PRESSURE DROP, LIQUID HOLDUP AND MASS TRANSFER IN A
GRAPHITE FIBRE BED WITH UPWARD CO-CURRENT GAS-LIQUID
FLOW

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We accept this thesis as conforming
to the required standard

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Date 15th Sept. 1943
Abstract

The pressure drop, liquid holdup and overall mass transfer capacity have been studied in a graphite fibre electrode of porosity 0.90 and fibre diameter 22 micrometer with cocurrent upward gas-liquid flow.

A U-tube mercury manometer was used to measure the pressure drop in a graphite fibre bed 356 mm long by 38 mm wide by 3 mm thick. The gas and liquid were oxygen and water. For gas load $0 \leq G \leq 0.43 \text{ kg/m}^2\text{s}$ and liquid load $1.46 \leq L \leq 7.30 \text{ kg/m}^2\text{s}$, the pressure gradient ranged from 0.24 and 2.09 bar/m. The correlation for the pressure gradient is

$$\Delta P = L \left[ 0.36 + 1.182 \left( \frac{G}{L} \right)^{0.618} \right]^2$$

where

- $\Delta P$ = pressure gradient\hfill bar/m
- $L$ = liquid load\hfill kg/m$^2$s
- $G$ = gas load\hfill kg/m$^2$s

The quick closing valve method was used to measure the total liquid holdup in a graphite fibre bed 356 mm long by 38 mm wide by 3mm thick. Oxygen and 1M aqueous sodium hydroxide were the fluids used for the total liquid holdup measurements. For gas load $0 \leq G \leq 0.35 \text{ kg/m}^2\text{s}$ and liquid load $1.53 \leq L \leq 7.62 \text{ kg/m}^2\text{s}$ the liquid holdup ranged from 0.44 to 1.0.

The correlation for the total liquid holdup is

$$h_L = 1 - 0.907 L^{-0.362} G^{0.301}$$
where $h_L = \text{liquid holdup}$

The overall mass transfer capacity was determined by the electrochemical reaction method with the electro-reduction of oxygen to peroxide. The electrochemical reactor used consisted of graphite fibre cathode bed of dimension 89 mm long by 38 mm wide by 3 mm thick. The cathode was separated from the anode by a cation membrane. Oxygen gas and 1M aqueous sodium hydroxide were the fluids used. For gas load $0.04 \leq G \leq 0.36$ and liquid load $3.05 \leq L \leq 7.62$ the overall mass transfer capacity ranged from 3.4 to 9.0 $\text{s}^{-1}$. The correlation for the overall mass transfer capacity is

$$K_a = 5.9 \, L^{0.371} \, G^{0.233}$$

where $K_a = \text{overall mass transfer coefficient} \quad \text{m/s}$
$a = \text{effective interfacial area for gas to solid} \quad \text{m}^{-1}$
$L = \text{superficial liquid load} \quad \text{kg/m}^2\text{s}$
$G = \text{superficial gas load} \quad \text{kg/m}^2\text{s}$
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CHAPTER 1

Introduction

Over the years one of the aims of electrochemical technology has been to increase the space-time yield of electrolytic cells. The need to design a cell with a high active surface area per unit volume of reactor has led to the design of new electrodes which differ from the classical plate electrodes.

Presently, in some processes, the classical plate electrodes are being replaced by porous electrodes. This is because recent studies have proved that porous electrodes have a higher active electrode area per unit volume of reactor and also a higher overall mass transfer capacity when compared to the classical electrodes. These porous electrodes have commonly been applied in the reaction of dilute species whose conversion depends on effective mass transfer. Typical examples are recovery or separation of dissolved metal ions, the oxidation of dissolved organic wastes and surfactants and the electrosynthesis of organic compounds. Also, they have been employed for separation of organic species by electrosorption, fuel cells and batteries [1], and peroxide synthesis. These porous electrodes have been constructed from wire screens, expanded metal, packed beds, metallic foams and felts [2]. They have been found to have high surface area per unit volume of electrode and to improve mass transfer by turbulent promotion when compared to the classical plate electrodes [2].
Chapter 1. Introduction

A trickle-bed electrode is a porous electrode through which a gas and an electrolyte solution are passed in cocurrent flow. Usually, the gas and liquid are passed cocurrently downward but sometimes the direction of the flow may be upward. For this study the trickle-bed will refer to an upward flow system. Just like all other porous electrodes, the effective electrode thickness of the trickle bed is limited to about one centimetre. This is because of the decrease in electrode potential which occurs in the direction of current flow. Usually a long electrode bed is required to be able to obtain useful chemical conversion per pass at high space-velocity and low voltage drop. The particle or fibre diameter for these electrodes is about two millimetres or less. This is lower than the size generally employed in trickle-bed thermochemical reactors [3].

In trickle-bed electrochemical reactors there is a simultaneous absorption of the reactive gas into the liquid phase followed by a further electrochemical reduction or oxidation of the dissolved gas. The reactant gas when introduced into the cell along with the electrolyte phase helps to improve the availability of the reactant to the electrode surface. It also affects the hydrodynamics of the flow in the porous electrode and thus its mass transfer capacity. Usually, high gas loads used in trickle-bed electrodes cause a decrease in the liquid holdup but increase the pressure drop [3].
Chapter 1. Introduction

According to Takahashi and Alkire [4], the few data that exist for gas-liquid flow over small packing of about two millimetres in diameter or less, suggest that their mass transfer behaviour is different from that of particles in the range of 2.6-76 mm. Thus, the many two-phase flow mass transfer correlations reported in the Chemical Engineering literature do not apply to porous electrodes. There is therefore the need for a more accurate knowledge of the fluid dynamics and models to be developed to predict the behaviour of porous electrodes, for example the graphite fibre electrode.

Since the fibre diameter (22 micrometer) of the graphite fibre trickle bed electrode is less than two millimetres it is likely that its pressure drop, liquid holdup and mass transfer rate will be different from the larger packings reported in the Chemical Engineering literature.

The objective of this study was to determine by experiment the pressure drop, the liquid holdup and the overall gas-solid mass transfer capacity for a range of gas-liquid co-current upward flow in a graphite fibre electrode. Also, to develop empirical correlations for pressure drop, liquid holdup and mass transfer capacity to assist in designing graphite fibre trickle-bed electrodes.
CHAPTER 2

2: Literature Review

2:1 Hydrodynamics

The transport processes within a packed bed depend on the prevailing flow regime. The flow regime primarily depends on the flow loads of the gas and liquid phases, together with the nature, size and status of the packing material.

The various flow regimes that exist for co-current gas-liquid downflow are trickle flow which is gas continuous, pulsed flow, spray flow and bubble flow. The flow regime for cocurrent upflow through a packed column is composed of various modes such as liquid-continuous, gas-continuous and slug flow. In the gas continuous regime, the liquid may flow partly as drops and partly as a film over the packing.

In the trickle-flow regime, the liquid trickles over the packing in the discontinuous shape of films, rivulets, and drops near a stagnant continuous gas phase. The trickle flow regime in downward flow is usually referred to as the poor interaction regime because there is very little interaction between the gas and liquid [5].
2:2 Gas-Liquid Flow

Gas-liquid flow in fixed bed thermochemical reactors has been widely used over the years. Some of the applications include coal liquefaction, catalytic hydrodesulfurization, selective hydrogenation, etc [6].

Countercurrent flow is often used to provide effective gas-liquid contacting for distillation towers and gas absorption columns. It is not usually applied in electrochemical cells because of the problem of flooding which limits the range of flowrates.

Fixed-bed electrodes with simultaneous co-current flow of gas and electrolyte solution have been investigated for electrochemical processes involving gaseous reactants like oxygen [7,8,9], sulphur dioxide [10], propylene [11]. Jansson and other workers have investigated the use of bipolar trickle towers to solve pollution control problems (scavenging of copper, silver, lead and calcium from dilute streams, oxidation of cyanide concentration by direct and indirect methods, etc.) [12-14].

Compared to the co-current gas-liquid downflow reactor, the upflow reactor gives higher pressure drop, better wetting of the catalyst and higher liquid holdup [15].
Chapter 2. Literature Review

2.3 Pressure Drop

Since pumping costs could play a significant part of the total operating costs in an electrochemical process it is important to consider the pressure drop in the design of electrochemical reactors. Significant pressure drop can also cause large undesirable changes in the partial pressure of the reacting gas within the reactor and mechanical problems in cell construction. There is little literature on pressure drop in electrochemical reactors.

Two main approaches are usually adopted to correlate the pressure drop for a two phase flow in trickle-bed reactors. One is the empirical approach, the other involves the modification of the single-phase flow Ergun equation to account for the two-phase flow.

Single-phase flow: The Ergun equation [16] has been widely accepted and used to predict the pressure drop for single phase flow through fixed beds. Ergun proposed universal constants of 150 and 1.75 for the two parameters in his equation. He assumed that the constants hold for all types of packings. He also proposed the use of an equivalent spherical particle diameter.

Macdonald et. al. [17] investigated the single phase flow model predictions by Ergun. After a thorough comparison of their data
with the Ergun equation they concluded that the functional form of
the Ergun equation represented their data well but discovered large
errors which were up to two orders of magnitude with the use of the
universal Ergun constants. They therefore suggested the need to
determine the constant for the packing of interest since the
universal constants do not hold for all packings.

Two-phase flow:— Sweeney [18] assumed the single-phase flow Ergun
equation for each phase in the two-phase flow. He also assumed that
the solid particle size remained unchanged and considered the
liquid as a solid boundary relative to the gas. He also stressed
the need to determine the constants for the packing under
consideration instead of using the ones suggested by Ergun in his
equation.

In arriving at their model for gas phase pressure drop in two-
phase flow Specchia and Baldi [19] considered the effect of phase
interaction in the trickle flow to be negligible. They recommended
that the coefficient in the Ergun equation be determined.

The Pittsburgh Energy Research Centre (PERC) [20] obtained their
pressure drop data in highly-pulsed and spray-flow regimes in a
10.2 cm internal diameter clear acrylic column. They obtained over
300 pressure drop data points for both 6.35 mm by 6.35 mm and 3.2
mm by 3.2 mm pellets. Their data fitted well with Tallmadge’s
correlation [21]. Sato et al [22] correlated their data using the
Lockhart-Martinelli type of relation. They also graphically
represented some pressure drop data in the bubble, pulse and spray (gas continuous) flow regimes. They used the Ergun equation to determine the single-phase flow for each phase for the two-phase flow. They assumed that the pressure drop of each phase flow through the restricted section is the same as that for a single-phase flow through the whole section. Thus they did not account for the interaction effects between the gas and liquid.

Empirical Correlations:—Turpin and Huntington [23] used data obtained from an air-water flow system and 51, 102 and 153 mm diameter columns. They used tabular alumina particles for their packing. The diameter sizes of the packings were 7.6 and 8.2 mm. They correlated their pressure drop data for co-current upflow empirically. They chose gas loads ranging from about 0.02 kg/m²s to 38.3 kg/m²s. The range for the liquid load was from 6.52 to 54.33 kg/m²s. Their correlation which is shown in Table 1 related the gas and liquid phase Reynolds numbers to the pressure gradients.

Ford [24] measured the pressure drop in beds packed with approximately one millimeter diameter particles. He presented empirical correlations for both two phase pore flow and single-phase pore flow. According to Ford [24] the transition from one flow regime to the other could occur at a liquid holdup of 0.43. Before his correlation can be used knowledge of the liquid holdup is required. He suggested that if the liquid holdup is less than 0.43 the equation for the two-phase pore flow should be used and
Chapter 2. Literature Review

when the liquid holdup is greater than 0.43 then the correlation for the single pore flow should be used to calculate the pressure drop. Saada [25] discovered some inconsistencies in Ford’s correlations. He repeated and extended part of Ford’s work. He studied the air-water system in a 45-mm internal diameter, 400-mm long column. For his examination he used three different size packings of glass-bottom spheres of nominal diameter 0.514-, 0.974 - and 2.064-mm. He came out with two different empirical correlations one for the single-phase pore flow and the other for the two-phase pore flow. He developed an empirical correlation (shown in Table 1) which related the pressure drop to the Reynolds number for the gas and liquid, the diameter of the column and packing. He arrived at a relation to determine the transition from single-phase pore flow to two-phase pore flow. His equation shows how the Reynolds number of the liquid and the diameters of the particle and the column can be used to obtain the minimum value of gas Reynolds number ($Re_G^*$). $Re_G^*$ is the minimum value of gas Reynolds number above which the flow will be in the two-phase pore-flow regime. The pressure drop correlation derived by Turpin and Hungtinton [23] has been found suitable for calculation of pressure drop in the bubble and pulse flow regimes. For small size packing the PERC [20] data or the data of Sato [22] for the pressure drop are recommended [5].

