Reactor Loading Procedure CTR

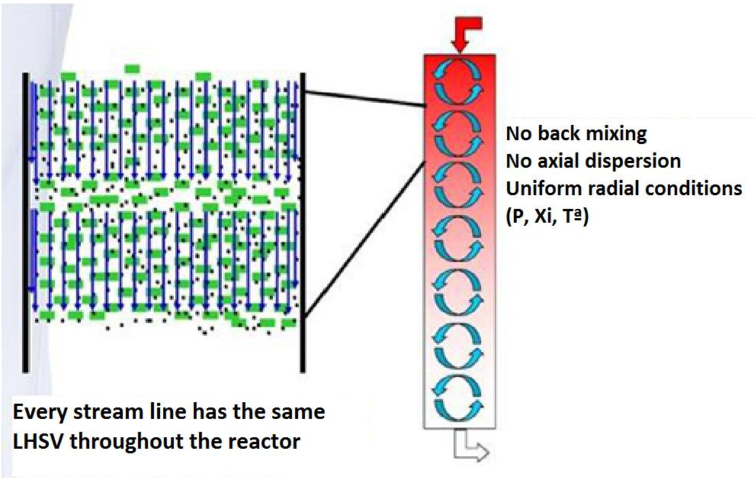
1st Haldor Topsoe Workshop

OUTLINE

1. Previous Considerations
2. Loading Procedure
   - Why loading is important?
   - Case of study
   - Optimal Load
   - Other consideration
3. Start up
4. Outcomes

Previous Considerations
Meeting Plug Flow Conditions (HDT)

- Repsol Catalyst/feedstock evaluation based on:
  - Same LHSV (h^{-1})
  - WABT Isothermal*
  - Same PP H_2
  - Same RHC
  - Flow model: Plug Flow



- Plug flow or Piston Flow
  - Every particle of fluid entering the reactor leave it without mixing it with particles before of after it
  - Every particle of fluid pass through the reactor at the same LHSV
  - The reactor can be divided in many layers whit uniformal radial conditions (P, Tª, Xi)

* There are some exception where we work forcing profile or semi-adiabatic reactor

Previous Considerations
Meeting Plug Flow Conditions (HDT)

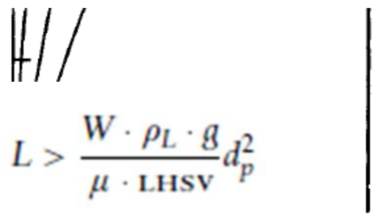
- *Wall Effect:* liquid velocity increase or decrease in the proximity of reactor wall producing desviation from Plug Flow.

D/Dbed > 25

- *Bed Lenght Effect* … ɛeping the LHSV constant mean lower fluid flo… ːy.

dim

$$L > 200 \cdot d_p \cdot n \cdot Ln\left(\frac{1}{1-X}\right)$$

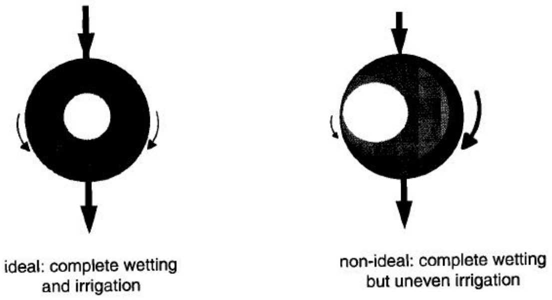


- *Complete Irrigatic* … ust contribute to the global conversion

$$L > \frac{W \cdot \rho_L \cdot g}{\mu \cdot \text{LHSV}} d_p^2$$

- *Particle diametei*
  - Catalys crushed: I … nass transfer limitations metal contamina
  - Inert dilution: hy

    inert dilution wh



$$d_p < 0{,}005 \cdot \frac{L}{n \cdot Ln\left(\frac{1}{1-X}\right)}$$

Previous Considerations

Meeting Plug Flow Conditions (HDT)

Reactor			
ID (mm)	17,48	Section (cm2)	2,328
OD (mm)	25,4	Vol (l)	0,211
Lreactor (mm)	907,2	L/D reactor	51,899
OD TW (mm)	3,02	OD Bed (mm)	17,217
Flow	Upflow		

