
10. Heat to dry

So, now I am going to concentrate mostly on Cyanide Waste Treatment, because for other elements most of them are heavy metals they can be precipitated very easily and acid and alkali metal alkalis are can be very comfortably treated with alkalis and acids and the most important treatment waste from electroplating is a cyanide waste treatment.

So, let us discuss the cyanide waste for electroplating industries. In this context I want to tell you that there are even though cyanide is a known toxin and carcinogenic it is a killer and all

that. The use in the electroplating industry is phenomenal and the toxicity of cyanide has never been a deterrent for people using cyanide as a chemical treatment chemical, they have gold cyanide and many other cyanides as an electroplating chemical.

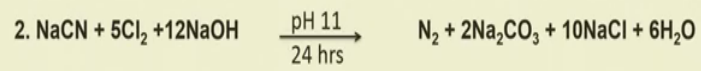
I personally have seen many people putting their hands like this in cyanide salts and then handling them without gloves and other things. Nowadays, such operations are almost unthinkable, but our teachers used to tell us that we must feel the chemicals, we must know how a chemical looks, we must know how a chemical feels. So, they used to encourage us to put our hands together in such chemicals and feel get a feel of the chemicals.

Nowadays, we will never advise students even to go near benzene, because when we want to prepare our benzene you would be very advised well to take it through a sucking arrangement rather than directly putting out from the mouth. So, such things are many of the common laboratory practices have gone out of their usual favor flavor, but still they are needed ok.

Coming back to the cyanide treatment there are more than 10 methods of cyanide treatment that are invoked and those include Gas chlorination treatment with Hypochlorites, chlorine dioxide treatment, ozonation conversion to less toxic cyanide compounds, acidification, lime sulphur method, ion exchange and heating to dryness many of these things are practiced even now depending upon the quantity of the cyanide to be treated.

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1. Chlorination is done in alkaline solutions Ca(OH)_2 / NaOH



About 6 kg NaOH and Cl_2 are required to oxidize 1kg of Cyanides.
Sometimes secondary chlorination may be necessary.

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CYANIDE WASTE TREATMENT

There are more than 10 methods of treatment:

1. Gas chlorination
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7. Acidification
8. Lime-sulphur method
9. Ion exchange
10. Heat to dry

A video inset shows a man with glasses and a light green shirt speaking.

So chlorination, let us discuss them one by one and at least in terms of chemical importance and first comes chlorination, chlorination is done in alkaline solutions. In acidic solutions whatever we do certain amount of cyanide gas will be HCN hydrocyanic acid will be generated which could be very dangerous. And of course HCN is not that dangerous as cyanide salt, but dangerous all the way. So, people can smell cyanide from a long distance and say there is some cyanide there and still fall back to safety and move away from the source of cyanide. But if you take cyanide internally in the body it is gone.

So, the chlorination is done in alkaline solutions where there is minimum chance of HCN gas being released. So, such alkaline solutions can be composed of calcium hydroxide or sodium hydroxide depending upon the requirement and the reaction should be approximately something like this. This is any salt of cyanide mostly sodium cyanide assuming that it is in the waste it is treated with chlorine gas and sodium hydroxide is added.

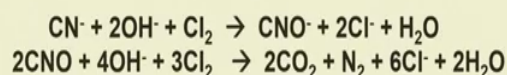
So, all in all alkaline pH is maintained around 11 and when it is treated with chlorine bubbling in the solution for almost 24 hours, we will have nitrogen, sodium carbonate and sodium

chloride and water. The equation has not been written with stoichiometric inputs, but the products would be there for same. So, about 6 kg of NaOH and chlorine are required to oxidize 1 kg of cyanide. Sometimes secondary chlorination may also be necessary, but again it depends upon the residual cyanide in the sample.

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2. 10-15% sodium hypochlorite

3. Stable bleaching powder



Cyanogen is not released under alkaline conditions. The second reaction takes place in 30-60 min.

So, we can also use 10 to 15 percent of sodium hypochlorite and we can use stable bleaching powder all these processes add chlorine to the cyanide solution. So, I can either go for chlorination or addition of sodium hypochlorite or simply add bleaching powder that is available in the market in hardware shops. So, it is very simple all you got to do is if you have cyanide somewhere around at home or something like that, you do not have to be afraid just go to a hardware shop get some bleaching powder mix it with that and that will degenerate into harmless sino CNO ions and nitrogen will also be liberated in alkaline conditions.

