

WATER PURIFICATION
BY FORCED-FLOW ELECTROPHORESIS

by
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TABLE OF CONTENTS

	Page
Chapter 1 INTRODUCTION	1
1.1 Statement of the Problem	2
1.2 Scope of the Problem	2
1.3 Objectives of the Research	2
Chapter 2 DEFINITIONS	3
Chapter 3 PROPERTIES OF COLLOIDAL SYSTEMS	5
3.1 Introduction	5
3.2 Types of Colloidal Systems	5
3.3 Stability of Colloidal Systems	5
3.4 Electrical Properties of Colloidal Systems	6
3.5 Electrophoretic Mobility	7
3.6 Determination of Electrophoretic Mobility	7
Chapter 4 PREPARATIVE ELECTROPHORESIS	10
4.1 Early Methods	10
4.2 The Bier Forced-Flow Electrophoresis Cell	13
4.21 Supplemental Equipment	15
4.22 Modification for Water Purification	15
Chapter 5 WATER SAMPLE COLLECTION AND ANALYSIS	18
5.1 Collection Sites	18
5.2 Collection of Samples	18
5.3 Analysis	19

	Page
Chapter 6 SEPARATION OF COLLOIDAL SILT FROM WATER	24
6.1 Background	24
6.2 Experimentation	26
6.3 Operational Considerations	27
6.4 Results	34
Chapter 7 SEPARATION OF ALGAL GROWTH FROM WATER	36
7.1 Background	36
7.2 Experimentation	37
7.3 Operational Considerations	39
7.4 Results	43
Chapter 8 SEPARATION OF DISSOLVED ORGANICS FROM WATER	47
8.1 Background	47
8.2 Operational Considerations	48
8.3 Results	51
Chapter 9 CONCLUSIONS AND SUGGESTIONS FOR FURTHER STUDY	57
APPENDIX	63
LIST OF REFERENCES	66

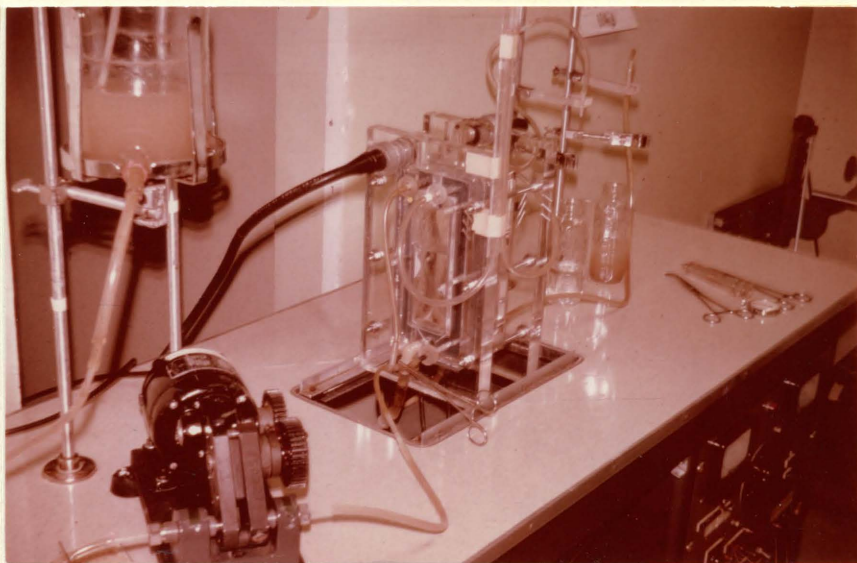
LIST OF TABLES

<u>Table</u>	<u>Title</u>	<u>Page</u>
6.1	Typical Values of Electrophoretic Mobility	25
7.1	Results of Algal Removal and Concentration Experiments	46
8.1	Results of Organic Removal Experiments (90% removal)	54
9.1	Cost-Benefit Ratios and Desalting Effects.	62

LIST OF FIGURES

<u>Figure</u>	<u>Title</u>	<u>Page</u>
3.1	Concept of the Zeta Potential Surrounding a Colloidal Particle	8
4.1	Schematic Picture of Electrodecantation Cell by Pauli .	12
4.2	Electrophoresis - Convection Method by Kirkwood	12
4.3	A Schematic Presentation of the Bier Forced-Flow Electrophoresis Cell	14
4.4	A Front View of the Forced-Flow Electrophoresis Equipment as Used for Selective Plasmaphoresis	16
4.5	A Front View of the Forced-Flow Electrophoresis Equipment as Used for Water Purification.	17
5.1	Standard Turbidity Calibration Curve for Silt Removal Experiments.	21
5.2	Spectral Curve for ABS.	22
5.3	Spectral Curve for 2,4-Dinitrophenol	23
6.1	Flow Diagram for Suspended Silt Experiments	28
6.2	2 Day Run for a Suspended Silt Removal Experiment . . .	30
6.3	Decline of Flow Rate with Reduction in Applied Voltage for Suspended Silt Removal.	31
6.4	Change in Flow Rate When No Cleaning is Employed . . .	33
6.5	Change in Flow Rate When Periodic Cleaning is Employed .	33
6.6	Solids Removal for Suspended Silt Removal Experiments @ 50 ml/min, and 50 Watts	35
7.1	Mobility of Human Blood Cells as a Function of $1/K$ @ pH 7.3 (from Furchogott and Ponder).	38
7.2	Mobility of Human Blood Cells as a Function of pH @ 0.172 Ionic Strength (from Furchogott and Ponder) . .	38

<u>Figure</u>	<u>Title</u>	<u>Page</u>
7.3	The Effect of Periodic Off-On Switching of Voltage on Flow Rate for Algae Removal	41
7.4	Time-Resistance Curves for Clogging Due to Algal Cells.	45
8.1	Variation of % Remaining With Applied Voltage for Removal of ABS.	52
8.2	Variation of Flow Rate with Voltage for 90% Removal of ABS.	52
8.3	Variation of % Remaining with Flow Rate and Applied Voltage for 2,4-Dinitrophenol (10 mg/l).	53
8.4	Variation of % Remaining with Applied Voltage for Xylene Cyanole (10 mg/l).	53
8.5	Spectral Curves in Ultraviolet Range	55
8.6	Comparison of Filtration to Forced Flow Electrophoresis for Kitt Peak Water	56
9.1	A Plot of Power Consumed vs. Specific Resistance of the Colloidal Solution	61



FRONT PLATE. A view of the Bier forced-flow electrophoresis equipment used for the study on Water Purification.

ABSTRACT

The movement of charged contaminants in an electrical field is adapted into a continuous flow system under carefully controlled conditions, resulting in a separation into two fractions, one relatively free of the contaminants and one in which they have been concentrated. An evaluation was made of the operating conditions required to obtain a desired product, the degree to which the type of contaminant and solvent characteristics affect the product and power input, and the future benefits to the water supply and waste-water treatment fields.

Experiments were conducted on the following systems:

(1) Inorganic suspended solids: Clarification of natural flood water of high silt content was accomplished, but had special problems of sludge buildup and handling of the concentrated fraction.

(2) Algal growths in stabilization pond effluent: Complete removal was obtained at rapid rates with a possible byproduct of concentrated algae, a potential protein supplement. It has an economic limitation due to the high conductivity of the effluent.

(3) Low concentrations of phenolics and other compounds: Phenolics were removed at very high rates of application with negligible electrical cost. Anionic detergents, even of high concentration, were removed to meet public health standards using low rates of application to the filter surface.

Chapter I

INTRODUCTION

1.1 Statement of the Problem

In recent years, rapid advancements have been made in the area of water purification including spectacular developments in saline water conversion and sewage reclamation. New processes applicable to both areas may soon become major production systems for consideration in areas now depleting their present fresh water sources. Most of the parameters of design and operation for equipment used in these new methods have been established through research and field experience. Other parameters, which are also quite important, are often underrated and usually estimated. These include maintenance requirements, costs, process control, and flexibility to adapt to changes in loading characteristics. In addition, the economy, efficiency, and practicality of a specific method of water treatment will dictate the extent of its application.

Many ideas and methods developed in other fields are now finding applications elsewhere. Such is the case of electrophoresis, which originated as a method of protein fractionation and isolation, and now is being used for analytical studies in water and sewage treatment. Its application to the field of water purification for actual separation of particles from water has received little attention due to physical limitations of the equipment. With the development of continuous flow-type electrophoresis systems, the applicability of this method should now be considered.

1.2 Scope of the Problem

Electrophoresis covers a broad range of analytical and separatory uses, few of which are applicable to water purification. However, it is believed that a few of the separatory, or preparative methods, have practical application and therefore this study has been directed toward evaluating one particular method, forced-flow electrophoresis. No precise evaluation of the process from the standpoint of secondary factors has been made. These include pathogenic survival, biochemical and chemical oxygen demand reduction, and removal of principal ions that affect water quality. The investigations have been limited to the following points:

- (1) The removal of a major contaminant from a possible water source.
- (2) The operational parameters involved in the contaminant removal and their effect on cost-benefit.
- (3) The mechanical parameters of the equipment in contaminant removal.

1.3 Objectives of the Research

It is hoped that this study will contribute to the knowledge required for development of a new and unconventional method of water treatment. Other specific applications in the field of water reuse or purification no doubt exist and should be searched out. The author believes that with a future potable water shortage being inevitable in many parts of the world, every means of economical production should be investigated.

Chapter II

DEFINITIONS

For purposes of convenience to the reader and limiting unnecessary repetition in succeeding chapters, commonly used terms are defined here.

(1) ABS - alkyl benzene sulfonate, a widely used surfactant in synthetic detergents, comprising about 18% of commercial products.

(2) Bier cell - a preparatory electrophoresis apparatus closely approximating a filter press with an electrical field superimposed across it.

(3) Cell pack - a series of anodic and cathodic chambers separated by cellulose membranes.

(4) Contaminant - any impurity causing water to be less than potable or below tolerable and aesthetic standards.

(5) Electrodecantation - the separation of charged fractions due to density differences caused by imposed electrical convection gradients.

(6) Electrodialysis - the removal of salt ions from water using electrical forces superimposed across cation and anion permeable membranes.

(7) Electrophoresis - the migration of dissolved or suspended charged particles under the influence of an electrical field.

(8) Electrophoretic mobility - the velocity of a charged particle in a potential gradient of one volt per centimeter.

(9) Isoelectric particle - one which possesses zero charge.

(10) Optical density - the absorbance of light by a substance at a particular wavelength. Beer's law states that the amount of light absorbed is directly proportional to the concentration.

(11) Sediment - normally defined as those impurities that will settle out of suspension, in time, when in a quiescent state.

(12) Semipermeable membrane - a membrane which allows passage of a solvent, inorganic salts, and organic compounds of low molecular weight. (Water was the only solvent used in this study.)

(13) Specific resistance (resistivity) - the electrical resistance of a solution, or solid, one centimeter in length and one square centimeter in cross-sectional area.

(14) Synthetic detergent - a surface active agent which will not react with hardness in water to produce insoluble compounds.

(15) Turbidity - impurities suspended in the water which impede passage of light and may be inorganic or organic in nature.

(16) Ultrafiltration - the filtration of colloids or very small particles using an extremely dense filtering media.

(17) Zeta potential - a measure of the electrokinetic charge which surrounds suspended material.

Chapter III

PROPERTIES OF COLLOIDAL SYSTEMS

3.1 Introduction

An understanding of the electrophoretic properties of colloids is best approached by the "classical" concepts of the early colloid chemists. A few of the important points concerning the stability, type, and special characteristics of colloidal systems are discussed in the following sections. It is quite evident that adequate coverage can not be given to the whole picture of colloidal particles and in this respect selected references relating to the specific uses of colloidal theory in this study have been suggested.

3.2 Types of Colloidal Systems

Colloids are generally classified as reversible and irreversible systems. Reversible systems, also called hydrophilic or lyophilic, can undergo a reversible phase change, and may or may not possess electrical charge. Irreversible systems are hydrophobic or lyophobic in character and possess an electrical charge in the form of an electrical double layer. These particles are also characterized by their movement in an electrical field (electrophoresis). Investigators of flocculation and sedimentation in water treatment have concerned themselves primarily with the irreversible type of colloid.

3.3 Stability of Colloidal Systems

The stability of a suspensoid is dependent on both its concentration and electrical charge. With an increase in concentration or density of like colloidal particles, there is a greater chance for

interaction or collision due to Brownian movement and Van der Waals forces, with a resulting agglomeration and sedimentation. Without increase in concentration, such a colloidal system may last for a period of many years before agglomeration takes place.

If particles of unlike charge are introduced into a colloidal suspension, there will be a neutralization of charge with flocculation and sedimentation.

The addition of a hydrophilic colloid to a hydrophobic colloidal system results in the phenomenon of "protective action," where the hydrophobic particles are surrounded by the hydrophilic ones. In this manner the hydrophobic particles acquire a greater stability and an increase in charge.

3.4. Electrical Properties of Colloidal Systems

The existence of electrophoretic velocities of colloid particles and the fact that the solution as a whole is electrically neutral leads to the theory of the electrical double layer. Each particle becomes surrounded with a thin layer of oppositely charged ions from the solution. Outside this layer, the ions are freely mobile and approach a neutral resultant charge some distance away from the interface. The potential of the layer at the interface is termed the zeta potential. This potential is governed by the ionic strength of the solution and will vary as pictured in figure 3.1. As the concentration of the electrolyte is increased, the zeta potential is reduced due to a greater number of oppositely charged ions within the adhering layer. Both the concentration and the valency of the electrolyte will affect the zeta potential

in this manner.

3.5 Electrophoretic Mobility

The charge and potential of the adhering layer give rise to a kinetic factor, electrophoresis. The resultant electrophoretic velocity of motion is described as:

$$v = A' D Z E / V \quad (3.1)$$

where A' = a constant, given a value of $1 / 4 \pi$ by von Helmholtz,

D = the dielectric constant of the solution.

Z = the zeta potential, in millivolts

E = the applied field strength, in volts/cm

V = the absolute viscosity of the solution

therefore:

$$M = v / E = D Z / 4 \pi V \quad (3.2)$$

The electrophoretic mobility, M , may also be evaluated directly from observed measurements by:

$$M = v / E = v A / I R_s \quad (3.3)$$

where: A = the cross-sectional area in square centimeters

v = the linear velocity in an electrical field in centimeters per second.

I = the applied amperage.

