

CHAPTER IV

APPARATUS & EXPERIMENTAL PROCEDURES

There are three principal methods used to carry out an electrochemical reaction.

1. Constant Voltage
2. Constant Current
3. Constant Potential

In this experiment, the constant current method was chosen because it is simple and can be easily set up in the laboratory.

APPARATUS

The electrical circuit for constant current method is shown in Fig 2

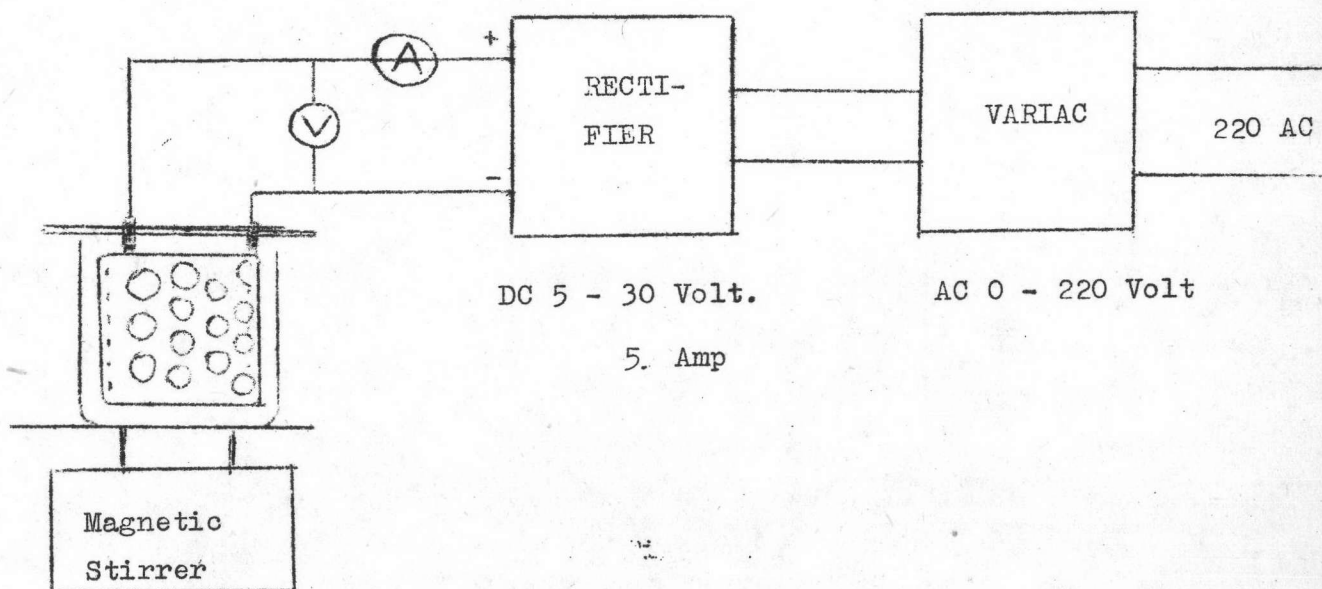


Fig. 2 Schematic illustration of Electrolysis Apparatus

The source of power supply is 220 v. AC, which is controlled by a variac. The current that passes through the rectifier will be converted to direct current. The volt and amp. meters are used for measurement of voltage and current that developed across the electrolytic cell. The electrolytic cell consists of a 1000 cc. plastic box open at the top to facilitate the introduction of wastewater and the insertion and removal of electrodes as shown in Fig. 3

