

# What is the Continuous Technology? An Overview

As the chemical industry looks for alternatives to stirred tank, reactors, continuous process in fact an area of global interest!!

Over the years, it has been successfully employed by the polyester industry for several decades. This proven technology has a number of distinct advantages:

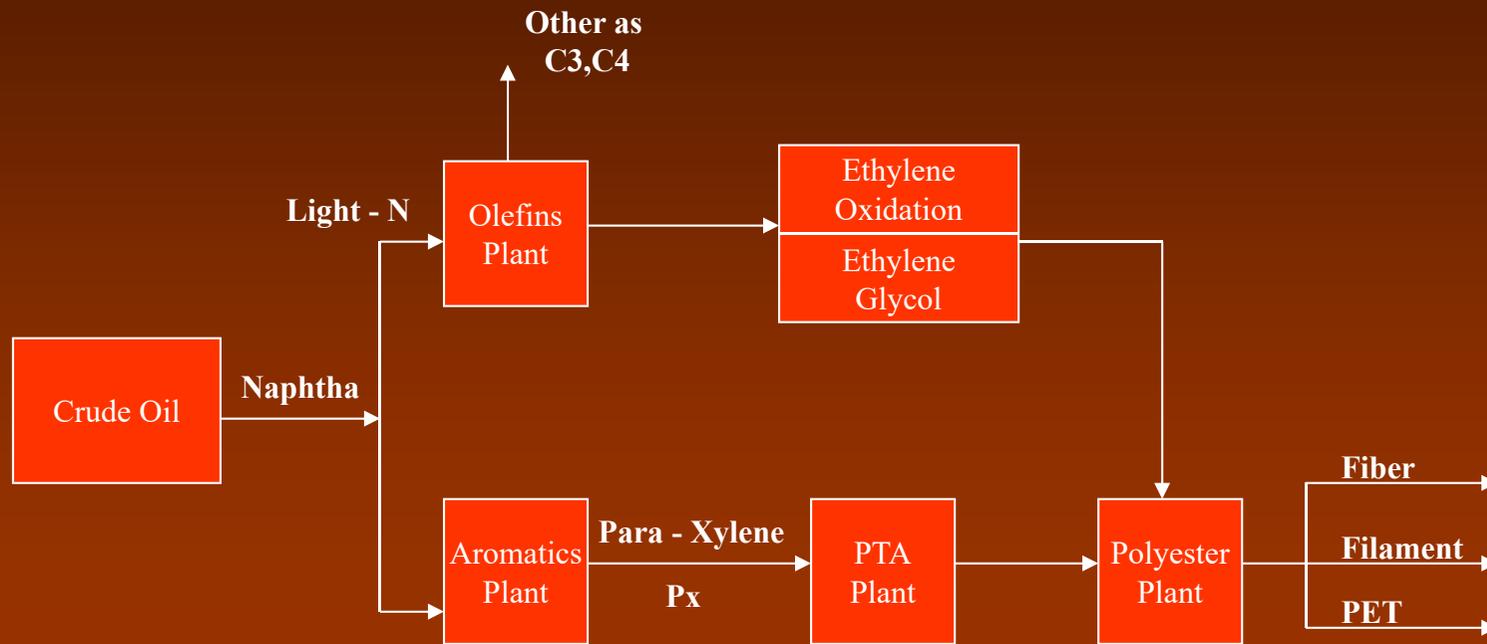
- ❖ Improved product quality & process yield.
- ❖ Low inventory & safe operation.
- ❖ Shorter development & scale up time.
- ❖ Lower capital & operating cost
- ❖ Environmentally friendly & less waste.

# What is Continuous Process?

All the polyester technology suppliers believe that continuous processing technology is most practical & cost effective way of achieving what is after referred to as process intensification ,by using continuous flow through reactors ;the simplest being a pipe containing a static mixer with reactant inlet an outlet and either heating or cooling along the pipe length, the same production rate can be achieved as when using reactors systems many order of magnitude larger.

This reaction in reactors size and different mode of operation is particularly advantageous when operating a faster or energetic process such as those often encountered the production of today complicated intermediates.

