Worksheet: Unsteady state material balances on a CVD reactor

Name(s): ____

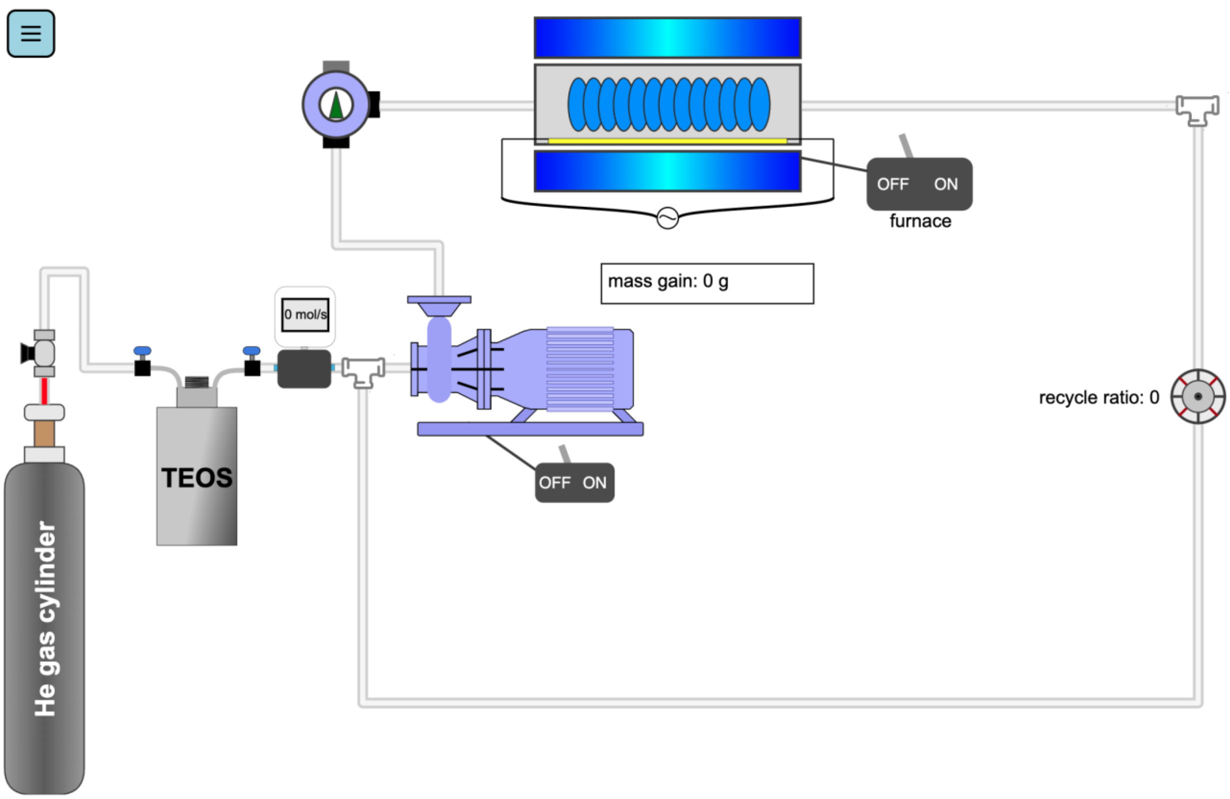
Fill in all sections – These are today's notes.

In this experiment, a stream of inert helium containing a low concentration of the reactant tetraethyl orthosilicate (TEOS, or $Si(C_2H_5O)_4$) at 0.050 mol% is fed at a constant rate into a high-temperature reactor, where it produces solid-phase SiO_2 and vapor-phase water and ethylene. Silicon dioxide (SiO_2) deposits on material surfaces inside the chemical vapor deposition (CVD) reactor. The reactor has recycle. Mass balances are calculated for the system.

Student Learning Objectives

1. Write material balances for a reacting system with accumulation terms.
2. Analyze how changing flow rates of recycle streams affects overall system behavior.

