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THE USE OF CELLULOSE FIBER FILTER AIDS AS PRECOATS IN THE FILTRATION OF COAL LIQUEFACTION INTERMEDIATES

A Thesis

Presented for the

Master of Science

Degree

The University of Tennessee, Knoxville

Harold T. Conner, Jr.

August 1978

136325

ACKNOWLEDGMENTS

The author wishes to express his appreciation to the Materials and Systems Development Department, Gaseous Diffusion Development Division, Oak Ridge Gaseous Diffusion Plant, Union Carbide Corporation and the University of Tennessee which made this investigation possible.

The writer especially wishes to express his appreciation to his major professor, Dr. D. I. Dunthorn for his guidance, encouragement, and patience throughout the course of this work.

The author would like to express his gratitude to C. G. Jones, J. H. Pashley, H. E. Trammell, and J. R. Merriman who allowed me the resources to perform this work and offered needed encouragement.

The author wishes to express his appreciation to the personnel of the K-1413 development facility for their patience during the course of this work. A special thanks is extended to W. E. McLendon for his help in supervising construction of the experimental apparatus and to T. H. Hamilton for his assistance in designing and providing the required instrumentation for the experimental unit. The writer also wishes to thank K. H. Cox for constructing the system and J. Fowler for adding the required instrumentation and control system, and to other mechanics who provided valuable assistance when required.

The author would like to extend his appreciation to M. E. Mitchell for examining the environmental impact of this work and providing disposal procedures and to T. C. Helms for providing needed information regarding industrial hygiene requirements.

The author wishes to thank Dr. Huffman of Southern Services, Catalytic Inc., for providing the material for testing and to B. W. Wright for providing the necessary information for sample characterization.

Special appreciation is extended to J. E. Attrill of the Oak Ridge National Laboratory for all sample analyses.

The writer also extends his sincere appreciation to Ms. Jane Higdon for her excellent typing and patience in the preparation of this manuscript.

Finally, the author expresses his deepest appreciation to his wife, Ann, for her encouragement and understanding during the course of this work and to his two sons, Harold and Brian, for their understanding.

ABSTRACT

A process is developed for removing the small micron-sized ash and unconverted coal material from a liquefied Emery coal. The separation is effected through constant pressure filtration using cellulose fiber filter aid as precoat. Depending upon the process and the application, the solids must be removed to avoid high sulfur concentrations in the coal-derived liquid, to minimize erosion and corrosion of equipment, and to prevent fouling and poisoning of catalysts in downstream processing, or sludge formation in storage or transportation. A fractional factorial experiment is developed for four variables and used to determine the best combination for removing the unliquefied coal and mineral matter from the liquid product. Some further tests are performed to explore the trends shown by the fractional factorial and find approximately optimum conditions for the filtration. This optimum is shown to be of interest when compared with other separation studies on an economic basis.

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CHAPTER I

INTRODUCTION AND SURVEY OF PREVIOUS WORK

A. INTRODUCTION

All coal liquefaction processes must face the problem of separating unliquefied coal and the mineral matter that entered the process as part of the coal from the liquid product stream. Separation is difficult because the liquid is highly viscous; the solids have extremely small particle sizes; and the density difference between the solid and the liquid is small. Depending upon the liquefaction process and the application, the solids must be removed to avoid high sulfur concentrations in the coal-derived liquid, to minimize erosion and corrosion problems in associated equipment, prevent fouling and poisoning of catalysts in downstream processing, or sludge formation in storage or transportation. The liquid-solids separation procedure depends upon the nature of the liquefaction process, the coal, the fuels to be produced and the allowable solids level in the products. Separation techniques[8] currently under study include filtration, centrifugation, hydrocloning, vacuum distillation, and agglomeration and settling.

In most of the coal liquefaction processes which are in various stages of development, rotary drum filtration[8] with precoat has been the preferred method for solids removal for two reasons: Precoating of the filter surface with finely divided filter aid materials enhances removal of suspended mineral matter and undissolved coal from liquefaction intermediates; and by maintaining thin cakes on the filter surface,

longer filtration cycles result. However, numerous mechanical problems[8] have resulted from this equipment due to the increased temperatures and pressures required for coal liquefaction processes.

Detailed economic information for the various liquid-solid separation techniques in coal liquefaction processes is limited; however, some general comments can be made. Precoat filtration, although advantageous from a solids removal aspect, represents the greatest capital cost[8] of any separation method. Centrifuges, hydroclones, vacuum stills, and settling basins require lower initial capital investment but do not provide satisfactory product clarity. These methods may be useful as preliminary solids concentration steps prior to filtration.

Rotary drum precoat filtration appears to be the method chosen by investigators for accomplishing the liquid solid separation, and the economics of this procedure should be explored. Some preliminary cost information[8] for a rotary drum precoat filter is given by researchers at the Oak Ridge National Laboratory. Their preliminary investigations revealed that the filtration step represents a sizable portion of the total process cost for a 23,000-bbl/da facility. An overall filtration rate of 6 gal/hr/ft^2 was assumed in this study, and the filtration cost was estimated as approaching $1.00 per barrel of oil produced. Coal liquefaction process costs, to be economically justified, must be competitive with imported crude oil prices[29, 25] of approximately 8 to 15 dollars per barrel; and thus filtration would represent 6 to 13% of maximum production costs. Precoat filter aid material itself was valued at $0.06/lb, and the usage rate corresponded to 5% of the solids in the prefiltered oil or about 0.25 to 0.3 pounds per hour of filter aid per square foot of filtration surface. The cost of precoat was estimated at

12.5% of the total filtration cost and thus would represent 1 to 2% of total production costs. If "body mix" were required, that is, addition of filter aid to the slurry prior to filtration with subsequent precoating of the filter surface, this cost could double; and a body mix may be necessary to prevent blinding of the precoat by colloidal particulates. Obviously, an improvement in filtration rate and/or filter aid usage would have a significant impact on the process economics.