# Relationship with Upstream & Downstream Process

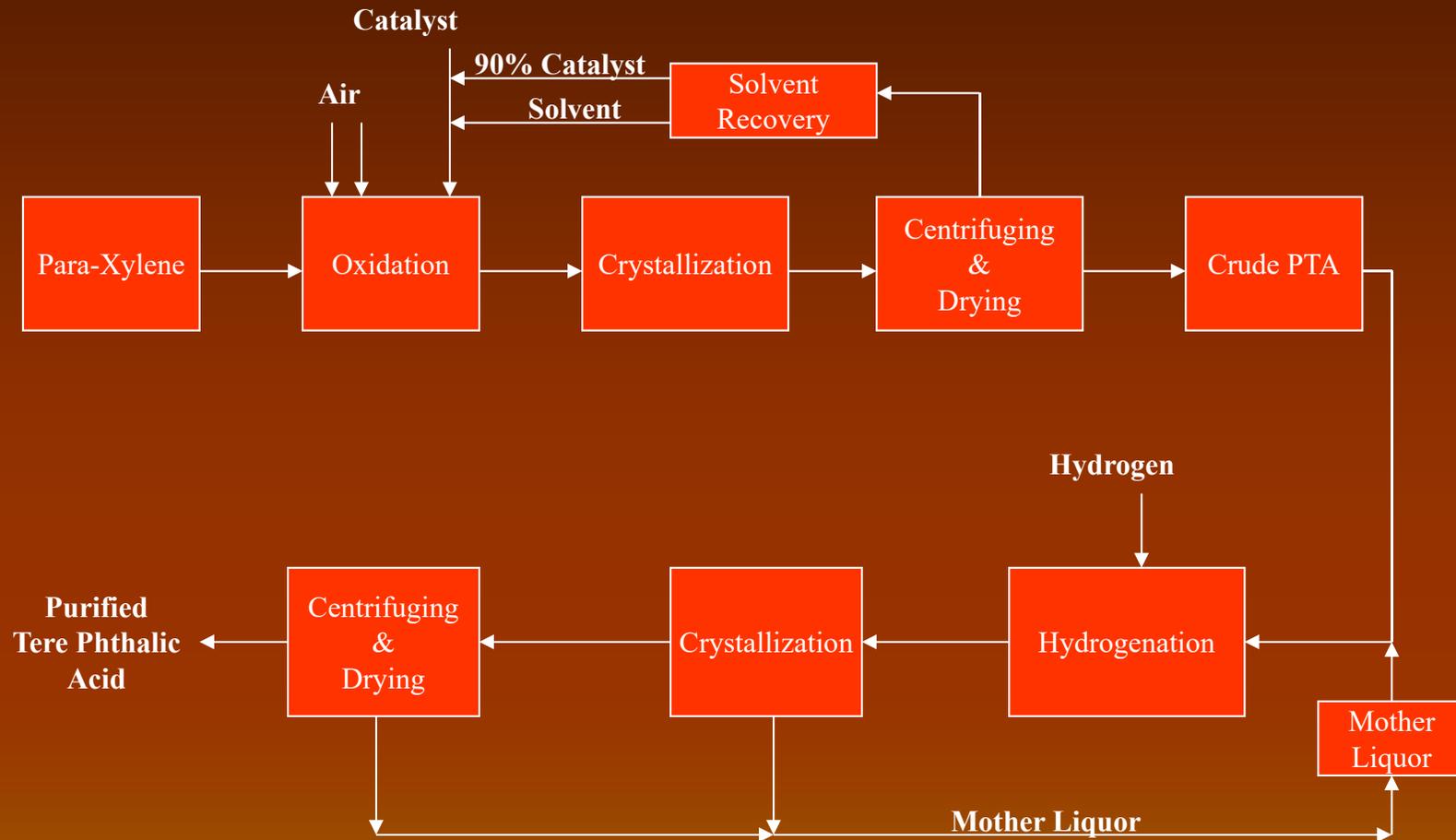


Oil Refinery

Petrochemicals Mid Stream

Fibers Down Stream

# PTA Manufacturing Process



# PTA Technology Suppliers

To name a few major suppliers of PTA around the world:

- ❖ Eastman (Integ-Rex technology)
- ❖ ICI
- ❖ Investa (Du pont)
- ❖ Kohap
- ❖ Tuntex
- ❖ Amoco
- ❖ Mitsubishi
- ❖ Interquisa
- ❖ Dow's

## Technology :polymer cp & ssp

- Now days many technologies are available in the market in polymer field for cp and ssp.