Catalyst		Silicon Carbide		Bed	
Shape	Trilobe	Shape	Sphere	Lenght (mm)	433
dp (mm)	1,3	Dilution	1:1	Dp bed (mm)	0,137
L (mm)	3	Dp	0,08	Dbed/Dp	160,91
D (g/cm3)	0,86	D (g/cm3)	2,2		
V (cm3)	70	V (cm3)	70		

Feedstock	
Density @15 ºC (g/cm3)	0,8531
Sulphur (ppm)	4010
Nitrogen (ppm)	138
Viscosity @ 40ºC (cSt)	3,013
Brome number (g/100 g)	7,28

Procces condition		kinetic data	
LHSV (h-1)	1	X Sulphur (%)	99,3
RHC (Nm3/m3)	200	X Nitrogen (%)	83,3
PPH2 (kg/cm2)	35	n HDS	1,4
WABT (ºC)	330-350	n HDN	1

Validation				
L min HDS (mm)	146,7	Wetting number "W"	2,24E-03	> 5E-06
L min HDN	38,3	Reynolds	4,77E-01	
L min Irrigation (mm)	300,67	Bo	0,04	
Dp min (mm)	0,316	Pe	161,869	> 54,82
Dbed/Dp	>25	N	80,9	

Loading Procedure
Why is loading important?

- Reproducibility of Lenght Bed

- Avoid segregation of Carbide and Catalyst
  - May lead to desviation from Plug Flow
  - Wall Effect influence
  - May lead to rivulets (bypassing catalyst bed)
  - Safety issues (Hot spots)

- Finally lead to an incorrect interpretation of RVA of catalyst

Loading Procedure
Case of study

- *Case 1: Premix catalyst/SiC*
  Carga en premezcla.mp4
- *Case 2: Concurrently Catalyst + SiC using funnel*
  Carga a la vez.mp4
- *Case 3: Leaned load premixing*
- *Case 4: Load using belt and concurrently Cat +SiC*
  Carga de la cinta.mp4
- *Case 5: Optimal Load*
  Carga óptima micro.mp4

Loading Procedure
Optimal Load

- Steam up the reactor (1h) to remove any contaminant of previous test
- Apply copper paste and fix the bottom VCR connection and the thermowell
- Place the reactor vertically
- Fill up the reactor within 70mm of wood fiber
- Fill up within 60 mm of SiC 0,2mm
- Split up the total catalyst amount in 8 portions of equal weight
- Split up the total SiC amount in 8 portions of equal weight
- Load one portion of catalyst and following load one portion of SiC
- Apply 25 vibration
- Repeat with other portion of catalyst followed by on portion of SIC and apply 25 vibration
- Apply 400 vibration when all the catalyst and SiC is loaded
- Measure the lenght of Bed
- Refill the reactor within SiC 0,2mm
- Place 70 mm of wood fiber at the top
- Apply copper paste and fix the bottom VCR connection