There is has been no previous work done on pressure drops in co-current two-phase upward or downward flow in fibre beds.
2:3 Liquid Holdup

The liquid holdup can be defined as the volume of liquid contained per reactor volume. However, it is usually defined on the basis of the void volume of the reactor and not the total reactor volume. The liquid holdup is one of the factors used to evaluate the performance of a reactor. The liquid holdup can be divided into two parts: dynamic holdup and the static holdup. The dynamic liquid holdup depends on the gas and liquid loads, the properties of the fluids and the packing material. The static holdup depends on the nature of the packing and the fluid properties. Quite a substantial number of studies on liquid holdup has been carried out on different packings.

Voyer and Miller [26] studied the liquid holdup in a column with screen packings. They observed that the liquid holdup decreases with the increase in gas velocity. Unfortunately they did not develop any correlation to represent their data.

The liquid holdup in an air-water system and 51, 102 and 153 mm diameter columns packed with tabular alumina particles of 7.6 and 8.2 mm in diameter was measured by Turpin and Huntington [23]. They correlated their total liquid holdup to the ratio of liquid to gas mass flux by an empirical relation. Their total liquid holdup was based on the column’s void volume. The ratio of the liquid to gas
mass flowrate ranged from 1 to 6. Their correlation model has been found not to be valid for \( \frac{L}{G} \) greater than 5.69 since it gives a total liquid holdup greater than unity.

Sato et al [22] correlated their data to the Lockhart-Martinelli parameter for the pressure drop. They accounted for the porosity of the packing in their correlation.

Hutton and Leung [27] proposed a theoretical model of the liquid holdup for cocurrent upflow through a packed column. They also correlated their liquid holdup to the pressure drop. They assumed the liquid holdup to be a function of the two-phase pressure drop and the superficial liquid velocity. Their theoretical prediction of the total liquid holdup by their model showed only a fair agreement when compared to the experimental data of Turpin and Huntington [23]. Hutton and Leung [27] also confirmed the fact that co-current upflow operation gives higher liquid holdup under the same gas and liquid loads. They arrived at this result using a theoretical model. This fact had earlier on been demonstrated experimentally by Turpin and Huntington [23].

The Pittsburg Energy Research Centre [20] studied the liquid holdup in a packed column with co-current gas-liquid upflow. They performed their measurements on air-water flow through a 10.2 cm internal diameter acrylic column. Three different packing sizes were examined. They are 19 mm x 19 mm, 6.35 mm x 6.35 mm and 3.2 mm x 3.2 mm cylinders. Most of their experiments were obtained in the bubble flow and pulsed flow regimes. They found the average
liquid holdup to be strongly dependent on the liquid velocity.

Stiegel and Shah [28] studied the characteristics of the liquid holdup using an air-water system. The dimensions of their column were 168 mm long by 20.6 mm wide and 1220 mm high. Polyethylene packing with 3.18 mm diameter was used. The air load ranged from 0 to 0.203 kg/m²s. Their results indicated that under the same flow conditions, the liquid holdups in a rectangular column were in general higher than those obtained in a cylindrical column of equivalent section.

Lamine et al [15] studied the liquid holdup using small particles of diameter one, two and three millimeters. The liquid load ranged from 1-11 kg/m²s and the gas load ranged from 0-0.3 kg/m²s. Achwal and Stepanek (29) also measured the liquid holdup. The diameter of the particles they used was about 6 mm. Both the correlations of Lamine et. al. and Achwal and Stepanek are shown in Table 1. The results of Achwal and Stepanek agree fairly well with the results of Lamine et. al. The experimental results of Lamine et al deviate by ±20 % from the empirical correlation of Achwal and Stepanek.

No previous work has been done on liquid holdup measurement in graphite fibre beds.
2:4 Gas-Liquid mass transfer for co-current upward flow

Quite a number of interesting studies have been carried out to determine the gas-liquid mass transfer capacity for upward co-current gas-liquid flow.

Mashelkar and Sharma [30] used columns of diameters 66, and 200 mm with a variety of packing to examine both the gas side and liquid side gas-liquid mass transfer coefficient. They measured the absorption of carbon dioxide in various electrolytes and non electrolytes. Their results showed that the volumetric gas-liquid mass transfer coefficient increased with the superficial gas velocity.

Using an 80 mm diameter packed column with three different types of packings: glass spheres, Berl saddles, and ceramic rings, Specchia et. al. [31] measured the liquid-phase mass transfer coefficient. The superficial gas and liquid velocities they used ranged from 0.14 to 2.21 m/s and 0.0025 to 0.043 m/s respectively. They correlated their liquid-side mass transfer coefficient to the energy parameter. This is shown in Table 1. Prior knowledge of the gas-liquid pressure drop is required before their correlation can be used. Higher $k_L$ values were obtained for upward compared to downflow for lower liquid velocities. Specchia et al [31] found that the $k_La_L$ values for upward flows were on the average 100% greater than the downflow in pulsed and spray flow regime. This was
because of the gravitational force which gave higher liquid holdup and pressure drop [5].

Snider and Perona [32] used a column packed with 2.9 mm alumina spheres coated with palladium catalyst to study the mass transfer coefficient for the hydrogenation of alpha-methyl styrene. They found that the mass transfer coefficient increased with the gas rate up to a gas-phase Reynolds number of about 50 and as the half power of the liquid load. The pulsating nature of the flow for higher gas rates accounted for the strong decrease in the mass transfer coefficient for higher gas rates.

Goto et. al. [33] measured the liquid-gas mass transfer coefficient for the desorption of oxygen from water into nitrogen. They used a 25.8 mm internal diameter glass tube packed with CuO-ZnO particles with diameter of 0.541 or 2.91 mm. The condition at which the data were obtained are 1 atm pressure, and 25°C temperature. The gas load ranged from 0.0027 through 0.0082 and the liquid from 0.478 to 5.738 kg/m²s respectively. They obtained their data in the bubble-flow regime as defined by Specchia et. al. [31]. Their results showed that the liquid-side mass transfer coefficient increased with both gas and liquid load. Compared to the co-current downflow data their results show that the upflow gave larger values of mass transfer coefficient only at high gas and liquid loads. They used the desorption of naphthalene from water to determine the gas side mass transfer. From their results they deduced that the gas-side mass transfer was a strong function...
of the gas load and only depended mildly on the liquid load.

Ohsima [34] used a column packed with 1-, 2.8-, and 4.3 mm glass beads to measure the liquid-phase mass transfer coefficient for the oxidation of sodium sulfite. The superficial liquid velocity ranged from 10 to 60 mm/s and that of the gas from 5 to 60 mm/s. They correlated the $k_{LaL}$ to the dynamic gas holdup.

Voger and Miller [26] studied the liquid-phase mass transfer coefficient by measuring the desorption of CO$_2$ from water. They performed their experiments in a column of internal diameter of 140 mm and heights ranging from 2.04 to 2.38 m. The column was packed with screens. The gas load ranged from 0.18 to 1.62 kg/m$^2$s and the liquid from 5.5 to 30 kg/m$^2$s. From their investigation they found that the mass transfer coefficient was independent of gas velocity but increased with liquid velocity. Also, that at short column heights the average mass transfer coefficient and capacity decreased with an increase in column height.

Alexander and Shah [35] measured the desorption of oxygen from water in a 60-mm diameter column. They used different shapes of packing which are normally used in gas-liquid-solid catalyst reactors. The gas load ranged from 2.14 to 44.3 kg/m$^2$s. The liquid load ranged from 0.9 to 6 kg/m$^2$s. These flow rates are the rates typically used in pilot scale catalytic hydrogenation processes. They correlated $k_{LaL}$ with the gas and liquid loads. They also correlated their data with the energy correlation of Reiss [36]. Prior knowledge of the two-phase pressure drop is required
before their equation can be used.

Charpentier [37] suggested that in the absence of any reliable data or correlation a value of 0.15 s$^{-1}$ could be used as a first approximation for the $k_La_L$ for pulse and spray flow regimes.

It is generally recommended that the correlation by Specchia et. al. [31] be used to determine the $k_La_L$ [5].

2:5 Liquid-solid mass transfer

Snider and Perona [32] used 3-mm alumina spheres coated with palladium catalyst to measure the volumetric liquid-solid mass transfer coefficient for the hydrogenation of alpha-methyl styrene. They performed their experiments in the bubble flow regime.

Mochizuku and Matsui [38] used the diffusion-current method to determine the liquid-solid mass transfer coefficient. The active platinium particle was 5 mm long and 5 mm in diameter. This particle was placed in a dummy particle-packed bed. The diameter of the column was 87 mm. The measurement of the diffusion current from the platinium anode particle was determined by streaming an equimolar solution of potassium ferro and ferricyanides through the bed. They kept the Schmidt number constant at 1170.

The validity of the equations above is questionable under the conditions of an actual reactor, where all particles are active and
under the conditions where the Schmidt number is not equal to 1170.

Goto et. al. [33] measured the liquid-solid mass transfer coefficient in the bubble flow regime for the dissolution rates of naphthalene in water. They used a glass column packed with particles of sizes 0.54 to 2.4 mm of naphthalene and CuO-ZnO. The column had internal diameter of 25.8 mm. The liquid load ranged from 0.5 to 5.74 kg/m$^2$s while the gas load ranged from 1.8 to 7.2 kg/m$^2$s. Their correlation can be found in Table 1. The temperature and pressure were 25°C and one atmosphere respectively. They found the mass transfer coefficient to be higher for upflow when compared to the downflow.

Takahashi and Alkire [4] used the electrochemical limiting current method to measure both the liquid to solid mass transfer and the overall gas to solid mass transfer capacity for a two phase co-current upflow in a packed bed electrode. In both cases they used 1.2 mm glassy carbon particles for their packing. They used ferricyanide reduction to measure the liquid-to-solid mass transfer capacity. Pure nitrogen was used as the inert gas. Oxygen reduction was used to obtain the overall gas to solid mass transfer capacity. The Reynolds number for the gas and liquid they chose for the overall gas to solid mass transfer capacity ranged from 10 to 300 and 11.7 to 59.3 respectively. They observed that the liquid to solid mass transfer rates depended mainly on the liquid flow rate. Surprisingly, their measurements showed that the mass transfer rates increased with decreasing liquid flow rate. The overall mass
transfer capacity they obtained ranged from 0.009 to 0.032 s\(^{-1}\). Unfortunately, they did not correlate their results for the gas to solid mass transfer though they presented it graphically.

Kinoshita and Leach [39] studied the mass transfer coefficient for a single phase flow using the limiting current measurement for the cathodic reduction of bromine. They used carbon felt electrode (fibre diameter about 25 micrometers) for their packing. The Reynolds number for the electrolyte ranged from 0.01 to 0.4. Their correlation related the Reynolds number of the liquid to the Sherwoods number. They used two electrodes of thickness 0.25 cm and 0.175 cm. The values of their mass transfer capacity for the electrode of thickness 0.25 cm ranged from 0.011 to 0.157 s\(^{-1}\) and for electrode of thickness 0.175 cm the Ka value ranged from 0.018 to 0.170 s\(^{-1}\). The specific surface area for the electrode thickness of 0.175 and 0.25 cm were 110 and 88 cm\(^{-1}\) respectively.

No previous work has been reported on mass transfer for two-phase upward or downward flow in graphite fibre beds.

### 2.4 Porous electrodes

Porous electrodes or three dimensional electrodes have been considered and studied because they yield high mass transfer performance. They help carry reactions to a high degree of completion. This is significant for slow electrochemical reactions.
Porous electrodes are preferred when dealing with reactive gases which have low solubility. These reactive gases may be dissolved and forced through these electrodes and kept at the surface. Compared to classical plate electrodes they provide a larger interfacial area per unit volume and higher space-time yield [40].

Unfortunately, excessive ohmic voltage losses occur within the porous electrode. The potential difference of the electrode and electrolyte varies through the electrode. This leads to non-uniform reaction rate distribution [40] thereby restricting the use of the whole internal surface area of the electrode. To be able to use the whole internal surface area of the electrode there is the need to minimize the ohmic influences. This can be achieved by maximizing the equivalent conductivity of the electrode.

The ohmic potential drop can be reduced by compressing the electrode to reduce the distance through which the current must flow and also increasing the electrode conductivity (i.e. increasing electronic conductivity of electrode).

The models used for the three-dimensional electrodes account for the essential features of the electrodes and not the very fine geometric details of the pores. Usually, the parameters used in the model are those which can easily be obtained experimentally without much difficulty for example, flowrates, porosity, pressure drop, liquid holdup, etc.

Knowledge of how the electrode processes occur and why they occur so non-uniformly through the depth of the electrode will be
a big step in designing porous electrodes with high performance.

The general requirement of three dimension electrodes are high effective conductivity, high porosity, high internal surface area and adequate mechanical strength.

2:6 Electrochemical reaction method for determination of mass transfer rates

The electrochemical measurement of the reduction of ferricyanide ions on a cathodic packed bed is commonly used to study the mass transfer characteristics of porous electrodes. The mass transfer coefficient is calculated from the limiting diffusion current. Cathodic reduction of bromine and oxygen have also been employed.