To name the major players:

- ❖ Zimmer (cp & ssp both).
- ❖ Udhe Inventa (cp).
- ❖ Du pont /Envesta (cp and ssp both) .
- ❖ Torrey (cp).
- ❖ Kohap (cp).
- ❖ Aquafil (cp).
- ❖ Eastman “Interg-rex technology” (cp & ssp both).
- ❖ Buhler (ssp).
- ❖ Sinco (ssp).

# Raw Material:

The basic raw material use to make bottle grade PET (Polyethylene Terephthalate) are:



MEG



DEG



Antimony Tri Acetate



Antimony Tri Oxide



Phosphoric Acid



Cobalt Acetate

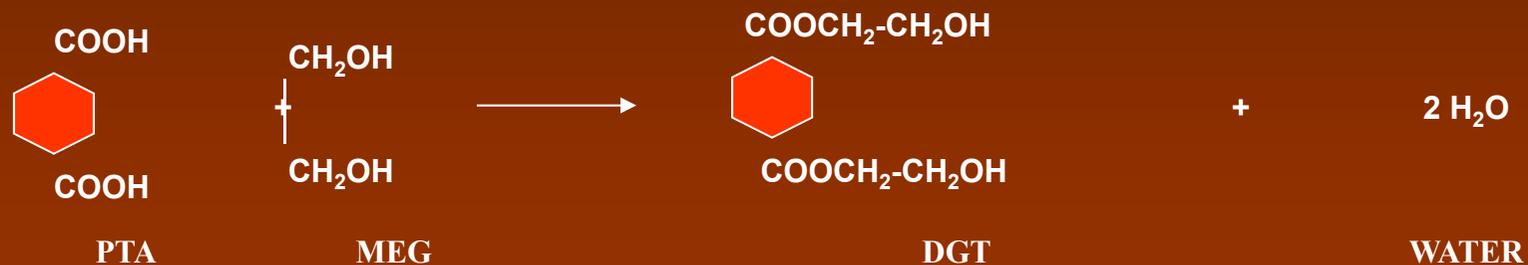


Blue Toner

# What kind of reactions occur during the process?

## Esterification:

The first reaction of the process is esterification reaction.



The main side reaction during the esterification reaction is formation of DEG, which is formed by removal of water from hydroxyl end groups.



The DEG content in the final product can therefore be controlled within certain limits by corresponding process alterations. The DEG molecules are built instead of MEG molecules in the polymer chain.

The optimization of DEG is limited because the degree of esterification and the temperature in the stages has to be in certain range.

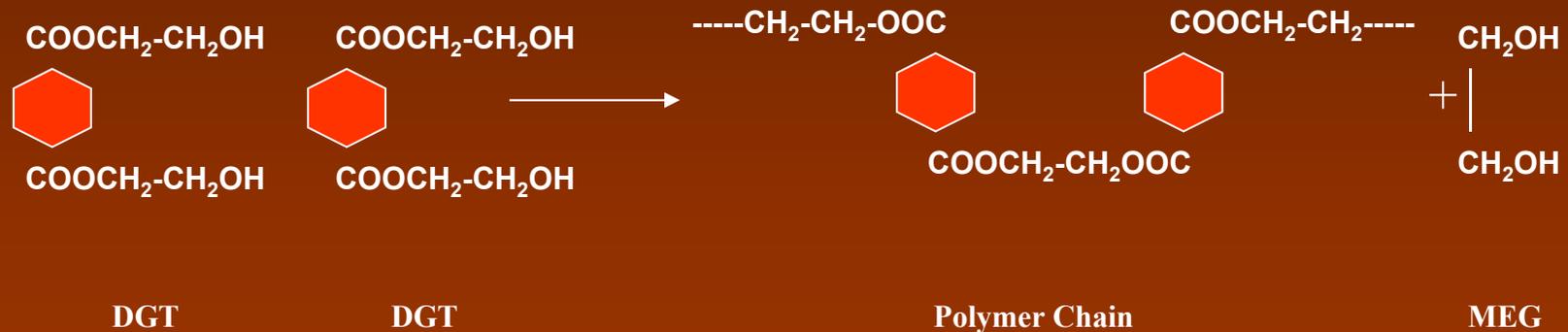
Another side reaction which is acid catalyzed is the dehydration of EG to acetaldehyde. This reaction occurs in the vapor phase of the esterification and prepolycondensation stages.



The main reaction of the formation of Acetaldehyde is during the polycondensation stage.

## Polycondensation:

The polycondensation reaction converts esterification product – so called monomer – in steps to long chain molecules while simultaneously splitting off low molecular weight molecules.