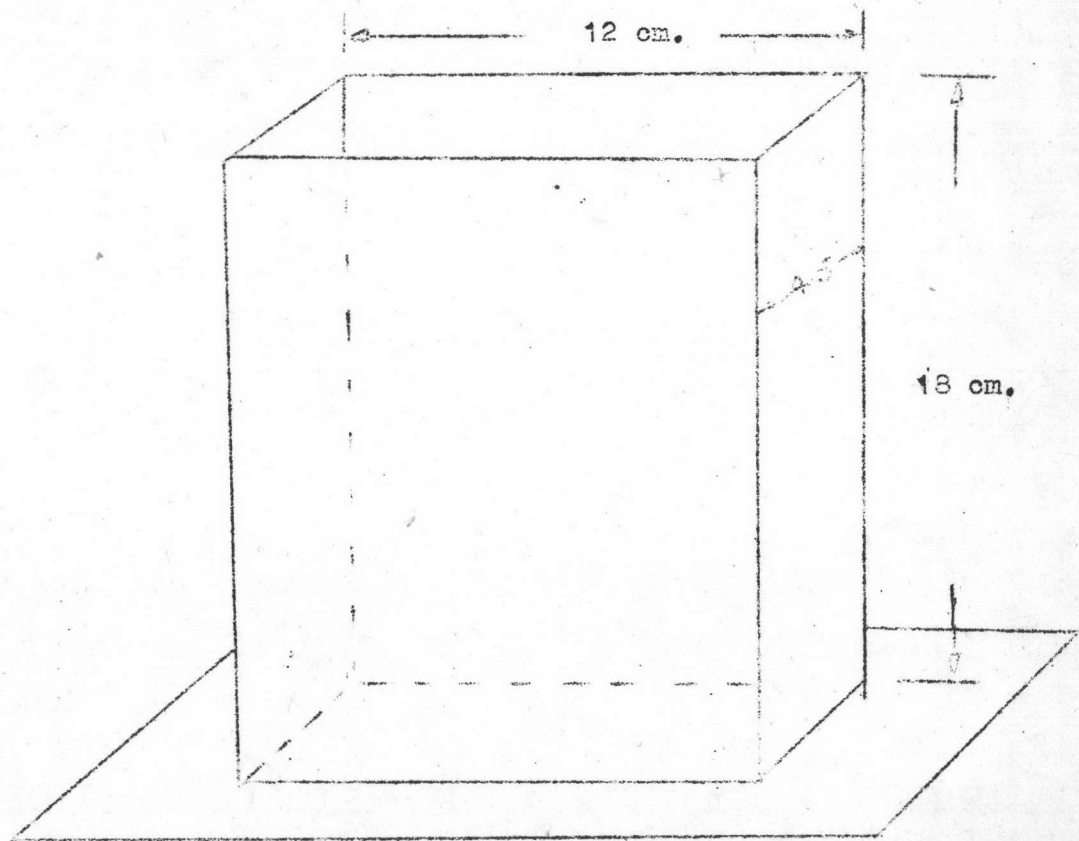


Fig. 3 Cell Container



The magnetic stirrer is used to stir an electrolyte to mix the content and to reduce polarization.

The two electrodes are lead and lead dioxide of the lead storage battery and each electrode composed of 2 plates as shown in Fig. 4.