Delaunay et. al. [6] found that the electrochemical technique, which is frequently used for a single phase flow is still applicable for two-phase flow with a large range of gas and liquid loads.
Table 1: Some experimental data cited in the literature for pressure drop, liquid holdup and mass transfer in packed beds

[VARIABLES DEFINED IN NOMENCLATURE 2]

<table>
<thead>
<tr>
<th>Pressure drop correlation</th>
</tr>
</thead>
<tbody>
<tr>
<td>Investigators: 1. Turpin and Huntington</td>
</tr>
<tr>
<td>System: Air-water Column size 51, 102 and 153 mm</td>
</tr>
<tr>
<td>Particle size 7.6 to 8.2 mm</td>
</tr>
<tr>
<td>Gas load 0.02 to 38.3 kg/m²s Liquid load 6.52 to 54.33 kg/m²s</td>
</tr>
<tr>
<td>Correlation: ( \ln f_{LG} = 8.0 - 1.12(\ln z_1) - 0.0769 (\ln z_1)^2 + 0.0152 (\ln z_1)^3 ) 0.3 ≤ z ≤ 500</td>
</tr>
<tr>
<td>where ( z_1 = (d_p G_g / \mu_g)^{1.167} / (d_p G_L / \mu_L)^{0.767} )</td>
</tr>
<tr>
<td>( f_{LG} = (\Delta P/\Delta z)_G ) ( d_e ) ( g_c / (2U_g^2 \rho_g) )</td>
</tr>
<tr>
<td>( d_e = \varepsilon_b V_p / [\rho_p (1-\varepsilon_b)] )</td>
</tr>
</tbody>
</table>

Investigator: 2. Saada

| System: Air-water Column diameter 45 mm |
| Particle size: 0.514, 0.914 and 2.064 mm |
| Correlation: Single-phase pore flow |
| \( 1/gp_L (\Delta P/\Delta z)_G = 0.024 \) \( Re_G^{0.39} Re_L^{0.60} (d_p/d_c)^{-1.1} \) |
| Correlation: Two-phase pore flow |
| \( 1/gp_L (\Delta P/\Delta z)_G = 0.027 \) \( Re_G^{0.51} Re_L^{0.35} (d_p/d_c)^{-1.15} \) |
**Chapter 2. Literature Review**

\[
\text{Re}_G^* = 0.44 \text{Re}_L^2 (d_p /d_c)^{0.38}
\]

**Investigator:** Sato et. al.

**System:** Air-water  
Column: 65.8 and 122 mm internal diameter  
Packing: 6 different glass spheres of diameters ranging from 2.59 - 24.3 mm.

\[
\Delta P_{LG} = \Delta P_L [1.3 + 1.85 (\Delta P_G /\Delta P_L)^{0.85}]^2
\]

**Liquid holdup**

**Investigators:** Lamine et. al.

**System:** nitrogen-water  
Gas load: 0 - 0.3 kg/m²s  
Liquid load 1 - 11 kg/m²s  
Particle size: 1, 2, 3, 4 and 5.9 mm  
Correlation: \( h_L = 1 - (1.3 + 0.3 L^{0.13} G^{-0.563})^{-1} \)

**Investigators:** Achwal and Stepanek

**System:** air-water  
Gas load 0 - 0.65 kg/m²s  
Liquid load 4 - 48 kg/m²s  
Porosity 0.4  
Column diameter 50 mm  
Packing diameter 6 mm  
Correlation: \( h_L = 1 - (1 + 0.59 L^{0.13} G^{-0.563})^{-1} \)

**Gas-Liquid mass transfer**
Chapter 2. Literature Review

Investigators: 1. Specchia et. al.

<table>
<thead>
<tr>
<th>System for mass transfer measurement:--</th>
</tr>
</thead>
<tbody>
<tr>
<td>Desorption of oxygen with air from a 2N solution of caustic soda previous saturated with oxygen</td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>System for interfacial area:--</th>
</tr>
</thead>
<tbody>
<tr>
<td>Chemical absorption of CO₂ in NaOH solution</td>
</tr>
<tr>
<td>superficial gas velocity 0.14 to 2.21 m/s</td>
</tr>
<tr>
<td>superficial liquid velocity 0.0025 to 0.043 m/s</td>
</tr>
<tr>
<td>diameter of packed column 80 mm</td>
</tr>
<tr>
<td>Packings: glass spheres, berl saddles, and ceramic rings</td>
</tr>
<tr>
<td>Packing sizes: glass spheres 6mm berl saddles 6mm ceramic rings 6mm</td>
</tr>
</tbody>
</table>

\[
10^3 \frac{k_L \varepsilon}{U_L} = 7.96 \left( \frac{\Delta \rho}{\Delta z} \right)_{LG} g_c \frac{\varepsilon}{(a_s \rho L U_L)}^{0.275} - 9.41 \\
\frac{a_L}{a_s} = 0.29 \left( \frac{\Delta \rho}{\Delta z} \right)_{LG} \varepsilon/a_s \right)^{1.17} + 0.61
\]

Investigators: 2. Takahashi K.M. and Alkire R.C.

<table>
<thead>
<tr>
<th>System: Desorption of oxygen from an air-saturated 0.70 M Na₂SO₄ solution into nitrogen bubbles</th>
</tr>
</thead>
<tbody>
<tr>
<td>Gas velocity 0.02 to 0.16 m/s</td>
</tr>
<tr>
<td>Liquid velocity 0.0051 to 0.056 m/s</td>
</tr>
<tr>
<td>Packing: glassy carbon particles 1.2 mm</td>
</tr>
</tbody>
</table>

| Correlation: ln \( k_L a_L \) = 73.5 \( U_G \) + 32.4 \( U_L \) - 3.49 |

Liquid to solid mass transfer capacity
<table>
<thead>
<tr>
<th>Investigators: l. Goto et. al.</th>
</tr>
</thead>
<tbody>
<tr>
<td>System: Air-water</td>
</tr>
<tr>
<td>Column diameter: 25.8 mm</td>
</tr>
<tr>
<td>Gas load 0-0.009 kg/m²s</td>
</tr>
<tr>
<td>Liquid load: 0.118 - 5.2 kg/m²s</td>
</tr>
<tr>
<td>Porosity 0.44</td>
</tr>
<tr>
<td>Particle size 0.54, 1.08 and 2.41 mm</td>
</tr>
<tr>
<td>Correlation: ( j_D = 1.31 , \text{Re}_L^{-0.436} )</td>
</tr>
</tbody>
</table>
3 Experimental Apparatus and Procedures

The experimental apparatus and procedure chosen to measure the pressure drop, liquid holdup and the overall mass transfer capacity for the graphite fibre trickle-bed electrode were simple and reliable. These procedures have been used by other authors for the studies of the parameters mentioned above in particulate beds. Other methods available for measuring the pressure drop, liquid holdup and overall mass transfer capacity are mentioned in this chapter.

3:1 Pressure drop measurement

Static pressure gradients can be measured by means of
- U-tube mercury manometer
- differential pressure gauge.

The U-tube mercury manometer was used for the determination of the pressure gradients in this study.

Apparatus

Cell: - A graphite fibre bed (UCAR 1/4" WDF Graphite Felt, Union Carbide Corp.) of dimension 356 mm long by 38 mm wide by 6.4 mm
(before compression) thick with graphite fibres of about 22 micrometer diameter packed to about 90% porosity after compression was used. This graphite felt was placed inside a neoprene gasket of dimensions 460 mm long x 64 mm wide x 3.2 mm thick to hold it in place. Details of the cell are shown in Figure 1. The gasket which contained the graphite felt was placed between two clear plexiglass sheets of size 505 mm long x 127 mm wide x 25 mm thick, bolted and compressed with a torque of 120 lb in. Sixteen stainless steel bolts (1/4") were used in all. Both the gasket and graphite bed were compressed to a thickness of 3 mm. To obtain a uniform bed thickness and prevent bulging of the plexiglass, stainless steel metal spacers of thickness 3 mm were placed on each side and the top and bottom of the gasket before compression.

The apparatus for measuring the pressure drop consisted of a feed tank, pump, rotameter, cell, U-tube mercury manometer, gas cylinders, associated piping, etc. The experimental setup is shown in Figure 2.

**Procedure**

The liquid used was de-ionized water and the gas was pure oxygen. The flow of the water and the gas were obtained respectively by a positive displacement pump and an oxygen cylinder under pressure. The gas flow rate was adjusted with the valve above the rotameter and the liquid flow with the pump. The oxygen and water were mixed at a simple 'tee' before entering the cell. The arms of the U-tube
mercury manometer were connected to the manometer taps on the side of the cell opposite to the side where the water and oxygen mixture enters the cell. Since the maximum pressure drop was 40 \% of the inlet pressure a linear pressure gradient was assumed in determining the static pressure gradient for the different sets of gas-liquid flowrates chosen. For each of the different liquid and gas flow rates the static pressure drop within the cell was detected by the change in the mercury levels in the U-tube manometer. The difference in the level of mercury in the manometer was equal to the pressure drop inside the cell for that flow condition. Two readings were taken for each flow condition. The average value of the two readings was taken as the pressure drop for that condition. The gas loads ranged from 0 to 0.43 kg/m$^2$s and the liquid from 1.46 - 7.30 kg/m$^2$s. This corresponds to gas and liquid flows of 0 - 739 ml/min at S.T.P and 10 - 50 ml/min respectively. The temperature ranged from 20°C to 25°C. The outlet pressure was one atmosphere absolute.
Chapter 3. Experimental Apparatus and Procedures

Figure 1 Detailed sketch of the cell
Figure 2 Apparatus used for the pressure drop measurement
Chapter 3. Experimental Apparatus and Procedures

3:2 Total liquid holdup measurement

The measurement of liquid holdup may be carried out by different methods:

(1) Weighing method
(2) Tracer response method
(3) Quick closing valve method

There is always the possibility of obtaining different results depending upon the method adopted for measurement. The method adopted here for the measurement of the overall liquid holdup was the quick closing valve method.

Apparatus

The equipment setup for this experiment is shown in Figure 3. It consisted of feedtanks, pump, rotameters, cell and oxygen cylinder. The cell used for this study was exactly the same as that used for the pressure drop study as shown in Figure 1.

Procedure

The gas and liquid used for this experiment were pure oxygen and a molar solution of sodium hydroxide in water (the density of water at 20°C is 998.2 kg/m³ and its viscosity is 1.04 kg/ms, the density
and viscosity of 1M NaOH hydroxide are 1042.8 kg/m³ and 1.11 kg/ms respectively, the surface tension of water is 73 dynes/cm and that of a molar solution of sodium hydroxide is 76 dynes thus the difference between the surface tensions of these two liquid is small such the switching from water to aqueous sodium hydroxide will not likely affect the fluid dynamics) respectively. The aqueous solution of sodium hydroxide was pumped upward through the cell along with the oxygen gas. The oxygen and sodium hydroxide solution were mixed at a 'tee' before entering the cell. The gas-liquid mixture was made to flow through the cell for about ten minutes to obtain a steady flow. After, which the inlet and outlet 3-way valves were closed simultaneously. Water was used to wash out all the sodium hydroxide captured in the cell column. Ten millilitres of the bulk washwater was taken and titrated with one molar solution of hydrochloric acid to determine the concentration of the washwater and thus the volume of sodium hydroxide solution trapped in the bed, channels, and inlet and outlet valves. To account for the liquid trapped both in the inlet and outlet valves and channels, the graphite felt was removed and the space it occupied was covered with solid neoprene which was 356 mm long x 33 mm wide x 3.0 mm thick after compression. This allowed a width of 5 mm for the fluid to flow through when the procedure above was repeated. The volume occupied by the inlet and outlet valves and channels was used to determine the volume of liquid that was held by the graphite electrode bed alone. Different sets of gas-liquid
flows were metered through the cell and their liquid holdups determined. For each flow condition two readings were taken. The gas and liquid loads ranged from 0 to 0.35 kg/m²s and 1.53 to 7.62 kg/m²s corresponding to 0 to 605 ml/min S.T.P. and 10 to 50 ml/min respectively. The temperature ranged from 20°C to 25°C. The outlet pressure was one atmosphere absolute.

Figure 3 Apparatus used for the total liquid holdup measurement
3.3 Measurement of mass transfer capacity

Electrochemical reaction method

The electrochemical reaction method was used for the overall mass transfer studies. The thickness of the electrode bed chosen for the study was 3 mm. It was assumed that the electrochemical reaction through the electrode volume was solely controlled by mass transfer and the total volume of the bed was electroactive (calculation was carried to support this -Appendix C (7)).

Apparatus

Electrolytic cell

The electrolytic cell consisted of a cathode bed of graphite fibre felt (UCAR 1/4" WDF). Two layers of stainless steel mesh screens (8-mesh) were used for the anode. The thickness of the anolyte chamber was about 3 mm. A cation selective membrane (Nafion 214) separated the anode from the cathode. The dimension of the cation membrane was 454 mm long by 68 mm wide. The dimension of each of the stainless steel mesh screens was 89 mm long by 38 mm wide by 1.5 mm thick. The graphite cathode bed dimension was 89 mm long by 38 mm wide by 3 mm thick with graphite fibres about 22 micrometer diameter packed to about 90% porosity.

