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ABSTRACT

A small, laboratory-sized loop has been designed for steady state radiolytic gas recombination studies. It consists of an electrolysis cell, which simulates the radiolytic gas formed in a reactor, and a bomb for containing slurry under test. The electrolysis capacity of the system is adequate for the study of slurries having recombination rates as high as 10 moles/hr/liter of slurry at the proposed slurry reactor operating conditions.

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Electrolysis Cell for Recombination Studies

1.0 Summary

A small, laboratory-sized loop has been designed for steady state radiolytic gas recombination studies. It consists of an electrolysis cell, which simulates the radiolytic gas formed in a reactor, and a bomb for containing slurry under test. The electrolysis capacity of the system is adequate for the study of slurries having recombination rates as high as 10 moles/hr/liter of slurry at the proposed slurry reactor operating conditions.

The condensate return from the bomb to the electrolysis cell avoids dilution of the slurry. Gravity feed of the condensate makes a circulating pump unnecessary.

The use of a separate cell for dissociation permits accurate control of the gas production rate under nonexplosive conditions. A trap in the gas line prevents the entrainment of electrolyte and interrupts the propagation of an explosion from one vessel to the other.

2.0 Description

The bomb has a volume of 30 cc and contains 10 cc of slurry. It is mounted at a 45° slope on a translational shaker which keeps the slurry in suspension. The bomb operates at a temperature ranging from 200° to 300°C. The steam in the vapor space above the slurry is condensed on cooling coils in the top of the bomb and returned to the electrolysis cell. The bomb is made of 347 stainless steel.

The nickel cylinder forming the outer shell of the electrolysis cell serves as one electrode and a central nickel rod serves as the other. The cell contains 100 cc of 25% KOH solution as an electrolyte and a small vapor space above the liquid, largely filled with a teflon baffle which serves as an entrainment separator and a liquid level control. It operates at 30°C and the loop pressure of 1500 psi.

The trap in the gas line is a stainless steel block with a wire mesh filter submerged in water. In case of an explosion, the water will absorb the heat and the wire mesh will obstruct the propagation of the shock wave.

The three components of the loop are connected with 35 mil stainless steel capillary tubing with Autoclave high pressure connectors.

Figure 1 shows a block flowsheet of the apparatus.

For more detailed drawings, refer to ORNL drawings: D-21296, D-29297 and D-29298.

3.0 Construction

All pressure connections are welded for high pressure capillary fittings. Silver soldering is used only on external connections in remote areas.

Upon completion of construction, each component is to be electro-polished to remove any sharp edges which might cause out-of-slurry recombination. This process consists of putting the pieces to be polished in a bath of concentrated phosphoric acid containing 10% sulfuric acid at 40° - 50°C. A current density of 1 to 3 amp/in.² (D.C.) is applied for 3 or 4 minutes with the piece being polished serving as the anode. The bomb and cell shells serve as their own bath containers. A carbon or stainless steel cathode is suspended in the center.

The electrolytic cell is next operated by itself for approximately two days with the 25% KOH solution at 5 amps and 2-3 volts (as required) to clean the cell. A soapy foam will appear on the surface at first. The electrolyte in the cell is then replaced to ready it for operation.

The bomb is to be mounted in the existing heater and shaker equipment with minor alterations to the hold-down assembly. The electrolytic cell needs to be mounted in a vessel which has a constant temperature bath and can be raised or lowered at least five inches to vary the liquid head in the condensate line. The normal liquid level difference is 6.0 in. plus the pressure drop through the trap, which must be determined experimentally.

The trap should be mounted beside the electrolytic cell with its liquid level approximately that of the electrolyte.

4.0 Instrumentation

The bomb temperature is measured by a thermocouple mounted in a thermowell extending to the bottom of the bomb. A thermocouple on the exit cooling line gives a Δt for the cooling coils.

A Baldwin cell records the system pressure through a pressure tap on the trap.

The electrolytic gas generation rate is controlled by the current consumption of the cell as indicated by the rectifier ammeter. The direct current is supplied by a Stevens Bench Rectifier, Model 725SLC or 750SLC. If the rectifier is connected in parallel with a storage battery, the current and voltage output will be smoothed considerably.

5.0 Operation

Prior to the hookup of the system, the bomb will be calibrated to determine the dynamic steam pressure corresponding to a given bomb temperature and cooling air ΔP .

After filling the vessels and condensate line, the capillary tubing is connected and the vapor spaces are purged with oxygen. Gas generation is then started in the electrolysis cell and heating of the slurry is begun. The oxygen atmosphere is bled off through the Baldwin cell connection as the system pressure rises. The temperature vs. steam pressure calibration data enables the operator to adjust the gas generation rate to maintain a small gas overpressure on the system as it is brought up to temperature. When the bomb has reached operating temperature, the desired gas production rate is set on the electrolysis cell and the electrolytic gas overpressure is allowed to stabilize at a steady state operating condition.