R_s = the specific resistance in ohm-centimeters

3.6 Determination of Electrophoretic Mobility

The movement of particles in an electrical field may be measured using a microscope and a special cell designed by Northrop

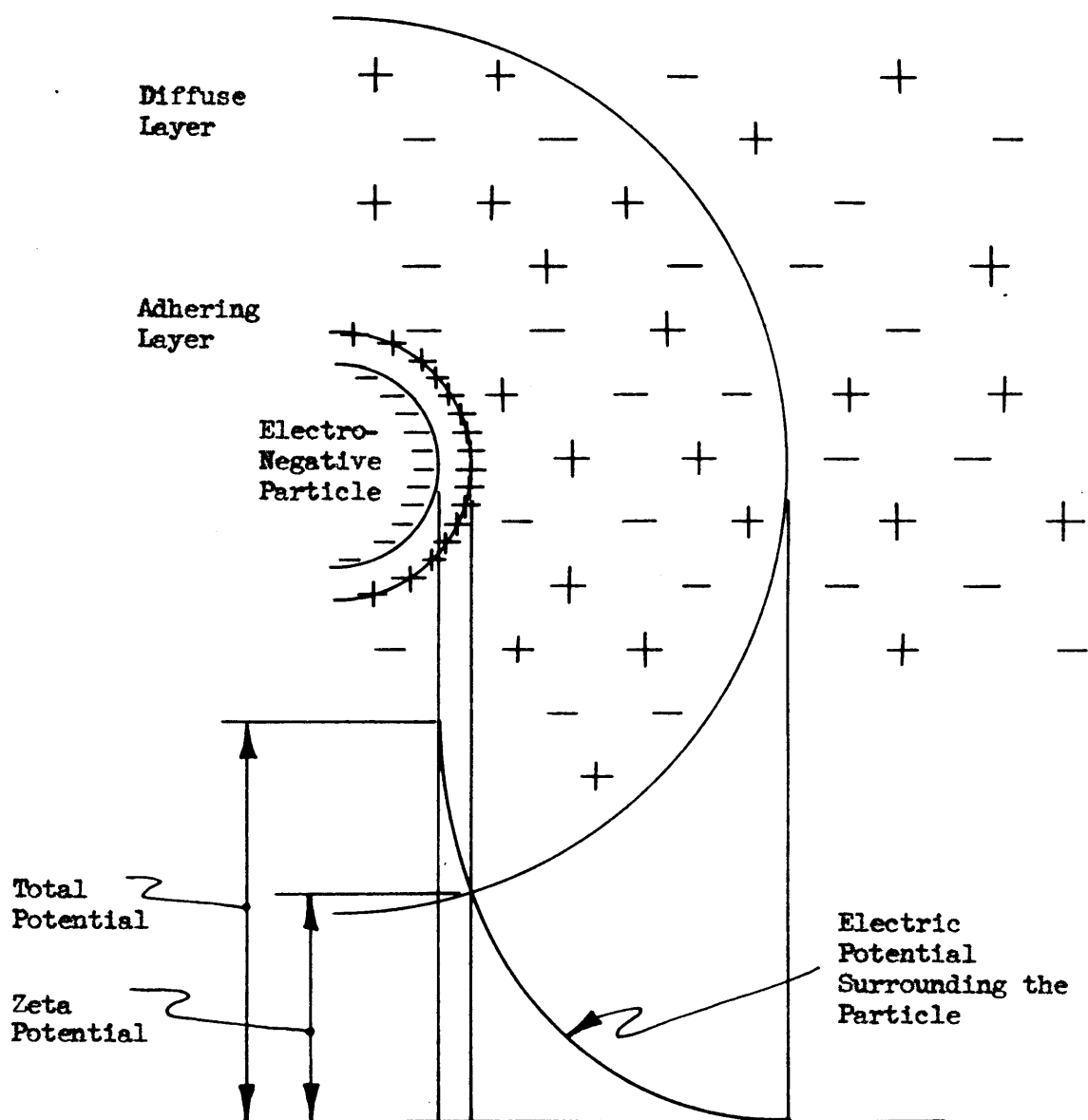


FIGURE 3.1. Concept of the zeta potential surrounding a colloidal particle.

and Kunitz, and modified by Abramson (1). This instrument rests directly on the microscope stage and is constructed of glass to permit observation of the suspended particles. Side arms contain the electrodes and facilities to admit the colloid solution. Due to the narrowness of the cell, electro-osmotic flow exists and requires a correction to yield the true migration velocity.

Whereas the microscopic method requires particle visibility, either directly, or indirectly through adsorption of smaller particles on larger particles such as silica crystals, there is another method where only the boundary of the mass is observed. This moving boundary technique employs a U-tube with the colloidal suspension in the bottom of the tube covered by a buffer solution (electrolyte). With the passage of electricity the mass of colloid moves in a particular direction and at a given rate dependent on its surface charge.

When observed under proper conditions and by a reasonable method, the electrophoretic mobility is a valuable and useful parameter for colloid studies.

Chapter IV

PREPARATIVE ELECTROPHORESIS

4.1 Early Methods

With the advent of the Tiselius apparatus in 1937, electrophoresis in various forms has been used for analytical work in the study of the composition of blood serum by electrophoretic separation. The analytical methods of microelectrophoresis, moving boundary, and zone electrophoresis, are one area of two generally noted in electrophoresis. The second area includes the preparatory methods of curtain, column, and paper electrophoresis, electrodecantation, and counter-current electrophoresis. Only a few of these have any large scale application. Most are limited by the requirements of a supporting media (paper, powder, or gels), a batch-type processing, extremely low flow rates, or adequate heat exchange.

The methods of preparative electrophoresis have been reviewed up to 1948 by Svensson (27) and later by Bier (3) and Moore (21). Three discoveries are especially important in discussing continuous methods of purification of colloidal suspensions: Pauli's electrodecantation process, developed in 1924, the electrophoresis-convection method of Kirkwood, 1941, and Bier's continuous flow preparatory apparatus which was used in this study.

Electrodecantation depends on an increase in concentration, due to the mobility of the colloid, to effect a fractionation. A

migration of colloids toward the anode causes an increase in concentration and induces settling because of higher density, while isoelectric components remain equally dispersed. If any cationic material is present it will migrate in the opposite direction. Figure 4.1 shows a schematic picture of the method. This procedure has been successfully used commercially in the separation of rubber latex on a continuous basis.

Kirkwood suggested a system such as that pictured in figure 4.2. Here a movement of the mobile fraction within the center column in the direction of the anode causes an increase in concentration resulting in a downward motion. Eventually all the mobile colloid is collected in the lower chamber while the upper chamber is free of the colloid. A buffer, circulated through the outer compartment, will remove electrically produced heat and prevent the colloidal solution from being affected by electrolysis.

The truly remarkable point about all of the variations of electrophoresis is that not one has been used to permanently separate colloidal wastes from water. In water, most particles, including bacteria, colloids, proteins, dissolved organic compounds, and insoluble droplets, acquire a charge which usually is negative. They therefore possess some mobility in an electrical field. However the migration of these charged particles is dependent on certain factors, particularly the ionic strength of the solution, the temperature, viscosity, particle size and shape, and in some instances pH. Thus,

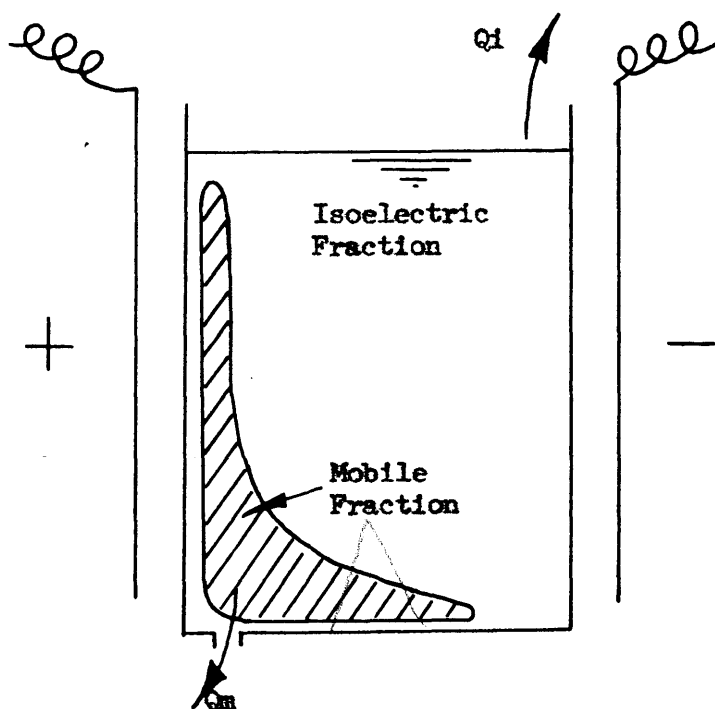


FIGURE 4.1. Schematic Picture of Electrodecantation Cell by Pauli

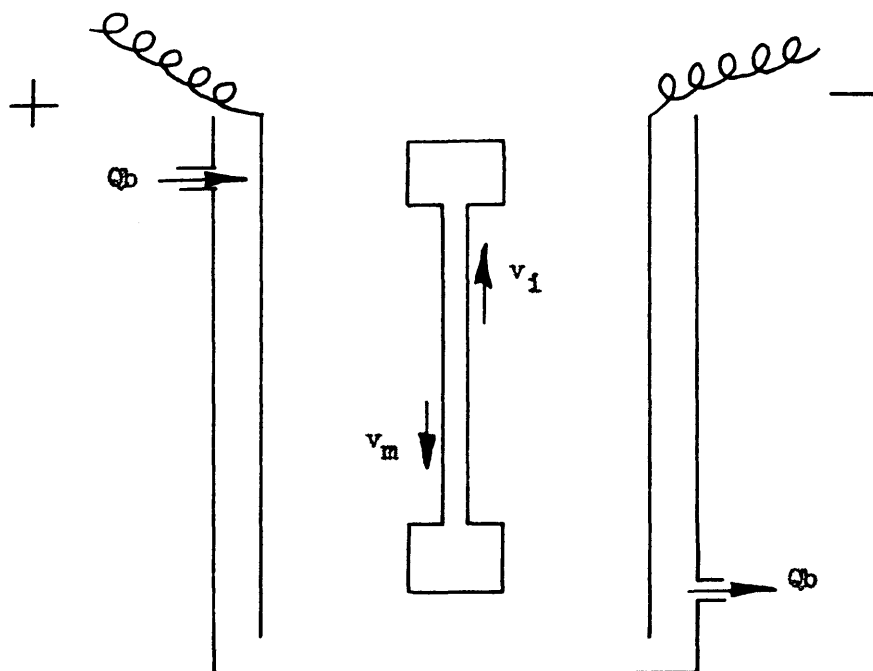


FIGURE 4.2. Electrophoresis--Convection Method by Kirkwood

the continuous separation of negatively charged particles from the rest of the solution is not easily accomplished.

4.2 The Bier Forced-Flow Electrophoresis Cell

This method has been discussed in several articles (2,3,4, 5). but warrants greater detail here, particularly since more recent modifications have been made on the apparatus. Only this equipment was studied for applications to water purification.

The theoretical operation of the Bier cell may be most easily explained by referring to figure 4.3. Introduction of a colloidal suspension, volume V_1 , under laminar flow conditions, into the input chamber results in a two-product separation: colloid free and concentrated fractions. If the induced horizontal flow, v_f , shown by solid arrows, is balanced by the velocity of migration, v_m , represented by broken arrows, the colloid is forced to remain in the input chamber while the isoelectric components and solvent will travel through and be removed at the top, volume V_3 . The concentrated colloid, volume V_2 , is pumped out the bottom of the input chamber.

The cell is composed of a filter, B, sandwiched between two membranes, A and A'. Filters of ordinary Whatman paper or porous polyvinyl chloride (PVC) material have been used. Membranes are standard visking dialyzing tubing purchased in a dry, flat state. Spacers of a plastic or rubber material, placed between each membrane and filter create the chambers. With connections in parallel, as many cells as desired may be placed in the same field. The whole assembly is tightened

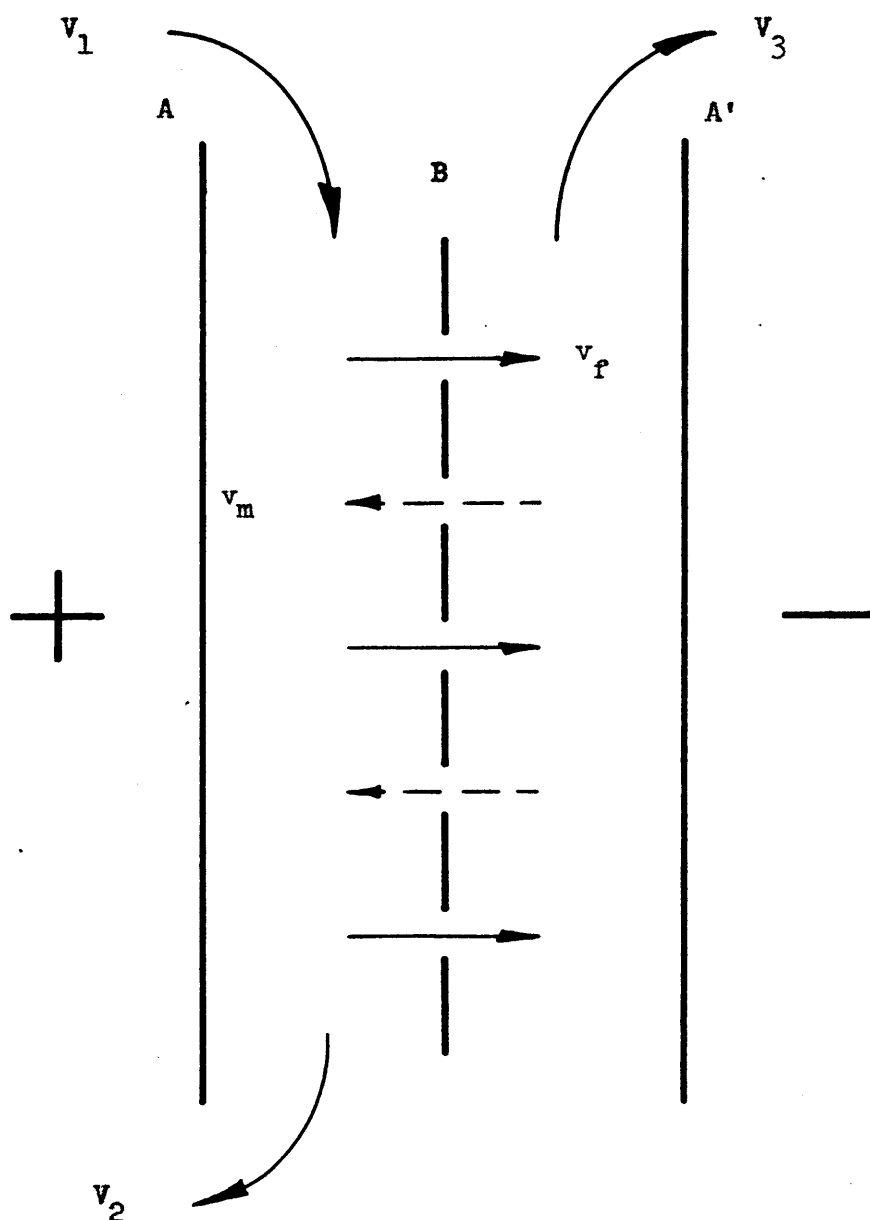


FIGURE 4.3. A schematic presentation of the Bier forced-flow electrophoresis cell. The velocity of flow is represented by the solid arrows and the migration velocity by the broken arrows; A and A' are membranes; B is the filter.

together by means of two end-plates made of plexiglass and bolts.

Platinum electrodes are housed within the end-plates.

4.21 Supplemental Equipment

To employ the Bier cell, several items of additional equipment are necessary. They include a variable D.C. power supply, electrical meters, buffer circulation apparatus, pumps, tubing and connectors, clamps, collection containers, supply bottles, and cell assembly tools. In the laboratory, the cell pack was mounted on a table console. (See front plate.)

4.22 Modification for Water Purification

The equipment used by the author differed from the usual set-up developed for blood fractionation. Modifications are shown in figures 4.4 and 4.5 and described in the footnotes below them.

The supply to the cell may be gravity fed, pumped by non-pulsing variable speed gear pumps, or under applied pressure, as long as laminar flow is provided within the cell.

Also a pump may be used for an output fraction, or both may be allowed to flow by gravity. Both systems were used by the author. The small gear pumps were deleted when working with silty water and sewage effluent due to probable clogging of the gears.

Two additional modifications included deletion of the internal buffer compartments used in the blood fractionation, and elimination of the refrigeration system for the external buffer. Neither heat exchange nor pH control were critical factors in the present application.

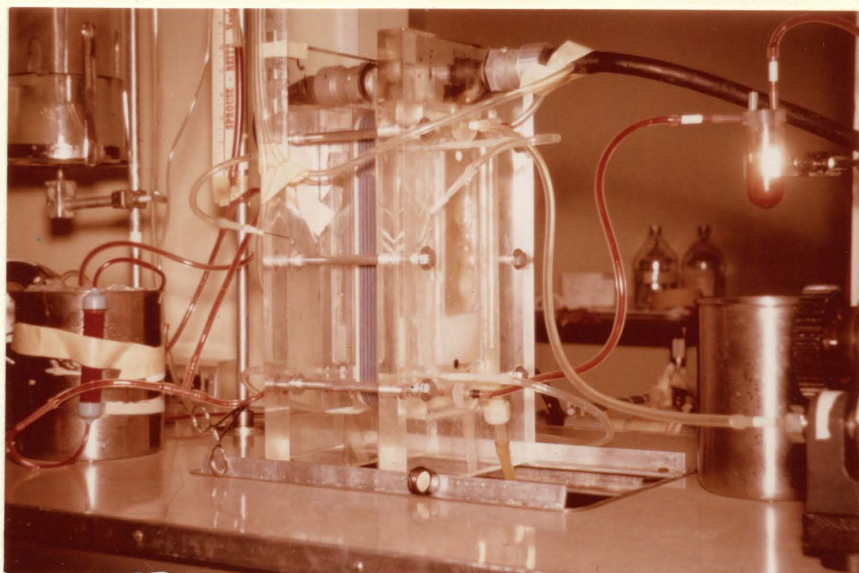


FIGURE 4.4. A front of the forced-flow electrophoresis equipment as used for selective plasmapheresis. Blood, directly from the donor is cooled in the metal beaker on left, passes through the vertical pulse trap, in rear, and enters cell by the upper left tubing (red). The lower left tubing (red) with a hemostat clamp is used to initially fill the cell. The upper right tubing (clear), leading to pump at lower right, contains the separated fraction, while the lower right tubing (red) contains the whole blood which drains into the reservoir on the right from which it is pumped back to the donor. Upper tubing, from each side of the cell (clear), fastened to the vertical scale indicate internal pressures. The blue center area is the cell pack. The right platinum electrode is visible within the plexiglass endplate. The refrigerated buffer solution enters through the large tube at bottom and leaves beneath the cell pack.