The reaction generates the polyester chain and EG split-off product which has to be removed from the melt phase to shift the reaction equilibrium to the polymer side. The polycondensation reaction is catalyzed by metal catalysts like Antimony, Titanium Germanium or Tin.

# What is the IPA Used For?

IPA is like PTA a Benzene Di Carboxylic Acid. The only difference is the position of carboxylic end group in benzene ring.

PTA molecules are replaced with the IPA molecules in the PET formation and more angles are built in the structure of PET chain. IPA reduces the melting point of the product, lowers the glass transition temperature and increases the crystallization temperature. IPA improves the clearness and crystallization of the product.

IPA lowers the dynamic viscosity of the polymer melt, while the chain length of the polymer and Intrinsic viscosity remains constant.

IPA is mixed in the range of  $1.5 \pm 0.5\%$  of the final product. The added quantity of IPA replaces the same quantity of PTA.

# What is the DEG Used For?

DEG is formed from EG by side reaction. DEG is produced during the process and extra DEG is added, if required.

Like IPA, DEG also reduces the melting point of the product, lowers the glass transition temperature and increases the crystallization temperature. improves the clearness and crystallization of the product.

DEG lowers the dynamic viscosity of the polymer melt, while the chain length of the polymer and Intrinsic viscosity remains constant.

DEG is mixed in the range of 0.5+/-0.1% of the final product.



# What is the Catalyst Used For?

Catalyst Antimony is used in form of Antimony Triacetate or Antimony Tri Oxide to accelerate the polymerization process without taking active part in the reaction. It does not influence the reaction equilibrium.

Antimony form Antimony Glycolate when mixed with glycol. If Antimony Tri Acetate is used as a catalyst, Acetic Acid is form and vaporized out of the catalytic solution. If Antimony Tri Oxide is used as a catalyst, the glycol is to be heated to  $150^{\circ}\text{C}$  to dissolve the Antimony Tri Oxide. Water is formed and also vaporized off the catalytic solution. After dissolving the Antimony Tri Oxide, solution need to be cooled down to  $50^{\circ}\text{C}$  to avoid thermal degradation of the solution.

Other catalyst which are under development phase are Germanium & Titanium based catalyst. Antimony Trioxide is more economical than Antimony tri Acetate.

# What is the Cobalt Acetate Used For?

Cobalt Acetate is used as the color additive. It improves the b-color of the product. It is dissolved with catalyst solution and injected in the Paste Preparation Section.

A certain ratio of Phosphoric Acid as inhibitor to eliminate the catalytic effect of the Cobalt, which gives negative influence to the b color of the product.



# What is the Phosphoric Acid Used For?

Phosphoric acid is used to heat stabilizer or inhibitor. Side reactions which are catalyzed by the metals ions have negative influence on the color of the product. The phosphorus reacts in a precipitation reaction with the metal ions like cobalt and deactivate the catalytic property of the metal ions.



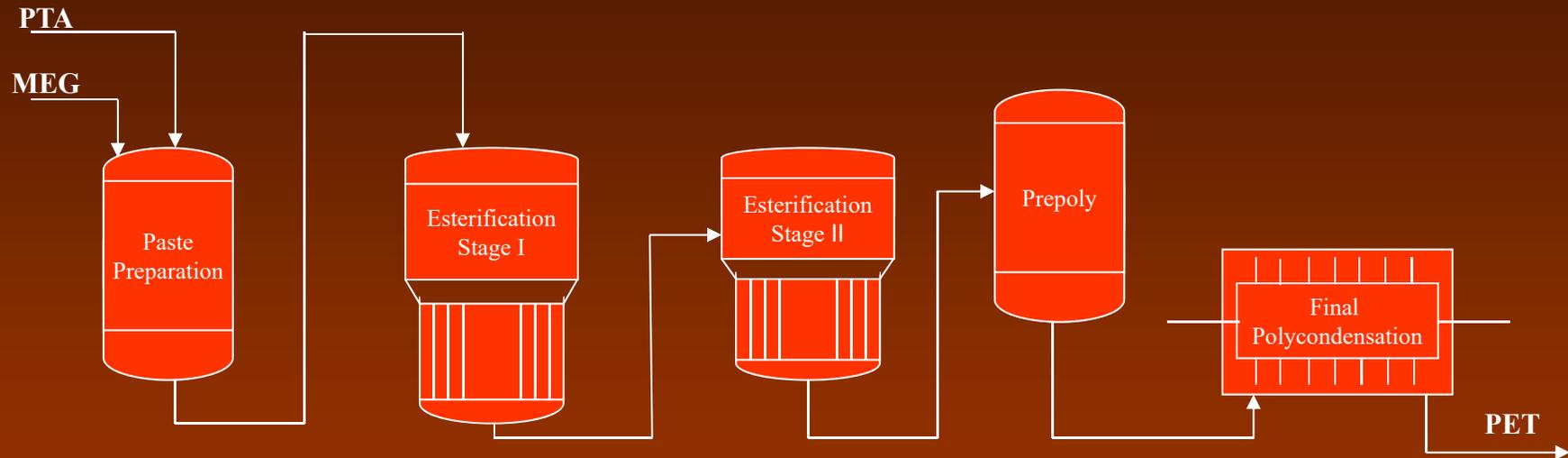
# What is the Blue Toner Used For?