In a report[14] issued by the Oak Ridge National Laboratory for lab-scale filtration of a Solvent Refined Coal (SRC) liquefaction process intermediate, an equivalent of 8.76 pounds per hour of filter aid was used for each square foot of filter area. The solvent refined coal process is a solvent extraction coal liquefaction process. In this process, dry pulverized coal mixed with recycle coal-derived solvent is heated with hydrogen to 815°F and 1000 psig to form a solution which is filtered to remove undissolved residues. The filtrate is vacuum distilled into recycle oil, middle distillates, light oil, and the SRC product oil. An Illinois No. 6 coal was used as feed to the process and contained 3.19% ash. After the precoat filtration, 0.15% ash remained. Johns-Manville Fibre-Flo[13] diatomaceous earth-asbestos filter aid was used in all experiments, and no attempt was made to optimize the usage rate. Even though the ash level was reduced considerably and sufficient clarity was obtained, the economics would be undesirable with such a high usage of filter aid for precoat.

B. LITERATURE SURVEY

Currently there are five major coal liquefaction processes in various stages of development. As background material, these processes

are reviewed relative to the general process steps and methods of separating the mineral matter and unliquefied coal from the product oil, after which a review is given of the general precoat and filter aid literature.

Coal Liquefaction Processes

Under development since 1962 is the Char-Oil-Energy-Development (COED)[8, 15, 26] process by the F. M. C. Corporation. In this process, pulverized coal is heated to successively higher temperatures in a series of fluidized bed reactors (600°F, 850°F, 1000°F, and 1500°F) at 6 to 10 psig. Hydrotreating of the oil yields high grade low sulfur syncrude (synthetic crude oil) which can be converted to gasoline and other products by refining techniques producing about 1 to 1-1/2 barrels per ton of coal. Part of the char produced by the pyrolysis is used to supply heat, and the product gas yield is 8,000 to 10,000 scf/ton of coal at a heating value of 550 to 650 Btu/scf gas. High efficiency separation of solids from the COED pyrolysis oil is required to permit final oil processing by fluid bed catalytic hydrotreatment. Rotary drum precoat filtration is being used to remove the solids. Depending on the coal used, the initial solids concentration ranges from 3 to 17% and solids in the filtrate range from 0.01 to 0.09%. Filtration rates are on the order of 4 to 9.5 gal/hr/ft^2. Relatively low filtration pressures (18 to 23 psig) are used in the COED process, and the filtration is carried out at 350°F. Viscosities are dependent upon the coal used but range from 19 to 140 centipoise at 200°F and 3 to 8 centipoise at 350°F. Greater than 95% of the solids in the prefiltered oil are smaller than 1 micron, indicating the need for precoat. Various grades of Johns-Manville Celite filter aids are used for the precoat, and there appears to be an optimum sized filter aid[28] for each type of coal liquefied.

The Synthoil[8, 15, 20, 38] process developed by the U. S. Bureau of Mines is a direct hydrogenation coal liquefaction process designed primarily for producing a utility fuel and is adaptable to the production of diesel fuel, naphtha, and gasoline. In this process, dry pulverized coal is added to a recycle oil and reacted with hydrogen rapidly in a turbulent flow-fixed bed reactor packed with a pelletized catalyst of cobalt molybdate on silica-activated alumina. The reaction is carried out at about 840°F and 2000 to 4000 psi. A phase separator is used to disengage the gases (H_2, NH_3, H_2S, and H_2O) from the liquid products containing the unreacted solids. The gas stream from the separator is purified to remove NH_3, H_2S, and H_2O; and the H_2 is recycled to the reactor. The liquid product is a desulfurized fuel oil or, at more severe reactor conditions, a desulfurized synthetic crude. About 3 barrels of oil per ton of coal are produced, and the hydrogen consumption is around 3000 to 4000 scf/barrel of oil. Mineral matter, sulfur, catalyst fines, and organic insolubles are removed from the oil by centrifugation and/or filtration. Typical ash concentration before centrifugation is 6 to 8% depending on reactor conditions, sulfur content around 5.5%, and the organic benzene insolubles 2 to 6%. The viscosity at 180°F is 10 to 98 centipoise, depending on reactor operating conditions and type of coal used. Centrifugation reduces sulfur levels in the product oil to 0.1 to 0.5%; and the centrifuge residues which amount to 12 volume percent contain 55% ash, 14 to 18% organic benzene insolubles, and 31% asphaltenes and oils. Centrifuged product liquids contain around 2% ash, 4% organic benzene insolubles, and 94% asphaltenes and oil. Filtration studies on the Synthoil process product liquids have been limited and were aimed at reducing the level of ash in the centrifuged liquid to that required for

oil turbine fuel, i.e., 0.005%. Filtration rates have been low, between 1 and 4 gal/hr/ft^2,[38] with rotary drum pressure precoat filters using Johns-Manville Celite (diatomaceous earth) precoat. Rates of about 9 lb/ft^2/hr were obtained with a Ronningen-Petter pressure filter with ash levels of 0.002%.