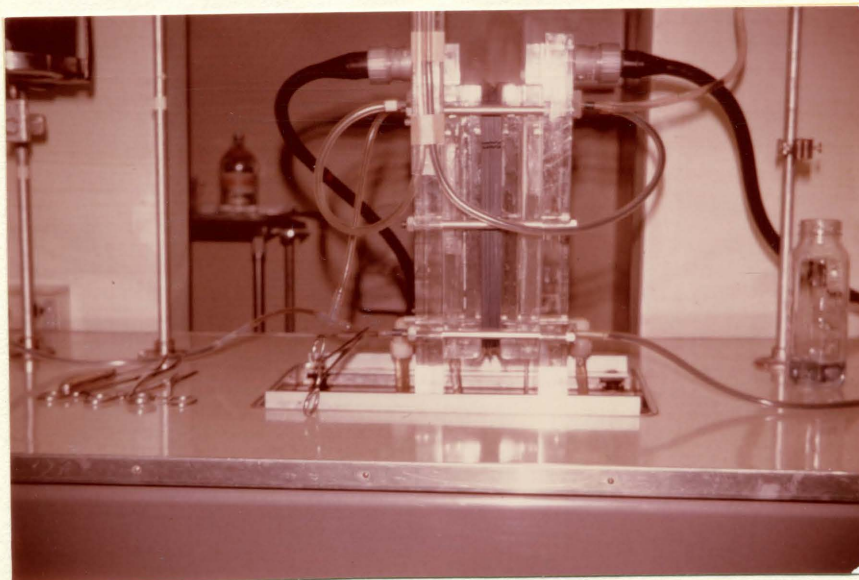


FIGURE 4.5 A front view of the forced-flow electrophoresis equipment as used for water purification. The sample supply water enters the cell through the upper left tubing (rear) with the adjoining lower tubing used for initial filling of the cell. The purified water leaves the cell by the upper right tubing (clear) and the concentrated fraction through the lower right tubing (blue). Manometer tubings are shown attached to the vertical scale. Cooling water enters from the outside bottom and leaves below the cell pack.

Chapter V

WATER SAMPLE COLLECTION AND ANALYSIS

5.1 Collection Sites

The sites used for water sample collection as well as the type of site sample collected were:

(1) Julian Wash Detention Basin. Located in Pima County, Arizona, northwest of the intersection of Ajo Way and Campbell Avenue in the City of Tucson. Collected silty runoff water.

(2) Sanitary District #1 Sewage Stabilization Pond. Located west of the Casa Grande Highway 5 miles north of the City of Tucson. Collected pond effluent.

(3) Kitt Peak Observatory Catchment Area. Located on Kitt Peak south of the Ajo Highway 50 miles west of Tucson. Collected sample of the captured precipitation.

All other water samples were artificially prepared in the laboratory.

5.2 Collection of Samples

A practice was established of collecting large quantities of water at one time in five gallon plastic containers and transporting them to the laboratory. This procedure was required since large volumes were needed for individual experiments, and it was impossible to take the equipment to the source in the field.

At the detention basin, grab samples were taken from the edge

of the pond. Settled material was not included in the sample collection.

Samples from the stabilization pond were taken from the discharge pipe for the final lagoon. Due to the late Fall season, the algae were gradually decreasing in concentration. This variation ranged from 0.1 - 0.05%, dry weight basis, during the testing period.

For the catchment area, samples were taken from the storage tank preceding rapid sand filtration and contained some returned water from filtering and chlorination treatment.

5.3. Analysis

In general, all measurements were quantitative and included the following procedures:

Measurement of turbidity. Using a sample of the collected water, successive 1:1 dilutions were made and consecutive readings were taken with a Beckman DU Spectrophotometer operating at 660 millimicrons in the visual range and with a Jackson Candle Turbidimeter (26). The data were used to prepare the calibration curve shown in figure 5.1. This curve then served as the basis for converting sample measurements to standard units.

Measurement of spectral absorbance. Samples of the collected water were placed in quartz cuvettes and measured for spectral absorbance in the Beckman DU Spectrophotometer over the entire ultraviolet range. Distilled water was used as a blank. The wavelength giving a prominent peak in the curve was selected for subsequent measurements

of concentration reduction. Figures 5.2 and 5.3 are special curves for ABS and 2,4-dinitrophenol.

Measurement of specific resistance. Direct readings were taken using a Leeds and Northrup Zero Reading Galvanometer equipped with an electrode-encased squeeze dropper. The reciprocal of the specific resistance equalled the conductivity of the solution with the instrument preset for conversion to standard conditions.

All additional quantitative examinations were conducted as outlined in Standard Methods (26).

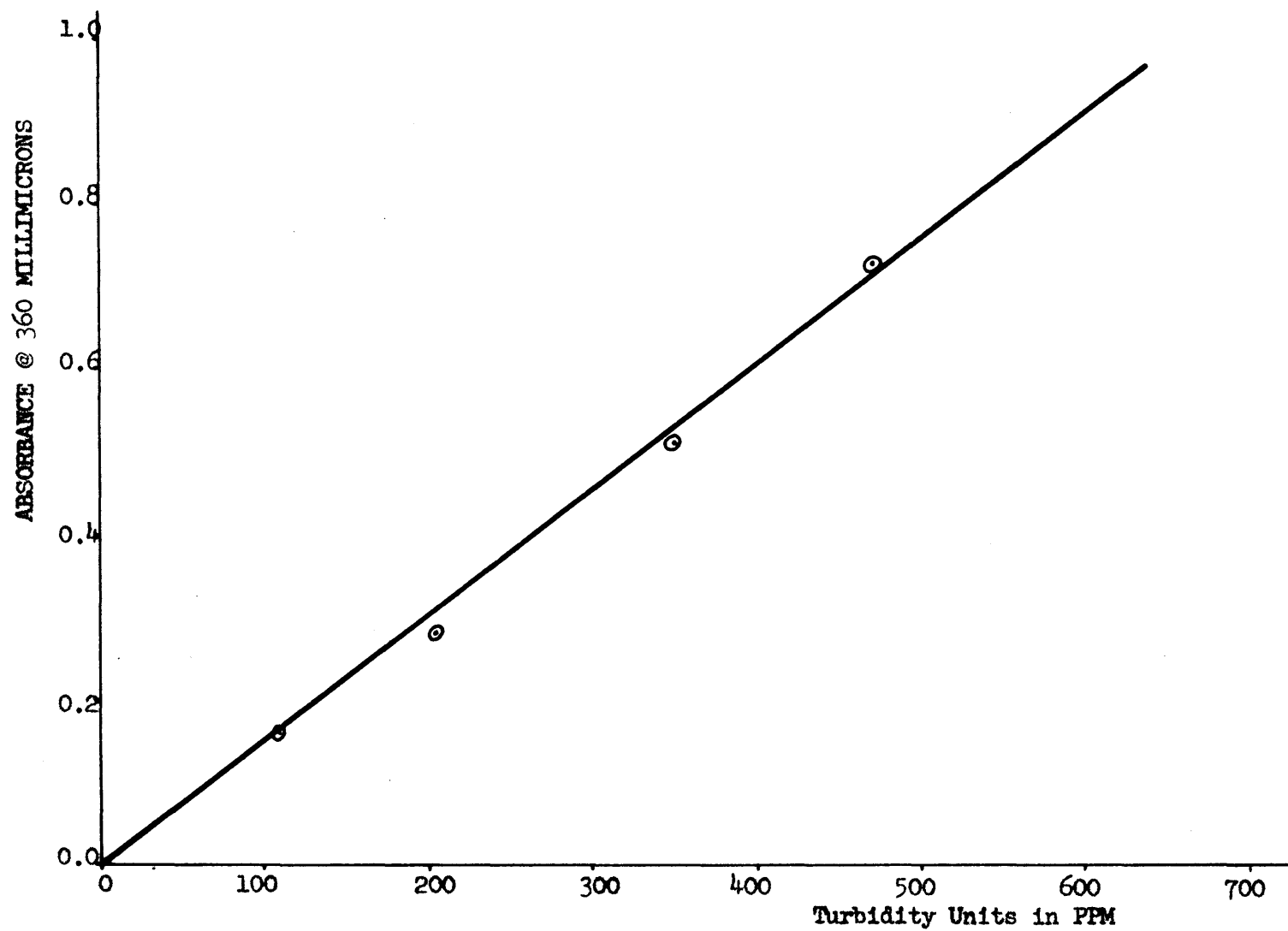


FIGURE 5.1. Standard Turbidity Calibration Curve for Silt Removal Experiments

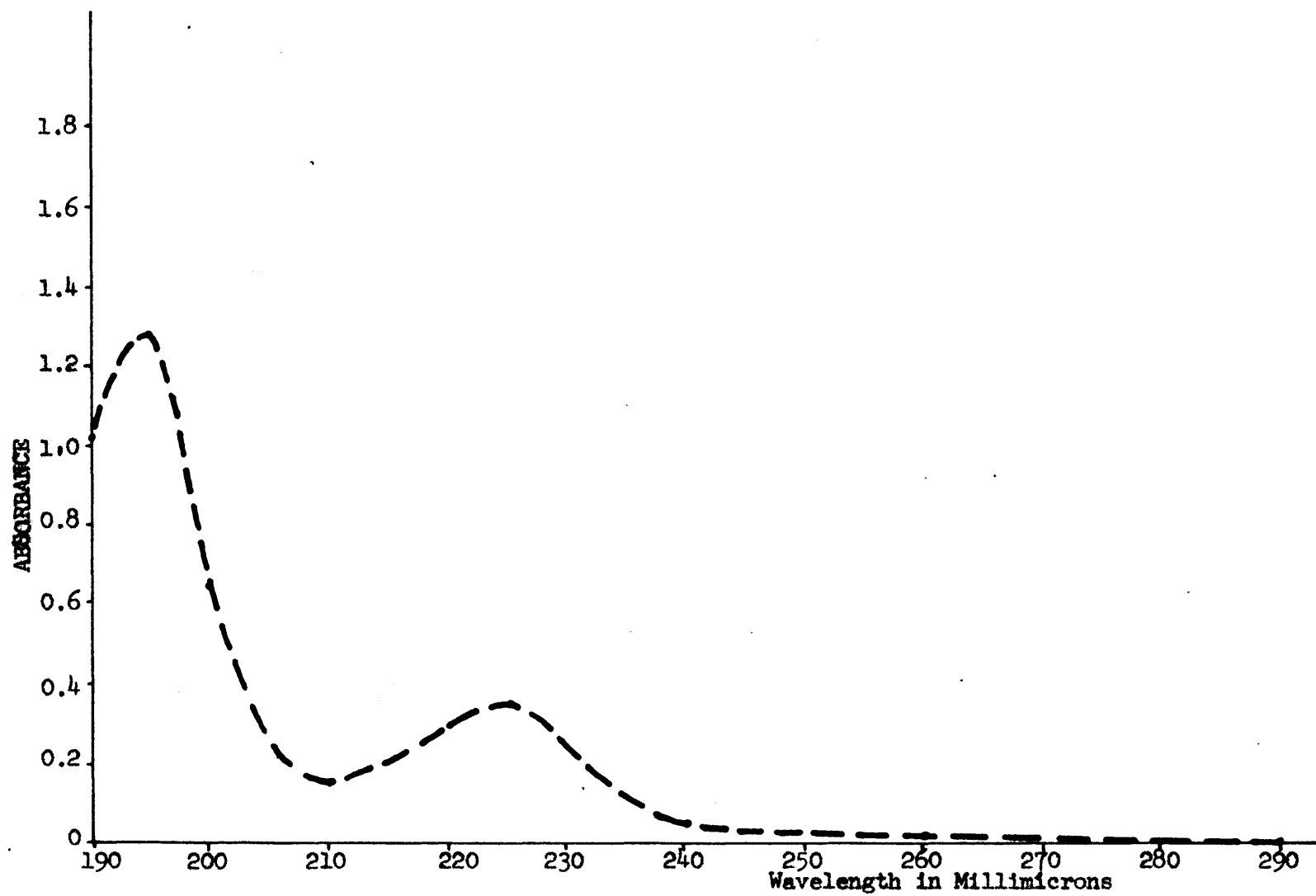


FIGURE 5.2. Spectral Curve for ABS.

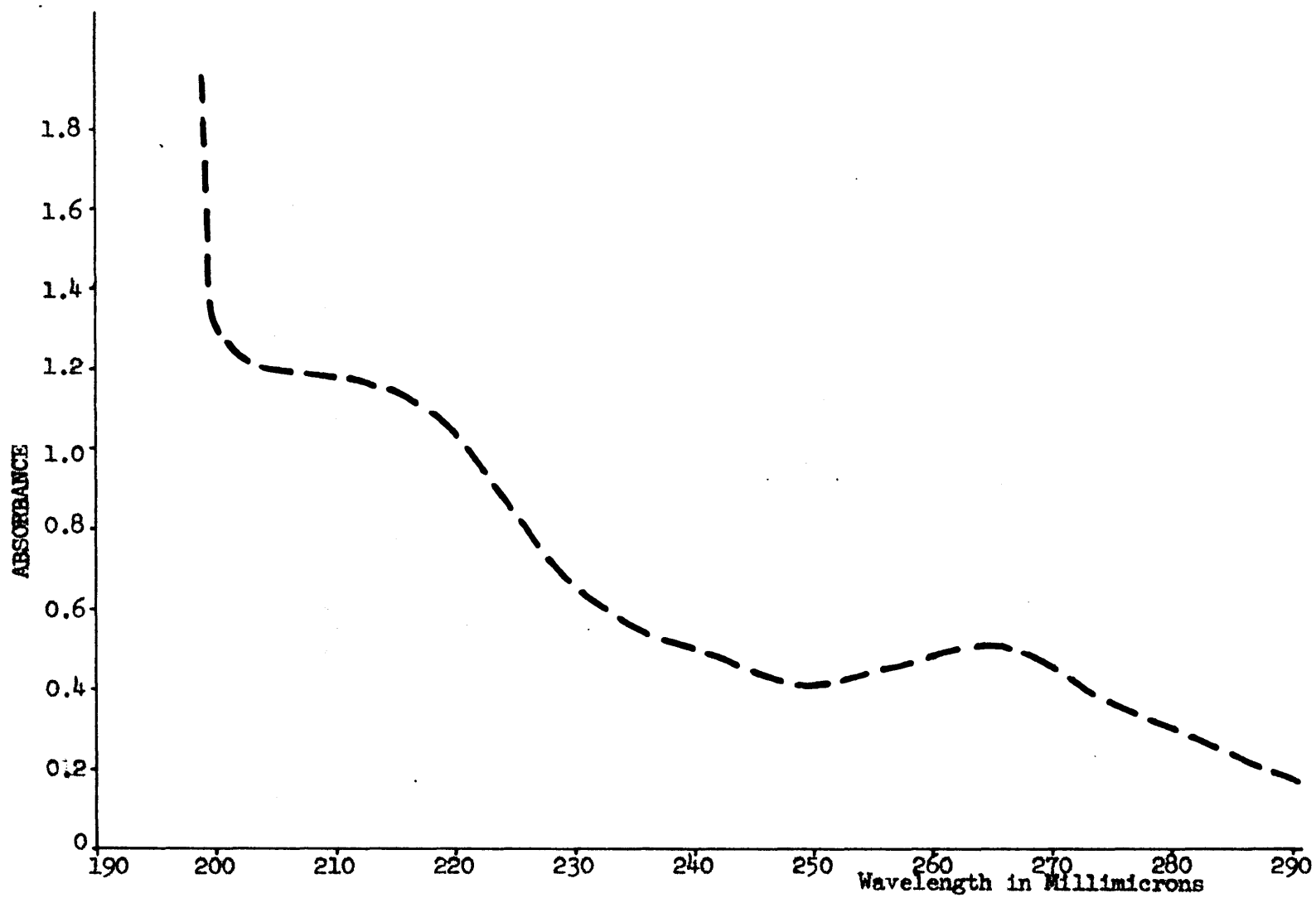


FIGURE 5.3. Spectral Curve for 2,4-Dinitrophenol.