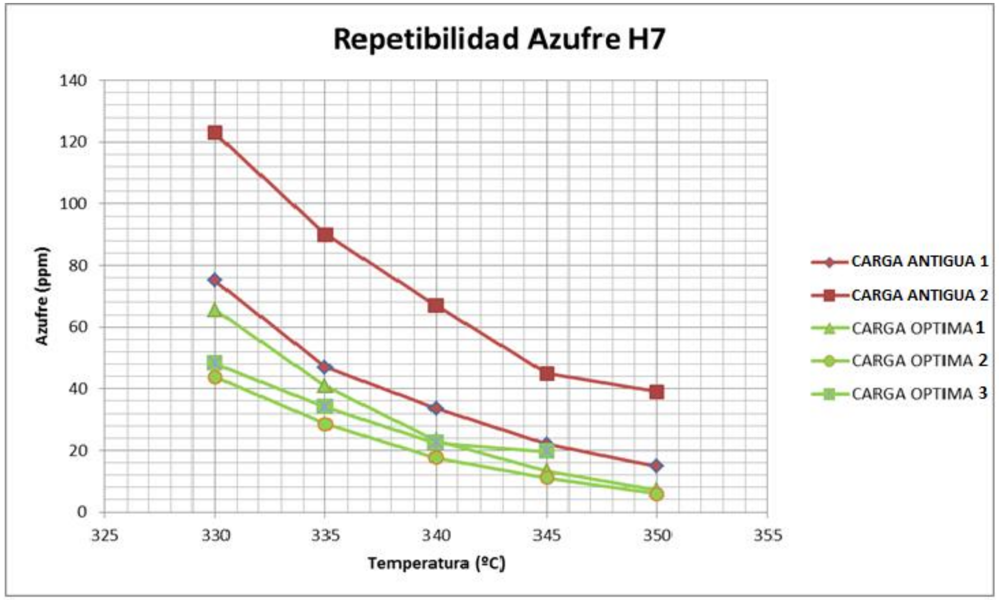
Loading Procedure
Others consideration

- Different shapes lead to different parameters of loading (portions, vibration…)
- The bed shall be located in the middle of the reactor leaving one furnace to preheat the mix (liquid +gas) assuring the first contact between them occur at the desired temperature.
- Catalys amount is measured in weight (precision weightscale is required)
- Catalys Bulk density shall be obtained in dry basis (repeat the value 3 times)
- Carbide is obtained in volume according the SiC:Cat used
- Check the plug flow criteria before start up
- Dry the catalyst before start up if necesary
- Wet the bed closing a hand valve downstream reactor before to start up (low pressure to avoid DP in the bed)
- Finally line-in the reactor and the unit

Start up
Sulphide and stabilize step

- Follow the vendor procedure to presulphide the Catalyst
- Stabilize the Catalyst with SR destillate (mesure de Brome Number to assure no olephines presente in the feed)
- Incresase the temperature by ramp (50º C/h) over 320ºC (5-10ºC/h) to avoid peak of temperature at the early hours
- Stabilize catalyst for minimum 72h to and follow the sulphur downstream the reactor
- If any unexpected shutdown occur during presulphide or stabilization step, shutdown the unit and load fresh catalyst
- If any unexpected shutdown occur during test repeat a previous temperature and decide if go on or load fresh catalyst
- Configured Interlocks
  - Safety reason
  - Protect catalyst if shutdown occur during unattended time