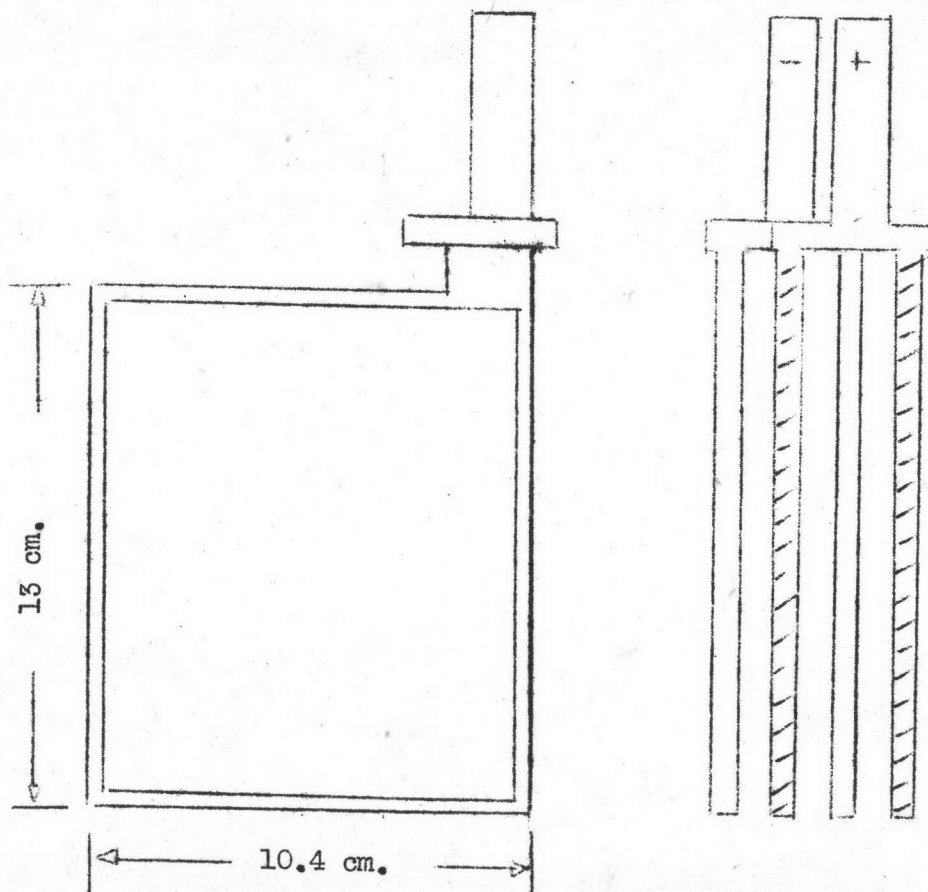


Fig. 4 Section of Electrodes

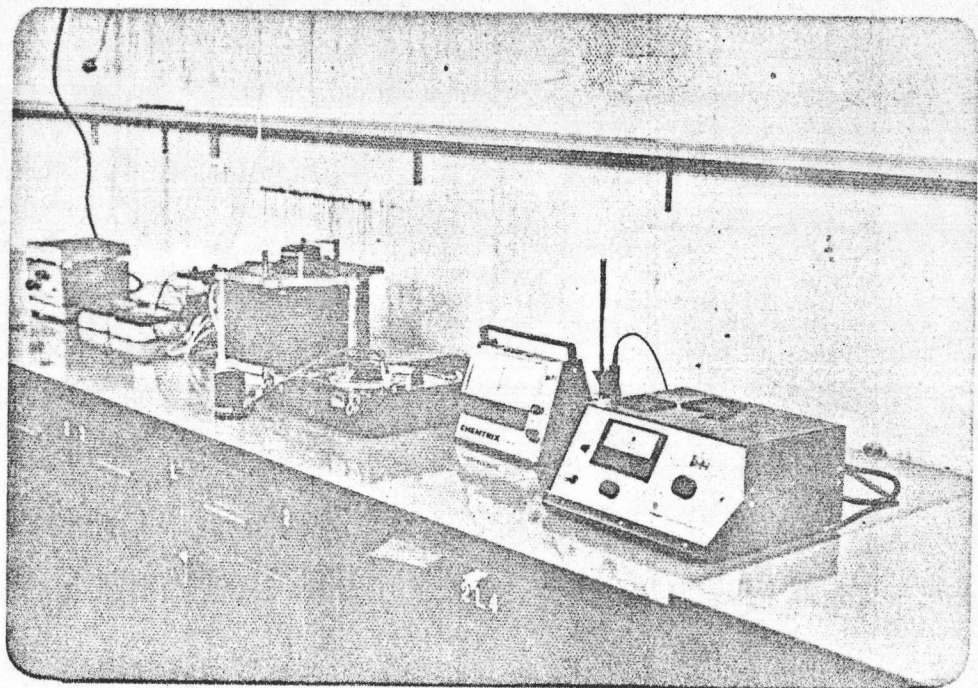


Fig. 5 Typical Apparatus



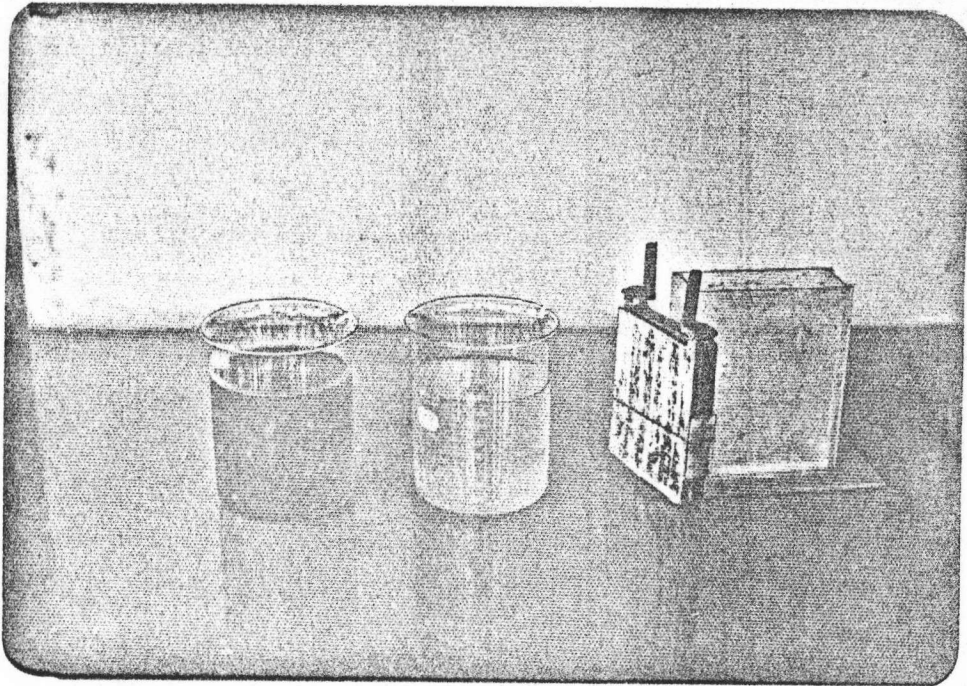


Fig. 6 Electrolytic Cell Dissassembled parts

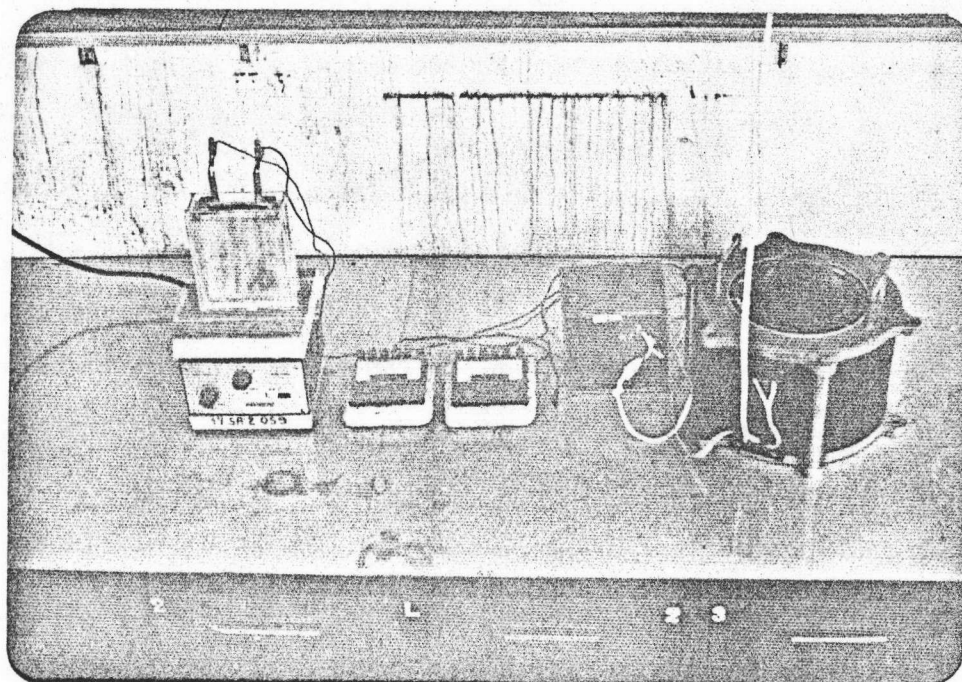


Fig. 7 Electrolysis Apparatus



### Electrolytic cell design

The basic design theories or principles concerned different electrode materials, electrode combination, retention time between plates and plate spacing. The shape, size, and many individual detail of an electrolysis cell are largely a matter of personal taste and of course, vary with the nature of the problem. To maintain a high current flow at a low voltage between electrodes, the two electrodes have to be placed as close together as possible. The most important in cell design is the ratio of the surface area of the electrodes to the volume of the cell, which should be made as large as possible.

The choice of electrode material for a given electrochemical reaction is not an easy task and often requires experience and knowledge of results for related systems. It is generally preferable to have the electrode chemically inert and high overvoltage in aqueous media. Electrode having greater overvoltages will yield higher current efficiency. The physical state of the electrode may often play an important role. A spongy electrode may perform more effectively than a smooth electrode although overvoltage on these rough electrodes is usually low. Apparently, the larger effective surface area and more active sites for reaction tend to compensate for the differences in overvoltage. In addition to pure metal electrodes, electrodes, electroplated metals, metal amalgams, alloys, and certain metal oxides, such as lead oxide have

been used. The number of materials useful as anodes is limited by the corrosion of many metals, platinum, graphite, and lead oxide are used as anodes.