Chapter VI

SEPARATION OF COLLOIDAL SILT FROM WATER

6.1 Background

The first observation of movement of colloidal silt or clay particles in an electrical field was made by Ruess in 1806. This movement was later measured by Quincke in 1861. With the development of the ion theory, knowledge of the forces of interaction, and the specialized work of such men as Smoluchowski, Helmholtz, Debye, Gouy, Chapman, and Huckel, this migration has been explained. The surface charge or zeta potential of the colloid determines the rate of migration within the electrical field.

A survey of the literature failed to provide information concerned with research on removal of a clay suspension from water using electrophoresis. However, there are numerous articles to be found on the study of zeta potential, particularly in the area of chemical coagulation.

A. P. Black and others (6,7,8,9,10,11), using the microscopic method by Briggs (12), have observed and measured the movement of various flocs in an electrical field with varied dosages of aluminum sulfate and ferric sulfate. Faust and Manger (16) used the same method for measuring migration of suspended material in sewage. It has also been used by Jorden (18) for studying the processes of filtration and the influence of the 'schmutzdecke' on filter efficiency. Table 6.1 gives some of the values of electrophoretic mobility which have been

TABLE 6.1

TYPICAL VALUES OF ELECTROPHORETIC MOBILITY

<u>Source</u>	<u>Particle Description</u>	<u>Electrophoretic Mobility</u>	
		M microns/sec/v/cm	
Black	CaCO ₃ sludge produced by lime softening	-0.90	to -1.00
Black	fullers earth	-0.70	to -1.40
Black	kaolinite	-1.00	to -1.50
Black	montmorillonite clay	-1.00	to -1.50
Black	fullers earth w/15 ppm alum	+1.00	to -1.70
Black	kaolinite w/15 ppm alum	+0.90	to -1.10
Black	montmorillonite clay w/15 ppm alum	-0.30	to -1.60
Faust & Manger	particulate matter in sewage	-0.55	to -3.75 mean -1.73
Faust & Manger	same at $K = 0.165 \text{ ohm}^{-1} \text{ cm}^{-1}$	-1.15	to -3.41 mean -1.97
Faust & Manger	same at $K = 0.285 \text{ ohm}^{-1} \text{ cm}^{-1}$	-0.75	to -2.19 mean -1.40
Jorden	montmorillonite clay	-0.78	to -2.24
Cooper	raw water sample (silt & clay - 500 ppm)	-1.3	to -2.6

determined by this method. (Plus and minus signs refer to positive and negative charge.)

It is quite likely that the wide variation in these measurements may be attributed to differences in ionic strength of the solution. This parameter was not reported in the literature, and as a result no comparison should be made between different particles as to values of electrophoretic mobility. This may be also true for the same particle for different tests.

6.2 Experimentation

With the cooperation of the Pima County Highway Department, samples of runoff water during the 1963 summer rainy season in Tucson were obtained from the Julian Wash Detention Basin. This basin has a drainage area of slightly less than 33 square miles and a ponding capacity of approximately 2000 acre feet. During the period of this study, there was no outflow from the detention basin and losses were by evaporation and seepage only. The sample water was found to have a silt content ranging from 500 to 1500 ppm (mg/l) upon collection, and laboratory studies indicated little further sedimentation in an additional 24 hours. High values occurred after fresh storm runoff entered the pond.

With the intention of studying field problems using the laboratory model, it was believed that these collected samples gave a better indication of actual operating conditions than if artificial suspensions had been used. If theoretical parameters were to be investigated, the use of artificial suspensions would be better since concentrations and particle mobilities could be carefully controlled. However, the runoff

water, if kept well mixed, showed actual characteristics that would be encountered in large scale site treatment.

Preparatory treatment consisted of simple filtration through a layer of cheese cloth to remove larger particles, floating material, and numerous fresh water crustacea which abounded in the pond. This procedure was adopted since the small entrance openings of the cell pack and tubing used would not allow passage of these materials.

A typical flow diagram used with these experiments is shown in figure 6.1. In certain instances the bottom fraction was recycled to the supply tank as shown. This was beneficial in studying conditions for prolonged runs. An automatic relay device was set to actuate the centrifugal recycle pump since its pumping capacity usually exceeded the rate of the concentrated flow from the cell.

The basic steps used for a laboratory run were as follows:

- (1) assembling a cell, including preparation of the membranes and filters, (2) installation of the assembled cell on the console with connection of the tubing, (3) filling the supply reservoir and calibration of the flow rates to be employed, and (4) running the experiment with collection and analysis of the samples.

6.3 Operational Considerations

With the colloidal silt, successful separation was dependent upon rigid operating requirements designed to eliminate filter clogging. Clogging would occur in only a few minutes at zero voltage but would remain clear for several days at 50 volts/ cm. Figure 6.2 shows a run

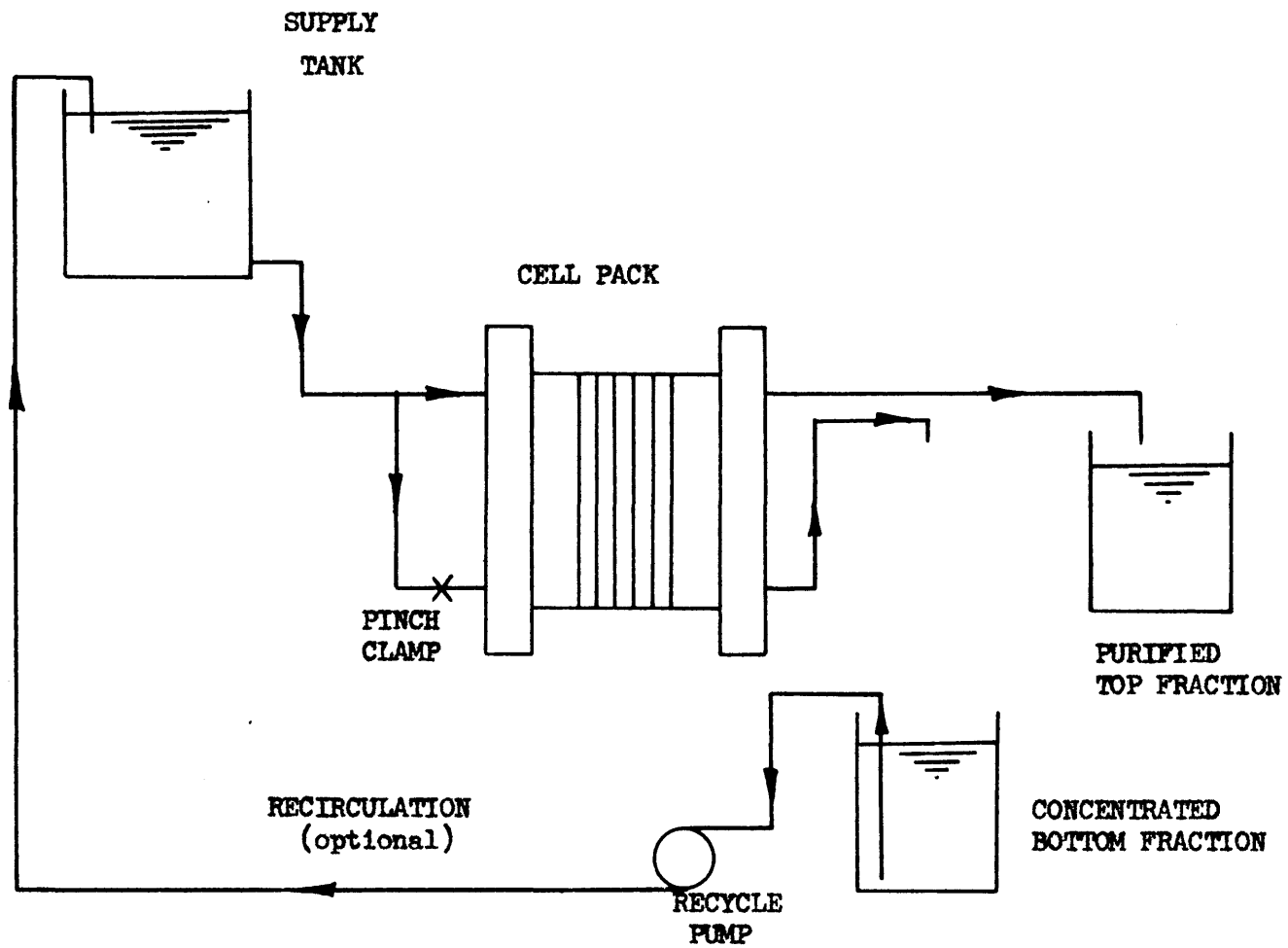


FIGURE 6.1 Flow diagram for suspended silt experiments.

where after processing of the original sample water both fractions were remixed with the supply. A stirring device was placed in the supply reservoir to prevent settling and assure a uniform supply nearing that of the detention basin. This run was stopped after 46 hours with only minor silting visible within the cell pack.

To evaluate the effect of clogging on the cell pack, the rate of decline of flow rate was measured with variation in voltage applied. With this data known, (see figure 6.3) safe operating conditions could be selected from curves. Below these conditions clogging quickly occurred while above them it would not. Since theoretically at the minimum safe operating conditions, the velocity of flow, v_f , should balance the velocity of migration, v_m , the mobility of the particles could be calculated by equation 3.3. The author used the applied voltage 33.3 volts/cm and flow rates of 60 and 90 ml/min, finding the value of M to be about -1.3 to -2.6 microns/sec/volt/cm. A linear and continuous drop of voltage potential across the cell pack was assumed.

A difficulty arose with long runs in that a sludge deposit accumulated in the input compartments which eventually reduced the flow through the cell pack. This sludge buildup was associated with two factors. First, as a result of migration of the colloidal material toward the negative pole, the particles came in contact with a membrane and became adsorbed to its surface before they could be removed through the bottom outlet. Upon disassembly of the cell pack after a run, it was always observed that this sludge deposit was on the membrane side away from the filter and not on the filter itself. The filters

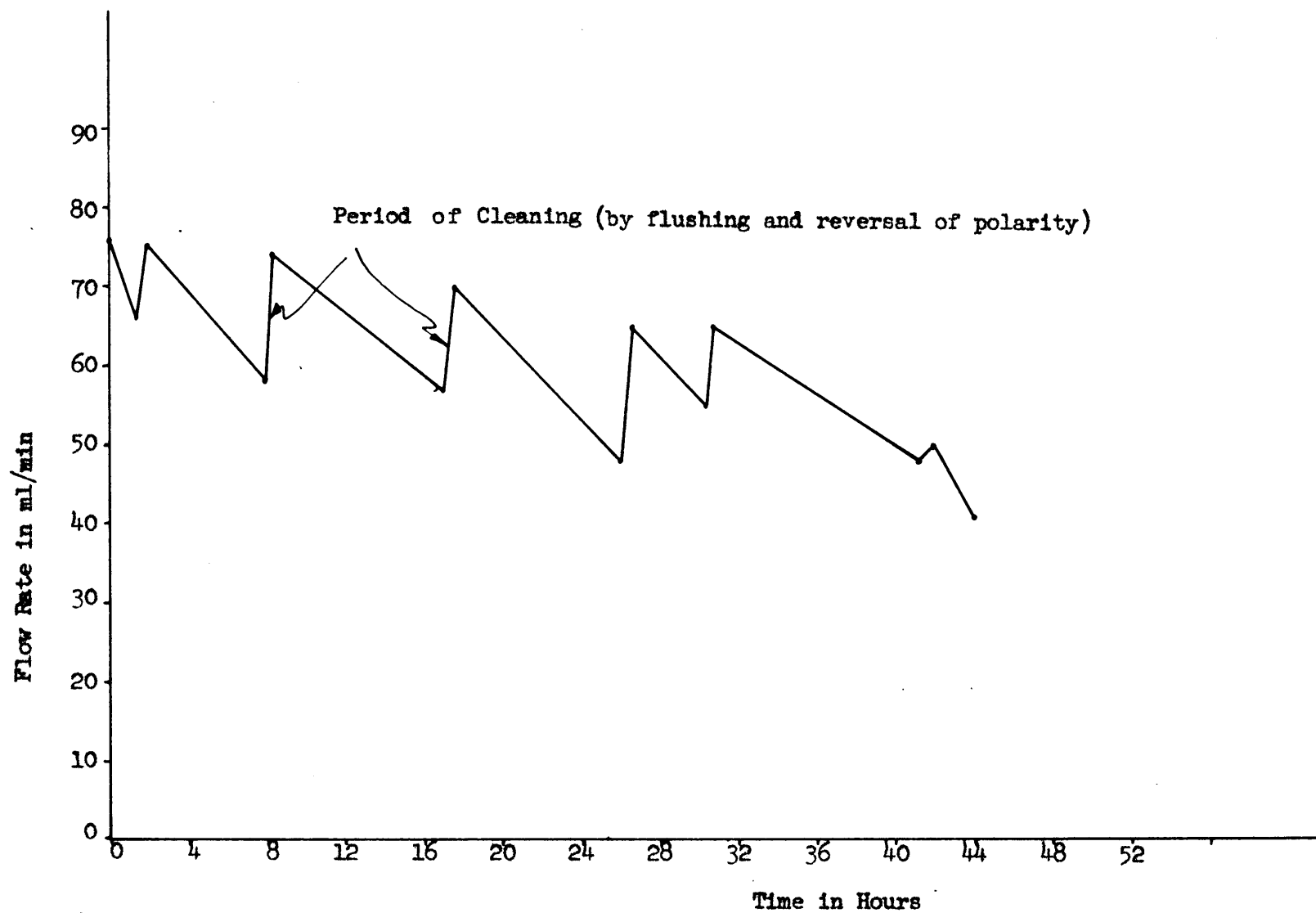


FIGURE 6.2. 2 Day Run For A Suspended Silt Removal Experiment

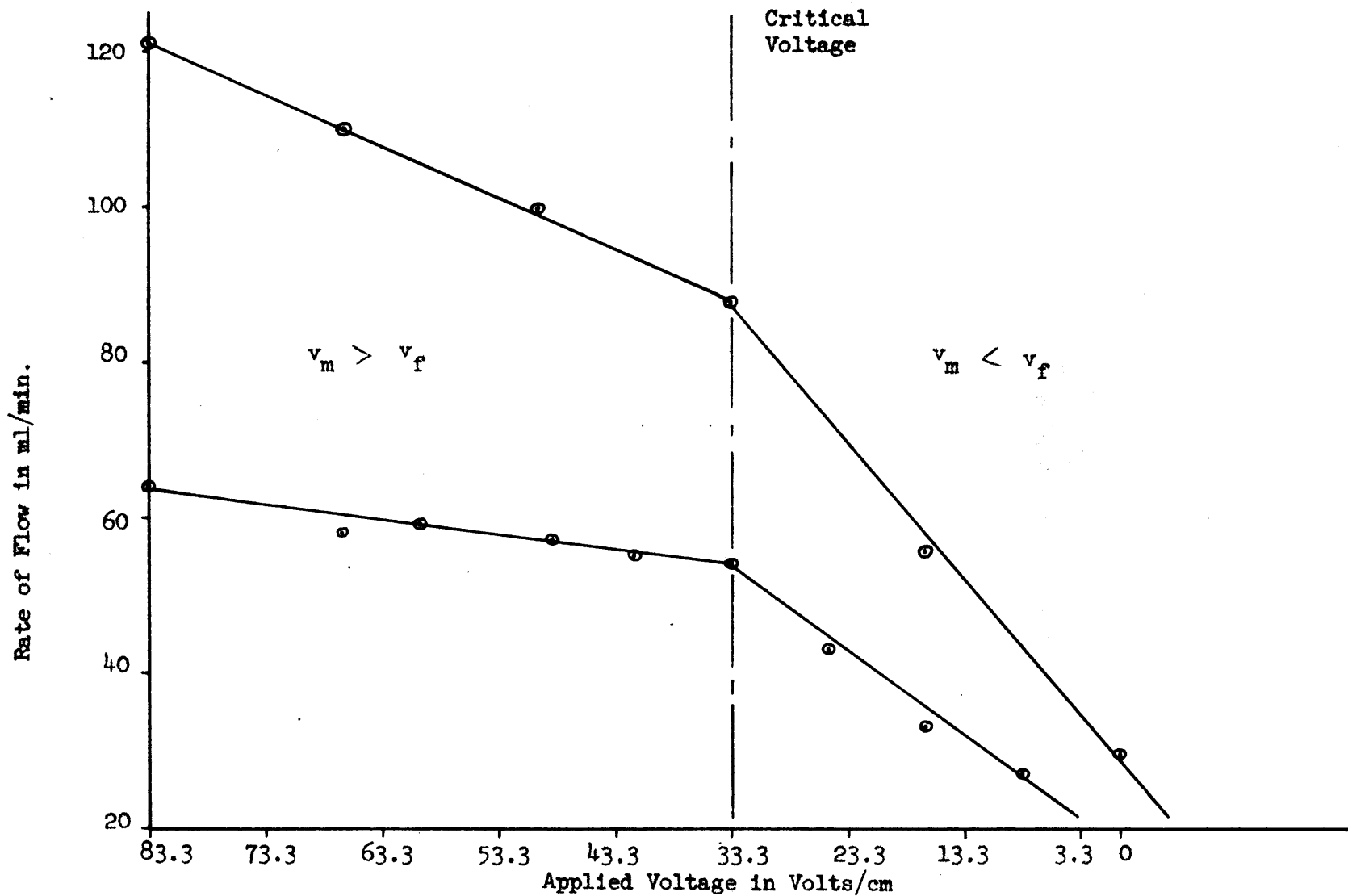


FIGURE 6.3. Decline of flow rate with reduction in applied voltage for suspended silt removal.

remained clean. Secondly, the physical size of the manifold entrance and exit openings restricted the size of particle passing through, and if the colloid grew in size due to concentration in the input compartments, the resulting agglomeration clogged these bottom manifold openings.