The experimental setup for measuring the overall mass transfer rate is shown in Figure 6. It consisted of feedtanks, pumps
rotameters, pressure gauges, thermocouples, direct current power supply source, ammeter, electrochemical cell, heat exchanger and gas-liquid separator.

Procedure

A one molar solution of sodium hydroxide containing 0.02% DTPA and the pure oxygen gas were metered into the cathode compartment of the electrolyte cell with the aid of the rotameter and pump. The sodium hydroxide and the oxygen gas were mixed together at a 'tee' before entering the reactor as shown in Figure 6. The inlet and outlet pressure and temperature were measured by pressure gauges and thermometers respectively.

A molar solution of aqueous sodium hydroxide was recycled through the anode compartment. The temperature of the catholyte product was kept in the range of 17 to 24°C by the introduction of a cooler in the anolyte loop.

Various currents were applied to the top of the 3 mm thick stainless steel feeder electrodes. The ammeter measured the current within the closed system. The alkaline hydrogen peroxide produced in the cathode compartment were sampled and the concentration of the sample determined using the potassium permaganate titration method [41]. Two samples were taken and analysed for each condition of gas and liquid flowrate chosen.

Graphs of concentration of alkaline hydrogen peroxide versus
applied current were plotted to determine the maximum concentration. The current at the maximum peroxide concentration was assumed to correspond to the maximum rate of oxygen reduction. This current (called the applied current in Appendix C-6) was used to determine the limiting current and thus the mass transfer capacity. Figure 7 shows a typical plot of concentration of alkaline hydrogen peroxide versus current.

The concentration of the hydrogen generated for a typical gas and liquid load was determined using gas chromatography (Appendix C).

Calculation of the mass transfer capacity from the experimental data is detailed in Appendix C - sample calculations 4, 5, 6 and 7.
Figure 4. Detailed sketch of the graphite fibre trickle-bed reactor.
Figure 5 Detailed sketch of the graphite fibre trickle-bed electrode
Figure 6 Apparatus used for the overall mass transfer capacity measurement
Chapter 3. Experimental Apparatus and Procedures

Fig 7: Peroxide concentration vs current

- Gas load: 0.351 kg/m²s
- Liquid load: 3.049 kg/m²s
CHAPTER 4

4:1 Results and Discussion

The aim of this project was to determine the pressure drop, liquid holdup and the overall mass transfer capacity of a graphite fibre trickle-bed electrode with co-current upward flow. Also, to develop satisfactory empirical correlations based on the experimental results obtained for the above-mentioned parameters.

This chapter reports the experimental results obtained, discusses results and their significance, and how well the empirical correlations fit the experimental results.

4:1 Pressure drop

The results of the pressure drop measurement for different sets of gas-liquid loads for the graphite fibre trickle-bed electrode are summarised in Table 3 (Appendix B). The variation of the static pressure gradients with the gas loads at constant liquid load is presented in Figure 8. Figure 9 shows the variation of pressure gradient with liquid load at constant gas load.

The general trend shows that the static pressure gradients increase with both the gas and liquid loads as shown in Figures 8 and 9. There happens to be a point of inflection at gas loads of 0.3 kg/m²s for the various liquid loads. Increasing the liquid load
caused a big change in the pressure gradient for the same gas load.

The pressure gradient obtained for both gas and liquid loads were lower than that given by Oloman [3] and Delaunay et. al. [6]. Oloman used downward flow in a fixed bed of graphite particles 0.1 to 1 mm. Delaunay et. al. used an upward flow. They used packings of diameter 4 mm. The graphite fibre bed used for the present study had a porosity of about 0.90 which is greater than that of the particulate electrodes used by the authors mentioned above. The porosity of the electrodes they used was 0.4. From the Ergun equation the pressure gradient decreases with the increase in the porosity. The results obtained confirmed this fact. The lower pressure gradients obtained was mainly influenced by the high porosity of the graphite fibre. It is possible that even lower pressure gradients could have been obtained if the flow system had been downward since in general downflow yields lower pressure gradients than upward flow [15]. Increasing the pumping power leads to an increase cost. Thus, by selecting a graphite fibre trickle-bed electrode instead of particulate electrode some savings can be made on the pumping cost for equivalent mass transfer capacity.

In developing an equation to fit the experimental results obtained it was assumed that the pressure gradient was a function of the liquid load and the gas load. Only the gas and liquid load parameters were used for the correlation. A number of different correlations were tried and the one that gave the highest rank correlation was chosen. This developed empirical correlation is of
Chapter 4. Results and Discussions

the same form as that of Sato's [22] two phase pressure gradient correlation. The pressure gradient was correlated as a function of liquid and gas load instead of as a function of the single phase pressure drop. The developed correlation from this study is shown below.

\[ \Delta P = L[0.36 + 1.182 (G/L)^{0.618}]^2 \]

where \( \Delta P \) - pressure gradient in bar/m

\( G \) - superficial gas load in kg/m\(^2\)s

\( L \) - superficial liquid load in kg/m\(^2\)s

The standard deviation is 0.10 bar/m and the rank correlation coefficient for the above non-linear regression is 99.4 %. This shows a good fit between the predicted results and the experimental results. The relationship between the experimental results and the correlated results are graphically shown in Figure 10.

The empirical correlation by Turpin and Huntington [23] was found to be unsuitable for the present experimental results obtained. Their correlation was suitable for values of \( \text{Re}_g^{1.167}/\text{Re}_l^{0.767} \) between 0.3 and 500. The \( \text{Re}_g^{1.167}/\text{Re}_l^{0.767} \) values obtained for this study were far below the required range (i.e. below 0.3). Their gas and liquid loads ranged from 0.02 kg/m\(^2\)s to 38.3 kg/m\(^2\)s and 6.52 to 54.33 kg/m\(^2\)s respectively. For the present study the gas and liquid loads ranged from 0 to 0.43 kg/m\(^2\)s and 1.46 to 7.30 kg/m\(^2\)s respectively. The diameters of the alumina particles used by Turpin and Huntington [23] were far larger than
the diameter of the graphite fibres. The graphite fibre has a diameter of 22 micrometer while that of the alumina particles studied by Turpin and Huntington [23] were 7.6 and 8.2 mm.

The correlation by Sato [22] which is generally recommended for small packings was also found to give a very poor fit of the experimental results obtained here.
Chapter 4. Results and Discussions

Figure 8: Pressure gradient versus gas load

- Liquid 1.46 kg/m²s
- Liquid 2.92 kg/m²s
- Liquid 4.38 kg/m²s
- Liquid 5.84 kg/m²s
- Liquid 7.30 kg/m²s

Gas load (kg/m²s)

Pressure gradient (bar/m)

Figure 8: Pressure gradient versus gas load
Chapter 4. Results and Discussions

Figure 9: Pressure gradient versus liquid load
Figure 10: Experimental versus correlated pressure gradient
4:2 Liquid holdup

The total liquid holdup for this study was defined as the total volume of liquid trapped divided by the void volume of the bed. The results of the experiments are summarised in Table 4 (Appendix B). Figure 11 shows the liquid holdup versus gas load at constant liquid load. The relationship between the liquid holdup versus the liquid load at constant gas load is shown in Figure 12. The liquid holdup decreases with increase in gas load but increases with increase in liquid load. At constant liquid load the liquid holdup decreases sharply at low gas loads (less than 0.1 kg/m$^2$/s) then gradually at high gas loads (greater than 0.1 kg/m$^2$/s). This can be seen in Figures 11 and 12.

The highest total liquid holdup obtained for all the different liquid loads chosen was 1.0. At the total liquid holdup of 1.0 the gas load was zero.

From the Bruggeman equation the presence of gas bubbles decrease the effective conductivity of the electrolyte which adversely affects the performance of an electrochemical cell. Thus, electrodes which are able to retain a lot of the solution leaving no or little space available for the gas will generally yield higher cell performance because of higher effective electrolyte conductivity. Also the equation by Neale and Nader [42] shows a relationship between the liquid holdup and the effective conductivity.
electrolyte conductivity. From their equation it can be implied that the higher the liquid holdup that can be achieved the higher the effective electrolyte conductivity. Figure 13 shows a plot of effective electrolyte conductivity of 1 M aqueous sodium hydroxide with temperature ranging from 20 to 25°C versus liquid holdup using the results obtained for the liquid holdup in present study and the equation by Neale and Nader to obtain the effective electrolyte conductivity.

For gas loads ranging from 0 to 0.35 kg/m²s and liquid load from 1.53 to 7.62 kg/m²s the liquid holdup obtained for present study ranged from 0.44 to 1.0 The empirical relationship between the liquid holdup and the superficial gas and liquid loads obtained in this study is shown below

$$h_L = 1 - 0.907 L^{-0.362} G^{0.301}$$  \hspace{1cm} Eqn (2)

where

- $h_L$ = liquid holdup
- $L$ = superficial liquid load kg/m²s
- $G$ = superficial gas load kg/m²s

The liquid holdup was taken to be a function of only the gas and liquid loads as done by Lamine et. al. [15] and Achwal and Stepanek [29]. Many different forms were tried and the one that gave the highest rank correlation was chosen. For this chosen correlation the standard deviation is 0.02 and the correlation coefficient for the above non-linear regression is 100 %. This shows a good fit between the correlated results and the experimental results. The
relation between the experimental results and the correlated results have been graphically shown in Figure 14.

The correlations by Lamine et. al [15] and Achwal and Stepanek [29] fit equation (2) quite well as shown in Figure 15. The correlation by Lamine et. al. deviates by a maximum of ±17% while that of Achwal et. al. by ±9%. The empirical correlation obtained from present study deviates by ±3% from the experimental results obtained. The standard deviations of the values obtained from Lamine et. al. and Achwal and Stepanek from the experimental results are 0.11 and 0.06 respectively.
Figure 11: Liquid holdup versus gas load

- Liquid load 1.53 kg/m\(^2\)s
- Liquid load 3.05 kg/m\(^2\)s
- Liquid load 4.57 kg/m\(^2\)s
- Liquid load 7.62 kg/m\(^2\)s
Chapter 4. Results and Discussions

Fig 12: Liquid holdup versus liquid load

- Gas load 0.0 kg/m²s
- Gas load 0.40 kg/m²s
- Gas load 0.08 kg/m²s
- Gas load 0.20 kg/m²s
- Gas load 0.35 kg/m²s

Liquid load kg/m²s

Liquid holdup
Figure 13: Effective electrolyte conductivity of 1M aqueous sodium hydroxide (20-25 °C) vs liquid holdup
Fig 14: Experimental versus correlated liquid holdup
Fig 15: Experimental vs correlated liquid holdup by different authors
4.3 Overall mass transfer capacity

The results showing the variation of the applied current and the concentration of alkaline hydrogen peroxide have been summarised in Tables 7 to 18 (Appendix B). Figure 16 shows the relationship between the overall mass transfer capacity (Ka) versus the gas load of the gas at constant liquid load. The Ka value increases both with the increase in gas and liquid loads.

The overall mass transfer capacity (Ka) obtained was high. This may be due to the following reasons:

- The graphite fibre increases the turbulence of the fluid. It has a high surface area per unit volume and because of this it allows for high-space time yield. The high porous nature of the graphite fibre trickle-bed allows for free flow of the solution.

- The reactive gas increases the mass transfer rates by increasing the velocity of the liquid. When the electrolyte surface is well wetted by the electrolyte the gases that reach the surface of the electrode do not block the surface like inert gases but rather readily react. Thus, there is always a strong drive for the gases to move to the electrode surface for reaction hence the high mass transfer.

- The gas and liquid loads were used correlate the overall mass transfer capacity. Different forms were tried and the one that gave the highest rank correlation was chosen. Below is the empirical
correlation obtained from the experimental results.

\[ Ka = 5.9 \ L^{0.371} \ G^{0.233} \quad \text{Eqn (3)} \]

where \( Ka \) = mass transfer capacity \( s^{-1} \)
\( L \) = Liquid load \( \text{kg/m}^2\text{s} \)
\( G \) = gas load \( \text{kg/m}^2\text{s} \)

The standard deviation is 0.60 \( s^{-1} \) and the standard correlation coefficient for the above non-linear regression is 99.5\%. This shows a good fit between the experimental results and the correlated results. The relationship between the experimental results and the correlated results has been graphically shown in Figure 17.

The experimental technique used to determine the overall mass transfer capacity in this study was also used by Takahashi and Alkire. They used larger spherical packing diameters of about 1.2 mm compared to 22 micrometer which was used for this study. Also the porosity of their packing was lower (0.4) than what was used in this study (0.90). The Reynolds number for the gas and liquid they chose were much higher than what was used in this study. The Reynolds number of gas they used ranged from about 15 to 210 and that of the liquid from 11.7 to 59.3. For the gas and liquid loads that they chose the Ka values they obtained ranged from 0.009 to 0.032 \( s^{-1} \). The Ka values obtained in this study ranged from 3.4 to 9 \( s^{-1} \).

