Diethyl Oxalate Process

In the first reactor (RX1, plug flow), the major fresh feed streams are oxygen and a mixture of ethanol and water. Small fresh feeds of nitric oxide and nitrogen are also fed into this reactor. There are two recycling streams. A large gas recycles stream contains mostly nitric oxide and nitrogen. The liquid recycles stream contains mostly ethanol and water.

2EtOH + 2NO +
$$1/2O_2 \rightarrow 2EN(ethyl nitrile) + H_2O$$

The RX1 reactor effluent is cooled before entering a flash drum. The liquid from the drum contains mostly unreacted ethanol, water and small amounts of light components. It is fed to a distillation column in which water product is removed and ethanol is recycled back to RX1. The liquid from flash drum FL1 is fed to a distillation column. The water produced in the reaction and the water in the mixture of ethanol and water fed in as fresh feed goes out the bottom. The liquid distillate is recycled back to reactor RX1. There is a small vapour purge stream to remove the light hard-to-condense components that come into the column in the liquid feed from the flash drum.

The vapour feed to reactor RX2(plug flow), 2 erections occur here,

2CO + 2EN
$$\rightarrow$$
 DEO(diethyl oxalate) + 2NO (majority) CO + 2EN \rightarrow DEC(diethyl carbonate) + NO (minority)

The RX2 reactor effluent is cooled in the feed-effluent heat exchanger and in a chilled-water cooler and fed to flash drum FL2. The liquid, which has diethyl oxalate, is fed to a distillation column for diethyl oxalate purification. The gas from the flash drum is split between a vent stream and a gas recycle stream. The major portion of the gas is compressed and recycled back to the RX1 reactor.

The liquid from flash drum FL2 is fed to a distillation column operating under vacuum conditions. The diethyl oxalate product goes out the bottom. The reflux-drum temperature produces both a vapour distillate and a liquid distillate that are sent to a downstream waste-disposal facility.

- Q.1) Draw a neat flowsheet clearly showing all the unit operations and material/energy streams as described in the process described above.
- Q.2) Clearly show all the control valves on the flow sheet.
- Q.3) What is the control and steady-state degrees of freedom for the process.
- Q.4) Draw a plantwide regulatory control system with the flowrate of oxygen as the throughput manipulator.
- Q.5) Draw a plantwide regulatory control system with the total mixed stream of ethanol coming from the distillate of column C1 and the fresh feed of the ethanol/water mixture as the throughput manipulator.

Ans.) Control degree of freedom = 18, Steady-state degree of freedom = 12
$$T_{C1}$$
, T_{C2} , T_{RX1} , T_{RX2} , $[L/F]_{C1}$, $[L/F]_{C2}$, F_{O2} , F_{NO} , F_{N2} , F_{EtOH} , F_{CO} , and F_{Vent}