In the H-Coal[8, 15] process by Hydrocarbon Research, Inc., dry pulverized coal in recycle oil is mixed with hydrogen preheated and reacted with catalyst at 850°F and 2700 psi. Reactor products are flashed at atmospheric pressure to give light hydrocarbon, and then flashed at lower pressures to yield vacuum distillate overhead and bottoms. Hydrogen consumption varies from 14,000 to 20,000 scf/ton coal processed, and liquid products vary from fuel oil to syncrudes. About 2 to 3 barrels of distillate and 1 barrel of heating oil, or alternatively 3 to 4 barrels of distillate in the gasoline range, per ton of coal are produced. In the preparation of syncrude by the H-Coal process, the heavy liquid product containing the unconverted coal and ash is charged to a vacuum distillation system where a 650 to 950°F distillate is removed overhead. A slurry consisting of ash and unconverted coal is the bottoms product. Filtration rates for the bottoms product have generally been around 10 gal/hr/ft^2,[5] as in the COED process for a rotary drum precoat filter. Filter aid consumption is 5% by weight of the solids to be filtered. Higher filtration rates have been experienced using solvent dilution of prefiltered oils with paraffins.

The Solvent Refined Coal (SRC)[2, 8, 14, 15, 26] process developed by the Pittsburgh and Midway Coal Mining Company is a solvent extraction coal liquefaction process. In this process dry pulverized coal mixed with recycle coal-derived solvent is heated with hydrogen to 815°F and

maintained at 1000 psig to form a solution which is filtered to remove undissolved residues. Gases from the dissolver are separated, scrubbed, and vented. A portion of the H_2 is recycled. Solids are removed by precoat filtration with a Funda filter (a batch-type pressure filter) using 0.1 pounds of diatomaceous earth per square foot of filter surface. About 0.5 pound of filter aid per pound of prefiltered solids is used for body mix. Filtration is carried out at 150 psig and 350 to 650°F, and typical rates are 12 to 15 gal/hr/ft^2. The filtrate is vacuum-distilled; and the overhead is condensed and fractionated into recycle oil, middle distillates, and a light oil fraction. The still bottoms are solidified and collected in storage bins. The SRC process is designed to produce a fuel that will meet EPA guidelines for sulfur level in boiler fuels (<1%). Most pyritic sulfur is eliminated as is about 60% of the organic sulfur. Ash levels are reduced to 0.1%.

Another solvent extraction coal liquefaction process of interest is the Consol Synthetic Fuel process (CSF)[8, 15] developed by the Consolidation Coal Company. In this process, dry pulverized coal is preheated to 450°F and slurried with a coal-derived hydrogen donor solvent and extracted at 765°F and 150 psig to yield an overhead and a slurry of insolubles and extract which is separated in hydroclones. The extract is fractionated, and the insolubles are carbonized at 925°F. Light oil from the fractionation and the bottoms from the carbonization are catalytically hydrotreated at 650 to 700°F and 1200 psig and distilled to yield donor solvent for recycle, distillate fuels, and product fuel oils. The required hydrogen is generated from the extraction residues. Three methods have been investigated for removal of insolubles: centrifugation, filtration, and hydrocloning. Multiple hydrocloning has

reduced ash contents to about 1% which is still higher than required for low sulfur synthetic crude oil or heavy fuel oil. Serious mechanical problems were experienced with both filtration and centrifugation, and these studies were discontinued.

Liquid-Solid Separation in the Coal Liquefaction Processes

In general, rotary drum precoat filtration has undergone the most extensive development for separation of the mineral residues and organic insolubles from liquefaction intermediates. Other methods such as hydrocloning in the Consol Synthetic Fuel Process, centrifugation in the Synthoil Process, solvent dilution, extraction, and vacuum distillation in the H-Coal process have also been investigated. It may be necessary as in the Synthoil process to combine some of the separation techniques above with rotary drum precoat filtration to successfully clarify the liquefaction product. Except for the lab-scale work at the Pittsburgh Energy Center[38] with a Synthoil process coal-derived liquid, very little attention has been given to optimizing the grade and type of filter aid materials for precoating. In most of the processes, 5% filter aid by weight of prefiltered solids was used for precoat; and body mix additions were on the order of 0.5 pound of filter aid per pound of prefiltered solids. Some experiments have been run at rates corresponding to 3 to 10 times the normal filter aid usage for liquid-solid separations as reported by vendors.[13] The mechanical problems associated with rotary drum precoat filtration have been substantial; but even if the mechanical problems can be solved, development of the optimum precoat filtration parameters would still be required for economical operation. Considerable work has been done by researchers at

the Oak Ridge National Laboratory[8, 14, 26] in which the solvent fractionation of COED and SRC unfiltered oils and precoat filtration of SRC unfiltered oils has been investigated. Characterization analyses of the unfiltered and filtered COED and SRC oils were also developed, consisting of physical and chemical properties and determinations of viscosity and particle sizes.

In the ORNL work[14, 26] a laboratory-scale filter was constructed to operate at elevated temperatures and moderate pressures. The effects of temperature, pressure, precoat, and filter cake thickness were studied systematically; and coal was investigated as a precoat material. As expected, the required filtration times for both COED and SRC liquefaction processes were strongly dependent upon temperature and pressure with higher rates being obtained at higher temperatures and pressures. The flows of previously filtered oil through precoat alone were 20 to 30 times larger than those through precoat plus filter cake, indicating the small resistance to filtration provided by the precoat material and an estimate of the maximum flow rate possible with zero filter cake thickness. The filter cake compressibility was determined to be 0.49 indicating only moderate increases in flow resistance with pressure. When coal was used as the precoat material rather than the Johns-Manville Fibre-Flo filter aid, the filtration rates were increased for similar precoat thickness. The use of the coal as precoat may reduce process costs considerably because of lower cost, greater availability, and elimination of disposal and contamination problems. Characterization studies indicate that nearly all of the ash is removed by precoat filtration, but some pyritic sulfur and soluble organic sulfur remain. Ninety volume percent of the particles was less than

3 microns for both SRC and COED unfiltered oils. Filtration rates using 8.76 lb/hr/ft^2 of Johns-Manville Fibre-Flow precoat and with a 0.11-inch deposited filter cake were around 6 gal/hr/ft^2.