So, stable bleaching powder reactions are shown here that is a CN minus plus 2 OH minus plus Cl₂ goes to CNO minus, that is cyanogen oxide 2 chloride 2 times chlorine Cl minus that is it could end up as NaCl. But we are not writing sodium because, we assume that in alkaline

conditions even sodium chloride will also be ionized and then water of course is a product of the reaction.

Then this cyanogen oxide can react with alkali again and chlorine gas at to produce carbon dioxide nitrogen chloride and water. Cyanogen is not released under alkaline conditions usually never. The second reaction in which three chlorine atoms are involved molecules it takes place in about 30 to 60 minutes quite often. The first reaction is always faster that is $\text{CN}^- + 2\text{Cl}_2 + 2\text{OH}^- \rightarrow \text{CNO}^- + 2\text{Cl}^- + \text{N}_2 + \text{H}_2\text{O}$ that is very fast.

In general it takes the moment you had and just stir it all the reaction is over, but the second part that is cyanogen to CO_2 and nitrogen that will take longer time. And the it has nothing to do with the chemical, but it has something to do with the kinetics. So, you does not mean that if you have very less it the reaction goes fast not at all, cyanogen it is not released under alkaline conditions so you are safe there and second reaction takes place 30 to 60 minutes, but still that can be managed.

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PRESENCE OF ACTIVATED CARBON, OXIDIZABLE MATERIALS, SIDE REACTIONS WITH N_2 TO NO_x , NCl_3 ETC., RESULTS IN HIGHER CHLORINE DEMAND THAN THE STOICHIOMETRIC REQUIREMENT.

4. ELECTROCHLORINATION

Pt/Ti Anode, Ti-cathode 250 ASE produces NaOCl liquor from NaCl

$\text{CN}^- \rightarrow \text{cyanate} \rightarrow \text{N}_2 + \text{CO}_2$

NO SLUDGE

When both chromate and CN^- are present, cyanide should be treated first and destroyed. Then chromate may be Recovered using cation exchange or precipitated as chromium hydroxide $\text{Cr}(\text{OH})_3$.

And then sometimes we allow we put a certain amount of activated carbon and then we also put some oxidizable materials and then side reactions also will take place with nitrogen. What else can happen? Nitrogen can get converted to NO_x and nitrogen in presence of chlorine will form NCl₃ trichloro nitrogen and then results such things results in higher chlorine demand than the stoichiometric requirement.

This is important because quite often it is our experience that the addition of a particular chemical in larger quantities always leads to faster reaction. That is something to do (Refer Time: 14:10) principle you can say, if the reactants are more the equilibrium will shift towards the products and if the product is added to a cauldron of the reactants and mixture and products, the product extra product will push the reaction backwards provided it is a reversible reaction.

So, electro chlorination is another process that has been developed recently and here we have the advantage of controlling the production of chlorine required to the required extent from electrochemical operations. So, we here it is a how do I produce chlorine, that is I take platinum and titanium anode and titanium cathode ok. So, among these 250 to ASE produces NaOCl sodium hypochlorite from NaCl, then that can react with cyanide to give you a cyanate that is also an oxidized product followed by nitrogen and carbon dioxide.

The beauty of this reaction is no sludge, so that is important. If there is sludge we usually do not know how a if a sludge is produced we usually do not know exactly how to handle the sludge, because before you handle the sludge you have to again recharacterize the sludge and such recharacterization can take lot of time and also quite often we expect the process to be very costly.

So in general if I go for electro chlorination, since there is no sludge the whole process becomes very very economical. Then when both chromate and cyanide are present cyanide should be treated first and destroyed that is in alkaline conditions and then chromate may be recovered using cation exchange or precipitation reactions as chromium hydroxide or something like that. That is a very standard operation in many electroplating industries whenever there are cyanides and chromium in the effluent.