Comparatively the graphite fibre trickle-bed electrode is better
than the 1.2 mm glassy carbon particles since it yields lower pressure gradients and higher mass transfer capacities. This qualities are necessary for high performance of an electrochemical cell.

The mass transfer capacity obtained by Kinoshita and Leach for a single-phase flow ranged from 0.011 to 0.157 and 0.018 and 0.157 s\(^{-1}\) for electrode thickness of 0.175 and 0.25 cm respectively. They used carbon felt of fibre diameter 25 micrometer. Their Reynolds number of the liquid ranged from 0.01 to 0.4. The range of mass transfer capacity obtained when the Reynolds number of the liquid for the present study is fitted into their equation is 0.329 to 0.593 s\(^{-1}\). The value of two phase mass transfer capacity obtained in the present study is about ten times that of the single phase. The difference is due to reactive gas which enhances the mass transfer.
Chapter 4. Results and Discussions

Fig 16: Mass transfer capacity versus gas load

- Liquid load 3.05 kg/m²s
- Liquid load 4.57 kg/m²s
- Liquid load 7.62 kg/m²s
Chapter 4. Results and Discussions

Correlated mass transfer capacity (s\(^{-1}\))

Fig 17 Experimental vs correlated mass transfer capacity
5: Conclusion and Recommendations

5:1 Conclusion

The pressure drop, liquid holdup and overall mass transfer capacity of a graphite fibre trickle-bed electrode have been determined experimentally. These experimental data have been correlated empirically.

For the gas load ranging from 0 to 0.43 kg/m$^2$s and liquid load ranging from 1.46 to 7.30 kg/m$^2$s the pressure gradient ranged from 0.24 to 2.09 bar/m. The empirical correlation for the pressure drop for the graphite fibre of porosity 0.90 and fibre diameter 22 micrometer was

$$\Delta P = L[0.36 + 1.182 \times (G/L)^{0.618}]^2$$ (standard deviation=0.10 bar/m)

This low range of pressure gradient obtained is due mainly to the high porosity (0.90) of the graphite fibre trickle bed electrode. Lower pressure gradient is advantageous to cell performance since the cost for pumping power will be less, compared to higher pressure gradients. The empirical correlation developed could be used to estimate the pressure gradient for graphite fibre trickle-bed electrode at porosity of 0.90 and fibre diameter of 22 micrometer operating at 20-25°C near atmospheric pressure with gas and liquid loads within the ranges of this study.
Chapter 5. Conclusions and Recommendations

The total liquid holdup determined for the gas and liquid load of 0 to 0.35 kg/m²·s and 1.53 to 7.62 kg/m²·s respectively ranged from 0.44 to 1.00. Such a high liquid retaining ability allows for high reaction rates and effective electrolyte conductivity, thus high cell performance. The developed empirical correlation for the graphite fibre electrode of porosity 0.90 and fibre diameter of 22 micrometer is shown below:

\[ h_L = 1 - 0.9071L^{-0.362}G^{0.301} \]  
(standard deviation = 0.03)

This correlation could be used to estimate the total liquid holdup for graphite fibre trickle-bed electrode operating at 20 - 25°C near atmospheric pressure with the same value of porosity and fibre diameter for the flow ranges of the study.

For the gas load ranging from 0.04 to 0.36 and liquid load ranging from 3.05 to 7.62 the overall mass transfer capacity ranged from 3.4 to 9.0 s⁻¹. This range of mass transfer capacity is remarkably high, suggesting the graphite fibre trickle-bed electrode to be a more suitable porous electrode than graphite particles. The mass transfer rates are strongly influenced by the gas load at low liquid load. The developed empirical correlation obtained in this study for the overall mass transfer capacity shown below:

\[ K_a = 5.9 \ L^{0.371}G^{0.232} \]  
(standard deviation = 0.60 s⁻¹)

could be used for fibre beds of porosity of 0.90 and fibre diameter 22 micrometer operating at 17 - 26°C near atmospheric
pressure instead of those reported in the Chemical Engineering Literature which do not apply to these fibre beds.

This study has been able to provide more knowledge about the fluid dynamics of the graphite fibre trickle-bed electrode and also its mass transfer properties. The empirical correlations developed will assist in designing and evaluating the performance of such electrodes.

5.2 Recommendations

This knowledge now made available can be applied in the electrosynthesis of alkaline hydrogen peroxide. One way of obtaining alkaline hydrogen peroxide is by the cathodic reduction of oxygen using graphite fibre trickle-bed electrodes.

Further studies need to be carried out to determine the individual mass transfer coefficients as well as the interfacial areas for the gas-liquid and liquid-solid sides for a graphite fibre trickle-bed electrode. The effects of pressure on pressure drop, liquid holdup and mass transfer capacity could also be investigated.

Graphite fibre trickle-bed electrodes with different porosities could be studied over a wider range of gas and liquid loads to determine the optimum porosity that gives the best electrode performance.
Chapter 5. Conclusions and Recommendations

The mechanical strength of these graphite fibre trickle-bed electrodes needs to be considered more critically and determined since there may be some problems encountered when constructing a series of these cells for industrial use.

In order to make maximum use of the graphite fibre trickle-bed electrode the thickness of fibre bed must be selected such that the whole bed is electrochemically active. As a first estimate the developed empirical correlations from this study could be used to determine the thickness required.

The measurement of pressure drop, liquid holdup and overall mass transfer capacity could be carried out for a two phase downflow to determine how different it is from upward flow. This will help in evaluating the two different configurations so that the better of the two system could be selected.
### Nomenclature 1

<table>
<thead>
<tr>
<th>Symbol</th>
<th>Description</th>
<th>Units</th>
</tr>
</thead>
<tbody>
<tr>
<td>A</td>
<td>cross sectional area of fibre bed (width x thickness)</td>
<td>$m^2$</td>
</tr>
<tr>
<td>a</td>
<td>specific surface area of fibre bed</td>
<td>$m^2/m^3$</td>
</tr>
<tr>
<td>CE1</td>
<td>current efficiency of hydrogen gas generated</td>
<td>%</td>
</tr>
<tr>
<td>CE2</td>
<td>current efficiency of peroxide produced</td>
<td>%</td>
</tr>
<tr>
<td>Co</td>
<td>bulk concentration of reactive species, (oxygen)</td>
<td>moles/$m^3$</td>
</tr>
<tr>
<td>d</td>
<td>fibre diameter</td>
<td>m</td>
</tr>
<tr>
<td>F</td>
<td>Faradays number, 96480</td>
<td>coulombs/mole of electron</td>
</tr>
<tr>
<td>G</td>
<td>superficial gas load</td>
<td>kg/$m^2s$</td>
</tr>
<tr>
<td>H</td>
<td>concentration of hydrogen peroxide</td>
<td>moles/$m^3$</td>
</tr>
<tr>
<td>H_f</td>
<td>final concentration of peroxide</td>
<td>moles/$m^3$</td>
</tr>
<tr>
<td>H_i</td>
<td>initial concentration of peroxide</td>
<td>moles/$m^3$</td>
</tr>
<tr>
<td>h_L</td>
<td>liquid holdup</td>
<td>-</td>
</tr>
<tr>
<td>I</td>
<td>theoretical current</td>
<td>A</td>
</tr>
<tr>
<td>I_1</td>
<td>current used in producing peroxide</td>
<td>A</td>
</tr>
<tr>
<td>I_2</td>
<td>current used in destroying peroxide</td>
<td>A</td>
</tr>
<tr>
<td>I_3</td>
<td>current used in producing hydrogen gas</td>
<td>A</td>
</tr>
<tr>
<td>i</td>
<td>limiting current density</td>
<td>A/$m^2$</td>
</tr>
<tr>
<td>j</td>
<td>interfacial electrochemical reaction current density</td>
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<tr>
<td>K</td>
<td>overall mass transfer coefficient</td>
<td>m/s</td>
</tr>
<tr>
<td>K_a</td>
<td>overall mass transfer capacity</td>
<td>s$^{-1}$</td>
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<tr>
<td>L</td>
<td>superficial liquid load</td>
<td>kg/$m^2s$</td>
</tr>
<tr>
<td>M</td>
<td>mass of graphite fibre felt</td>
<td>kg</td>
</tr>
<tr>
<td>n</td>
<td>number of moles of electrons per mole peroxide</td>
<td></td>
</tr>
<tr>
<td>ΔP</td>
<td>pressure gradient</td>
<td>bar/m</td>
</tr>
</tbody>
</table>
\( Q_g \) gas flow rate at 1 atm and 21\(^\circ\)C \( \text{m}^3/\text{s} \)

\( Q_l \) liquid flow rate \( \text{m}^3/\text{s} \)

\( S \) concentration of electrolyte (1M NaOH) \( \text{moles/m}^3 \)

\( T \) thickness of graphite fibre bed \( \text{m} \)

\( t \) final thickness of graphite fibre felt (after compression) \( \text{m} \)

\( t_0 \) initial thickness of graphite fibre felt (before compression) \( \text{m} \)

\( V \) bulk volume of graphite fibre felt \( \text{m}^3 \)

\( X \) distance across the fibre bed \( \text{m} \)

\( Y \) total length of the fibre bed \( \text{m} \)

\( Z_+ \) charge on positive ion

\( \epsilon \) working porosity of graphite fibre felt (after compression)

\( \epsilon_0 \) initial porosity of graphite fibre felt (before compression)

\( \kappa \) effective conductivity of electrolyte (1M NaOH) \( \text{mho/m} \)

\( \kappa_0 \) conductivity of electrolyte \( \text{mho/m} \)

\( \Lambda \) equivalent conductance \( \text{mole cm}^2/\text{equiv} \)

\( N_+ \) number of positive ions in molecule

\( V_g \) kinematic viscosity of oxygen \( \text{m}^2/\text{s} \)

\( V_l \) kinematic viscosity of 1M NaOH \( \text{m}^2/\text{s} \)

\( \rho \) density of graphite electrode \( \text{kg/m}^3 \)

\( \rho_L \) density of liquid \( \text{kg/m}^3 \)

\( \rho_G \) density of gas \( \text{kg/m}^3 \)
<table>
<thead>
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<th>Unit</th>
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<td>$\sigma$</td>
<td>effective conductivity of graphite electrode</td>
<td>mho/m</td>
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<tr>
<td>$\sigma_o$</td>
<td>conductivity of graphite electrode</td>
<td>mho/m</td>
</tr>
<tr>
<td>$\phi$</td>
<td>electrode potential difference</td>
<td>Volt</td>
</tr>
<tr>
<td>$\phi_m$</td>
<td>electric potential in matrix</td>
<td>Volt</td>
</tr>
<tr>
<td>$\phi_s$</td>
<td>electric potential in solution</td>
<td>Volt</td>
</tr>
<tr>
<td>$\phi_o$</td>
<td>electrode potential when $x=0$</td>
<td>Volt</td>
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**Nomenclature 2** (Specifically for the correlations in Table 1)

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<thead>
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<th>Symbol</th>
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<tr>
<td>(a_L)</td>
<td>gas-liquid interfacial area</td>
<td>m&lt;sup&gt;2&lt;/sup&gt;/m&lt;sup&gt;3&lt;/sup&gt;</td>
</tr>
<tr>
<td>(a_s)</td>
<td>specific packing surface area</td>
<td>m&lt;sup&gt;2&lt;/sup&gt;/m&lt;sup&gt;3&lt;/sup&gt;</td>
</tr>
<tr>
<td>(D)</td>
<td>diffusivity</td>
<td>m&lt;sup&gt;2&lt;/sup&gt;/s</td>
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<tr>
<td>(d_c)</td>
<td>column diameter</td>
<td>m</td>
</tr>
<tr>
<td>(d_e)</td>
<td>equivalent packing diameter</td>
<td>m</td>
</tr>
<tr>
<td>(d_p)</td>
<td>diameter of packing particle</td>
<td>m</td>
</tr>
<tr>
<td>(f_{LG})</td>
<td>two-phase friction factor</td>
<td></td>
</tr>
<tr>
<td>(G)</td>
<td>superficial gas load</td>
<td>kg/m&lt;sup&gt;2&lt;/sup&gt;s</td>
</tr>
<tr>
<td>(g)</td>
<td>acceleration due to gravity</td>
<td>m/s&lt;sup&gt;2&lt;/sup&gt;</td>
</tr>
<tr>
<td>(g_c)</td>
<td>gravitational constant</td>
<td>m/s&lt;sup&gt;2&lt;/sup&gt;</td>
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<td>(h_L)</td>
<td>liquid holdup</td>
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<tr>
<td>(j_D)</td>
<td>Chilton-Colburn factor (Sc^{2/3} K_s /U_L)</td>
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</tr>
<tr>
<td>(K)</td>
<td>overall mass transfer coefficient</td>
<td>m/s</td>
</tr>
<tr>
<td>(K_L)</td>
<td>liquid-side mass transfer coefficient</td>
<td>m/s</td>
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<tr>
<td>(K_s)</td>
<td>liquid-solid mass transfer coefficient</td>
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<td>(L)</td>
<td>superficial liquid load</td>
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<tr>
<td>(Re)</td>
<td>Reynolds number (d_p G/v)</td>
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<tr>
<td>(Sc)</td>
<td>Schmidt number (\mu_L /\rho_L D)</td>
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<tr>
<td>(S_p)</td>
<td>surface area of packing</td>
<td>m&lt;sup&gt;2&lt;/sup&gt;</td>
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<tr>
<td>(U)</td>
<td>superficial velocity</td>
<td>m/s</td>
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<tr>
<td>(V_p)</td>
<td>volume of packing particle</td>
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<tr>
<td>(Z)</td>
<td>axial distance</td>
<td>m</td>
</tr>
<tr>
<td>(Z_1)</td>
<td>(Re_L^{1.167} /Re_G^{0.767})</td>
<td></td>
</tr>
<tr>
<td>(\varepsilon_b)</td>
<td>bed porosity</td>
<td></td>
</tr>
</tbody>
</table>
\( \nu \)  kinematic viscosity \( m^2/s \)

