

Name(s): _____

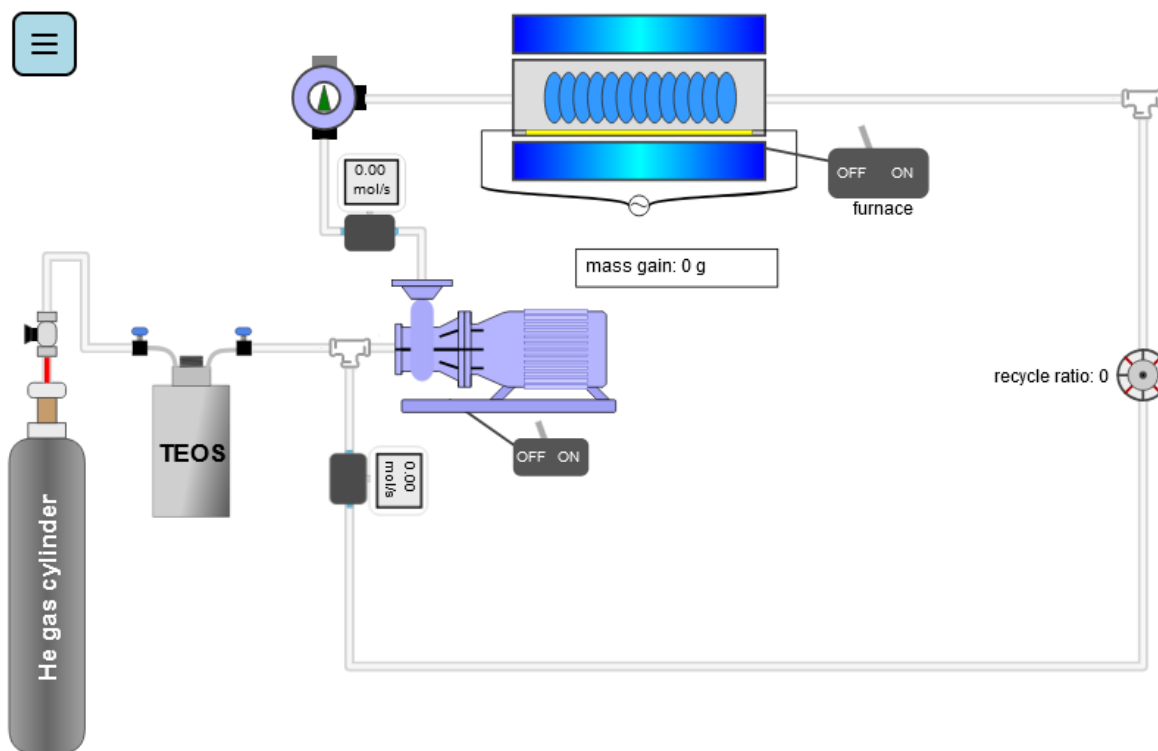
Fill in all sections – These are today's notes.

In this experiment, a stream of inert helium containing 0.34 mol% of the reactant tetraethyl orthosilicate (TEOS, $\text{Si}(\text{C}_2\text{H}_5\text{O})_4$) 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 a recycle stream and the recycle ratio can be changed. Mass balances are calculated for the system.

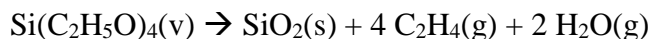
Student Learning Objectives

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

Equipment



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



Only a portion of the TEOS reacts. The stream exiting the reactor flows out of the process unless a valve is clicked opened and a recycle ratio R (ratio of recycle flow to fresh feed flow) is set so that a portion of the exit stream from the reactor mixes with the fresh feed stream.

Worksheet: Unsteady state material balances on a CVD reactor

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 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 ratio should be set to zero.
2. Open the He gas cylinder by clicking on the valve.
3. Turn on the feed pump.
4. Click on the 3-way valve so the TEOS/He stream flows through the reactor instead of to the hood.
5. Turn on the reactor furnace.
6. After flow in the system stabilizes, collect mass gain data as a function of time and record in Table 1. Repeat the measurement.
7. Click on the valve, set the recycle ratio to 1.0, and repeat the measurements and record in Table 1.
8. Repeat the measurements at recycle ratios of 2.0 and 5.0 and record in Table 1.
9. Calculate the average rate of mass gain at each recycle ratio.

Worksheet: Unsteady state material balances on a CVD reactor

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				

What trend do you notice in mass gain as the recycle ratio increases?

After the experiment:

1. Analyze the system in the absence of recycle. Here, the flow rates of 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 maintains 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

Worksheet: Unsteady state material balances on a CVD reactor

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 Table 2. Does the system exhibit first-order kinetics?

Table 2

	Molar flow rate of	Molar flow rate	
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Worksheet: Unsteady state material balances on a CVD reactor

Recycle ratio	TEOS into reactor (mol/s)	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 Table 3.

Table 3

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?