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Common plating baths			
Bath formulas	Metallic + cyanide concentrations, ppm	Rinse concentration, ppm	
		0.5 gph drag-out*	2.5 gph drag-out*
Nickel			
40 oz/gal nickel sulfate	82,000 Ni	171 Ni	855 Ni
8 oz/gal nickel chloride			
6 oz/gal boric acid			
Chromium			
53 oz/gal chromic acid	207,000 Cr	431 Cr	2155 Cr
0.53 oz/gal sulfuric acid			
Copper (acid)			
27 oz/gal copper sulfate	51,500 Cu	107 Cu	535 Cu
6.5 oz/gal sulfuric acid			
Copper (cyanide)			
3.0 oz/gal copper cyanide	12,400 Cu	2.8 Cu	14 Cu
4.5 oz/gal sodium cyanide	28,000 CN	58 CN	290 CN
2.0 oz/gal sodium carbonate			
Copper (pyrophosphate)			
4 oz/gal copper (as proprietary mix)	30,000 Cu	62 Cu	310 Cu
29 oz/gal sodium pyrophosphate			
0.4% ammonia (by volume)			
Cadmium			
3.5 oz/gal cadmium oxide	23,000 Cd	48 Cd	240 Cd
14.5 oz/gal sodium cyanide	57,700 CN	120 CN	600 CN
Zinc			
8 oz/gal zinc cyanide	33,800 Zn	70 Zn	350 Zn
5.6 oz/gal sodium cyanide	48,900 CN	102 CN	510 CN
10 oz/gal sodium hydroxide			
Brass			
4 oz/gal copper cyanide	21,000 Cu	44 Cu	220 Cu
1.25 oz/gal zinc cyanide	5,250 Zn	11 Zn	55 Zn
7.5 oz/gal sodium cyanide	47,500 CN	99 CN	495 CN
4 oz/gal sodium carbonate			
Tin (alkaline)			
16 oz/gal sodium stannate	53,000 Sn	110 Sn	550 Sn
1 oz/gal sodium hydroxide			
2 oz/gal sodium acetate			
Silver (cyanide)			
4 oz/gal silver cyanide	24,600 Ag	51 Ag	255 Ag
4 oz/gal sodium cyanide	21,800 CN	45 CN	225 CN
6 oz/gal sodium carbonate			

*Drag-out is the amount of solution carried out of the bath by the material being plated and the racks holding the material. Rinse rate is assumed to be 4 gpm.

Here are some common plating baths, it has nothing to do with the waste characterization, but you will get an idea of what kind of things are expected from the electroplating wastes. For example, for nickel what is the plating bath it is 40 own source of gal per gallon of nickel sulfate nickel chloride and boric acid.

So, in the wash out and in the rinsing stage and in the drag out stage we can expect all these chemicals that is nickel sulfate, nickel chloride and boric acid. Usually we expect all the three in the effluents. Now, in the next column I have here metallic waste as well as cyanide concentrations, they are all in ppm level. You can imagine what could be the level what could be the level of cyanide and nickel in such systems, sometimes we do use cyanide sometimes we do not use cyanide.

But the rinse concentration you will get an idea of how much of nickel you will be losing and in the drag outs. So, if it is 0.5 gph drag out you get about 171 ppm of nickel followed by if it is 2.5 gph drag out then I get around 855 ppm almost 1 milligram per liter.

So, when you are handling several thousands of effluent containing 1 milligram per liter that becomes unignorable. Similarly look at chromium we have 53 ounce per gallon of chromic acid and 53 ounce per gallon of sulfuric acid that is the bath for chromium. And what do I end up with 207000 ppm in the effluent of carbon chromium and then in the drag outs again the quantity lost is not insignificant either that will be 431 and 2155 ppm.

Similarly, we have we can calculate and write down the numbers for copper which is 107 and 535 and copper cyanide if it is there the limits would be 2.8 and 58 of cyanide 14 of Cu and 290 of cyanide. So, like that there are other things I am not going into details of each of the processes, but you can definitely get an idea of all these cyanides can be metals and anions that are coming in the drag out and in the rinsing operations.

So, the drag out just to give you an idea of what is a drag out, I have not explained to you what is drag out. So, drag out is that amount of solution carried out of the bath by the material being plated and the racks holding the material. So, rinse rate is assumed to be approximately 4 gallons per minute whereas, drag out for a drag out there is no limit, in such possibilities drag out is a real waste that ends up in the rinse waters.

Because drag out is something I dip in and by the time I take out and put it in the water, so much of solution is lost. So, what we normally do is we pull it out of the bath hold there for some time, before I move the job to the next washing tank or rinsing tank ok.