Schematic and Dimensions



A quartz crystal microbalance (QCM) measures the mass gain by the samples onto which the SiO_2 film deposits. The reaction is:

$$Si(C_2H_5O)_4(v) \rightarrow SiO_2(s) + 4\ C_2H_4(g) + 2\ H_2O(g)$$

Only a fraction of the TEOS reacts. The stream exiting the reactor flows out of the process, unless a valve is opened to allow recycle of a fraction of the stream at a fixed volumetric flow ratio R to the process outlet.

Questions to answer before starting the experiment

1. How is the mass gain of the solid surface related to the number of moles of TEOS reacted?

2. If the flow rate processed by the pump is kept constant, how will increasing the recycle ratio affect the total flow rate of the inlet and exit streams?

Running the experiment:

1. The 3-way valve at the reactor inlet should be flowing to the hood. The recycle valve should be closed.
2. Open the valve to the helium gas cylinder
3. Turn on the feed pump.
4. Switch the 3-way valve so the TEOS/He stream flows through the reactor. After flow in the system stabilizes, collect mass gain data as a function of time and record in Table 1. Repeat the measurement.
5. Change the recycle ratio ((volumetric flow rate of the recycle stream)/(volumetric flow rate of fresh feed stream)) to 1.0. Collect mass gain data as a function of time and record in the Table.
6. Repeat these measurements for recycle ratios of 2.0 and 5.0.
7. Calculate the average rate of mass gain.

Table 1

Recycle ratio	Mass gain	Time	Rate of mass gain (g/s)	Average rate of mass gain (g/s)
0				
0				
1.0				
1.0				
2.0				
2.0				
5.0				

5.0				

Compute the average mass gain per time at each recycle rate and record in the Table. What trend do you notice?

After the experiment:

1. First, analyze the system in the absence of recycle. Here, the stream leaving the bubbler and the stream entering the reactor are identical. Compute the molar flow rates of TEOS at the reactor inlet and reactor outlet by conducting a material balance on the reactor using the experimental information on rates of mass gain in the reactor.

2. When recycle is introduced, the molar flow rate of TEOS into the entire system (from the bubbler) is **not** equal to the molar flow rate entering the reactor. To account for this, set up additional material balances at the split point and at the mixing point. The inlet molar flow rate to the reactor will thus include the TEOS molar flow rate from the bubbler plus the TEOS molar flow rate from the recycle stream. In this system, the pump is controlled to maintain a constant volumetric flow rate of the gas stream through the reactor, which provides an additional constraint. Develop an expression for the molar flow rate out of the mixing point in terms of the recycle ratio R, the molar flow rate of TEOS out of the reactor, and the *original* molar flow rate of TEOS when R was set to zero.

$$\dot{n}_{TEOS}^{reactor-in} = \dot{n}_{TEOS}^{bubbler} + \dot{n}_{TEOS}^{recycle}$$

$$\dot{n}_{TEOS}^{reactor-in} = \frac{1}{R+1}\dot{n}_{TEOS\_no\_recycle}^{bubbler} + \frac{R}{R+1}\dot{n}_{TEOS}^{reactor-out}$$

Where $\dot{n}_{TEOS\_no\_recycle}^{bubbler}$ is a constant value that represents the flow from the bubbler when R = 0. When you specify that value (which is fixed) and R, the two unknowns are $\dot{n}_{TEOS}^{reactor-in}$ and $\dot{n}_{TEOS}^{reactor-out}$}

3. In this system, the pump maintains a constant volumetric flow rate of the gas stream. At constant temperature, the outlet molar flow rate of TEOS can be modeled as being a constant fraction of the inlet molar flow rate of TEOS. In other words, the reaction is expected to follow *first-order kinetics*. Use the mole balance from part 2 together with a material balance around the reactor (which should provide two equations with two unknowns) to complete the following table. Does the system exhibit first-order kinetics?

Recycle ratio	Molar flow rate of TEOS into reactor (mol/s)	Molar flow rate of TEOS out of reactor (mol/s)	(Flow rate out)/ (Flow rate in)
0.0			
1.0			
2.0			
4.0			

4. Complete a mole balance on the entire process and use it to complete the following table.

Recycle ratio	Molar flow rate of TEOS from bubbler	Molar flow rate of TEOS from process (exiting hood)	(Flow rate out)/ (Flow rate in)
0.0			
1.0			
2.0			
4.0			

Are these ratios constant? Why do you think they have the trend that they do?