Research[38] at the Pittsburgh Energy Center has indicated that for the Synthoil process the unfiltered oil ash content can be reduced from 5 to 6% to less than 0.1% using precoat filtration. Two different Johns-Manville Celite precoat materials were used.

Review of General Precoat and Filter Aid Literature

Filtration, in general, is the separation of undissolved particulate or fibrous solids from a liquid by passing through a septum impermeable to the solids. Many factors[23] must be considered in the selection of suitable filtration equipment. The important factors are generally the slurry character, production level, process conditions, required results, and the materials of construction. The cake-forming characteristics of the slurry indicate whether or not continuous filtration is applicable. Continuous filtration is usually not practical if a 0.125-inch cake cannot be formed under vacuum in less than 5 minutes. High production rates generally indicate the need for continuous filters. Process conditions restrict the choice of filter in that vacuum operation may not be possible due to temperature or vapor pressure limitations. In this case, continuous pressure filtration with or without precoat may be used. Continuous precoat pressure filters are typically used for slurries that are very dilute and slow filtering, while vacuum filters are used for slurries that are medium to fast filtering. Chemical resistance and temperature requirements limit the choice of filter medium. The filter medium may be constructed of metals, plastics, or

fibrous materials. Shoemaker[30] and Smith[32] discuss the importance of the filter medium. The filter septum supports the filter medium. Filter aid materials when admixed with the unfiltered liquid (prefilt) provide porosity in the filter cake. Hutto[11] and Culver[6] describe some of the practical general rules of filter aid and precoat use. A filter aid must be a relatively incompressible solid of high porosity with particle size somewhat greater but generally of the same order of magnitude as the suspended solids to be removed in the prefilt. Two methods are generally used in filtration work to employ filter aids, either as precoat or body feed. Precoating is the process of adding to the surface of the filter media a quantity of filter aid equivalent to 2 to 15 pounds per 100 square feet. The precoat serves two purposes:

1. It improves the initial clarity of the filtrate by presenting a septum of filter aid particles rather than the more coarse filter medium.

2. It protects the filter media from fouling with the prefilt solids.

Body mix or body feed is added to the prefilt to maintain flow through the filter cake. Without body feed, the cake may rapidly lose its permeability and greatly decrease the filter throughput, and for a rotary filter, reduce the amount of solids collected per revolution. Diatomaceous earth, perlite, cellulose, carbon, and asbestos are a few common filter aid materials in use today. These materials are rated as to their ash contents, relative permeability, and density. Water content of these materials is usually in the range of 3 to 5%, and the densities range from 5 to 20 lb/ft^3. Filter aid costs are generally in the range of 6 to 40 cents per pound.

Lloyd[16, 17] describes the effect of prefilt suspended solids particle size on filtration efficiency. The degree of filtration is strongly dependent upon particle size in that the finer the particle size the more difficult the separation problem, and the quantity of solids per unit volume strongly influences the separation. Particle shape tends to affect the separation in that irregular or nonspherical particles are more easily filtered because they tend to bridge and form flow channels to a greater extent. In some extreme cases with very fine particle sizes, pretreatment of the prefilt can become necessary. Thomas[34] and Tiller[35, 37] indicate typical pretreatment operations. This pretreating consists of the addition of agglomerating or floccing agents to the prefilt to increase the particle size and filterability. Reducing the solution viscosity will also increase the filtration rate.

Miller,[18] Silverblatt,[31] Purchas,[24] and Hoffing[10] describe the resistances to filtration. In correlating filtration data, the major resistances to filtration usually lie in the filter cake, increasing with cake formation, and in the filter medium dependent upon the pressure applied. These resistances are inversely proportional to the filtration rate and are characterized by a specific resistance or cake resistance determination. The filter cake compressibility is calculated from the relationship between cake resistance and filtrate rate. The objective of a filtration test is to minimize these resistance terms through the use of suitable pretreatment steps or by adjustment of operating conditions.

Unsatisfactory performance in pressure filters usually results from incorrect selection of filter type and filter medium, inadequate type or amount of precoat or body mix, poor flow characteristics in

filter causing settling of adhered cake, and incorrect pressure-time relationships for materials based on cake-compressibility patterns. Possible solutions to these problems are well documented.[1]

C. FILTRATION DATA TREATMENT

Filtration data can usually be correlated using a form of the Poiseuille[21] equation for flow through parallel capillaries. Precoat filtration proceeds by deposition of an open porous precoat material followed by filtration of a concentrated solids-liquid stream. Initially solids penetrate the precoat bed, but rapidly bridge pores to produce surface filtration by the filter cake solids. Cake filtration can be described as streamline flow through cake channels by the use of Poiseuille's equation,

$$\frac{dV}{Ad\Theta} = \frac{\Delta P}{\mu\,[\alpha W(V/A) + r]} \qquad (1)$$

where V = volume of filtrate collected,

A = filter surface area,

Θ = time,

α = average specific cake resistance,

r = total resistance of filtering medium,

μ = filtrate viscosity,

W = mass of accumulated solids per unit volume,

ΔP = pressure drop across filter cake and filter media.