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So, the flowchart for some of the common plating baths can be this can be very common and they can be classified depending upon the plating operations. Here I am presenting you four plating bath treatment that is the copper, nickel, chrome plating and zinc plating. In copper plating I have electro cleaner that is cathodic cleaner and then I have a rinse and then a hydrochloric acid dip followed by a rinse again followed by a copper cyanide strike.

So, strike is something which is a flash dip you can say and then we can after the flash dip we have some running rinse one more running rinse and then copper pyrophosphate solution, followed by running rinse, hot rinse and finally the drying oven. So, in all these cases you can see that there are more than 1 or 2 rinse operations involved before the job moves on to the next operation.

Same thing is true with nickel plating also, in nickel plating we have electro cleaner that is cathodic again followed by electro cleaner at the anodic electrode that they are all regular mechanical operations and then we can have running rinse and then acid dip followed by again

running rinse and bright nickel solution, followed by a running rinse soap dip hot running disk and finally from that drying oven.

If they got out of the solution only when there is a dip in the concentration which is no more practical for plating operations and now look at the chrome plating. Again we have an electro cleaner, running rinse, sulfuric acid dip, running rinse, chrome solution, recovery mist, spray rinse, running rinse, hot still dip and all these things rinsing operations keep on adding pollutants, which will finally end up in the treatment plant as total combined effluents.

Rarely we will look at the effluents coming from each operation for treatment it is hardly done, because there are so many dips and drag outs and rinses and washes and all these things it is not practical to remove from each tank separately and treat them. Rather than you most since concentration keeps on diluting it is better to collect all of them at the end and subjected to resource recovery if the concentration is less. If there concentration is more if it is not economically valuable simply precipitate it and filter it out and let the effluents run their course after precipitation.

So, zinc plating is again a very very popular operation, which involves electro cleaning, running rinse, sulfuric acid dip followed by running rinse and then zinc cyanide solution again running rinse, spray rinse, brightener dip and running rinse and all these things are automatically there involved. So, the longer the operation lining the more would be the pollutants more would be the operations.

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PLATING-WASTE CONCENTRATIONS								
Plant	pH	Cu ppm	Fe ppm	Ni ppm	Zn ppm	Cr, ppm +6 Total		CN, ppm
A	3.2	16	11	0	0	0	1.0	6
A	10.4	19	3	0	0	0	0.5	14
B	4.1	58	1.2	0	0	204	246	0.2
C	2.8	11		0.2		3	7	1.2
D	2.0	300	10	0	82	0	0	0.7
E	2.4	35	8			555	612	1.2
E	10.7	14	4	19		32	39	2.0
F	10.5	6	2	25	39			10
G	11.3	18	18	26		36		15
G H	11.9	23	21	32		95		13

So, normally we end up with a very exhaustive concentrations of the plating ways and here are some high chromium plating waste that is the hard chromium plating waste. So, many industries are there, but you can see here most of them are having ph approximately 3.2 and 3.2 to 11.9. In several plants depends upon the each of the each plant and then how they do their operations.

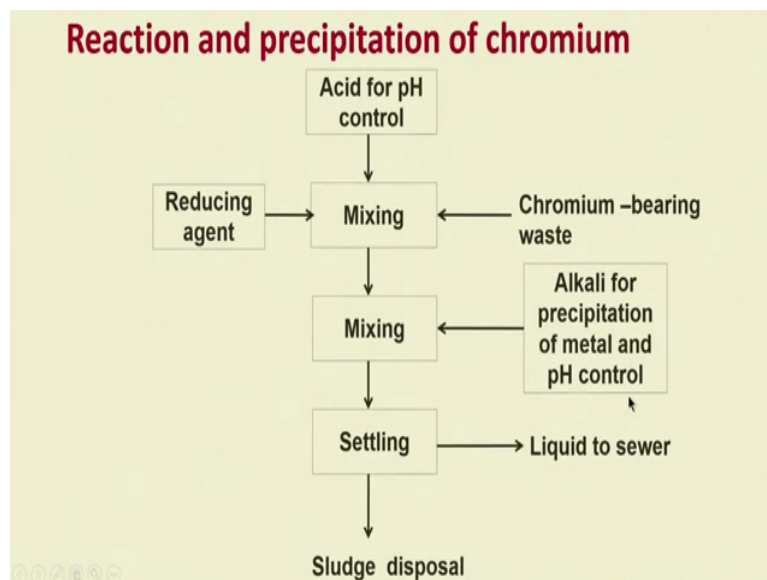
So, this is only a random list this is taken from mytra handbook of electroplating industry and the data is here for copper iron nickel zinc chromium and cyanide and this plus 6 represents hexavalent chromium otherwise they would write plus 3. So, 3.2 16 ppm of copper and 11 ppm of iron and this iron comes from the main job that is on it is a base plate on which coating is being done.