\( \rho_L \)  density of liquid \( kg/m^3 \)

\( \rho_G \)  density of gas \( kg/m^3 \)

\( \mu \)  viscosity \( kg/m.s \)

\( \frac{\Delta P}{\Delta z}_{LG} \)  two-phase pressure gradient \( kg/m^2s^2 \)

\( \Delta P \)  pressure gradient \( kg/m^2s^2 \)

subscript

G  gas phase

L  liquid phase

LG  mixed phase flow condition
Bibliography


Bibliography


in Packed Beds: Pressure Drop and Liquid Holdup Studies" Periodica Polytechnia-Chemical Engineering, 19, 317 1975


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Bibliography


[44] Perry's Chemical Engineers' Handbook Sixth Edition

Appendix A

Calibration Curve and Analytical Method

Table 2: Calibration of the gas rotameter, at R.T.P

<table>
<thead>
<tr>
<th>Float Elevation</th>
<th>Gas flow in ( \text{cm}^3/\text{min R.T.P.} )</th>
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<td>Stainless steel</td>
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<td>10</td>
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<td>20</td>
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<td>40</td>
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<td>60</td>
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<td>80</td>
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<tr>
<td>100</td>
<td>512</td>
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<tr>
<td>120</td>
<td>652</td>
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</table>

Analysis of Alkaline hydrogen peroxide

The reaction which occurs when potassium permanganate solution is added to hydrogen peroxide solution and acidified with dilute sulphuric acid is

\[
2\text{KMnO}_4 + 3\text{H}_2\text{SO}_4 + 5\text{H}_2\text{O}_2 = \text{K}_2\text{SO}_4 + 2\text{MnSO}_4 + 8\text{H}_2\text{O} + 5\text{O}_2
\]
Appendix A

Procedure

- A two ml of the sample solution was collected.
- 30 ml of distilled water was added.
- Two ml of two molar sulphuric acid was then added to acidify the solution. The solution was then shaken thoroughly.
- The solution was then titrated with standard 0.1 N potassium permaganate to the first permanent, faint pink colour.
Fig 18: Rotameter calibration for oxygen gas (at 20°C, 1Atm., absolute pressure)
Appendix B

Experimental Results
Table 3: Results of pressure drop measurements in fibre bed

Bed dimensions: 356 mm x 38 mm x 3 mm thick

Liquid: water  Gas: oxygen  Configuration: Upward flow

Porosity: 0.90  Temperature 20-25 °C.

Fibre diameter: 22 µm  Outlet pressure: 1 atm absolute

<table>
<thead>
<tr>
<th>Gas load kg/m²s</th>
<th>Liquid load kg/m²s</th>
<th>liquid load kg/m²s</th>
<th>Liquid load kg/m²s</th>
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<td>0.74</td>
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<td>0.46</td>
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<td>0.81</td>
<td>1.01</td>
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<tr>
<td>0.08</td>
<td>0.64</td>
<td>0.82</td>
<td>0.89</td>
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<td>0.13</td>
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<td>1.63</td>
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<td>1.23</td>
<td>1.48</td>
<td>1.71</td>
<td>1.95</td>
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</tbody>
</table>
Appendix B. Experimental Results

Table 4: Results of the liquid holdup in fibre bed

Bed dimension: 356 mm long x 38 mm wide x 3 mm thick

Liquid: 1 M NaOH  
Gas: oxygen  
Configuration: Upward flow

Porosity: 0.90  
Temperature: 20-25°C

Fibre diameter 22 μm  
Outlet pressure: 1 atm absolute

<table>
<thead>
<tr>
<th>Gas load kg/m²s</th>
<th>Liquid load kg/m²s</th>
<th>Liquid load kg/m²s</th>
<th>Liquid load kg/m²s</th>
<th>Liquid load kg/m²s</th>
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<tr>
<td>0.20</td>
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<tr>
<td>0.35</td>
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</table>
Appendix B. Experimental Results

Table 5:— Results of the overall mass transfer capacity measurements in fibre bed.

Bed dimensions: 89 mm long x 3.8 mm wide x 0.3 mm thick
Porosity: 0.90    Liquid: 1M NaOH    Gas: oxygen
Fibre diameter: 22 μm    Outlet pressure: 1 atm absolute

<table>
<thead>
<tr>
<th>Liquid load kg/m²s</th>
<th>Gas load kg/m²s</th>
<th>Applied Current A</th>
<th>Lim. Current A</th>
<th>Average Temp. °C</th>
<th>Sol. O₂ mol/m³</th>
<th>Mass Transf. (Ka)s⁻¹</th>
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<tbody>
<tr>
<td>3.049</td>
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<td>10</td>
<td>6.4</td>
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<td>0.954</td>
<td>3.43</td>
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<td>0.205</td>
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<td>5.69</td>
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Table 6: Results of the gas and liquid loads and mass transfer capacities.

Bed dimension: 89 mm long x 38 mm wide x 3 mm thick
Liquid: 1 M NaOH  Gas: oxygen  Fibre diameter: 22 µm
Porosity: 0.90  Outlet pressure 1 atm absolute

<table>
<thead>
<tr>
<th>Liquid load (kg/m²s)</th>
<th>Gas load (kg/m²s)</th>
<th>Mass Transfer Capacity (Ka) s⁻¹</th>
</tr>
</thead>
<tbody>
<tr>
<td>3.049</td>
<td>0.037</td>
<td>3.43</td>
</tr>
<tr>
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<td>0.081</td>
<td>4.81</td>
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<td>0.205</td>
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<td>0.037</td>
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<tr>
<td>7.623</td>
<td>0.355</td>
<td>9.03</td>
</tr>
</tbody>
</table>
Appendix B. Experimental Results

Table 7: Results of peroxide produced at various currents, run 1
Bed dimension: 89 mm long x 38 mm wide x 3 mm thick
Porosity : 0.90
Outlet pressure 1 atm abs.
Liquid load : 3.049 kg/m²s
Pressure drop 0.059 bars
Gas load : 0.037 kg/m²s
Fibre diameter 22 μm

<table>
<thead>
<tr>
<th>Average Temp (°C)</th>
<th>Current (A)</th>
<th>Concentration (M)</th>
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</thead>
<tbody>
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<tr>
<td>20.5</td>
<td>16</td>
<td>0.049</td>
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</table>
Appendix B. Experimental Results

Table 8: Results of peroxide produced at various currents, run 2.

Bed dimension: - 89 mm long x 38 mm wide x 3 mm thick

Porosity : 0.90
Gas load : 0.080 kg/m²s
Fibre diameter 22 μm

Pressure drop 0.073 bars
Liquid load 3.049 kg/m²s
Outlet pressure 1 atm abs.

<table>
<thead>
<tr>
<th>Average Temp. (°C)</th>
<th>Current (A)</th>
<th>Concentration (M)</th>
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</thead>
<tbody>
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<tr>
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</tr>
<tr>
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<td>20</td>
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</tr>
</tbody>
</table>
Appendix B. Experimental Results

Table 9: Results of peroxide produced at various currents, run 3

Bed dimension: 89 mm long x 38 mm wide x 3 mm thick
Porosity: 0.90
Outlet pressure 1 atm abs.
Fibre diameter: 22 μm
Pressure drop: 0.094 bars
Gas flow: - 0.204 kg/m²s
Liquid flow: 3.049 kg/m²s

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<th>Average Temp (°C)</th>
<th>Current (A)</th>
<th>Concentration (M)</th>
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</tbody>
</table>
Appendix B. Experimental Results

Table 10: Results of peroxide produced at various currents, run 4.

Bed dimension: 89 mm long x 3.8 mm wide x 0.3 mm thick
Fibre diameter 22 \( \mu \text{m} \)  
Porosity: 0.90
Liquid load: 3.049 kg/m\(^2\)s  
Gas load: 0.3541 kg/m\(^2\)s
Pressure drop 0.124 bars  
Outlet pressure 1 atm abs.

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<th>Average Temp. ( ^{\circ} \text{C} )</th>
<th>Current (A)</th>
<th>Concentration (M)</th>
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<td>6</td>
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</tr>
<tr>
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<td>8</td>
<td>0.034</td>
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<tr>
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<td>10</td>
<td>0.038</td>
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<tr>
<td>20.5</td>
<td>12</td>
<td>0.040</td>
</tr>
<tr>
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<td>0.045</td>
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<td>0.050</td>
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<td>26</td>
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</tbody>
</table>
Table 11: Results of peroxide produced at various currents, run 5

Bed dimension: - 89 mm long x 3.8 mm wide x 3 mm thick
Fibre diameter 22 μm
Porosity : 0.90
Pressure drop: 0.072 bars
Outlet pressure 1 atm abs.
Liquid flow : 4.574 kg/m²s
Gas flow : 0.037 kg/m²s

<table>
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<th>Average Temp. (°C)</th>
<th>Current (A)</th>
<th>Concentration (M)</th>
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<tbody>
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<td>18.5</td>
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<td>20.0</td>
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<tr>
<td>22.5</td>
<td>18</td>
<td>0.046</td>
</tr>
</tbody>
</table>
Appendix B. Experimental Results

Table 12: Results of peroxide produced at various currents, run 6

Bed dimension: - 89 mm long x 38 mm wide x 3 mm thick

Fibre diameter 22 μm
Porosity : 0.90
Pressure drop 0.079 bars
Outlet pressure 1 atm abs.
Liquid load : 4.574 kg/m²s
Gas load : 0.080 kg/m²s

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<th>Average Temp. (°C)</th>
<th>Current (A)</th>
<th>Concentration (M)</th>
</tr>
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<td>22.0</td>
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<td>24.0</td>
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<tr>
<td>23.5</td>
<td>18</td>
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</tr>
</tbody>
</table>
Table 13: Results of peroxide produced at various currents, run 7

Bed dimension: 89 mm long x 38 mm wide x 3 mm thick
Fibre diameter: 22 µm
Porosity: 0.90
Pressure drop: 0.101 bars
Outlet pressure: 1 atm abs.
Liquid load: 4.574 kg/m²s
Gas load: 0.204 kg/m²s

<table>
<thead>
<tr>
<th>Average Temp. (°C)</th>
<th>Current (A)</th>
<th>Concentration (M)</th>
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<tbody>
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<td>20.0</td>
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</table>
Table 14: Results of peroxide produced at various currents, run 8

Bed dimension: 89 mm long x 38 mm wide x 3 mm thick
Fibre diameter: 22 \( \mu \text{m} \) Porosity : 0.90
Pressure drop 0.135 bars Outlet pressure 1 atm abs.
Liquid load : 4.574 kg/m\(^2\)s Gas load : 0.354 kg/m\(^2\)s

<table>
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<th>Average Temp. (°C)</th>
<th>Current (A)</th>
<th>Concentration (M)</th>
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<td>0.046</td>
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</tbody>
</table>
Appendix B. Experimental Results

Table 15: Results of peroxide produced at various currents, run 9

Bed dimension: 89 mm long x 38 mm wide x 3 mm thick
Fibre diameter: 22 μm
Pressure drop: 0.115 bars
Outlet pressure 1 atm abs.
Liquid Flow: 7.623 kg/m²s
Gas load: 0.037 kg/m²s

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<th>Current (A)</th>
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<tr>
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</table>
Appendix B. Experimental Results

Table 16: Results of peroxide produced at various currents, run 10

Bed dimension: 89 mm long x 38 mm wide x 3 mm thick
Fibre diameter: 22 μm
Porosity: 0.90
Pressure drop: 0.132 bars
Outlet pressure: 1 atm abs.
Liquid load: 7.623 kg/m²s
Gas load: 0.080 kg/m²s

<table>
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<th>Average Temp. (°C)</th>
<th>Current (A)</th>
<th>Concentration (M)</th>
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</table>
Appendix B. Experimental Results

Table 17: Results of peroxide produced at various currents, run 11

Bed dimension: 89 mm long x 38 mm wide x 3 mm thick
Fibre diameter: 22 µm
Porosity: 0.90
Pressure drop: 0.164 bars
Outlet pressure: 1 atm abs
Liquid load: 7.623 kg/m²s
Gas load: 0.204 kg/m²s