The cake compressibility and particle size are usually related to the filtration rate by

$$\alpha = \alpha' (\Delta P)^{s} , \qquad (2)$$

where α' is a constant depending on particle size, and s is the cake compressibility. Integration of equation (1) at a constant pressure drop gives

$$\frac{\Theta}{V/A} = K_p \frac{V}{A} + C \ , \tag{3}$$

where $K_p = \frac{\mu\alpha W}{2(\Delta P)}$ and $C = \frac{\mu r}{\Delta P}$.

Time versus filtrate volume plots allow K_p and C to be determined as the slope and intercept, respectively. The value of r, the resistance of the filtering medium excluding the cake resistance, can be determined from the known viscosity and pressure drop. The average specific cake resistance, α, can be calculated from the known viscosity, pressure drop, and solids concentration.

D. PLANT SCALE PROJECTIONS FROM LABORATORY FILTRATION TEST DATA

The filtration equations[21] are useful in predicting the effect of a change in any variable if the constants are determined from data taken with the actual feed solution. Filtration rates are affected by temperature, pressure, solids content, particle size, viscosity, cake thickness, and type of filter medium. For a given solids separation process the feed solution particle size, solids concentration, and type of filter medium is usually constant. Consequently, filtration flow rate is usually governed by system pressure and temperature considerations.

If constant pressure filtration tests are run with temperature and solids content remaining constant, plots of $\frac{\Theta}{(V/A)}$ versus V/A can be

made from time and filtrate volume values. These plots enable "α", the average specific cake resistance, and "r", the filter medium resistance, to be determined from the slope and intercept, respectively. Since the cake resistance is

$$\alpha = \frac{2\ \Delta P}{\mu W} \text{(slope)} \ , \tag{4}$$

and the filter medium resistance is

$$r = \frac{\Delta P}{\mu} \text{(intercept)} \ , \tag{5}$$

the effect of a change in any variable except ΔP which affects α and r can be estimated. In many cases r does not vary appreciably with pressure; in which case, an average value can be used at all pressures.

A test must be run to evaluate α at two or more pressure conditions for determining the effect of pressure on α. A plot of $\ell n\ \alpha$ versus $\ell n\ \Delta P$ yields s, the compressibility factor, as the slope and α', the particle size constant, as the intercept. Subsequently, α and r can be determined for any pressure condition and, with information on the variation of viscosity with temperature filtration rates, can be estimated for any combination of variables.

The effect of increasing temperature is to lower solution viscosities, thereby resulting in proportionately higher filtration rates. Pressure effects are obtained from the compressibility determinations mentioned above, and pressure changes usually provide less than proportional flow rate changes. A perfectly incompressible filter cake

(s = 0) provides flows that are directly proportional to the applied pressure. However, for fully compressible cakes (s = 1) the rate is independent of pressure. In most situations s lies between 0.1 and 0.8 and results in moderate flow changes with pressure.

E. MATERIAL PROPERTIES

The health and safety requirements for exposure to polynuclear aromatics are very stringent. Before any filtration studies could be done, approvals were required from the Oak Ridge National Laboratory Medical Department, the Oak Ridge Gaseous Diffusion Plant Industrial Hygiene Section, and the Environmental Section. The toxic properties, physical properties, and the health and safety requirements are listed here for their general usefulness.

Physical Properties

A list of physical properties is listed in Table I for the Solvent Refined Coal unfiltered oil solution. These data were furnished by the Oak Ridge National Laboratory operated by Union Carbide Corporation for the Department of Energy. Due to the large suspended solids present in the feed (∿8%) and the relatively high viscosity (60 CP), separation is extremely difficult.

Toxic Properties

Coal liquefaction product oils are categorized as polynuclear aromatic hydrocarbons (PNA's) and do pose a detriment to long-term health if suitable precautions are not taken. Skin cancers are the primary hazard in working with PNA's. These cancers are readily treated and are usually only realized after repeated exposure over several

TABLE I

PHYSICAL PROPERTIES OF SOLVENT REFINED COAL UNFILTERED OIL

Property	Value
Molecular Weight	273 g/g mole
Pyridine† Insolubles	6.28%
Ash Content	1.45%
Volatile Matter	84.7%
Heat Content	16,309 Btu/lb
Density	1.10 g/cc
Viscosity (120°F)	60 centipoise
Carbon	86.84%
Hydrogen	7.58%
Nitrogen	0.95%
Sulfur	0.43%
Iron	0.074%

†Pyridine insolubles is a common method of analyzing for unconverted coal material in coal liquefaction streams.

months. The threshold limit value[12] (TLV) for coal tar pitch volatiles, benzene soluble fraction, as representative of material containing polynuclear aromatic hydrocarbons is 0.2 mg/m^3 in occupational settings. Currently, polynuclear aromatic hydrocarbon TLV's do not exist. There has been suggested by the Standards Advisory Committee on Coke Oven Emissions, a maximum allowable concentration for 3,4 benzpyrene of 0.0002 mg/m^3.

Industrial Hygiene

To control PNA hydrocarbon exposure, the Medical and Industrial Hygiene Departments of the Oak Ridge Gaseous Diffusion Plant in conjunction with the Oak Ridge National Laboratory require a skin examination prior to work with the material in which photographs of the face, neck, hands, and arms are taken. A sputum cytology is also taken to ensure no medical pathology. Follow-up exams are required. The Industrial Hygiene Department furnishes an air sampling program in the experimental work area and makes recommendations regarding respirators, special clothing, and special barrier creams for each experimental operation, and periodically, by the use of ultraviolet light, verifies the presence or absence of PNA hydrocarbons on exposed portions of the body or on tools and equipment.

Employee's hands and face must be washed thoroughly with soap and water after exposure. Good housekeeping must be maintained. All spills must be cleaned up at once with rags and disposed of in designated waste drums. Control areas must be posted as such, and smoking and eating are prohibited.