Several methods for breaking up and removing this sludge were tried. Probably the best method involved shutting off the top flow and surging the bottom flow. This caused extreme turbulence in the input compartment with a breaking up of the sludge deposit, clearing of the manifolds, and withdrawal of some of the material. This method, it is feared, would present difficult problems on application to large pieces of equipment. A second cleaning method involved reversing the polarity of the field thus causing two things to happen: (1) the sludge became dislodged and broken up due to a change in the internal cell pressure resulting from immediate clogging of the filters, and (2) the colloid material migrated in the opposite direction. By continuing the flow through the input compartment and alternating the direction of the field, accumulated silt was removed.

Figures 6.4 and 6.5 show the effect of cleaning on the rate of decline of flow rate due to clogging. Figure 6.4 involved a run at 50 watts with no cleaning. The flow rate was set at an initial value and periodically observed, then reset by increasing the supply head. A simple linear relation was assumed for calculating the slope although the actual case may approach a parabolic relationship. In figure 6.5

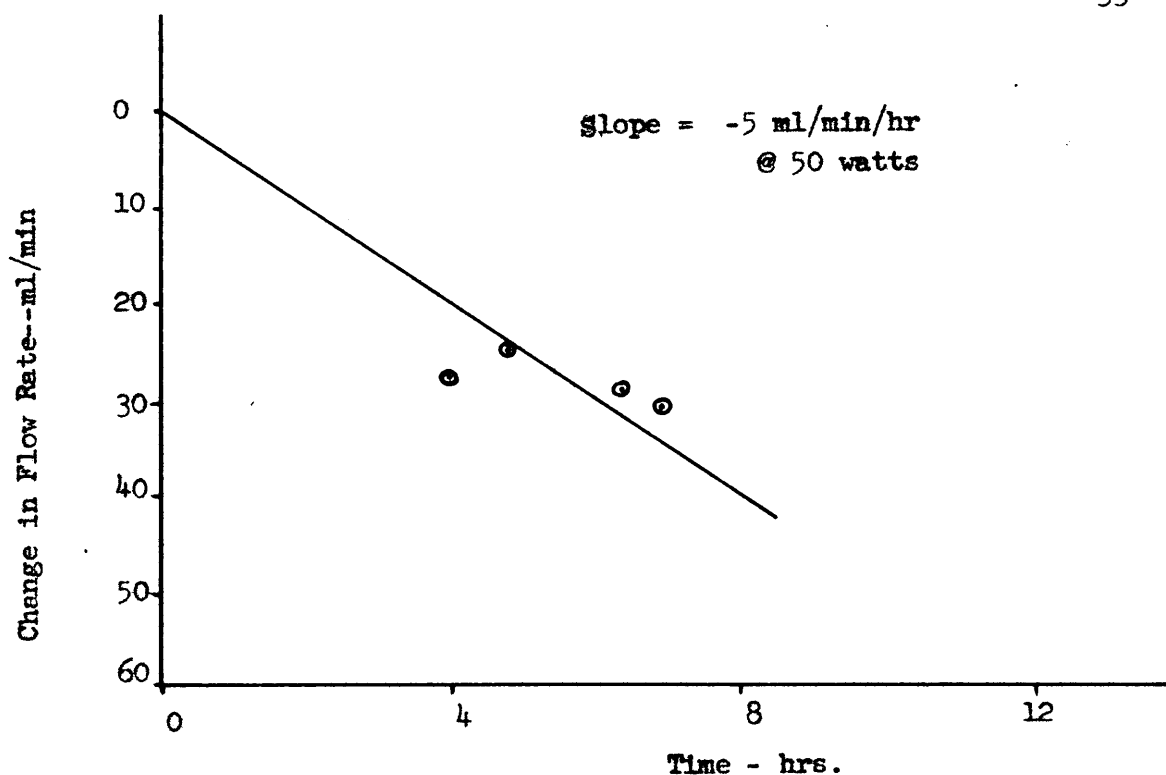


FIGURE 6.4. Change in flow rate when no cleaning is employed.

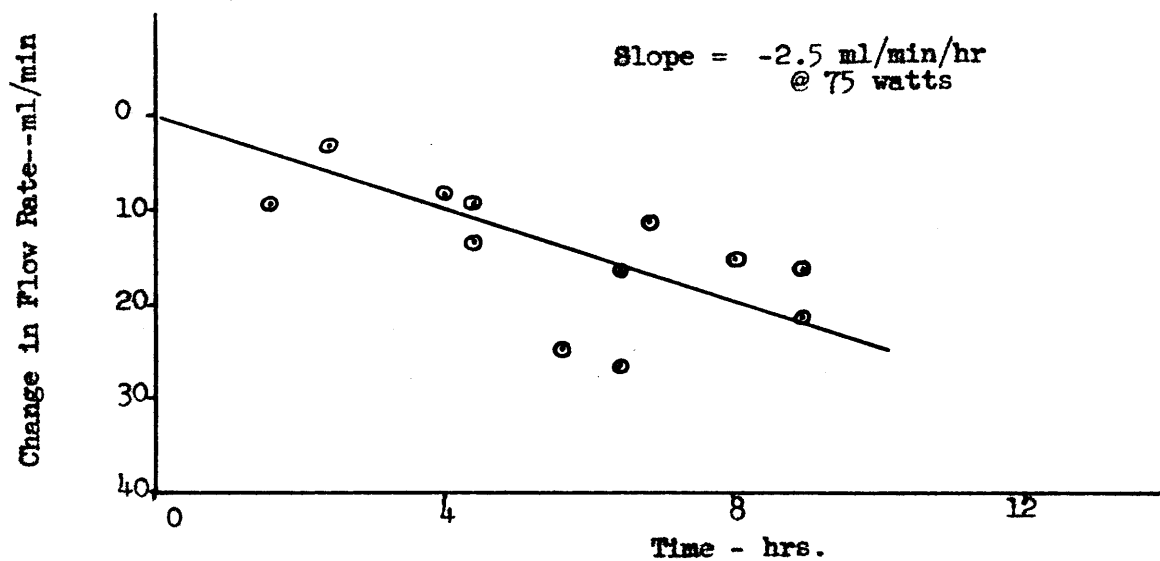


FIGURE 6.5. Change in flow rate when periodic cleaning is employed.

the effect of cleaning the cell by polarity reversal and flushing is apparent. Note that if a decline in flow rate of no more than 5 ml/min is permissible, cleaning is required every 2 hours, or for a decline of 10 ml/min, every 4 hours.

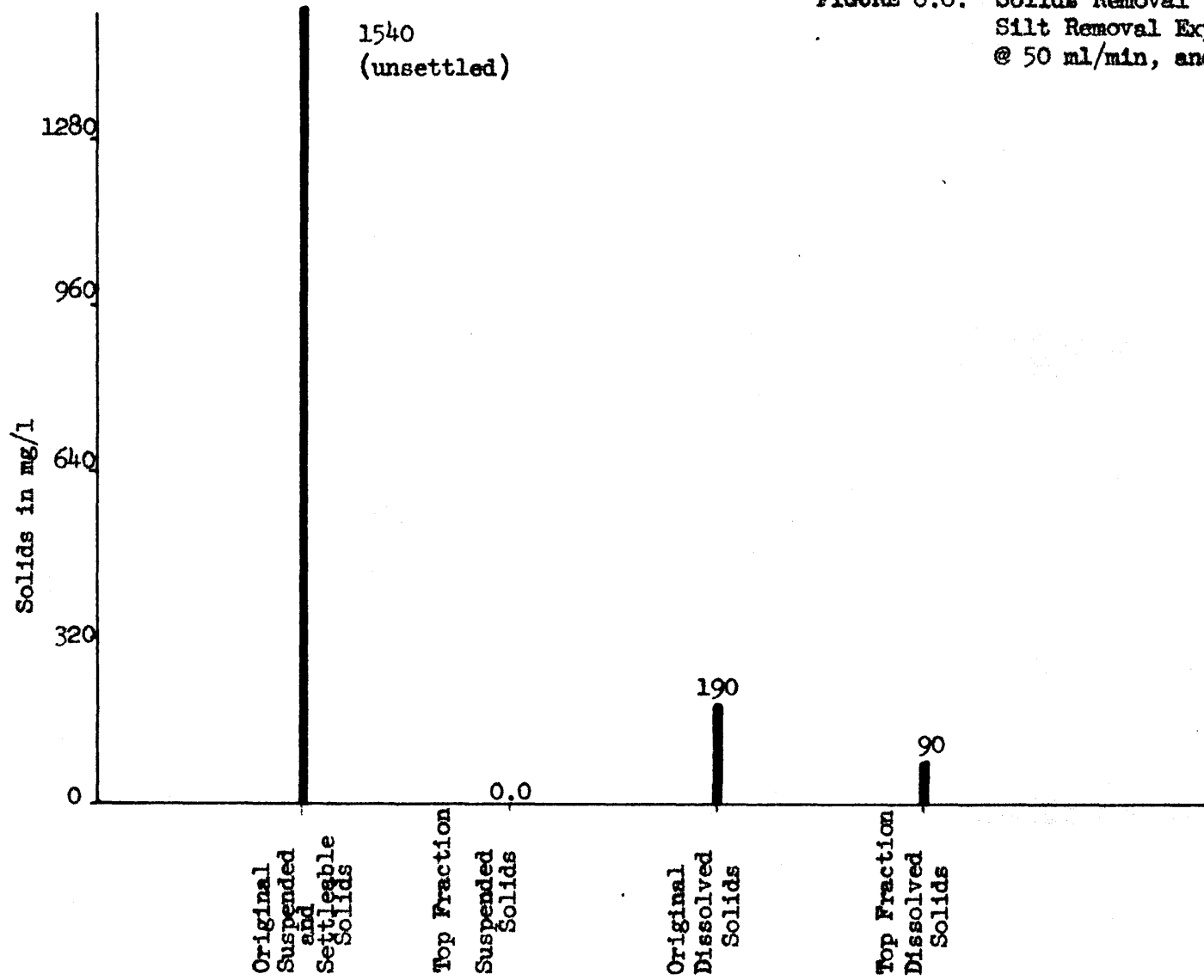
It was found that while the field was reversed, finer colloid material migrated through the filter causing contamination of that fraction with resulting rejection of the top fraction during a cleaning period. Increasing the supply rate was found to speed up the removal of the silt deposit.

Another deleterious effect observed on long runs was the deposit of a scale of calcium on the cathode end of the buffer compartment. Although this caused no trouble in operation and was also removed to some extent by the cleaning operation previously mentioned, it illustrates the fact that with water of moderate to high hardness, scaling problems can be expected to affect the life of membranes and filters and be a factor in maintenance requirements.

6.4 Results

Examination of water samples was primarily by spectroscopy with calibration against the Jackson Candle apparatus. Typical removals are shown in figure 6.6. In all cases, 100% of the suspended material was removed using the Reeves filters.

FIGURE 6.6. Solids Removal for Suspended
Silt Removal Experiments
@ 50 ml/min, and 50 Watts.



Chapter VII

SEPARATION OF ALGAL GROWTH FROM WATER

7.1 Background

Based on the evidence that algae possess a charge and thus behave similar to colloidal silt in an electrical field, a series of experiments were conducted using stabilization pond effluent containing a fairly high concentration of algal growth.

An efficient method of removal of algae from water may prove to be necessary where water reuse or algal concentration is attempted. Oswald (23) has given evidence that sewage grown algae may have some market value if harvested as a food nutrient. It has also been evident that the economic value of the water may be as high as the algal product in arid regions.

Algae have proven both a benefit and a nuisance in sewage and water works for many years. They play a vital role in stabilization ponds used for sewage or industrial wastes but impose an undesirable characteristic of adding turbidity and organics to the effluent.

In water works algae often become a nuisance factor when they grow in reservoirs and lakes used for water supplies. Color and sometimes taste and odor are then evident in the water system.

The method of algal growth removal commonly used is microstraining, a form of filtration. Chemical precipitation is also known to effectively remove them with the normal water treatment. Centrifugation

has also been investigated for large scale use but is probably the least economical due to equipment costs. Turre (28) reported on a microstraining unit installed at Denver which obtained an average removal of all microorganisms of 93%, and other units installed elsewhere which obtained from 62 - 86%. Frequent backwashing (every two hours) was found to be necessary in seasons of heaviest growth.

The author is not aware of any electrophoretic mobility measurements being made on algae, however information has been obtained on viruses, bacteria, and some cells. In size, bacteria fall within the colloidal range previously stated, 5 angstroms to 10^4 angstroms. Viruses will range smaller, and cells usually larger. The green algae, belonging to the euglenoid group, observed in the pond effluent ranged in size from 10^3 to 3×10^5 angstroms (.1 to 30 microns). The mobility of these particles is greatly affected by the pH and ionic strength of the solution and these relationships have been determined for some biological particles. For human red blood cells the mobility-ionic strength found by Furchogott and Ponder (15) varied as shown in figure 7.1, for a pH of 7.3. They also showed a relation between pH and mobility as shown in figure 7.2 The euglenoids could exhibit similar relationships.

7.2 Experimentation

Sewage effluent containing algae was processed with the Bier equipment in a similar manner to the silt experiments. Fresh sample water was obtained frequently and kept in 5 gallon plastic containers.

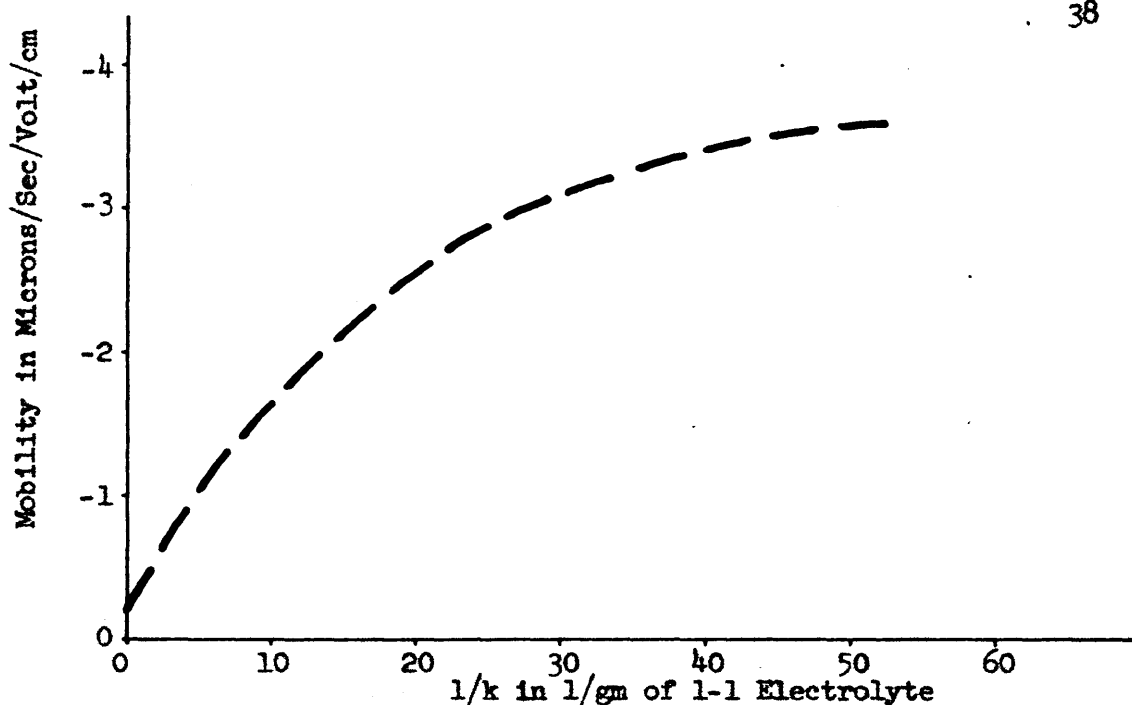


FIGURE 7.1. Mobility of Human Blood Cells as a Function of $1/k$ @ pH 7.3 (from Furchogott and Ponder).