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<th>Current (A)</th>
<th>Concentration (M)</th>
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</table>
Appendix B. Experimental Results

Table 18: Results of peroxide produced at various currents, run 12

Bed dimension: 89 mm long x 38 mm wide x 3 mm thick
Fibre diameter: 22 μm
Porosity: 0.90
Pressure drop: 0.186 bars
Outlet pressure: 1 atm abs
Liquid load: 7.623 kg/m²s
Gas load: 0.354 kg/m²s

<table>
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<tr>
<th>Average Temp. (°C)</th>
<th>Current (A)</th>
<th>Concentration (M)</th>
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</tr>
<tr>
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</tr>
</tbody>
</table>
Appendix C

Sample Calculations
Sample Calculation One

FLUID LOADS AND PRESSURE GRADIENT

Liquid (water)

Density of water at 20°C = 998.204 kg/m³
Area of fibre bed = 3.0 E-3 x 38 E-3 m²

Conversion of water flowrate from cc/min to kg/m²s

\[(\text{cc/min}) (\text{1 min/60s}) (998.204 \text{ kg/m}^2\text{s}) (1/1.14 \text{ E}-4 \text{ m}^2) (1 \text{ m}^3/1 \text{ E}-6)\]

1 cc/min = 0.1459 kg/m²s

<table>
<thead>
<tr>
<th>flow rate (cc/min)</th>
<th>liquid load (kg/m²s)</th>
</tr>
</thead>
<tbody>
<tr>
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<tr>
<td>20</td>
<td>2.92</td>
</tr>
<tr>
<td>30</td>
<td>4.38</td>
</tr>
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<td>40</td>
<td>5.84</td>
</tr>
<tr>
<td>50</td>
<td>7.30</td>
</tr>
</tbody>
</table>

Gas (oxygen)

Density of oxygen at 0°C, 1 atm = 1.4289 kg/m³ [44]
Density of oxygen at 21°C 1 atm = 1.33 kg/m³
Flow area of electrode bed = 1.14 E-4 m²

Conversion of the oxygen flowrate from cc/min at 760 mm Hg and 21°C to kg/m²s

\[1 \text{ cc/min} = (\text{cc/min}) (\text{1 min/60 s}) (1.33 \text{ kg/m}^3) (1/1.14 \text{ E}-4 \text{ m}^2) (1\text{E}-6)\]

= 1.944 E-4 kg/m²s

From the calibration curve for stainless steel float
### Scale Reading

<table>
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<th>Scale Reading</th>
<th>cc/min at 760 mmHg and 21°C</th>
</tr>
</thead>
<tbody>
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<tr>
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<tr>
<td>140</td>
<td>792</td>
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</tbody>
</table>

The pressure of the gas in the rotameter

\[= 100 \text{ lb/in}^2 \text{ gauge}\]

The pressure gauge

\[= (100)(4.448)(1/2.54 \times 2.54 \times 10^{-4})(1/1.01325 \times 10^5)\]

\[= 6.804 \text{ atm}\]

The absolute pressure

\[= 6.804 + 1 \text{ atm}\]

\[= 7.804 \text{ atm}\]

The actual flowrate

\[= (\text{absolute pressure/atmospheric pressure})^{1/2} \times \text{calibrated}\]

\[= 2.794 \times \text{calibrated value}\]
### Appendix C. Sample Calculation

<table>
<thead>
<tr>
<th>Calibrated value</th>
<th>Actual flow rate (cc/min, 760 mmHg and 21°C)</th>
<th>Gas load (kg/m²s)</th>
</tr>
</thead>
<tbody>
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<td>32</td>
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</tr>
<tr>
<td>68</td>
<td>199.0</td>
<td>0.04</td>
</tr>
<tr>
<td>148</td>
<td>413.5</td>
<td>0.08</td>
</tr>
<tr>
<td>248</td>
<td>692.9</td>
<td>0.13</td>
</tr>
<tr>
<td>376</td>
<td>1050.5</td>
<td>0.20</td>
</tr>
<tr>
<td>512</td>
<td>1430.5</td>
<td>0.28</td>
</tr>
<tr>
<td>652</td>
<td>1821.7</td>
<td>0.35</td>
</tr>
<tr>
<td>796</td>
<td>2224.0</td>
<td>0.43</td>
</tr>
</tbody>
</table>

1 in Hg (60°C) = 3.37685 E3 N/m²

1 bar = 1 E 5 N/m²

1 atmosphere = 1.01325 E5 N/m²

Conversion of pressure from inches mercury to bars

1 inch Hg = (3.37685 E3)(E-5)

1 bar = 3.37685 E-2 bar

Length of graphite packing = 0.356 m

Conversion of pressure drop to pressure gradient

1 bar = 9.4855 E-2 bar/m
Appendix C. Sample Calculation

Sample Calculation Two

LIQUID HOLDUP

Liquid (1M NaOH)

Density of 1M NaOH at 20°C = 1.0428 g/cc

Cross sectional area of electrode = 1.14 E-4 m²

Conversion of the flow rate of NaOH from cc/min to kg/m²s

\[(1/60)(1042.8)(1/1.14 \times 10^{-4})(1 \times 10^{-6})\]

\[1 \text{ cc/min} = 0.1525 \text{ kg/m²s}\]

<table>
<thead>
<tr>
<th>Flowrate (cc/min)</th>
<th>Liquid load (kg/m²s)</th>
</tr>
</thead>
<tbody>
<tr>
<td>10</td>
<td>1.53</td>
</tr>
<tr>
<td>20</td>
<td>3.05</td>
</tr>
<tr>
<td>30</td>
<td>4.57</td>
</tr>
<tr>
<td>50</td>
<td>7.62</td>
</tr>
</tbody>
</table>

Calculation of liquid holdup for liquid and gas loads of 1.53 kg/m²s and 0.04 kg/m²s respectively.

Volume of one molar aqueous sodium hydroxide trapped in graphite fibre bed, channels and inlet valves = 31 ml

Volume of one molar aqueous sodium hydroxide trapped in channels and valves = 5 ml
Therefore the volume of one molar sodium hydroxide trapped in graphite bed alone = 26 ml

The void volume of graphite electrode = 0.9x3.8x0.3x35.6 = 36.53 cm³

Therefore the total liquid holdup which is equal to the volume of liquid trapped in the bed divided by the void volume of bed for the gas and liquid load under consideration =26/36.53 =0.71

Sample Calculation Three

PEROXIDE CONCENTRATION

Calculation of the concentration of the alkaline hydrogen peroxide

Basis: 0.75 ml of 0.1N KMnO₄ used to titrate 2 ml of the sample solution (peroxide)

1 ml of 0.1 M KMnO₄ = 0.001701 g of H₂O₂  [41]

therefore 0.75 ml of 0.1M KMnO₄ = 0.75 ml x 0.001701 g of H₂O₂

since 2 ml of H₂O₂ was used for the titration analysis this implies that 2 ml of H₂O₂ will contain 0.75 x 0.001701 g

therefore

1000 ml of H₂O₂ will contain = 1000 x 0.75 x 0.001701/2

Molecular weight of H₂O₂ = 34 g

therefore 34 g of H₂O₂ = 1 mole /litre
Appendix C. Sample Calculation

1000 x 0.75 x 0.001701/2 of H₂O₂ = 1000 x 0.75 x 0.001701 / (34 x 2)
mole / litre
= 0.0188 M
approximately 0.019 E-3 moles/m³

Sample Calculation Four

THEORETICAL CURRENT

Calculation of the theoretical current required for peroxide generation

O₂ + H₂O + 2e⁻ \rightarrow OH⁻ + HO₂⁻

The theoretical current required for production of peroxide

= Q₁ n F (Hₐ - Hᵢ)

where Q₁ = catholyte flowrate m³/s
n = number of moles of electrons per mole peroxide
F = Faradays number coulombs/mole of electron
Hₐ = final concentration of the H₂O₂ moles/m³
Hᵢ = initial concentration of the H₂O₂ moles/m³
I = theoretical current A

Catholyte flowrate = 20 ml/min
= 20 ml/min x 1 min/ 60 s x 1 E-6 m³/ml
= 3.333 x 1 E-7 m³/s
Appendix C. Sample Calculation

\[ H_f = 0.0425 \times 10^3 \text{ moles/m}^3 \]
\[ H_i = 0 \text{ moles/m}^3 \]
\[ I = 3.333 \times 10^{-7} \times 2 \times 96480 \times 0.0425 \times 1 \times 10^3 \]
\[ = 2.73 \text{ A} \]
\[ = 2.7 \text{ A} \]

Sample Calculation Five

CURRENT EFFICIENCY
Calculation of hydrogen generated and its current efficiency

The electrochemical reactions that takes place in the cell are:-

**Cathode**:-
\[ O_2 + H_2O + 2e^- \Rightarrow OH^- + HO_2^- \] \[ \text{I1} \]
\[ HO_2^- + H_2O + 2e^- \Rightarrow 3OH^- \] \[ \text{I2} \]
\[ 2H_2O + 2e^- \Rightarrow H_2 + 2OH^- \] \[ \text{I3} \]

**Anode**:-
\[ O_2 + 2H_2O + 4e^- \leftrightarrow 4OH^- \]

liquid flow rate \[ = 10 \text{ ml/min} \]
\[ = 1.667 \times 10^{-4} \text{ l/s} \]

gas flowrate \[ = 20 \text{ rotameter reading} \]

From the gas rotameter calibration (Appendix A) this corresponds to
Appendix C. Sample Calculation

a reading of 68 ml/min
Pressure within the gas rotameter = 100 psi
the total pressure = 100 + 14.7
= 114.7 psi
therefore the gas flowrate = 68 (114.7/14.7)^0.5
= 3.167E-3 l/s

22.4 litres of gas is contained in one mole of a gas at STP
at temperature of 21°C the number of litres will be 24.12
The amount of hydrogen generated was 0.52 % by volume
therefore the amount of hydrogen in moles will be
= 0.0052 x 3.167 E-3/24.12
= 6.859 E-7 moles/s

One mole of hydrogen will be produced by 96480 x 2 Faradays.
The amount of current that will generate 6.859 E-7 moles
= 6.859 E-7 x 96480 x 2
=0.13 A
approximately 0.1 A

Current efficiency (CE) = \text{theoretical current required \over \text{Applied current}}

the theoretical current for hydrogen generation = I3
applied current = I1 + I2 + I3
CE 1 - the current efficiency for hydrogen generation
CE 2 - the current efficiency for peroxide generation
Appendix C. Sample Calculation

Below are some typical results calculated for the different current $I_1$, $I_2$ and $I_3$ and the current efficiencies for the hydrogen generated and the peroxide produced for a gas load of 0.04 kg/m²s and liquid load 1.53 kg/m²s.

<table>
<thead>
<tr>
<th>$I_1+I_2+I_3$</th>
<th>$I_1$ ($A$)</th>
<th>$I_2$ ($A$)</th>
<th>$I_3$ ($A$)</th>
<th>C.E. 1 (%)</th>
<th>C.E. 2 (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>2.2</td>
<td>1.9</td>
<td>0.2</td>
<td>0.1</td>
<td>4.5</td>
<td>73</td>
</tr>
<tr>
<td>4.4</td>
<td>3.3</td>
<td>0.9</td>
<td>0.2</td>
<td>4.5</td>
<td>48</td>
</tr>
<tr>
<td>8.8</td>
<td>6.1</td>
<td>1.8</td>
<td>0.9</td>
<td>10.0</td>
<td>38</td>
</tr>
<tr>
<td>11.0</td>
<td>7.3</td>
<td>2.5</td>
<td>1.2</td>
<td>11.0</td>
<td>32</td>
</tr>
<tr>
<td>13.2</td>
<td>8.4</td>
<td>3.3</td>
<td>1.5</td>
<td>11.4</td>
<td>27</td>
</tr>
</tbody>
</table>

When $I_3$ is neglected the calculated value of the mass transfer capacity will be higher than the true value by up to about 6%.
Sample Calculation Six

LIMITING CURRENT FOR OXYGEN REDUCTION

Calculation of the limiting current for oxygen reduction. At the cathode some of the current applied to the system goes to destroy the perhydroxyl produced. The total applied current was taken to be equal to $I_1 + I_2$ ($I_3$ was neglected in this calculation).

The theoretical current required to produce $H_2O_2$ will then be $I_1 - I_2$.

For an applied current of 10 A, $I_1 + I_2 = 10$ A (a)

The theoretical current required for peroxide generation from sample calculation Four $I_1 - I_2 = 2.7$A (b)

therefore $I_2 = I_1 - 2.7$A

Substituting (c) into eqn (a) gives

$$2I_1 - 2.7A = 10 \text{ A}$$

therefore $I_1 = (10A + 2.7A)/2$

$= 6.35 \text{ A}$

the limiting current for oxygen reduction

$I_1 = 6.4 \text{ A}$

Sample Calculation Seven

MASS TRANSFER CAPACITY

Calculation of mass transfer capacity ($K_a$)
Appendix C. Sample Calculation

Since the electrochemical reaction is controlled by the mass transfer rates, the mass transfer coefficient will be related to the current density by the equation

\[ i = n F K C_o \]

where
- \( i \) = limiting current density involved in reaction 1.
- \( n \) = number of electrons involved in reaction
- \( K \) = overall mass transfer coefficient
- \( C_o \) = bulk concentration of reactive species, that is the oxygen

The pressure drop throughout the packing was less than 0.19 bars. The \( C_o \) was considered to be a constant.