All equipment must be cleaned with suitable solvent and checked with UV light before being removed from a control area and covered with

plastic. If there are leaks in piping, flanges, and valves, containment of the escaping vapors shall be accomplished by local exhaust ventilation.

Disposal Procedures

Currently, no ultimate disposal method is available for these materials and the environmental section has approved containing cleaning solutions and all waste solutions in drums that are labeled accordingly.

CHAPTER II

EXPERIMENTAL APPARATUS

A. INTRODUCTION

The experimental data were obtained using a batch filtration system consisting of a Niagara bench-scale vertical pressure leaf filter, unfiltered material feed tank, filtrate receiver, precoat tank, feed and product pumps, and the necessary interconnecting pipe and instrumentation. All tanks, pumps, lines, valves, and the batch pressure leaf filter were of stainless steel 316 construction for corrosion resistance. The filter leaves were constructed of tubular metal frames and 4 mesh center drainage wire covered with a 24 x 110-mesh stainless steel filter cloth.

Temperatures were maintained in the tanks, valves, and lines using resistance heaters and monitored with calibrated thermometers. The filtrate receiver tank was calibrated for accurate volume measurement. Feed pressure and pressure drop across the filter were continuously monitored. Average flow rates were determined from total filtrate volume measurements for specified time intervals.

B. EQUIPMENT

Figure 1 illustrates the laboratory constant pressure filtration system. A Model LF-1 Niagara vertical leaf pressure filter[3] having 0.69 ft^2 of filtering surface and 62.5 cm^3 of filter cake volume was used. The filter was constructed of 316 stainless steel and contained a vent valve, drain line with bottom inlet, pressure gauge fitting, two

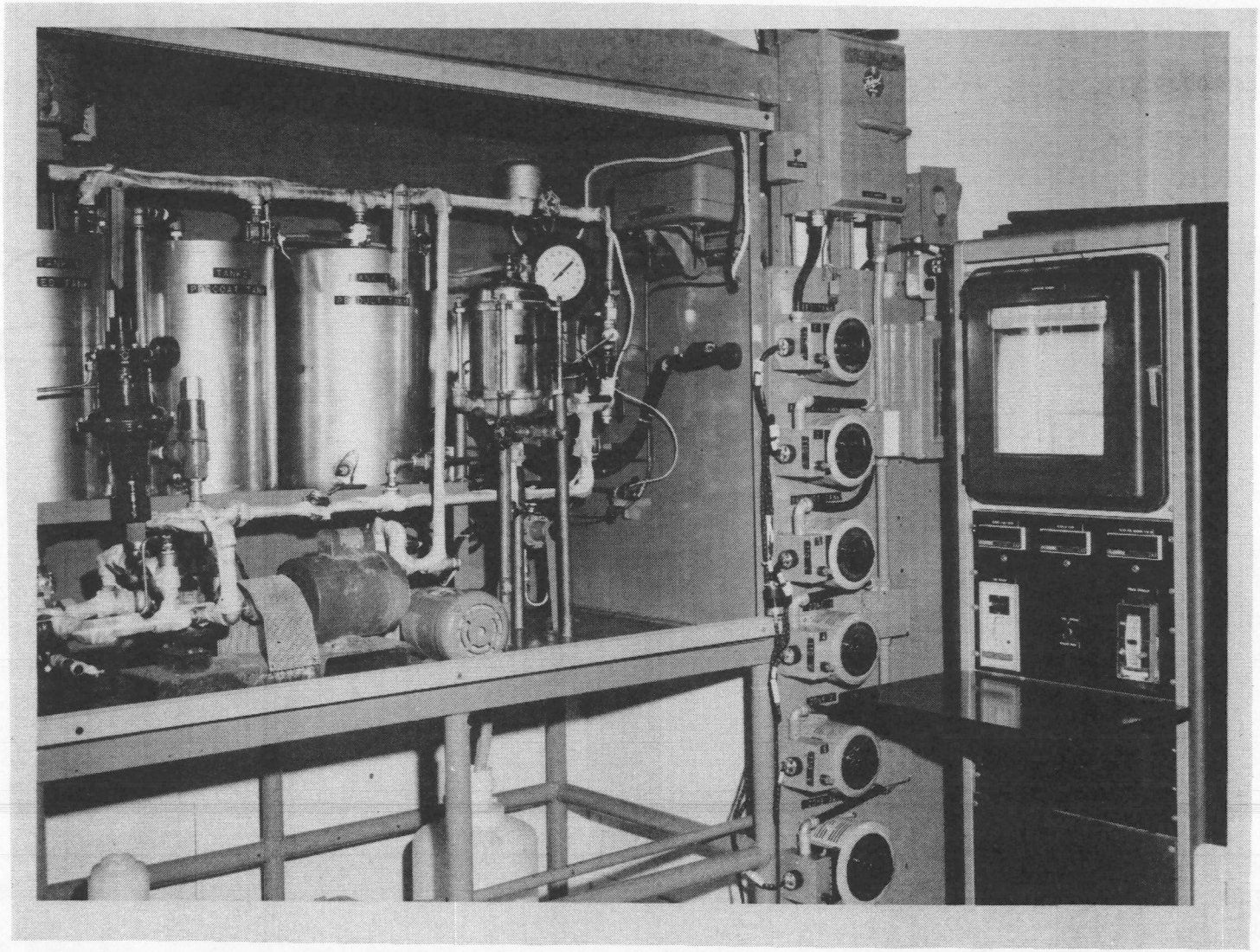


Figure 1. CONSTANT PRESSURE FILTRATION SYSTEM

auxiliary shell side connections, and a filtrate outlet manifold. The volume of the filter was approximately 1.4 gallons. Three filter leaves were provided having a total filtration surface of 0.69 square feet and constructed of stainless tubular metal frames and 4-mesh center drainage wire covered with a 24 x 110-mesh stainless steel filter cloth having a nominal 0.008-millimeter opening.

