Propositions for the Distillation of TEOS

Summary

As demand for semiconductors continues to grow, so does the demand for ultra-pure silicon products. To keep up with this demand, optimizations in the process of purification are always being looked for. We were tasked by EMD Electronics to look into the design of a continuous distillation process and compare it to the currently utilized batch distillation process. This distillation is of a tetraethyl orthosilicate (TEOS), a precursor to thin film deposition used in EMD's production of semiconductors.

For the continuous distillation process, we found that it is possible to meet all but one of the desired specifications. This was achieved by utilizing two distillation columns. One was used to remove the components which boiled off at a lower temperature than TEOS while the other was used to condense out the components which boil at a higher temperature than TEOS. The unmet specification was component C. To meet this specification in future iterations, we propose removing more of the light components in the first distillation columns and then adding them back to the final product stream as needed.

For the batch distillation process, we also found it possible to meet all but one of the desired specifications, being the same component C. This is achieved with two main operating steps, which acted to first remove the light key impurities in the feed, then to capture the product which would meet the specifications. With component C not meeting specifications, further optimization could be performed in the first operating step order to reduce the concentration of component C in the product.

In the end we determined that continuous distillation is the best option to move forward with. The gross yield of the batch was 23.97% of the initial feed, while the continuous process managed to have a gross yield of around 90.0%. This and economic analysis clearly shows that if the continuous distillation can be reliably run, it greatly improves the physical and financial output of EMD.

Motivation

TEOS is a precursor to silicon oxide which is the main source for thin film deposition of doped and undoped silicon dioxide films. This is an important process in the creation of electronics. EMD has tasked us with purifying TEOS to desired specifications that are needed for their processes seen below.

		Feed	Product
Product	TEOS	99.80%	99.99%
Light Impurities	Ethanol	1500	35
	Water	75	5
	Comp C	75	1
	Comp D	25	1
Heavy Impurities	Comp B	20	1
	Comp E	100	45

Economics

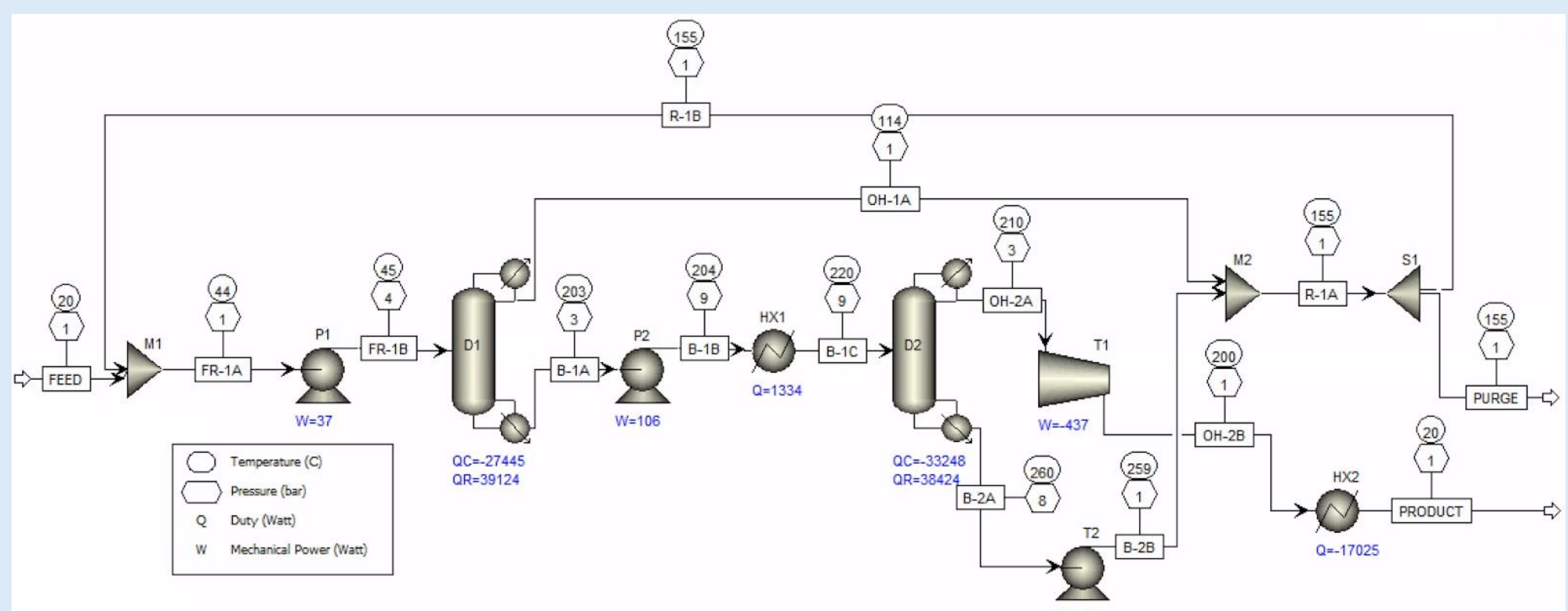
Continuous Distillation:

Compoenet Name	Total Direct Cost (USD)	Equipment Cost (USD)
P2	51,600.00	19,600.00
P1	35,400.00	5,300.00
D2	528,800.00	284,700.00
D1	416,400.00	194,900.00
	Total Capital Cost (USD)	5,693,648.85
	Total Operating Cost (USD/year)	1,253,418.96

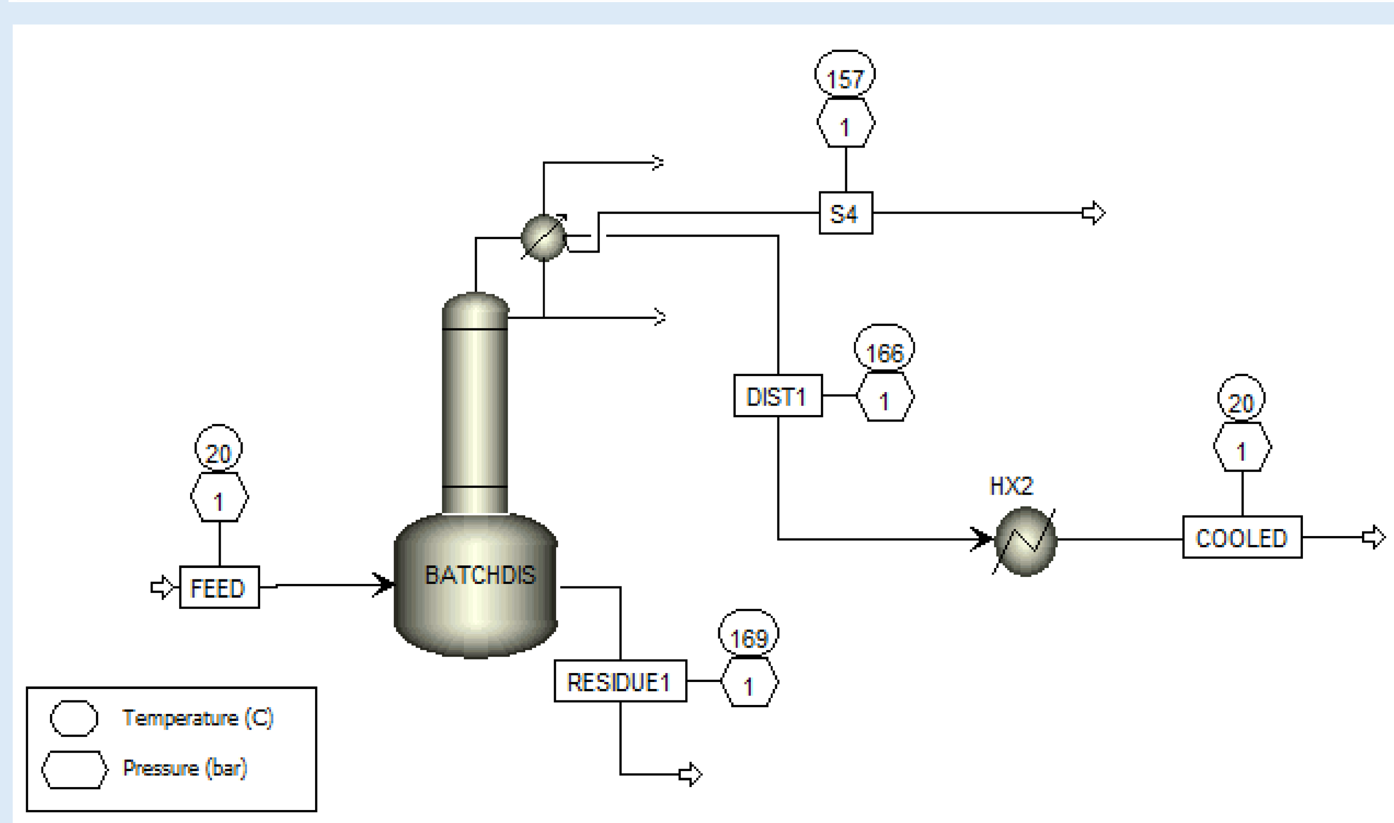
Batch Distillation:

Total Capital Cost (USD)	2,378,160.00
Total Operating Cost (USD/year)	954,497.00

Process Flow Diagrams



Cont. Mass Fraction	Feed	Product	Purge
TEOS	0.998	0.999933899	0.999933899
ETHANOL	0.001662971	0	0
WATER	8.31E-05	0	0
COMPB	3.22E-05	7.39E-10	7.39E-10
COMPC	8.31E-05	3.74E-05	3.74E-05
COMPD	2.77E-05	0	0
COMPE	0.000110865	2.87E-05	2.87E-05



Batch Mass Fraction	Feed	Product	Residue
TEOS	0.998	0.999974	0.999647
ETHANOL	0.001662971	9.29E-11	0
WATER	8.31E-05	3.63E-11	0
COMPB	3.22E-05	6.91E-10	7.92E-05
COMPC	8.31E-05	2.61E-05	5.55E-07
COMPD	2.77E-05	1.49E-10	0
COMPE	0.000110865	3.12E-07	0.000272826

Methods

Data for this model was collected using Aspen Plus V14. The properties of the relevant components were from Aspen's library and a template file provided by EMD. This file uses Peng-Robinson with Boston-Mathias modifications for the thermodynamic model. Distillation columns were modeled using the DSTWU method. Pumps were modeled with the ASME polytropic centrifugal model. To analyze the batch process, Aspen's batch separation model was used with 2 separate receivers.

Continuous Distillation:

There are two primary unit operations in the proposed continuous distillation process as can be seen in the PFD. In distillation column D1, the light impurities are removed through the distillate while the TEOS exits the bottom with heavy impurities. This bottom stream is then fed to distillation column D2 which removes the heavy impurities. This then produces a distillate with a purity better than the desired 99.99% TEOS.

Besides the two main distillation columns, there are many other operations to support the process. Pumps P1 and P2 pump up the pressure of the feed stream for D1 and D2 respectively to ensure flow is going in the right direction. Heat exchanger HX1 heats up the feed for D2 to decrease the heat duty of the column and improve separation. Heat exchanger HX2 cools down the overhead stream from D2 to prepare the final product for storage. Turbines T1 and T2 are both meant to recoup some energy as the process fluid is depressurized. Lastly, a recycle stream is used from the distillate of D1 and the bottoms of D2. This recycle stream increases the gross yield from around 81% to about 92%.

Batch Distillation:

For the batch distillation process, there is just one primary unit operation in the batch distillation column. The feed stream is fed at atmospheric pressure and 20°C into the batch distillation column, where the distillation product is fed to two different receivers. The first receiver will be taking any product from distillation from the startup and the initial distillation, which runs at a lower heat duty which will end up being a stream of mostly TEOS and lighter key impurities. Then the ideal product will be distilled and put into the second receiver, with the residue containing more heavy key impurities remaining in the pot as residue, which can be pumped out and removed. A heat exchanger will be utilized to bring the product stream down to an appropriate temperature. The residue can be pumped out and recycled, potentially by combining with other batch residues, or even with the product from the light key distillation in receiver 1, from where another batch distillation can be run to further purify the recycled feed.

Conclusion

Analysis of the product stream of the continuous process reveals that we hit all the desired specifications except for component C which ended up at 37 ppm. This is much greater than the desired 1 ppm and future modification to the continuous process should take this into account. To get closer to this goal of 1 ppm more optimization must be made with the first distillation column. In this model, more component C removal would decrease the ppm of the other light components. One solution to this is getting component C to the desired ppm and adding back the required component to compensate for the loss. In addition, another separator could be added in to specifically remove component C from the solution. This however would increase the cost of the plant significantly.

When examining the stream results of the batch process, Comp C reached 26 ppm with Comp E 0.3 ppm. Compared to provided target specifications of Comp C at 1 ppm and Comp E at 45 ppm, it could be said that the allocated contaminant concentration for Comp E was in Comp C instead. If the purity is not the goal and instead the desired specification for contaminants is the priority, then further optimization will be required to reduce the impurity concentration of Comp C, potentially at the cost of an increase in Comp E. Other than this specification, all other components were underneath the allowable impurity concentration, which shows promise in the overall performance of the process.

When taking all factors into consideration, our recommendation would be to use a continuous distillation process for the purification of TEOS. Strictly from a composition standpoint, both processes came the specified purity, with some variations in the impurity concentration. However, while the batch process only recovers, as pure product, 23.97% of the initial feed, the continuous process manages to have a recovery of 92.2% pure product from the initial feed. From an efficiency standpoint, too little of the feed is recovered through the batch process despite the purity for the batch process to be economically and ecologically sustainable. Meanwhile, the continuous column has a relatively high recovery from the initial feed and with a 10 year timeline, a NPV of 7340.644 MM$. Ultimately, through our analysis, the continuous distillation column has multiple advantages over the batch distillation column, leading us to recommend a continuous distillation process for the purification of TEOS.