A mass balance on a differential element in the flow direction (y) gives (discounting loss of peroxide by secondary reaction, decomposition or transfer to the anolyte.)

\[ \frac{d[H]}{dy} = C_o K \alpha A/Q \]

where
- \( H \) = concentration of peroxide moles/m³
- \( H_f \) = final concentration of peroxide moles/m³
- \( H_i \) = initial concentration of peroxide moles/m³
- \( C_o \) = solubility of oxygen in 1M aqueous sodium hydroxide moles/m³
- \( Y \) = total length of fibre bed m
- \( K \) = overall gas-solid mass transfer coefficient m/s

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Appendix C. Sample Calculation

\[ a = \text{effective interfacial area for gas - solid mass transfer} \quad \text{m}^{-1} \]

\[ A = \text{cross sectional area of bed (width times thickness)} \quad \text{m}^2 \]

\[ Q_i = \text{liquid flow rate} \quad \text{m}^3/\text{s} \]

For a plug flow of total length \( Y \)

\[ \frac{H_f - H_i}{Y} = C_o K a A/Q_i \]

From Faraday's law

\[ I = n F Q \left[ H_f - H_i \right] \]

\[ \frac{I}{(nFQ_i)} = H_f - H_i \]

\[ \frac{I}{(nFQ_i)} = C_o K a A/Q_i \]

\[ K a = \frac{I}{(C_o n F A Y)} \]

\[ n = 2 \text{ Faradays/mole peroxide} \]

\[ F = 96480 \text{ Coulombs/mole electron} \]

\[ A = 0.003 \times 0.038 \text{ m}^2 \]

\[ Y = 0.089 \text{ m} \]

\[ C_o = 0.954 \text{ mole/m}^3 \quad [44] \]

\[ I = 6.4 \text{ A (sample calculation 6)} \]

Therefore \( K a \)

\[ = \frac{6.4}{(2 \times 96480 \times 0.003 \times 0.038 \times 0.089 \times 0.954)} \]

\[ = 3.43 \text{ s}^{-1} \]
Sample Calculation Eight

POROSITY OF GRAPHITE FELT

Determination of the porosity of the graphite fibre felt

Graphite fibre felt with dimension 250 mm long by 76 mm wide by 6.35 mm thick was used. The mass of the sheet when weighed was 8.8868 g. The density of the fibre as given by the Manufacturer is 1500 kg/m³. The initial porosity of this sheet was calculated using the formula

\[ \varepsilon_0 = \frac{(V - M/d)}{V} \]

where
- \( V \) = bulk volume of graphite fibre matrix m³
- \( M \) = mass of the graphite fibre felt kg
- \( d \) = fibre density kg/m³

The fibre density was taken to be 1500 kg/m³

\[ \varepsilon_0 = \frac{(250 \times 76 \times 6.35 \times 10^{-9} - 8.8868 \times 10^{-3}/1500)}{(250 \times 76 \times 6.35 \times 10^{-9})} \]

\[ = 0.951 \]

The working porosity was calculated using the equation below

\[ \varepsilon' = 1 - \varepsilon_0 \left( 1 - \varepsilon \right) / t \]
\[ \varepsilon' = 1 - 6.35 \times 10^{-3} \left( 1 - 0.951 \right) / 3 \times 10^{-3} \]

\[ = 0.90 \]
Sample Calculation Nine

ELECTRODE POTENTIAL

Calculation of the electrode potential difference across a graphite fibre trickle-bed electrode

The electrode potential difference is related to the conductivities of the electrolyte and electrode by the well known differential equation given below

\[
\frac{d^2}{dx^2} (\phi_m - \phi_s) = -aj \left( \frac{1}{\kappa} + \frac{1}{\sigma} \right)
\]

Let \( \phi = \phi_m - \phi_s \)

Then

\[
\frac{d^2\phi}{dx^2} = -aj \left( \frac{1}{\kappa} + \frac{1}{\sigma} \right)
\]

for pure mass transfer control

\[ j = n F K C_o \]

Therefore

\[
\frac{d^2\phi}{dx^2} = -an K C_o F \left( \frac{1}{\kappa} + \frac{1}{\sigma} \right) + \text{constant}
\]

at \( X = 0 \)

\[
\frac{d\phi}{dx} = \frac{i}{\kappa}
\]

at \( X = X \)

\[
\phi_x = -an K C_o F \left( \frac{1}{\kappa} + \frac{1}{\sigma} \right) X + \frac{i}{\kappa} \phi_o
\]
Appendix C. Sample Calculation

\[ \phi_0 - \phi_X = \alpha n F K C_0 a (1/k + 1/\sigma) X^2 /2 - i X/K \]
\[ i = a j T \]
for a graphite bed of thickness \( x = T \) where the potential at \( X=0 \) is denoted by \( \phi_0 \).

the electrode potential difference can be written as

\[ \phi_0 - \phi_X = \alpha n F K C_0 (1/k + 1/\sigma) X^2 /2 - \alpha n F K C_0 T X/K \]

The electrode conductivity for graphite fibre can be obtained using the correlation by Oloman et. al. [46]

\[ K = 10 + 2800 \left( 1 - \varepsilon/\varepsilon_o \right)^{1.55} \]

the working porosity \( \varepsilon \) of the graphite fibre trickle-bed electrode is related to its thickness by the equation below

\[ \varepsilon = 1 - t_o \left( 1 - \varepsilon_o \right) /t \]

Specifications of the graphite fibre trickle-bed electrode used for the study were

\[ t_o = 6.35 \times 10^{-3} \text{ m} \quad t = 3 \times 10^{-3} \text{ m} \quad \varepsilon = 0.90 \quad \varepsilon_o = 0.951 \]

therefore \( \sigma = 10 + 2800 \left( 1 - 0.90/0.951 \right)^{1.55} \)

\[ = 40.04 \text{ mho/m} \]

From Neale and Nader equation [42] the effective electrolyte conductivity can be determined by the relation below

\[ \kappa = 2 \kappa_o \varepsilon h_L / \left( 3 - h_L \varepsilon \right) \]
\[ \kappa_o = 1 \times 10^{-4} Z_+ N_+ S \Lambda \]

where \( Z_+ \) = charge on positive ion
\( N_+ \) = number of positive ions in molecule
\( S \) = concentration of electrolyte (1M NaOH) moles/m^3
\( \Lambda_v \) = equivalent conductance of electrolyte mole
Appendix C. Sample Calculation

\[ \Lambda_v \text{ for } 1 \text{M NaOH} = 158.4 \text{ mho equiv}^{-1} \text{ cm}^2 \]

\[ S = 1 \times 10^3 \text{ moles/m}^3 \quad Z_+ = 1 \quad N_+ = 1 \]

\[ \kappa_0 = 1 \times 10^{-4} \times 1 \times 10^3 \times 1 \times 1 \times 158.4 \]

\[ = 15.84 \text{ mho/m} \]

From this study the total liquid holdup was found to range from 0.44 to 1.0

choosing the least value of the liquid holdup ie 0.42

\[ \kappa = 2 \times 15.84 \times 0.90 \times 0.44 / (3 - 0.44 \times 0.90) \]

\[ = 4.82 \text{ mho/m} \]

when the liquid holdup is 1.0

\[ \kappa = 13.58 \text{ mho/m} \]

Electrochemical Reactions

Cathode:

Primary reaction: \( \text{O}_2 + \text{H}_2\text{O} + 2\text{e}^- \Rightarrow \text{HO}_2^- + \text{OH}^- \quad V^0 = -0.076 \text{V} \ (1) \)

Secondary reaction \( 2\text{H}_2\text{O} + 2\text{e}^- \Rightarrow \text{H}_2 + 2\text{OH}^- \quad V^0 = -0.828 \text{V} \ (2) \)

\[ V = -0.828 - (-0.076) = -0.752 \text{V} \]

From the study the mass transfer capacity ranged from 3.4 to 9.0 s\(^{-1}\).

Recall

\[ \phi_0 - \phi_X = \text{anFKC}_O(1/\kappa + 1/\sigma)X^2/2 - \text{anFKC}_O\text{TX}/\kappa \]
Appendix C. Sample Calculation

case 1
when \( \kappa = 4.82 \text{ mho/m} \) and \( \text{Ka} = 3.4 \text{s}^{-1} \)
\( C_o = 0.954 \text{ mole/m}^3 \) \( T = 3 \text{E}^{-3} \text{ m} \)
then
\[
\phi_o - \phi_x = 96480 \times 3.4 \times 0.954 \frac{X^2}{(1/4.82 + 1/40.04)} \\
- 2 \times 96480 \times 0.954 \times 3.4 \times 3 \text{E}^{-3} \times \frac{X}{4.82}
\]
\[
\phi_o - \phi_x = 72741.58 X^2 - 389.56 X
\]
when \( X = 0.003 \text{ m} \)
\[
\phi_o - \phi_x = -0.51 \text{ V}
\]
The potential difference obtained in this case is less than the reversible potential for peroxide production but greater than that required for hydrogen generation thus only reaction (1) will occur.

case 2
when \( \kappa = 4.82 \text{ mho/m} \)
\( \text{Ka} = 9.0 \text{s}^{-1} \)
\( C_o = 0.871 \text{ mole/m}^3 \) \( T = 3 \text{E}^{-3} \text{ m} \)
\[
\phi_o - \phi_x = 96480 \times 9 \times 0.871 \frac{(1/4.82 + 1/40.04)X^2}{(1/4.82 + 1/40.04)} - 2 \times 96480 \times 0.871 \times 9 \times 0.003 \times \frac{X}{4.82}
\]
\[
\phi_o - \phi_x = 175798.89 X^2 - 941.46 X
\]
when \( X = 0.003 \text{ m} \)
\[
\phi_o - \phi_x = -1.24 \text{ V}
\]
The potential difference is less than the standard reversible potential for both reactions (1) and (2) thus hydrogen and peroxide will be produced.
Appendix C. Sample Calculation

**case 3**

when $\kappa = 13.58 \ \text{mho/m}$ \quad $\text{Ka} = 3.4 \ \text{s}^{-1}$

$C_o = 0.954 \ \text{mole/m}^3$ \quad $T = 3 \ \text{E-3 m}$

$\phi_o - \phi_X = 96480 \times 0.954 \times 3.4 \ (1/13.58 + 1/40.04) \ x^2$

$-96480 \times 0.954 \times 3.4 \times 0.003 \ x /13.58$

when $X = 0.003 \ \text{m}$

$\phi_o - \phi_X = -0.14 \ \text{V}$

Under these conditions only reaction (1) will occur.

**case 4**

when $\kappa = 13.58 \ \text{mho/m}$ \quad $\text{Ka} = 9.0 \ \text{s}^{-1}$

$C_o = 0.871 \ \text{mole/m}^3$ \quad $T = 3 \ \text{E-3 m}$

$\phi_o - \phi_X = 96480 \times 0.871 \times 9 \ (1/13.58 + 1/40.04 )x^2$

$-2 \times 96480 \times 0.871 \times 9 \times 0.003 \ x /13.58$

$\phi_o - \phi_X = 74581.47 \ x^2 \ - \ 334.16 \ x$

when $X = 0.003 \ \text{m}$

$\phi_o - \phi_X = -0.33 \ \text{V}$

Only reaction (1) will occur for this case.
Table 19: Experimental errors

<table>
<thead>
<tr>
<th>Variable</th>
<th>Error</th>
</tr>
</thead>
<tbody>
<tr>
<td>Temperature (°C)</td>
<td>± 0.5 °C</td>
</tr>
<tr>
<td>Current (A)</td>
<td>± 0.5 °C</td>
</tr>
<tr>
<td>Pressure drop (inHg)</td>
<td>± 0.5 inHg</td>
</tr>
<tr>
<td>Burette reading (ml)</td>
<td>± 0.05 ml</td>
</tr>
<tr>
<td>Bed dimensions (mm)</td>
<td>± 1 %</td>
</tr>
<tr>
<td>Pressure gradient (bar/m)</td>
<td>± 6 %</td>
</tr>
<tr>
<td>Total liquid holdup</td>
<td>± 5 %</td>
</tr>
<tr>
<td>Mass transfer capacity (s(^{-1}))</td>
<td>± 12%</td>
</tr>
<tr>
<td>KMnO(_4) concentration</td>
<td>± 5%</td>
</tr>
<tr>
<td>Peroxide concentration</td>
<td>± 2%</td>
</tr>
<tr>
<td>Aqueous sodium hydroxide conc.</td>
<td>± 0.05M</td>
</tr>
</tbody>
</table>