So, some of the ion is inevitably lost during plating operations, because of acid cleaning alkaline cleaning etcetera etcetera. And then nickel of course it comes from plating waste plating operations that is why you end up sometimes with to 0 if nickel is not there. Otherwise

you will end up with a certain amount of some numbers, here you can see that in E F G H I think it should be H not G and I can correct it that should be H.

And you can see that here it means there are two different kinds of operations going on in the same plant. And here you can see the chromium and other things in quite large quantities which warrant recovery of the metals by some operation or rather, it is too valuable to be to let it go and economically it may be lost. But cyanide is always there along in all these operations you can see in some former rather and their concentrations range from 0.7 to 14 15 ppm.

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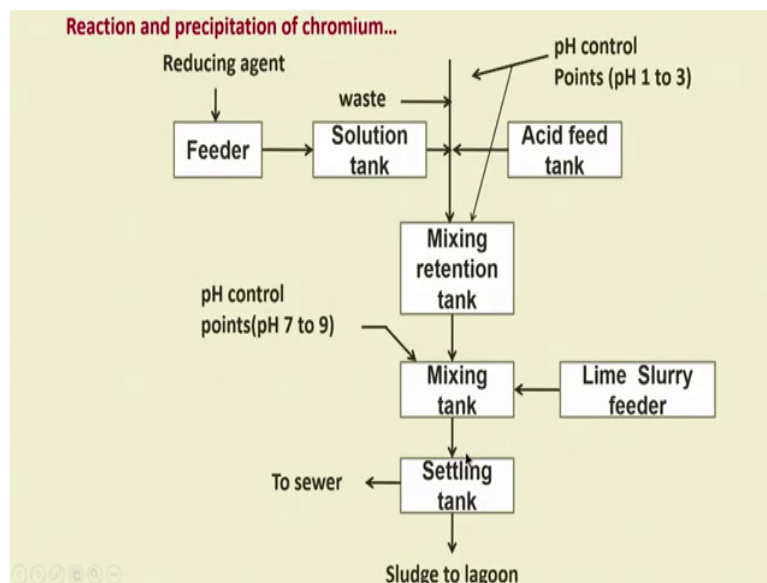


So, Reaction and the precipitation of chromium, the after cyanide treatment I want to concentrate a little on the treatment of chromium, because chromium hexavalent chromium is supposed to be carcinogenic and it is also toxic and it is not good for health also.

So, acid for what is normally involved is acidification for pH control and then I have to add a reducing agent like ferrous ion, which can reduce chromium 6 to chromium 3 and then

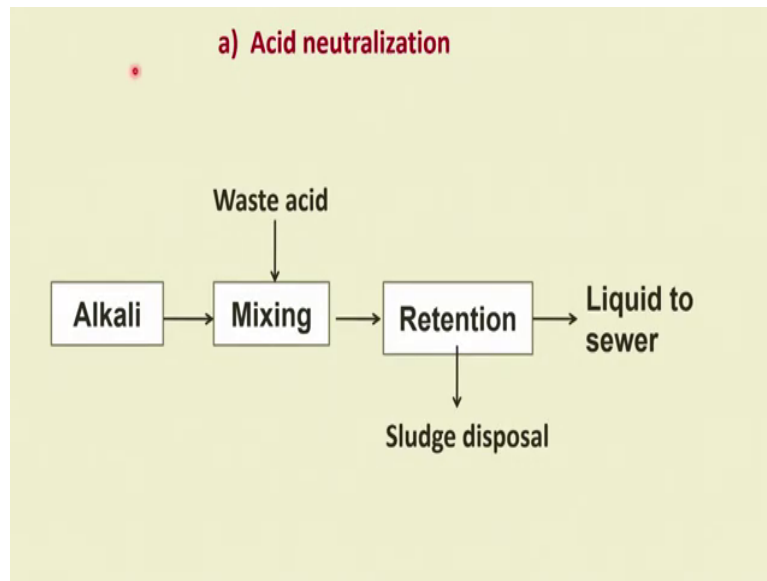
chromium bearing waste is brought in continuously and reducing agent is also brought in continuously. And there will be alkali for pH adjustment pH control followed by mixing and then settling the settled sludge contains chromium 3 which is not as toxic as chromium 6 and the layer then here after filtration the liquid can go to waste.