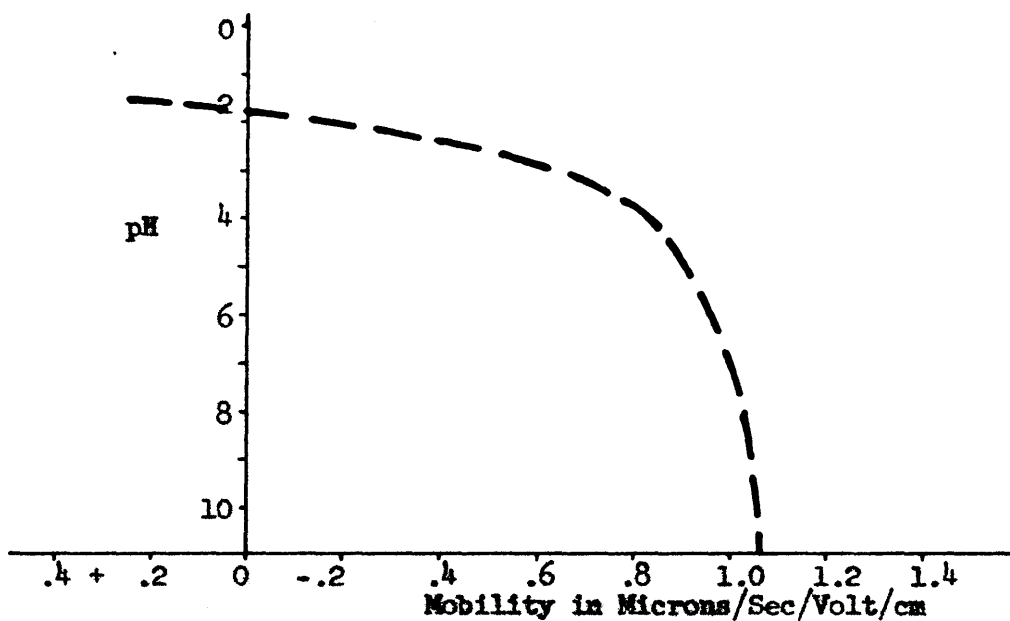


FIGURE 7.2 Mobility of Human Blood Cells as a Function of pH @ 0.172 Ionic Strength (from Furchogott and Ponder).

The fact that this water contained both live and dead cells in variable quantities did not cause a significant variation in results.

The flow system was altered by elimination of the gravity or pumped supply flow which was replaced by a pressurized flow. This procedure allowed the use of higher flow rates but changed the theoretical operation of the cell considerably from what has been previously described. As with the silt, the concentrated fraction could be returned to the supply or discarded.

7.3 Operational Considerations

It was found that algae could be concentrated with the equipment by two methods: (1) by repeated processing of the bottom fraction, or (2) by stopping the bottom flow and allowing buildup within the cell pack. In the latter method the concentrated algae was scrapped from the membranes upon disassembly of the cell pack.

Early experiments showed that algae could be removed from the solution only at low flow rates using the filters previously employed (Gelman type and Reeves PVC). Clogging of these filters was unavoidable and resulted in permanent damage to them because they could not be completely cleaned by any method.

Millipore filters with 0.45 and 3.0 micron pore sizes gave much better results. These filters allowed no passage of algae at higher rates, but again were quite susceptible to clogging. However, the flushing of the input compartments and polarity reversal revived their efficiency.

When pressure in the range of 5-15 psig was applied to the supply flow, the millipore filters were quickly ruptured. In order to provide support, a sheet of Eaton-Dikeman dacron paper, grade #3100 was placed behind each filter. This material has outstanding tensile strength and worked well in this situation.

If the Bier cell was operated with no electrical current and with the bottom fraction pinched off, the operation very much resembled a filter press. Under such conditions a reduction in flow, dropping to one third of the initial flow, occurred in only a few minutes. Upon discharge of the electrical current through the cell pack a noticeable increase in flow rate was observed. Usually the initial flow rate was again obtained. Since the applied pressure has increased the flow velocity much higher than the limit for balance against electrophoretic migration, this phenomenon must have another explanation.

One explanation is that the electrical current follows the path of least resistance which is through the pores of the filter rather than transferring through the filter structure or algal cells. In doing so, the effective area is reduced to about 10% at the filter from that in the compartments. Therefore at the filter, the current density is greater and forces the algal cells away from the pores thus clearing them. In figure 7.3 the effect of periodic off-on switching of the voltage is quite apparent. It was noted that the flow rate decreased to some extent even though the voltage was on, but that it stabilized with time.

In order to show a relationship between the clogging of the

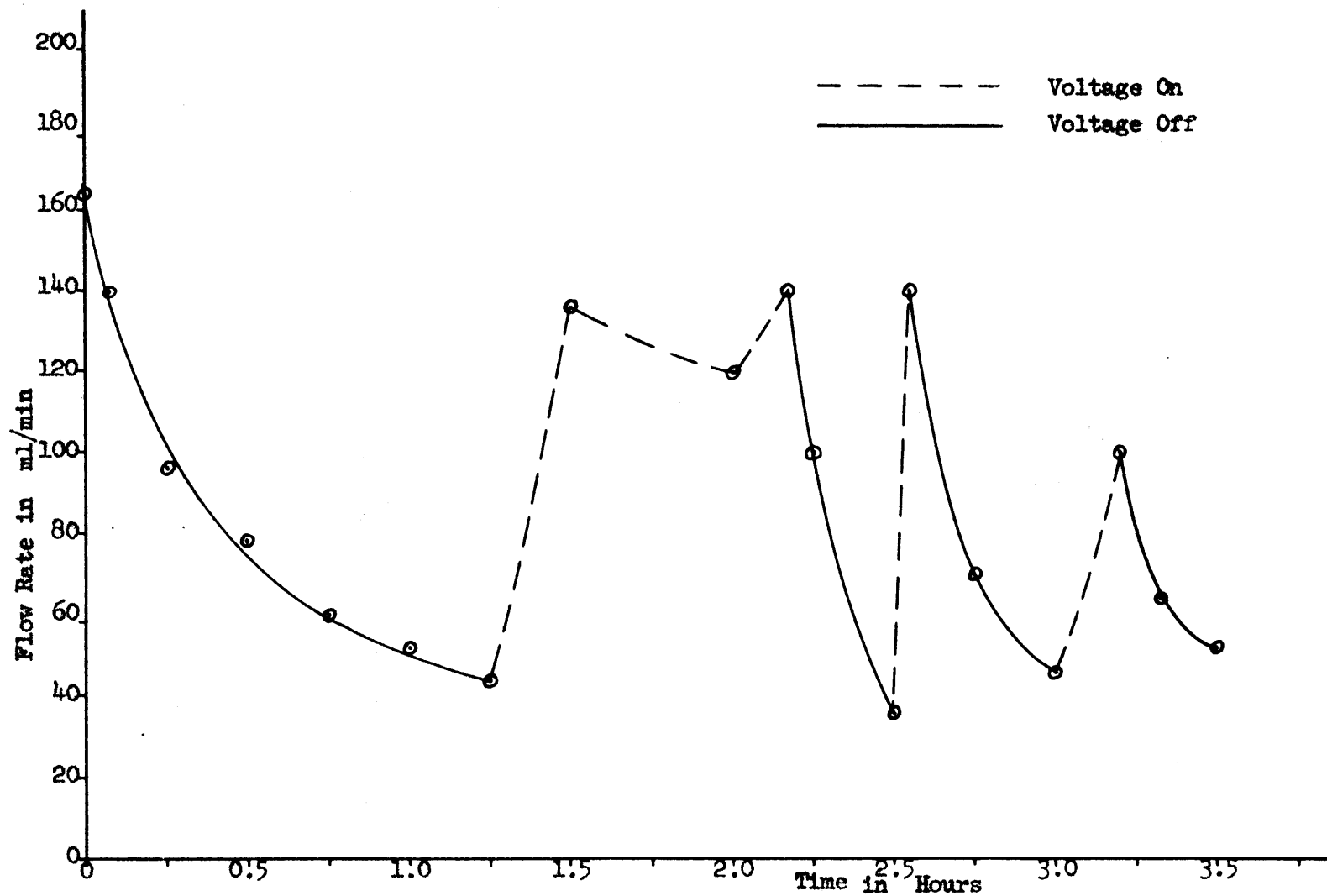


FIGURE 7.3. The Effect of Periodic Off-On Switching of Voltage on Flow Rate for Algae Removal.

filters, the applied voltage, and time, the author compared the cell to a soil permeameter and assumed analogous parameters. By Darcy's law:

$$q = K I A$$

$$\text{or } K = Q L / t h A$$

where K is the permeability coefficient ,

$q = Q/t$, the volume of flow per unit of time ,

$I = h/L$, the head loss per unit of distance ,

and A is the cross-sectional area .

If the resistance to flow through the cell is expressed as:

$$R = 1 / K = t h A / Q L$$

$$\text{or } R_v = (A h / q L) \quad (7.1)$$

where R_v is the resistance to flow in cm/ ml/ min. at the voltage V measured in volts/cm.

A is the cross-sectional area in sq. cm.

h is the head loss measured in cm.

L is the cell thickness in cm.

q is the flow rate in ml / min.

v is the voltage setting in volts / cm.

In an electrical field, this resistance will be slightly reduced due to electroosmotic flow. If R is evaluated for this factor alone and subtracted from the total calculated resistance, that

which is due to the clogging alone may be found. Any resistance to flow contributed by constrictions in tubing and entrance losses would also be included in the electroosmotic factor.

The resultant equation may then be written as:

$$\Delta R_v = R_v - R_v^1 \quad (7.2)$$

$$\Delta R_v = A h / q L - R_v^1 \quad (7.3)$$

where ΔR_v is the resistance due to clogging

R_v^1 is the resistance due to electroosmotic
flow and frictional factors.

By selection of four different voltage values and measurement of necessary parameters, this relationship was evaluated and is presented in figure 7.4. The stabilization of the resistance and thus the flow is clearly shown for each voltage. After a long period of time the situation may be disrupted due to the influence of some other factor such as slime formation or a sludge buildup on the opposite membranes adding constriction to flow. The shape of the curves would probably be different for other materials or other concentrations of the same material.

7.4 Results

Variation in the removal of the algal growth occurred with the pressure applied, the type of filter employed, and the applied voltage. Results of the investigation of these parameters are shown in table 7.1. It may be noted that the larger pore size filters gave good results at low flow rates but had to be replaced by finer material for the higher rates.

As the pore size of the filter was reduced, greater efficiency was still obtained because the electrical field prevented the pores from clogging.

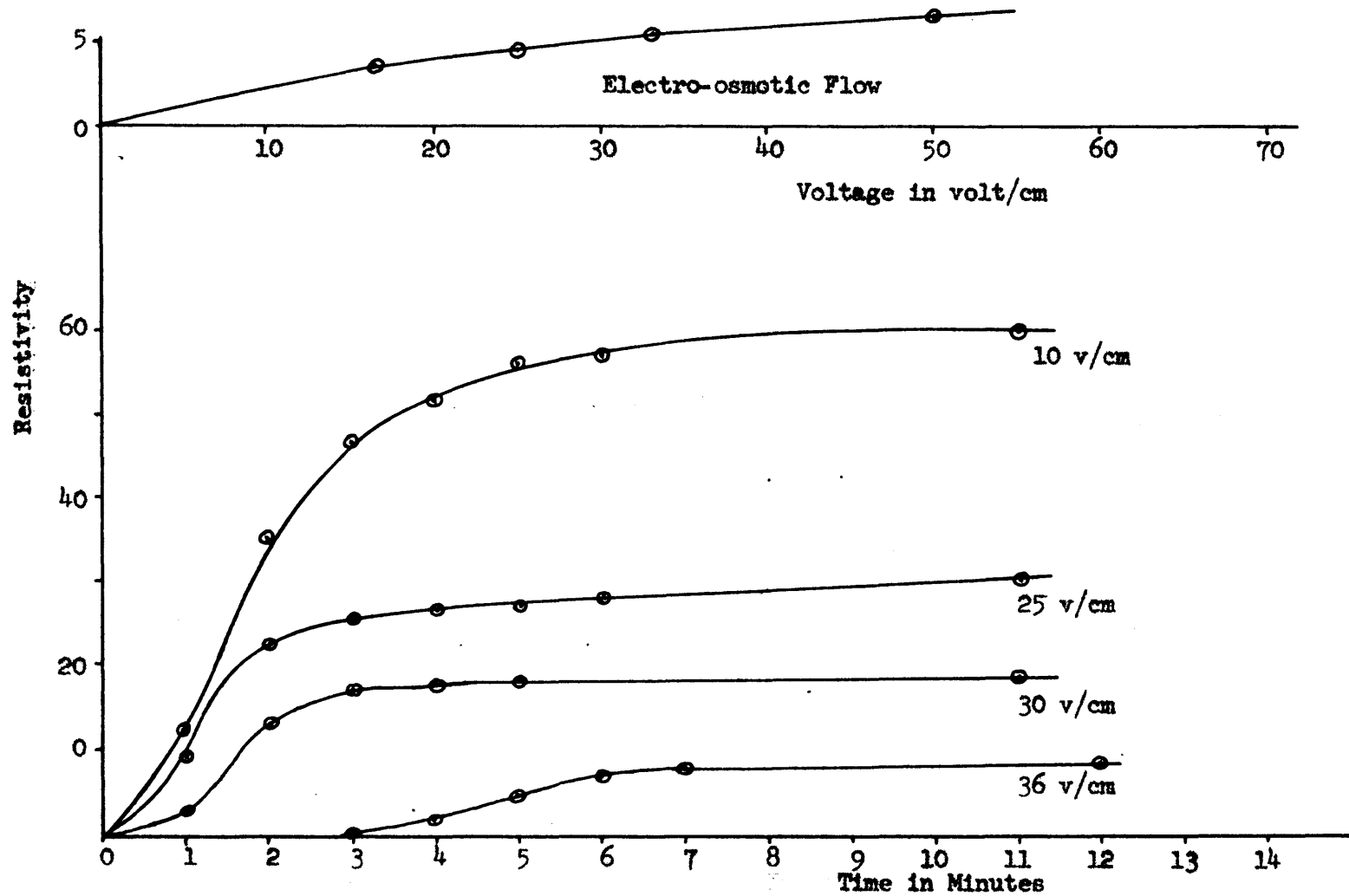


FIGURE 7.4. Time-Resistance Relationships for Clogging Due to Algal Cells.

TABLE 7.1

Results of Algal Removal and Concentration Experiments

<u>Removal</u>			
Method	Flow rate in ml/min	Power in watts	%algae remaining
Reeves PVC filter (5 microns)	20	100	0.0
Reeves PVC filter (5 microns)	75	108	0 - 10
Reeves PVC filter (5 microns)	135	100	20 - 30
Gelman filter, weak base (5 microns)	75	100	0 - 10
Millipore filter (0.45 microns) w/dacron support @ 5 psig.	70	110	0.0
Millipore filter (3.0 microns) w/dacron support	25	68	0.0
Millipore filter (3.0 microns) w/dacron support @ 5 psig.	90	100	0 - 10
Gelman "Versapor" filter @ 5 psig.	85	300	0.0
Gelman "Versapor" filter w/o pressure	50	150	0.0
<u>Concentration</u>			
Method	Flow rate in ml/min	% solids, dry basis	
Recycling, one time @ 150 watts	50	0.08	
Recycling, two times @ 150 watts	50	0.12	
Recycling, three times @ 150 watts	50	0.14	
Recycling, four times @ 150 watts	50	0.21	
Buildup, 10 hours @ 150 watts	50	70 - 80	

Chapter VIII

SEPARATION OF DISSOLVED ORGANICS FROM WATER

8.1 Background

Taste, odor, and color often result from the presence of organic contaminants in a water source. They may be naturally occurring or man-caused, and their removal may be a very real problem, particularly if they are of a refractory nature or unaffected by conventional treatment methods. Many organics are bio-degradable, however some are resistant or only slightly affected by biological processes. Other methods of removal such as solvent extraction, ozonation, adsorption, chlorination, and chemical coagulation have been used with some success but do not approach 100% removal of the thousands of organic materials appearing in our water, most of which are unidentified.