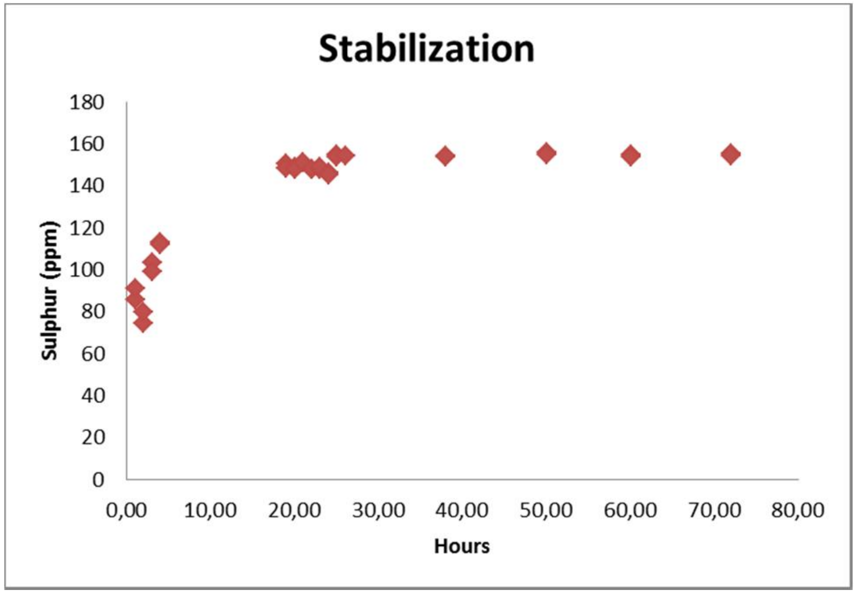
Outcomes

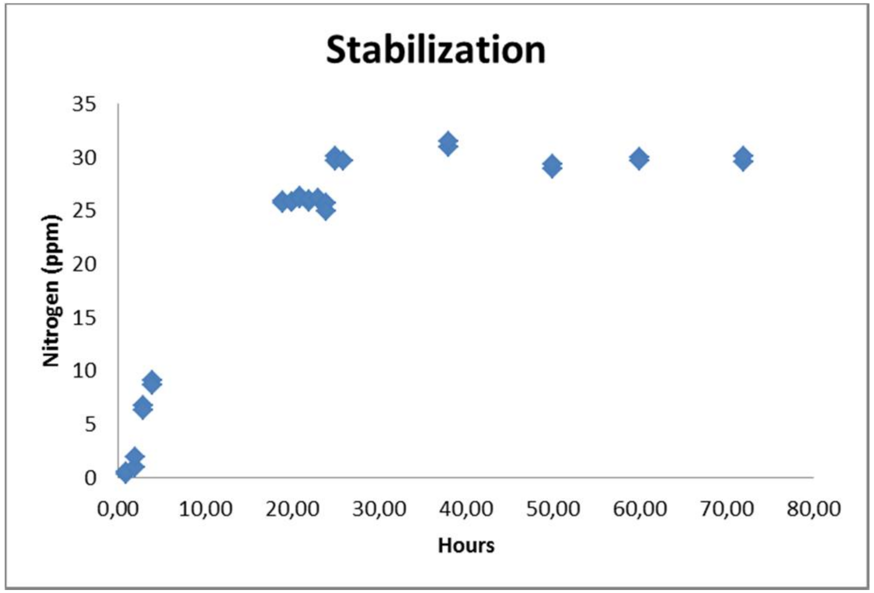


Case	L bed (mm)
Suboptimal	1013
Suboptimal	935
Suboptimal	910
Optimal	810
Optimal	884
Optimal	785
Optimal	835
Optimal	820
Optimal	827
Optimal	790
Optimal	821
Optimal	850