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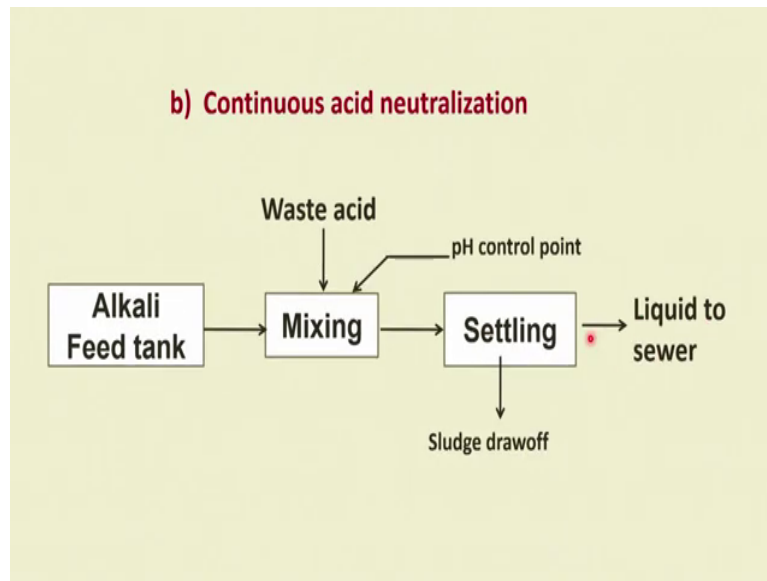
So, I can have some sort of a schematic diagram for the treatment of chromium waste, that is a reducing agent with a feeder and then waste solution is there then acid feed also can come from other side. So, acid feed and another pH control. So, in all these three when they are mixed together I can have some sort of a retention tank for taking all controls to make a uniform solution homogenous solution. Followed by a pH control with mixing and the lime slurry is added and then after settling the precipitate it can go to settling tank and then sludge can be taken to lagoons ok, so that is another kind.

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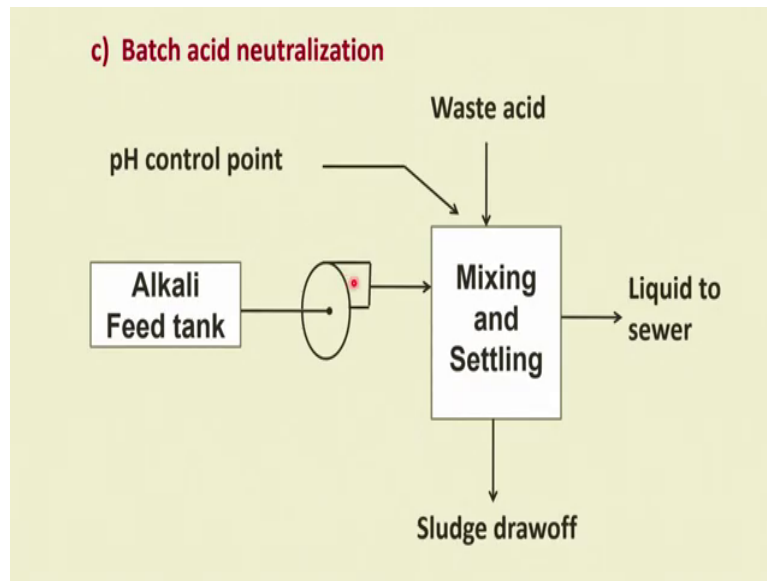
Third is the Acid neutralization here I just have an alkali based acid is coming and then they can be mixed together, it may generate some sludge. If you are using alkali lime of calcium hydroxide lime, if you use sodium hydroxide you may not find the sludge and then after retention and figure it will go to sewer.