Numerous investigators have analyzed tap waters and river water sources for high-molecular weight organic materials. Bunch and others (13) reported secondary sewage effluents containing a chemical oxygen demand, the usual measure of organic pollution, from 55.6 to 145.6 mg/l and detergents alone ranging from 1.5 to 12.5 mg/l. Similar figures would be found for heavily polluted river waters. Black and Christman (7), while investigating the characteristics of colored waters, found COD's from 24 to 142 mg/l and standard color units varying from 68 to 424. They determined that materials responsible for color were primarily colloidal and less than 10 millimicrons in particle

size. This was proven by the reduction of zeta potential during coagulation tests indicating an initial possession of the surface charge.

The field of synthetic detergent investigation has been extensive. Due to its non-biodegradability, well discussed by McKinney and Symons (20), it is an extremely great nuisance factor, both to treatment plant operators and the household water-user. However the newly developed degradable detergents will soon reduce this problem. The extent to which synthetic detergents, in particular ABS, were affected by an electrical field was not known and therefore several experiments were made using the electrophoresis equipment.

Phenolic compounds quite often remain unaffected by such treatment processes as filtration and chemical coagulation but have been removed by ozonation, adsorption, or solvent extraction. These processes, however, are expensive. Extended bio-oxidation also has had some success. Nenich (22) stated that biological removal of phenols from wastewater was significantly affected by the ammonia content, pH, temperature, and the uniformity of the feed wastewater. Phenolics also vary enormously in the amount of odor, color, and taste due to the many types of compounds possible. For instance reduction of phenolics by chlorination only results in many new forms of chloro-phenolic compounds.

8.2 Operational Considerations

The experimentation with dissolved organics differs from the silt and algae removal tests for several reasons. First there was no problem of clogging. In fact, there existed an opposite problem. Due

to low molecular weight of some pollutants, they easily diffused through the membranes separating adjacent cells, causing reduction in removal efficiency. The problem was that of finding the best possible filter-membrane combination. As with previous contaminants, this would be the point of balance of migration and flow velocities. It was found that the exact point could not be discovered due to leakage across membranes. Although the membranes employed would only diffuse a negligible amount under static conditions, this diffusion greatly increased with the application of electrical current.

Selection of the best operating conditions for a particular filter-membrane consisted of conducting runs with either constant power and variation of flow rate or constant rate of flow and variation of power. The percent removal of contaminant was measured for either case. If the first case was chosen, increasing power settings were used until a family of curves relating flow rate and percent removal were obtained. By choosing a limit for the percent of organic remaining in solution, a single curve of power against flow rate was plotted.

8.3 Results

With reference to figures 8.1 and 8.2 for ABS removal, one can note the increasing difficulty in obtaining removal with rise in flow rate. The flow rate of 10 ml/min corresponds to a loading of 1.5 gal/hr/sq.ft. In this case the filter employed was the Whatman #54 and the membrane was standard visking dialyzing tubing. The efficiency was increased by 24% by using a battery separator material from Gelman Instrument Co., in place of the Whatman filter, and further increased by 30% using the E.S.B. Reeves PVC filter material.

Similar studies for 2,4-dinitrophenol, shown in figure 8.3, gave best results with the Reeves filter.

Of the artificial solutions tested, xylene cyanol proved to be the most difficult to remove. Removal required more than 10 times the power needed for 2,4-dinitrophenol at the 90% level. (See figure 8.4).

Summaries of operating conditions for 90% removal of various contaminants investigated are listed in Table 8.1. Since the Reeves filters gave the best results for ABS and 2,4-dinitrophenol, they were used for all other tests.

Dissolved in sewage effluent, the dinitrophenol was removed, but a much higher amperage and more power were required. Whereas the artificial solution in tap water yielded power requirements of 0.15 amps at 150 volts, in sewage this was increased to 0.90 amps at 150 volts. This higher amperage was due to the greater salt content of the sewage effluent. There was also a 53% reduction in spectral absorption for the sewage effluent itself.

Similarly, very little power was required for removal of the contaminant from the Kitt Peak water (see figure 8.5). It had a very low salt content, being derived from precipitation. These results point out that the salt content has a very important effect on the cost of the operation. This is discussed further in Chapter 9.

The Kitt Peak water is probably an excellent example of an area of usage for this method. Since the water contained only a trace of suspended solids, was low in salts, but high in colored organics, good

efficiency of operation and excellent efficiency of removal should be obtained in large scale production. Although qualitative tests were not run on this water for positive identification of the organic contents, there was strong indication that they were phenolic compounds and thus have been expressed as an equivalent phenol concentration by spectral methods. Figure 8.5 shows the spectral curves of the Kitt Peak water as compared to other phenolic compounds.

For liquified phenol, even at a low concentration of 1 mg/l, no removal was observed. This was expected and agrees with the assumption that the molecular weight was too small and the organic ion possessed little or no zeta potential in an aqueous solution. This observation provides evidence that forced-flow electrophoresis will not remove every organic found in a waste-water. The author suggests that specific tests be made using the actual waste-water to be treated to see if the desired result can be obtained. Certain industrial wastes may be considerably reduced in COD loading to be imposed on a municipal treatment plant or stream if one or two organic refractories were removed.

Figure 8.6 shows a comparison of the electrophoretically processed Kitt Peak water to attempts to filter colored matter out. These results were highly significant.

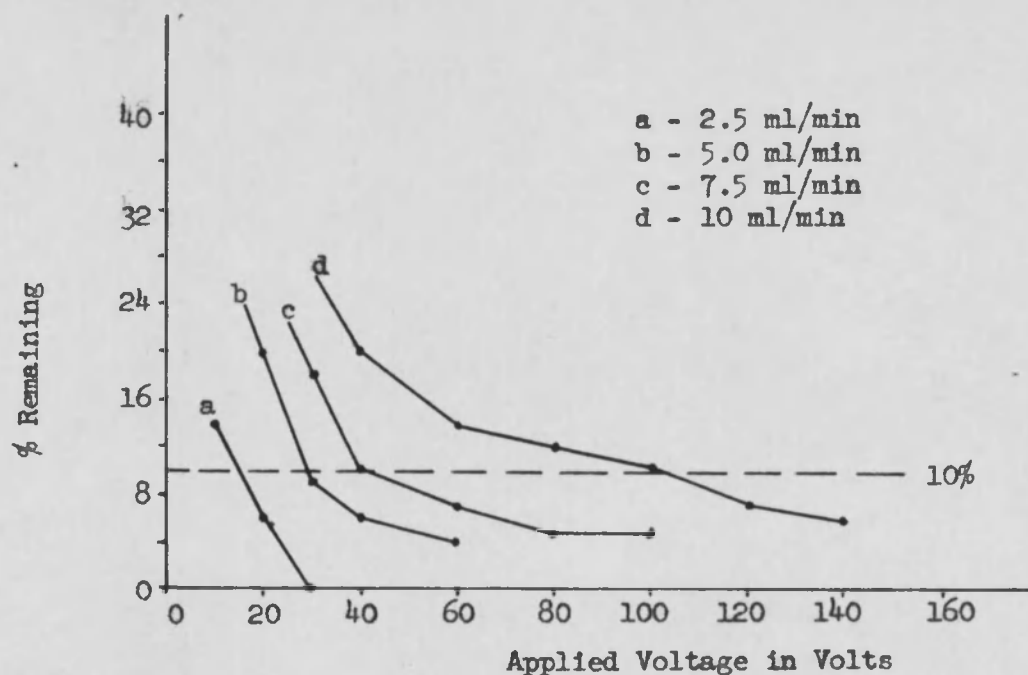


FIGURE 8.1. Variation of % Remaining with Applied Voltage for Removal of ABS.

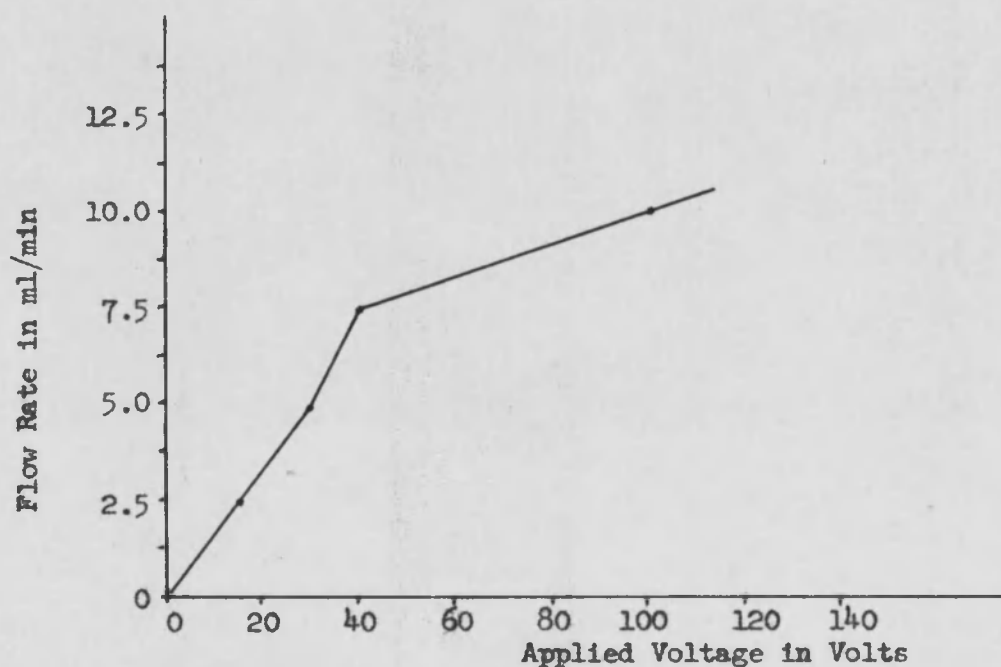


FIGURE 8.2. Variation of Flow Rate with Voltage for 90% Removal of ABS.

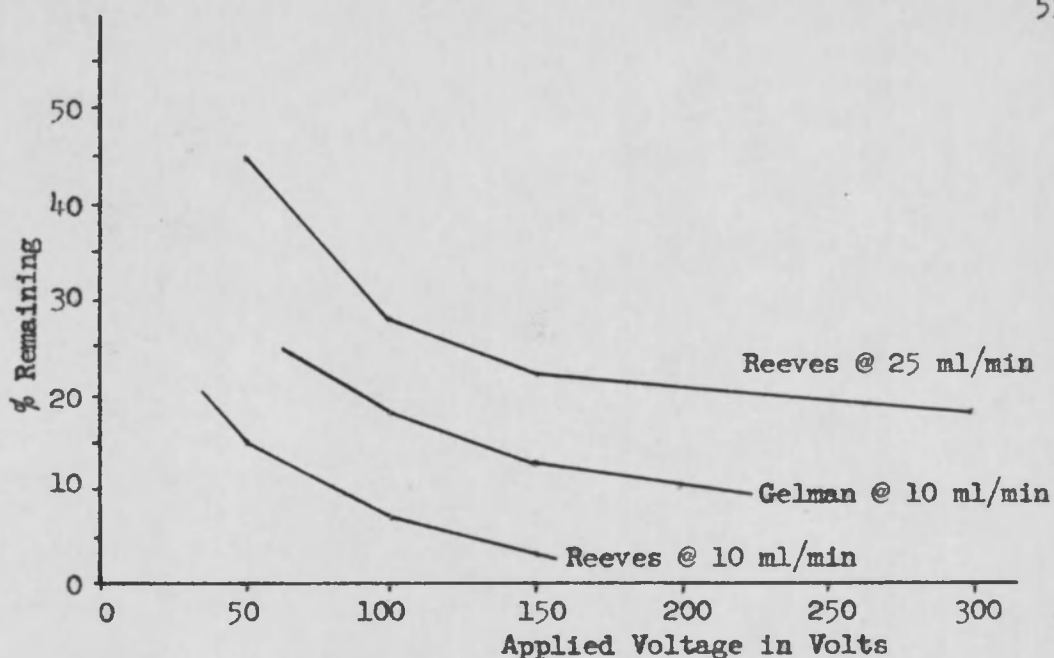


FIGURE 8.3. Variation of % Remaining with Flow Rate and Applied Voltage for 2,4-Dinitrophenol (10 mg/l).

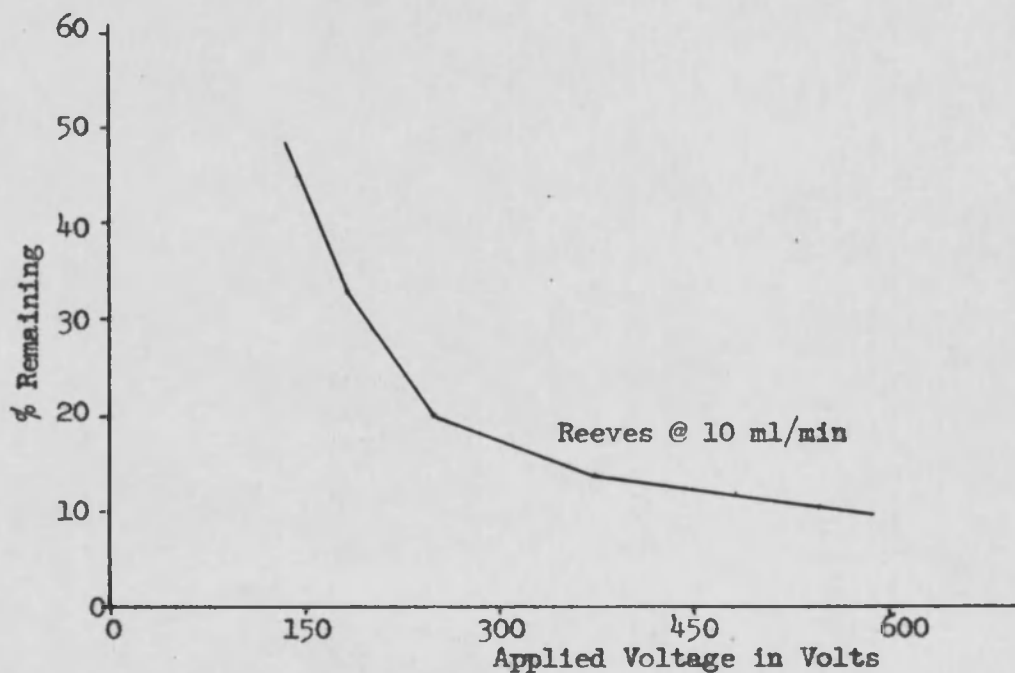


FIGURE 8.4. Variation of % Remaining with Applied Voltage for Xylene Cyanole (10 mg/l).