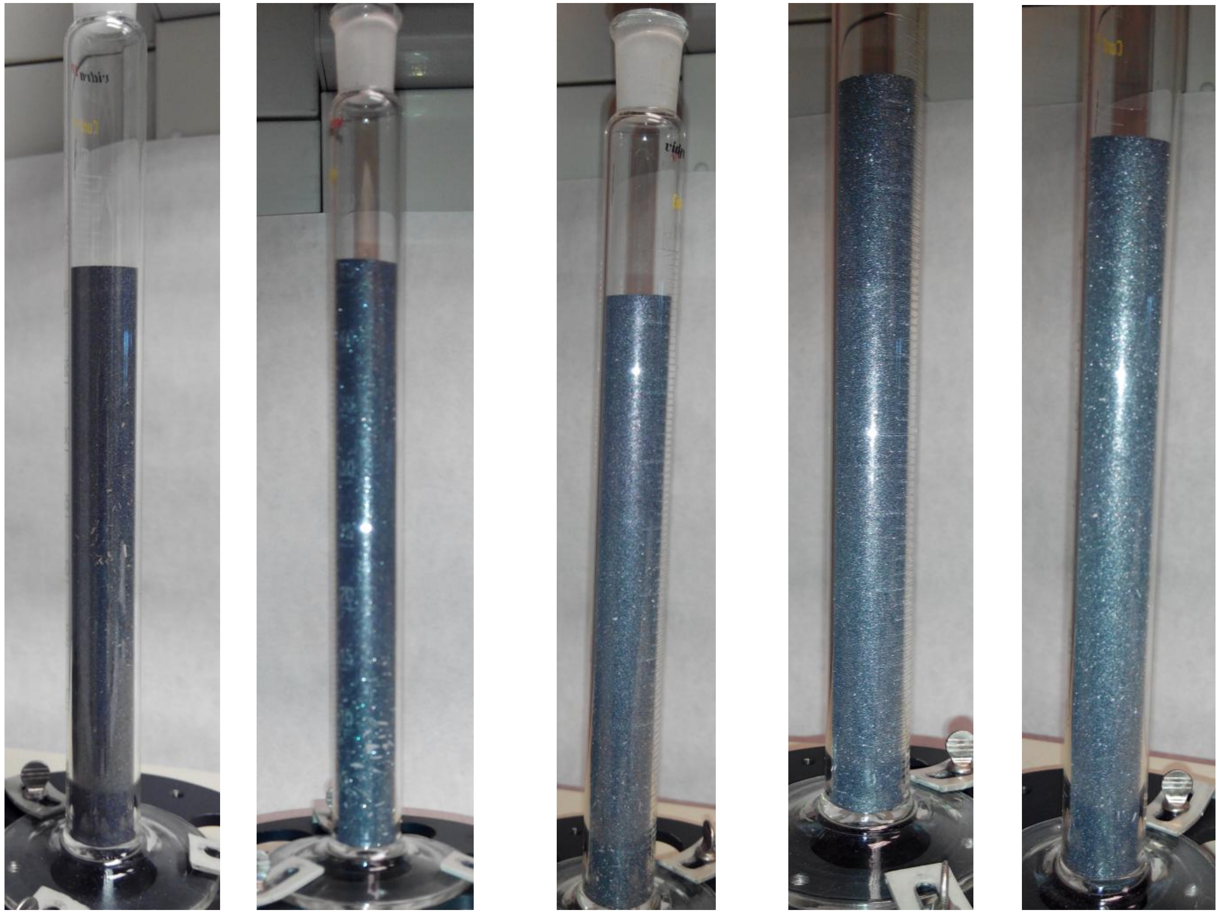
THANK YOU

Stabilization





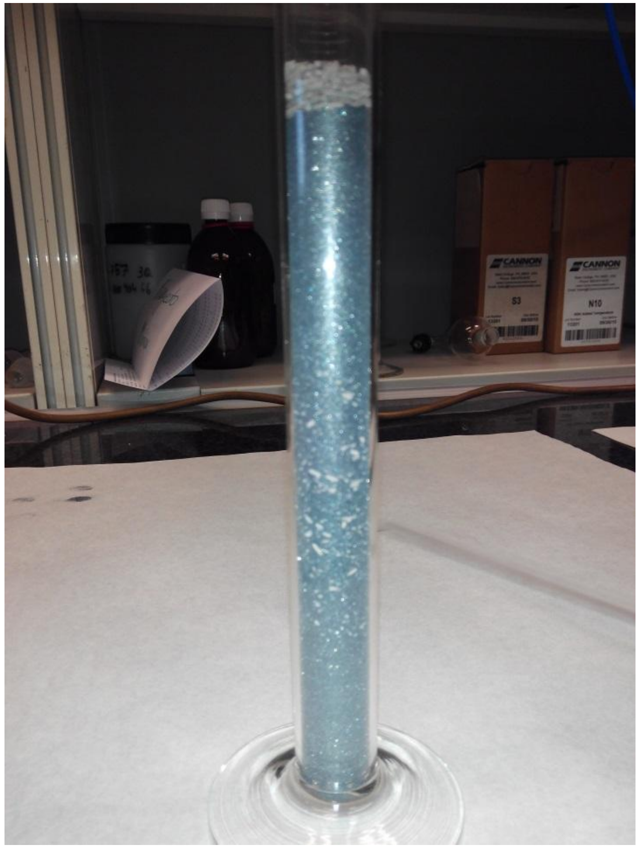
Loading Procedure
Case 5: Optimal load



2 4 6 8 10

Loading Procedure

Case 4: Load using belt and concurrently Cat +SiC

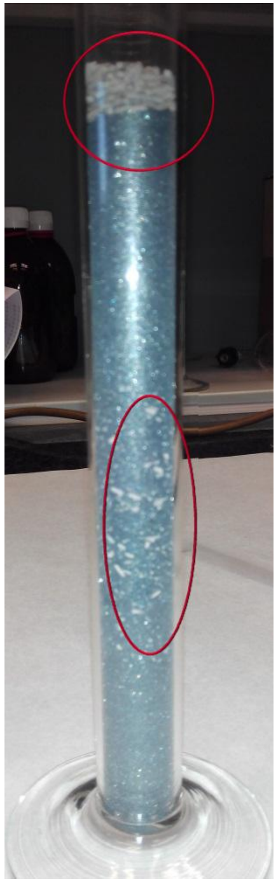


Loading Procedure

Case 3: Leaned load premixing

Loading Procedure

Case 2: Concurrently Catalyst + SiC using funnel



Loading Procedure

Case 1: Premix catalyst/SiC