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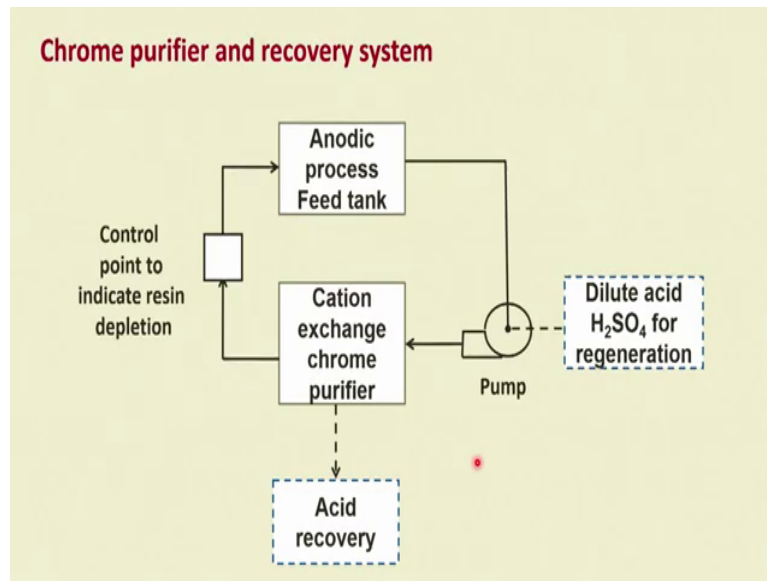
And Continuous acid neutralization also can be designed for the same operation with a alkali feed tank and mixing and settling and mixing should have waste acid and pH control point.

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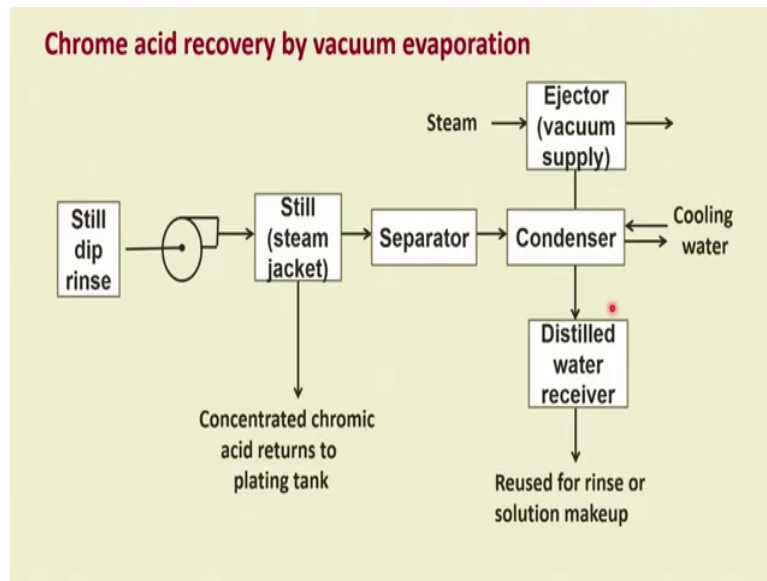
So, Batch acid neutralization is also almost always preferred in very small quantities. Whenever the waste are in smaller quantities here we have an alkali feed tank mixing, settling and waste acid is added and sludge is drawn off.

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And chrome purifier essentially same thing anodic process, cation exchange chrome purifier acid recovery followed by sulfuric acid for regeneration. And this is for chrome acid recovery by vacuum evaporation and here the rinse operations are involved.

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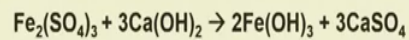
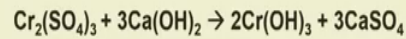
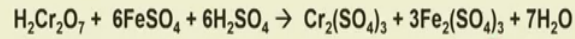


So, still dip rinse is there and then still steam jacket a separator followed by condenser and distilled water receiver the condenser can get input from cooling tower also and steam is available from vacuum supply.

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REDUCTION AND PRECIPITATION

Chromium reduction by FeSO_4 , SO_2 or NaHSO_3 in presence of free mineral acid around pH 3.0 followed by an alkali to precipitate hydroxides.



1 ppm Cr, 16 ppm CuAs, 6 ppm H_2SO_4 , 9.5 ppm lime produces 2 ppm $\text{Cr}(\text{OH})_3$, 0.4 ppm $\text{Fe}(\text{OH})_3$, 2 ppm CaSO_4 .

1 mole of chromium produces 454 g. sludge!

So, reduction and precipitation reactions are there for chromium, we will study these reactions in detail in our next class.

Thank you very much.