Feed, precoat, and filtrate tanks were constructed of stainless steel and provided around 5 gallons of holding capacity. Variable speed laboratory stirrers with propeller-type agitators were provided in the feed and precoat tanks for thorough mixing. A temperature probe was inserted in each tank for accurate temperature measurement and recorded on a strip chart recorder. Heating was provided by thermostatically controlled resistance heaters clamped to the outside of tanks.

The filtrate receiver was calibrated for accurate volume measurements using demineralized H_2O at operating temperature.

All block valves and piping were 1/2 inch and constructed of stainless steel 316 for adequate corrosion resistance. Serpentine heaters were wrapped around all valves and lines to maintain temperature.

A positive displacement gear pump with a stainless drive gear and glass impregnated Teflon idler gear, manufactured by Liquiflo Pump Corporation, was used for filter feed and precoat liquor pumping requirements. A Research control valve was placed in a recycle line around the pump to provide the required pressure and flow conditions. Filtrate liquor was recycled for sampling using a small capacity magnetic drive seal-less centrifugal pump manufactured by Serfilco Corporation .

C. PRESSURE MEASUREMENTS

Proper instrumentation is a necessity for obtaining maximum operating efficiency. Accurate measurement and control of the filter pressure drop (ΔP) are necessary requirements for a constant pressure filtration test. These requirements were satisfied by the pressure measuring and control system described below and shown schematically in Figure 2.

Feed Pressure

The feed pressure was sensed by a Taylor electronic pressure indicating transmitter with an input range of 0 to 60 psig. An output signal of 4 to 20 mA dc was displayed on a Digitec 4 to 20 mA dc panel meter. Calibration of the transducer was accomplished by adjustment of span and zero for referenced input signals varying from 0 to 60 psig. A plot of mA dc reading versus psig is shown in Figure 3.

Filter Pressure Drop Control

The difference in filter inlet and outlet pressures was sensed by a Taylor pneumatic differential transmitter with pressure taps located close to the inlet and outlet of the filter shell for accurate readings. The input range for the transmitter was adjusted for a 0 to 60 psi. differential. A Robertshaw pressure-to-current transducer (P/I) was used to convert the output signal (3 to 15 psi.) from the Taylor pressure transmitter to a 1 to 5 mA dc signal that was displayed on a 1 to 5 mA dc Digitec panel meter. The output signal from the Taylor electronic feed pressure transmitter was sensed by a Taylor Pressure Controller with a set point reference. Adjustment of the filter inlet pressure was accomplished by regulating recycle flow around the filter

