FLOW ELECTROLYZERS. VII.*

DETERMINATION OF RADIAL DISTRIBUTION COEFFICIENT OF AMALGAM FOR VERTICAL TOWER DECOMPOSER

S.RAJASEKARAN**, I.ROUŠAR and V.CEZNER

Department of Inorganic Technology,
Institute of Chemical Technology, 166 28 Prague 6

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The radial distribution coefficient of amalgam and mercury for a bed of graphite balls of 1 cm in diameter was determined. Its values for amalgam correspond to those found earlier for other liquids on a similar bed.

During last years, amalgam electrolyzers for a load of 100 kA or more have been exploited industrially. The produced amalgam is decomposed mostly in tower decomposers. In engineering calculations of the decomposer parameters, besides kinetic equations for the calculation of the rate of decomposition also the process of amalgam distribution in the reactor bed is essential, which is connected with a perfect utilization of the whole bed. In calculations of the decomposition tower there are two limiting factors with respect to the rate of amalgam decomposition: The first one is drowning of the tower with amalgam at high specific loads of the decomposer – the reaction rate drops to zero owing to a decrease of the free surface area of the graphite bed. The second limiting factor is that with a decomposer of unproper design a considerable amount of the amalgam flows down along the wall and thus escapes the decomposition reaction, which again results in a decrease of the average rate of amalgam decomposition. This effect can occur even with small rates of flow of the amalgam through the tower and can be suppressed by injecting the amalgam prevailingly into the center of the bed or by using a conical tower. To describe quantitatively the amalgam distribution in the bed, the coefficient of radial distribution of the amalgam must be known besides other data. The determination of this coefficient was not described in the literature. The aim of the present work is to determine experimentally the coefficient of radial distribution of the amalgam in a tower filled with graphite balls of a diameter of 1 cm.

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** Present address: Indian Institute of Technology, Kanpur, India.

THEORETICAL

A statistical treatment of the amalgam distribution in a fixed bed reactor was performed by Cihla and Schmidt[1]. Their equation valid for an axially symmetrical system is

$$D\left(\frac{\partial^2 f(r,z)}{\partial r^2} + \frac{1}{r}\frac{\partial f(r,z)}{\partial r}\right) = \frac{\partial f(r,z)}{\partial z}, \qquad (1)$$

where D (cm) denotes coefficient of radial distribution of the liquid phase, $f(r, z)$ density of wetting (cm^3/cm^2 s), r radial coordinate, and z axial coordinate. In view of the determination of the value of D, it is advantageous to assume that the liquid enters the center of a semi-infinite bed ($r \to \infty$) of a finite height, z, *i.e.* the wall of the tower has no influence on the distribution of the liquid. Thus, the boundary conditions have the form

$$r = 0: f(r,0) = \infty\,; \qquad 0 < r < \infty: f(r,0) = 0\,;$$

$$\lim_{r\to\infty} f(r,z) = 0\,, \qquad Q = 2\pi \int_0^\infty r f(r,z)\,\mathrm{d}r\,, \qquad (2)$$

where $Q(cm^3/s)$ denotes volume rate of liquid flow. These boundary conditions are fulfilled if the experiment is arranged so that in a bed height H and diameter a the liquid does not reach the tower wall at $z = H$. The solution of Eq. (*1*) is

$$\frac{f(r,z)}{Q/\pi a^2} = \frac{1}{4\,\mathrm{To}} \exp\left(-\frac{r^2}{4a^2\,\mathrm{To}}\right), \quad \mathrm{To} = DH/a^2\,. \qquad (3), (4)$$

It is convenient to determine experimentally the rate of flow of amalgam, $Q_{1/2}$, through an annular area between two circles of radii r_1 and r_2 with centers on the tower axis in a plane perpendicular to the axis and located under the bed. From Eqs (*2*) and (*3*) we obtain

$$Q_{1/2}/Q = \exp\left(-r_1^2/4HD\right) - \exp\left(-r_2^2/4HD\right). \qquad (5)$$

Hence, from experimentally determined values of $Q_{1/2}$, Q, r_1, r_2 and H the value of D can be found by iteration from Eq. (*5*), which was used in the present work.

EXPERIMENTAL

Apparatus. The coefficient of radial distribution of the amalgam or mercury was determined on a semi-pilot scale decomposition tower in the outlet of which four annuli were symmetrically

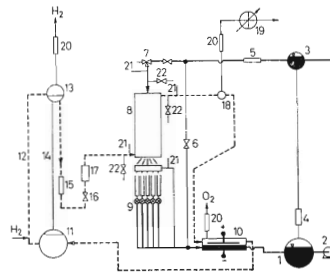

FIG. 1

Experimental Setup for Measurement of Radial Distribution Coefficient

1 Mercury reservoir, 2 mercury pump, 3 overflow vessel for mercury, 4 overflow tube, 5 heat exchanger, 6 regulation stopcock enabling to shunt the decomposition tower, 7 regulation stopcock, 8 decomposition tower, 9 calibrated separating funnel, 10 electrolyser, 11 sodium hydroxide reservoir, 12 air lift for electrolyte, 13 overflow vessel for electrolyte, 14 overflow tube, 15 heat exchanger, 16 flow regulator, 17 rotameter, 18 bubble separator, 19 exhaustor, 20 hydrogen cooler, 21 thermometer, 22 outlet for taking samples.