TABLE 8.1

Results of Organic Removal Experiments (90% removal)

Contaminant	concentration in mg/l	filter	flow rate in ml/min	voltage in volts	power in watts	specific resistance in ohm cm.
A B S	50	Whatman 54	10	105	51	1850
A B S	50	Gelman	10	80	40	1850
A B S	50	E.S.B. Reeves	10	50	30	1850
2,4-dinitrophenol	10	Gelman	10	200	36	1800
2,4-dinitrophenol	10	E.S.B.Reeves	10	40	2.5	1800
2,4-dinitrophenol in sewage effluent	10	E.S.B.Reeves	10	230	270	1000
xylene cyanole	10	E.S.B.Reeves	10	500	150	1800
Kitt Peak water	Orig.	E.S.B.Reeves	50	100	2.0	15,600
tetrachlorobis- phenol	10	E.S.B.Reeves	10	150	15	1800
liquified phenol	1.0	E.S.B.Reeves	no removal			1800

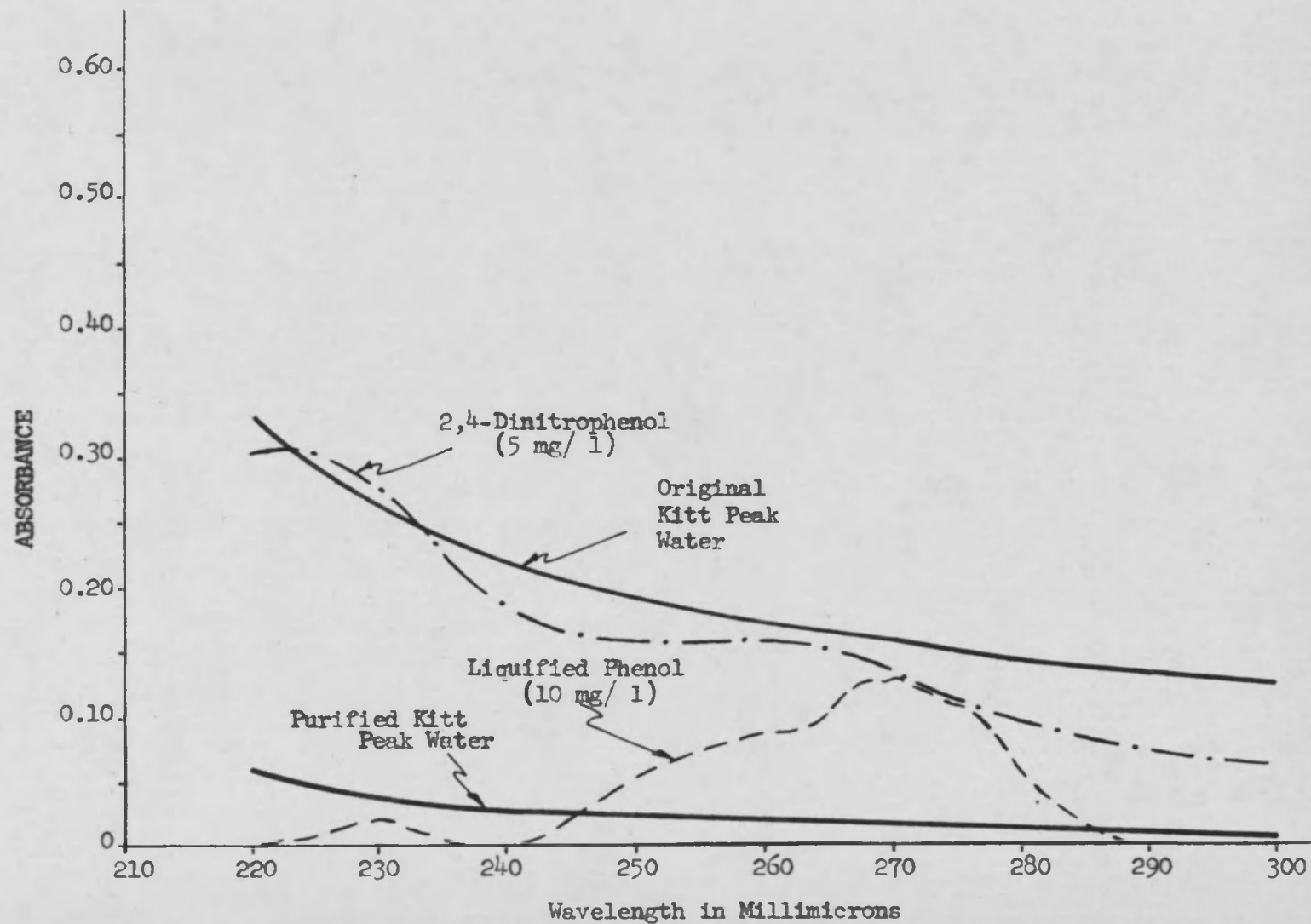
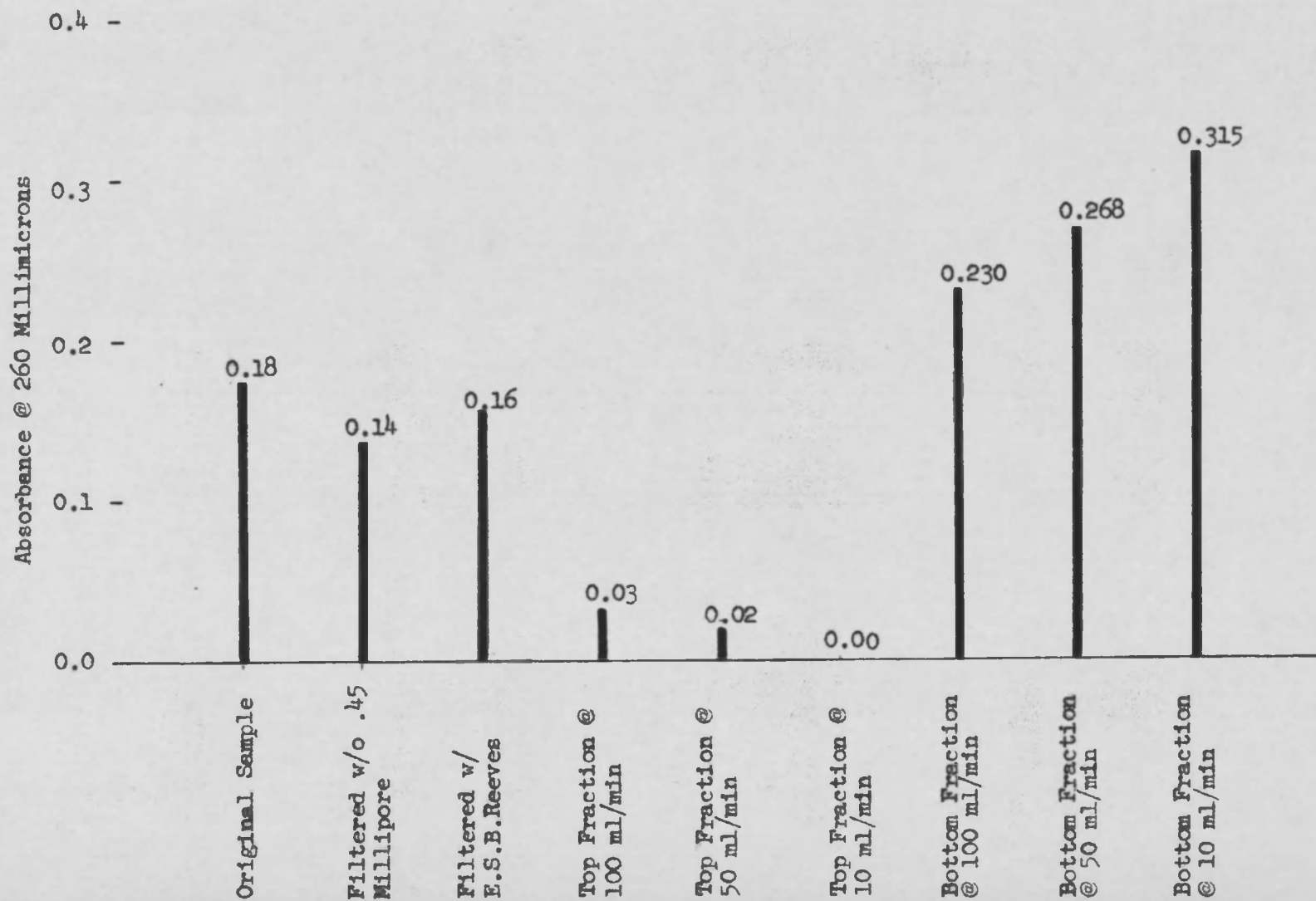


FIGURE. 8.5. Spectral curves in ultraviolet range.

FIGURE 8.6. Comparison of Filtration to Forced Flow Electrophoresis for Kitt Peak Water.



Chapter IX

CONCLUSIONS AND SUGGESTIONS FOR FURTHER STUDY

A very significant point to be made about the cost of treatment by forced-flow electrophoresis is the fact that the salt content or conductivity level of the untreated water has an important effect. Of the water sources used in the experiments, conductivities varied greatly and gave rise to large differences in power consumptions. It may be seen that if the water contains low salt concentrations, it creates a greater resistance to the transfer of electrical current and by Ohms' law requires a lower amperage where voltage remains constant. Since power varies directly as the product of resistance and amperage squared, the cost of operation will be considerably lower when applying the method to waters of low salt content. With data taken from the three areas of investigation, a plot was made of the power consumed against the specific resistance of the solution. (see figure 9.1).

Although no ion selective membranes were employed in the process some desalting was observed. This was indicated by the increase in specific resistance of the top fraction, as shown in table 9.1. Two previous analyses of processed sewage effluents showed similar results. This data is presented in the appendix.

The average cost of removal for the different elements was calculated and tabulated in table 9.1 assuming a cost per kilowatt-hour of power to be approximately \$0.02. Due to the size of the laboratory

model, only about 30% of the power was actually used for removal. The cell pack made up only about one third of the total volume between the electrodes and the remainder was buffer compartments. In any larger installation, where economy would be required, this factor would be increased to about 90% by placing more cells between the electrodes and reducing the percent volume taken up by the buffer compartments. These adjusted figures are listed in the last column of table 9.1.

Although the major part of this study was concerned with the removal of various materials from waste waters, concentration of the contaminants was also investigated and showed some interesting results:

(1) While a concentrated fraction was routinely obtained with each pass through the cell pack, the number of repeated passes became too large for concentration of algal cells on an economically sound basis.

(2) In using the cell compartments as concentration chambers for algal cells, processing times were increased due to loss of effective filter area, or if this were to be prevented, required continual increase of power.

(3) For the concentration of organics, the process was satisfactory only when the initial solution was already reasonably high in concentration.

As a result, the application of the method to solutions of low concentration is poor. Since 5 passes should be required to obtain a concentration 10 times that of the original, a concentration of only 10^{-3} would require 15 passes to raise it to 1.0. This is undesirable

from the standpoint of time and the large initial volume required for processing. However modifications of the equipment could make it highly advantageous for certain types of concentration. This could be accomplished by reducing the volume of the concentrated fraction, for example, by using a stagnant compartment into which the material is forced rather than forcing the water out of the concentrate. Also an adsorption surface could be placed in front of the electrode.

The results of this study indicate two areas of encouraging use. The method can be an efficient, economical means of converting flood runoff water to clean water suitable for municipal use or direct ground-water recharge. The employment for removal of organics causing tastes, colors, and odors is highly recommended both for the economy and automatic control of the operation.

There were many problems brought about by this study which require further investigation:

(1) Under conditions where high efficiency is required the problem of leakage across membranes is important. This rate of leakage is known to be related to the loading rate and the voltage potential and is also a resultant of simple diffusion. Better membranes, if they can be found, may solve this problem.

(2) The use of a two-step process may yield higher removal efficiencies at lower costs. It may also be possible to introduce ion selective membranes into one of the steps to reduce the salt content. This would be highly advantageous when processing sewage effluent.

(3) Design factors and control features can be better obtained from scale-up or pilot plant studies.

(4) Applications of the method are numerous and many are yet to be explored. Very likely separation of oil emulsions, colored chemical wastes, or activated carbon-adsorption mixtures could be accomplished.

(5) Concentration of contaminants for analytical purposes are possible and should be studied. Monitoring virus and insecticide content of drinking water represent two techniques that might be developed using a method of preparatory electrophoresis.

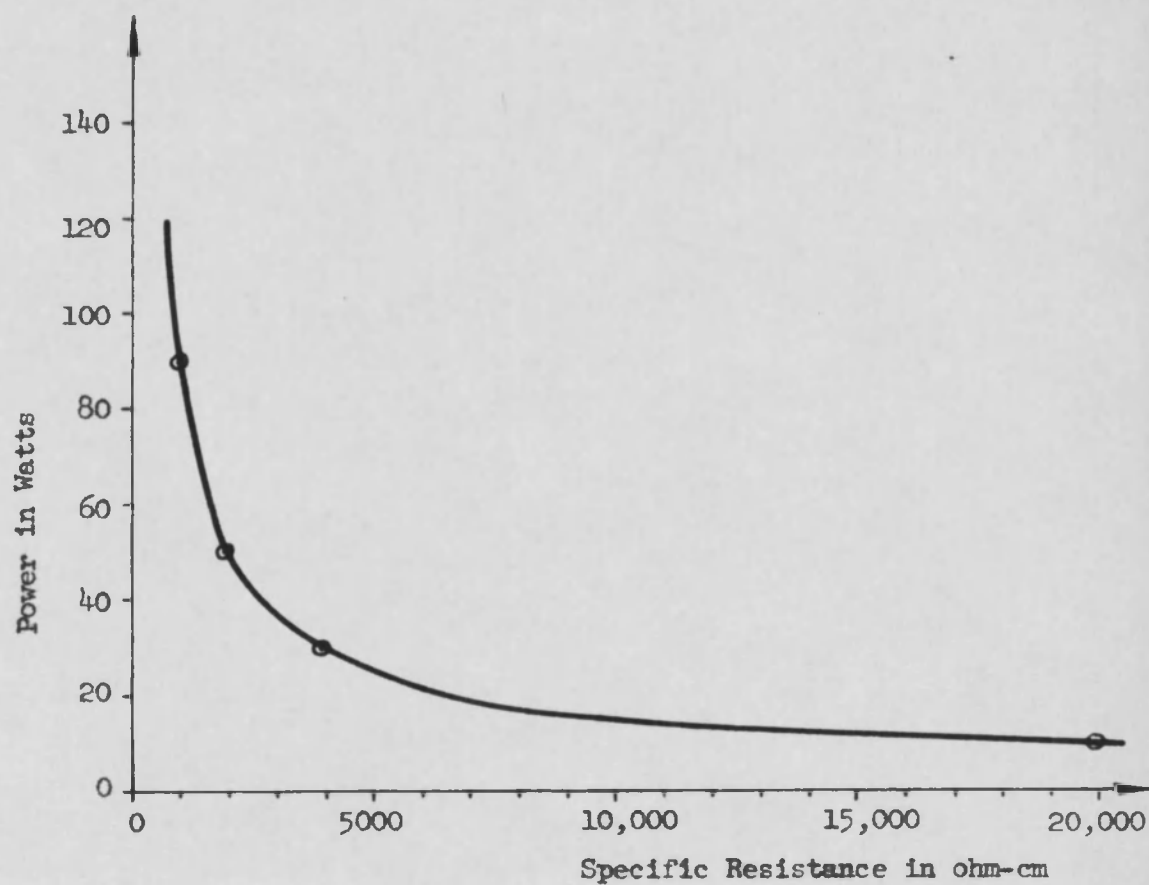


FIGURE 9.1. A Plot of Power Consumed vs. Specific Resistance of the Colloidal Solution.

TABLE 9.1

Cost-Benefit Ratios And Desalting Effects

	Orig. Sp.Resis.	Final Sp.Resis. ¹	Power In Watts	Rate In ml/min	Cost/1000 gal. Produced	Projected Cost/ 1000 gal. Produced ²
Colloidal Silt Separation	4,000	15,000	50	50	\$1.24	\$ 0.41
Algae Separation (sewage effluent)	900	2,200	165	64	3.08	1.03
ABS (tap water)	1,850	10,000	15	10	1.86	0.62
Kitt Peak Water	15,600	67,000	2	10	0.24	0.08

1. Top Fraction

2. Increase From 30% to 90% Power Efficiency

APPENDIX

The following data was taken from two personal reports on treatment of sewage effluents by forced-flow electrophoresis.

1. Treatment of activated sludge effluent. Analysis by Infilco, Inc., division of General American.

Assay	Original	Top Fraction	Bot. Fraction
pH	7.3	8.6	8.5
CO ₂	20	---	---
Cl ⁻	114	32	112
SO ₄ ⁻	140	80	260
Total Alkalinity	234	102	316
Ca ⁺⁺⁺	184	40	128
Mg ⁺⁺	26	2	10
Turbidity	8	1	5
Color	15	3	19
Total PO ₄ ⁻⁻⁻	7.8	1.3	7.3
C O D	45.5	22.8	68.3
Specific resistance	8000	10000	6000

2. Treatment of synthetic sewage consisting of phenol gelatine, NaCl, ABS, and calcium nitrate. Analysis by Taft Sanitary Engineering Center.

Assay	Original	Top Fraction	Bot. Fraction
Phenol	5	4	5
A B S	5	1	10
Kjeldal N	34	4	64
Cl ⁻	48	21	70
Na ⁺	30	19	89
Ca ⁺⁺⁺	28	3	6

3. Treatment of sewage effluent. Analysis by Taft Sanitary Engineering Center.

Assay	Original	Top Fraction	Bot. Fraction
C O D	112	57	153
Cl ⁻	48	52	93
Kjeldal N	19	6	16
Ammonia N	14	6	13
Na ⁺	68	43	129
K ⁺	19	7	18
A B S	10	5	15
Anthrone	3	1	2

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