Blue toner is only organic pigment used as delusterant agent for PET. The effect of blue toner is it reduces the b color and L color of the product. The result is a slightly blue product. The blue toner does not reacts with the polymer chain, it is only physically attached to it. Blue toner is always used in the combination of the Cobalt Acetate.

Blue toner is powder and is suspended in the EG in order to enable a continuous dosing in the process. Blue toner is added in the paste preparation section.



# Typical Main Operating Parameters



Parameter	Unit	Paste Preparation	Esterification Stage I	Esterification Stage II	Prepoly Condensation	Final Poly Condensation
Prod. Temp.	°C	50 ~ 60	250 ~ 260	260 ~ 273	273 ~ 280	280 ~ 286
HTM Temp.	°C	--	275 ~ 280	275 ~ 280	280 ~ 290	286 ~ 294
Pressure	bar / mbar	Atmospheric	1.1 ~ 1.6	0 ~ 0.5	12 ~ 20	0.8 ~ 1.5
Residence Time	min.	90 ~ 150	80 ~ 100	70 ~ 90	70 ~ 90	80 ~ 100
Agitator Speed	Rpm	25 ~ 30	120 ~ 160	--	6 ~ 8	1.0 ~ 2.4
MR/Conversion	- / %	1.4 ~ 1.6	88 ~ 92	97 ~ 98	99.5	99.8
DP (PN)	-	--	3	5	20 ~ 25	90 ~ 108

# Influence of main parameters on Properties

Trend		DEG		COOH		Melting Point		Intrinsic Viscosity		Color		Reaction Time		Heat Consum.		Through put	
		h	l	h	l	h	l	h	l	g	w	lg	s	h	l	h	l
Mole EG	h	x			x		x						x	x			x
	l		x	x		x						x			x	x	
Monomer React. Temperature	h	x		x			x				x		x	x		x	
	l		x		x	x				x		x			x		x
Monomer React. Pressure	H	x		x			x						x		x	x	
	l		x		x	x						x			x		x
Monomer React. Time	lg	x			x		x				x				x		x
	s		x	x		x				x					x	x	
Reboiler Reflux Temperature	h	x					x				x			x			
	l		x			x				x					x		
Polymer React. Temperature	h	x		x			x	x			x		x	x		x	
	l		x		x	x			x	x		x			x		x
Polymer Vacuum	h		x		x	x		x		x			x			x	
	l	x		x			x		x		x	x					x
Polymer React. Time	lg	x		x			x	x			x		x	x		x	
	s		x		x	x			x	x					x		x

h = high    g = good    w = worse    l = Low    s = short    lg = Long

•Color is mainly influenced by catalyst, stabilizers and other additives.

•Reaction times are influenced by type and quantity of catalyst.

# Suggestions – For better operation and safety

- ❖ Keep the set points list of different throughput CP& SSP in the Control room.
- ❖ Keep the trouble shooting manual always in the Control room.
- ❖ Emergency telephone no list should be present in the control room always.
- ❖ Inform immediate to the plant manager if big abnormality is in the plant.
- ❖ Each shift two rounds should be taken by shift engineer. One in the beginning, of the shift and one at the end of the shift of **“whole plant.”**
- ❖ Schedule job should be followed as per planned schedule.
- ❖ Safety rules should be followed during any work that is under progress.
- ❖ Preventive maintenance schedules should be followed strictly, and timely review meetings should be held to improve upon them.