In this study the cell is designed for the main purpose of oxidation of organic matter without much regards for other properties produced by electrolytic reaction.

#### Pretreatment of the electrodes

The two electrodes are taken from lead storage battery. Before use, they are immersed in a beaker containing 20 % sulphuric acid solution. The electrodes are then connected to a D.C. power supply, and 0.5 amp. current is passed through these two electrodes. The anode lead surface will be oxidized to lead dioxide and cathode lead surface will become sponge lead. After activation, they were washed thoroughly with water.



CELL DETAIL

cell container	1000 c.c. plastic box
cell capacity	675 cc.
anode material	Pb/PbO <sub>2</sub>
anode dimension	13 x 10.4 x 0.2 cm.
cathode material	Pb
cathode dimension	13 x 10.4 x 0.2 cm.
anode and cathode spacing	0.5 cm.
surface area of electrode: volume	1 : 1.66

Cell resistance calculate from

$$R = \frac{1}{kA}$$

l = electrode spacing  
k = specific conductance of electrolyte  
A = cross - section area of electrode

Cell voltage calculate from

$$E_{cell} = E_a + E_c + O_{Va} + O_{Vc} + IR$$

E<sub>a</sub> = anode potential  
E<sub>c</sub> = cathode potential  
O<sub>Va</sub> = over voltage at anode  
O<sub>Vc</sub> = over voltage at cathode  
IR = voltage drop

oxidation potential at anode = 1.72 Volt

reduction potential at cathode = 1.51 "

Procedures

All the samples of wastewater used in the experimental work were grab sample obtained from Thai Nam Tip Co., Ltd. Huamark, Bangkok. As sample couldnot be finished for an experiment within aday, so they were kept in a refrigerator for two for each set of experment. Analysis of five typical batches are shown in Table

A schematic diagram of the laboratory appartus is shown in fig. 2. The study programs consisted of a series of bathch experiments. Volume of 675 cc. pf wastewater was added into the cell equipped with the two electrodes. The assembly was then placed on a magnetic stirrer plate. The applied current was adjusted by a variac to an appropriate value, and rechecked at 5 minute intervals.

AS according to the scope of the study, which was divided into three parts. For each part the procedures were performed as follows.

1. Current Effects Various amount of electricity was passed dirrectly into the wastewater. Four sets of detention time were chosen. and each set was carried out in five successive runs.

Run code A	deteneion time	30 minute
	electrical charge	5400-9000 coulomb
Run code B	detention time	45 minute
	electrical charge	6750-12150 coulomb
Run code C	detention time	60 minute
	electrical charge	7200-14400 coulomb

Table 1 Raw wastewater characteristics

Dimension	Unit	2/5/75	8/5/75	10/5/75	16/5/75	20/4/75
pH	-	9.0	9.5	8.4	9.4	9.1
Conductivity	micro-mho	$0.85 \times 10^3$	$1.45 \times 10^3$	$0.97 \times 10^3$	$0.9 \times 10^3$	$0.85 \times 10^3$
Temperature	C	32	32	32	32	-
TS	mg/l	2050	1756	2096	1670	1660
SS	"	48	32	60	40	26
BOD	"	1100	960	1525	700	1150
COD	"	1665	1150	1890	1150	2119.0
Alk.as $\text{CaCO}_3$	"	1480	920	1200	1620	1500
Acid.as $\text{CaCO}_3$	"	0	0	0	0	0
Amonia - N	"	0	0	0	0	0
Organic - N	"	8.9	5.88	9.95	6.7	10.0
Nitrate - N	"	0	0.05	0	0.9	0.1
Orthophosphate	"	0	0	0	0	0
Chloride	"	28	76	41	29.2	21.5

Run code D detention time = 75 minute  
electrical charge 6750-15750 coulomb

2. Effect of Initial pH Initial pH of the wastewater was adjusted by NaOH and  $H_2SO_4$  and same amount of electricity was passed through: each sample.

Run code E detention time = 60 minute  
electrical charge = 14400 coulomb

3. Effect of NaCl addition Sodium chloride is added to the wastewater to increase the conductivity and the chemical reaction. The tests are carried out in five successive runs with constant electricity was dosed.

Run code F detention time = 60 minute  
electrical charge = 14400 coulomb

In the experiments, measurement should be made in ampere, voltage time and other parameters of interest to sanitary engineers, all analytical methods were based on Standard methods (APHA, AWWA, WPCF, 1970).