6.0 Calculations

Electrolysis Cell

Gas Production

$$\text{Rate: } 0.1 \frac{\text{mole}}{\text{hr}} = \frac{70 \text{ cc}}{\text{hr}} \text{ at } 1500 \text{ psi, } 300^\circ\text{C}$$

Current Requirement:

$$1 \frac{\text{mole}}{\text{hr}} \neq 2 \times 96,500 \frac{\text{coulombs}}{\text{hr}}$$

$$\text{or } 2 \times 26.8 \text{ amp}$$

$$0.1 \frac{\text{mole}}{\text{hr}} \neq 2 \times 26.8 \times 0.1 \text{ amps} = 5.36 \text{ amps}$$

$$A_{\text{Electrode}} = \pi \times 1 \text{ cm} \times 35 \text{ cm} = 110 \text{ cm}^2$$

Condensate Return

$$0.1 \frac{\text{mole}}{\text{hr}} = 1.8 \frac{\text{gms H}_2\text{O}}{\text{hr}} = 0.00396 \frac{\# \text{ H}_2\text{O}}{\text{hr}}$$

$$\text{at } 1500 \text{ psi, } 300^\circ\text{C } 0.00396 \frac{\# \text{ H}_2\text{O}}{\text{hr}} = 2.5 \text{ cc } \frac{\text{H}_2\text{O}}{\text{hr}}$$

$$\text{at } 1500 \text{ psi, } 30^\circ\text{C } 0.00396 \frac{\# \text{ H}_2\text{O}}{\text{hr}} = 1.8 \text{ cc } \frac{\text{H}_2\text{O}}{\text{hr}}$$

System Pressure Drop

$$\text{Capillary Pressure Drops from Poiseville Eq. } \frac{\Delta P}{L} = \frac{0.000034 \mu W}{\rho d^4} :$$

$$\Delta P_{O_2 + H_2} = 0.0034 \frac{\text{psi}}{\text{ft}}$$

$$\Delta P_{AV \text{ H}_2\text{O}} = 0.005 \frac{\text{psi}}{\text{ft}}$$

Assume length of capillary tubes = 3 ft:

l = liquid head in the condensate line due to elevation

Sp. Gr. of Electrolyte = 1.24 Sp. Gr. Slurry = 1

Density of Condensate = $50 \frac{\#}{ft^3}$ Sp. Gr. Gas = 0.07

$$l = h_{L \text{ gas line}} + h_{L \text{ slurry}} + h_{L \text{ condensate line}} + 16" \cdot (\rho_{KOH} - \rho_{H_2O}) + (l \frac{1}{12}) \rho_{gas}$$

Head loss through capillaries:

$$0.0034 \times 3 \text{ psi} + 0.005 \times 3 \text{ psi} = 0.0152 \text{ psi} = .52 \text{ in. of } H_2O$$

Gas head

$$(l - 1.5")(0.07) = 0.07 l - 0.10"$$

Electrolyte head

$$16"(1.24 - 1) = 3.8"$$

Slurry head

$$1" (1) = 1"$$

$$l = .52" + 0.07 l - 0.10" + 3.8" + 1"$$

$$l = 5.84"$$

Cooling Coils

$$h_{fg} = 592 \frac{\text{Btu}}{\#} \text{ at } 300^\circ\text{C, } 1500 \text{ psi}$$

$$M = .00396 \frac{\#}{\text{hr}}$$

$$\text{Required heat rate} = .00396 \times 592 = 2.34 \frac{\text{Btu}}{\text{hr}}$$

The condensation rate will be well above the required $.00396 \frac{\#}{\text{hr}}$ to keep the condensate line full and to wash slurry off the walls of the bomb. An arbitrary heat removal rate of $20 \frac{\text{Btu}}{\text{hr}}$ will be used in further calculations for this reason.

$Q = 20 \text{ Btu}$ Use 12" capillary for cooling

$$A_{\text{Inside}} = 0.005 \text{ ft}^2$$

$$A_{\text{Outside}} = 0.00875 \text{ ft}^2$$

$$\text{Coolant: } 90 \text{ psig air } 80^\circ\text{F}, \frac{c_p}{k} = .77, \rho = .523, c_p = .240$$

$$\gamma = 100 \text{ fps} \quad Q = 100 (.00000219)(3600) = .79 \text{ Cfh}$$

$$\Delta T = \frac{20}{(.79)(.523)(.24)} = 190^\circ\text{F}$$

$$Re = \frac{V d_p}{\mu} = \frac{(100)(3600)(.00167)(.523)}{.0505} = 6,230$$

$$n_i = .023 \frac{K}{d} (Re)^.8 (Pr)^.3 = (.023)(9.46)(1,100)(.924) = 221$$

$$\text{Assume } U_o = 200 \frac{D_i}{D_o} = 200 \frac{20}{35} = 114$$

$$Q = U_{o,o} A_o (T_{av} - T_{av}) = (114)(.00875)(577-170) = 402 \frac{\text{Btu}}{\text{hr}}$$

$$\Delta P = f \frac{L}{d} \frac{V^2}{2g} \frac{\rho}{144} = (.035) \left(\frac{1}{.00167} \right) \left(\frac{10,000}{64.4} \right) \left(\frac{.523}{144} \right) = 11.8 \text{ psi}$$

The cooling coils will have a throttling valve on the downstream side to regulate the heat removal. It may be necessary to use a preheater on the coolant or some other means of reducing heat removal.

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RSC/nr

FIGURE 1

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