# Questionnaire for the operator

- ❖ What is the procedure of change over EG circulation pump ?
- ❖ What are the procedure of PTA and IPA off loading system?
- ❖ What is the procedure of IPA one ton bag off loading ?
- ❖ What is the procedure of primary pump change over ?
- ❖ What are the immediate actions you will take, if PTA and IPA conveying line got chocked ?
- ❖ What are the procedure of sampling ,EST1,EST2, PP and CUTTER chips ?
- ❖ What are the procedure of heat exchanger change over ?
- ❖ What are the procedure of vent pot venting ?
- ❖ What are the procedure of additive and catalyst filter change over ?
- ❖ What are the procedure of EG off loading system ?

# Questionnaire for engineer and dcs supervisor :During higher capacity of plant

- ❖ What are the check points required during PTA and IPA off loading ?
- ❖ What are the following raw material contents in the final polymers ? IPA, antimony tri-oxide, cobalt acetate,, phosphoric acid,, DEG ?
- ❖ What are the following properties of final polymer ,if not what action will be taken to maintain IV, end group, melting point, aldehyde content, chips size, acid no and color ?
- ❖ What are the immediate action required in case of paste agitator failure?
- ❖ What are the immediate action required in case of both paste pumps failure ?
- ❖ What are the immediate action required in case of both pre polymer pumps failure?
- ❖ What are the immediate action required in case of product pump failure?
- ❖ What ate the immediate action required in case of one cutter/two/three cutter failure. ?
- ❖ What are the immediate action required in case one HTM heater/two/three failure ?
- ❖ What are the immediate action required in case of cooling water ,DM water raw water, steam and N2 failure ?
- ❖ What are the immediate action required vacuum failure of PP and FINISHER ?
- ❖ What are the procedures and action required in case of power failure ?

# How to Optimize the Process?

## ❖ Sudden decrease in capacity

➤ Try to bring the parameters like temp., pressure and level etc. as earliest at lower capacity to avoid loss in color, acid no. and other properties.

## ❖ Normal increase & decrease in capacity

➤ Parameters should start changing from Esterification Stage 1 towards Final Polycondensation and production should start increasing as soon as you get the margin in the DRR.



# What to know during shift change?

- ❖ Problem concerning raw material unloading like PTA, MEG
- ❖ Problem concerning the Primary HTM Section
- ❖ Problem concerning the utilities like cooling water, air, nitrogen etc.
- ❖ Problem concerning product handling
- ❖ Problem concerning individual process equipments
- ❖ Housekeeping required in the plant
- ❖ Water or any other leakages in the plant

# What to check during plant round?

- ❖ Checking the pressure on the pump gauges.
- ❖ Checking for any abnormal sound on pump motors, agitator motors, gear boxes, etc.
- ❖ Check for leakages in the plant, especially the floor, if you can see any liquid droplet on the floor.
- ❖ Check for the hidden places, where people don't like to go regularly
- ❖ Band filter paper movement, spray water on the cutter, chips cutting, chips distribution on the vibrating screen, over length chips collection.
- ❖ Filter cleaning area
- ❖ Additive charging area for cleanliness and proper handling
- ❖ IPA & PTA leakages
- ❖ MEG leakages.
- ❖ Cooling water flow to different section of the equipments.
- ❖ Keep eyes and ears open for any abnormality!!!

## **Purpose and Scope:**

To clearly define the authorities and responsibilities of process department

## **Responsibility and Authority:**

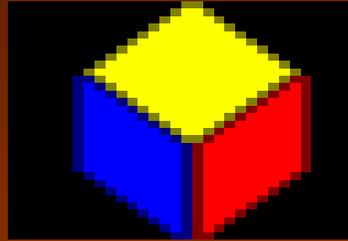
It is the responsibility of department head (process) to ensure smooth running of the plant with the emphasis on quality and target on production with the help of shift engineers (operation) and DCS supervisor (process). He shall coordinate with service departments for smooth running of the plant and at the same time ensure availability of service department help for any problems arises from time to time and 24hours a day. At the same time he can delegate the responsibilities to the staff under his administrative control

## **Procedure:**

The complete procedure can be divided into following heads.

- Production planning and control
- Product development
- Manpower planning and control
- Materials planning
- Maintenance planning
- Reporting system
- Work Instructions

# CHECK LIST AND SUGGESTIONS



Microsoft Office  
Excel Worksheet

**THANK YOU !!!**