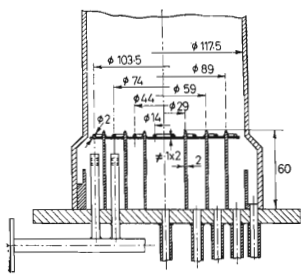

FIG. 2

Lower Part of Decomposition Tower

located as shown in Fig. 1. The functioning of the apparatus is as follows. The amalgam is pumped from reservoir 1 by pump 2 into overflow vessel 3 by means of which a constant flow through the tower is maintained. The excess amalgam flows back into the reservoir. From the overflow vessel the amalgam comes into heat exchanger 5 where it acquires the necessary temperature (measured by a thermometer). The rate of flow is controlled by stopcock 7. Before the amalgam enters the decomposer 8 it is possible to take samples and to measure approximately its rate of flow with the aid of a three-way stopcock 22. The rate of flow is measured exactly at the tower outlet. The amalgam flows from each annular section of the tower through a U-shaped

tube to separate the electrolyte and through calibrated separating funnels 9 into electrolyzer 10 and back into the reservoir.

The decomposition tower has the form of an iron cylinder, 42 cm in height and 11·75 cm in inner diameter, the lower part of which is conically widened at an angle of 15° and provided with a flange (Fig. 2). Four concentric tubes are welded to the bottom of the tower forming four collectors from which the amalgam is led into the U-shaped tubes. Above the bottom there is a lattice which supports the bed of the decomposer. The amalgam flowing downwards through the bed falls into the collectors, and from the rates of flow of the amalgam through each section the coefficient of radial distribution of the amalgam can be calculated. The portion of the amalgam flowing along the tower wall is measured separately; it must be equal to zero during measurement of the distribution coefficient. A tube of an inner diameter of 5 mm located in the axis of the tower serves as amalgam inlet.

An electrolyzer in the form of a horizontal channel of dimensions of 6 × 3 × 40 cm was made of organic glass. Its top part contains platinum anodes. An electric contact to the mercury cathode is provided by an iron plate with two welded current leads which pass through the bottom of the electrolyzer. The electric current is taken from a semiconductor rectifier of variable performance (maximum 680 A at 8 V). The amalgam enters the electrolyzer at the bottom, flows along the bottom and exits through an overflow so that the iron plate is always covered by an amalgam layer even if the supply of mercury to the electrolyzer is stopped. The sodium hydroxide solution flows countercurrently with respect to the amalgam. A separator of oxygen is built-in in the central part of the electrolyzer lid. The evolved oxygen is led through coolers into the atmosphere.

The sodium hydroxide solution is pumped from reservoir 11 (Fig. 1) by air lift 12 into overflow vessel 13. The air lift is fed with electrolytic hydrogen which is led through cooler 20 into the atmosphere. The electrolyte flows from the overflow vessel through heat exchanger 15, flow regulator 16 and rotameter 17 into the decomposer 8. The mixture of hydrogen and electrolyte is led from the decomposition tower through bubble separator 18 into electrolyzer 10 and back into the reservoir 11.

Method of measurement. The measurements were performed on a bed of graphite spheres of a diameter of 1·093 cm (Tohoku Kyowa, Japan). The bed was 10·9 cm in height and 11·75 cm in diameter (inner diameter of the tower). Three cases were considered: Mercury on dry bed at 25°C, mercury in the presence of 37% NaOH at 80°C, and amalgam containing 0·15% Na

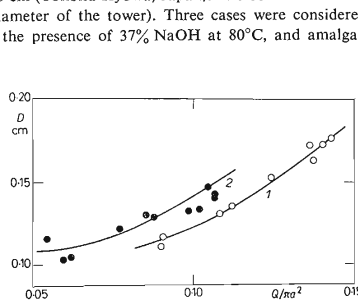

FIG. 3

Dependence of D(cm) on Mean Wetting Density $Q/\pi a^2$ (cm^3/cm^2 s)
1 Mercury in 37% NaOH at 80°C, *2* 0·15% Na amalgam in 37% NaOH at 80°C.

in the presence of 37% NaOH at 80°C. Before every measurement, the rate of flow was followed periodically and when a stationary state was reached the rate of flow of mercury or amalgam was measured with the aid of calibrated separating funnels. To eliminate the influence of the structure of the graphite bed on the value of D the tower was emptied after every series of measurements and refilled with the bed. The outer radii of the four concentric annular sections at the bottom of the tower were 1·40, 2·95, 4·45 and 5·87 cm.

RESULTS

The values of D for mercury in the absence of the electrolyte showed a large scatter and their dependence on the rate of flow, $Q/\pi a^2$, varying from 0·05 to 0·14 cm^3/cm^2 s could not be reliably determined. The average is $D = (0{\cdot}134 \pm 0{\cdot}015)$ cm at 25°C. The values of D for mercury in the presence of 37% NaOH at 80°C are shown graphically in Fig. 3, curve *1*; the case of substituting 0·15% Na amalgam for mercury is illustrated by curve 2. Each point is an average from 8 meassurements. It can be estimated that for $Q/\pi a^2 \to 0$ the value of D is about 0·1 cm for pure mercury and 0·11 cm for the amalgam.

Kolář and Staněk[2] found values of D for water, 0·2% aqueous solution of a detergent (Sapon), 45% aqueous glycerin, and 7% aqueous n-butyl alcohol in the range 0·1 – 0·17 cm. Our values of D (0·1 – 0·15 cm) for mercury and 0·15% Na amalgam in the medium of 37% NaOH lie in the same range and their increase with increasing rate of flow is also in accord with the previous findings[2].

REFERENCES

1. Cihla Z., Schmidt O.: This Journal *22*, 896 (1957); *23*, 569 (1958).
2. Staněk V., Kolář V.: This Journal *33*, 2636 (1968).

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