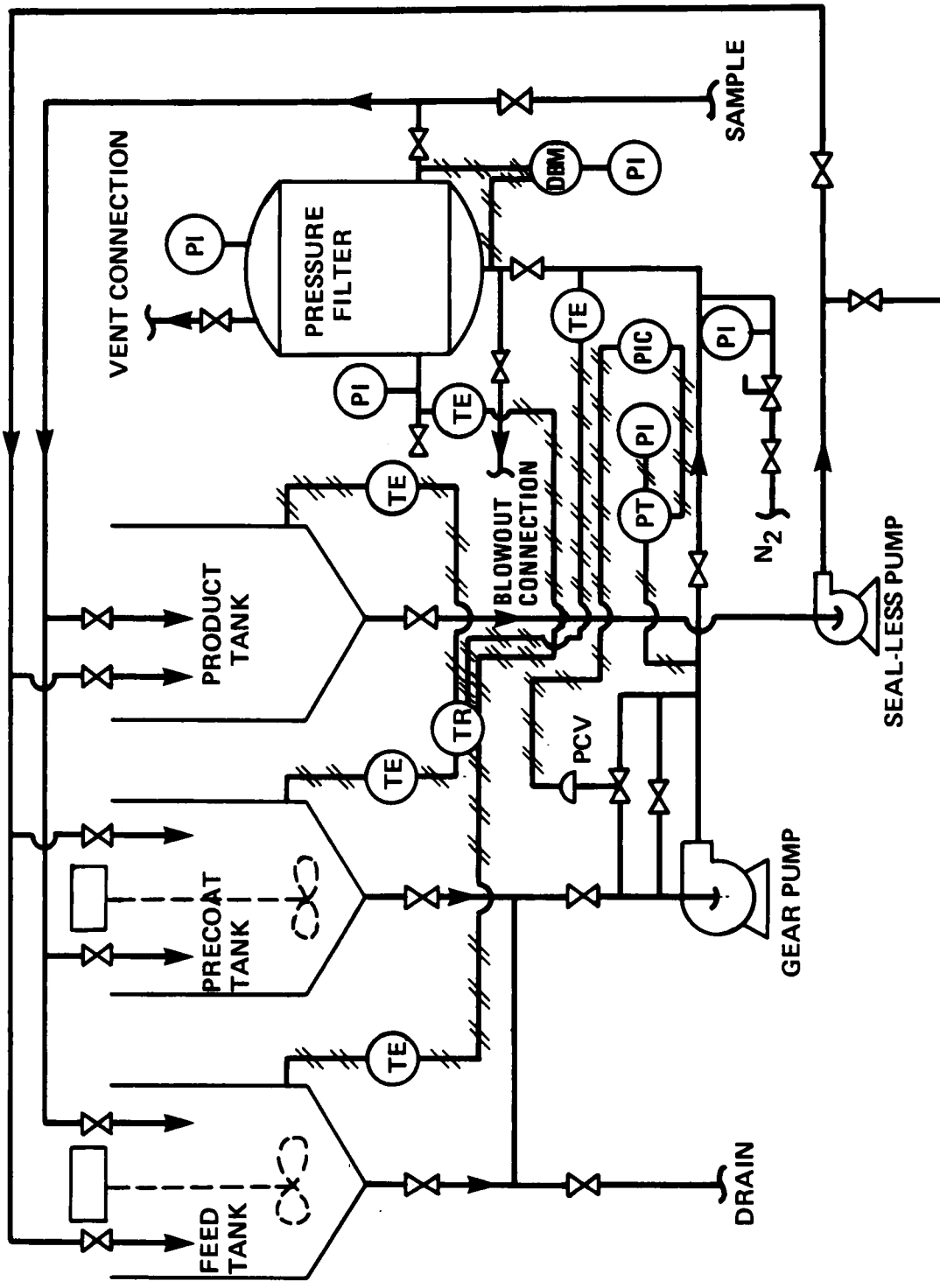


Figure 2. CONSTANT PRESSURE FILTRATION SYSTEM – INSTRUMENTATION AND FLOW SCHEMATIC

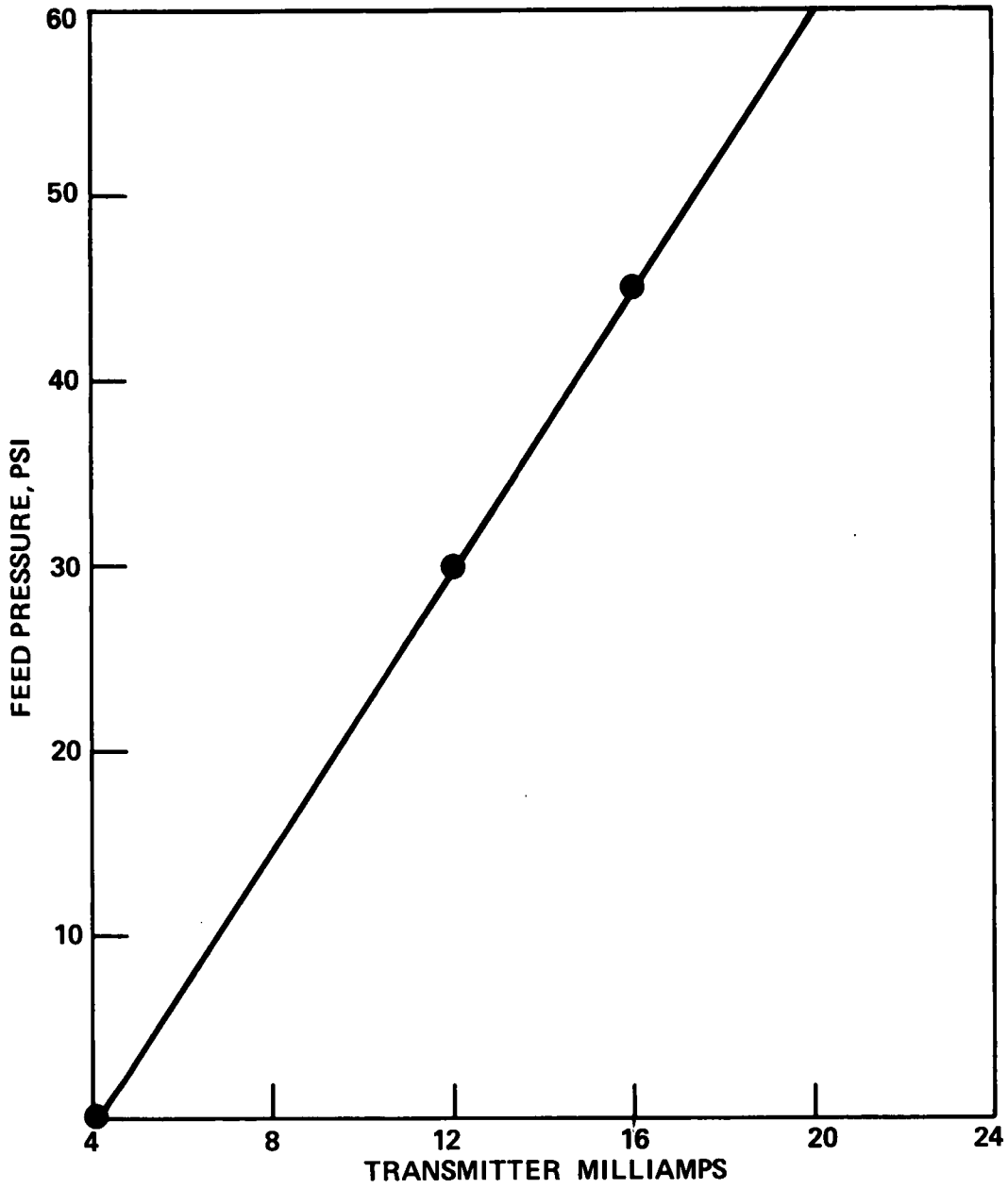


Figure 3. FEED PRESSURE VERSUS TRANSMITTER MILLIAMPS

feed and precoat liquor pump with a Research Control valve. This control system provided constant pressure drop as filtration proceeded and filtrate flow rate diminished. The calibration curve for the differential pressure transmitter is shown in Figure 4.

D. TEMPERATURE MEASUREMENTS

Maintenance of elevated temperatures in the constant pressure filtration system is necessary due to the high viscosity of the SRC liquefied coal intermediate. As can be seen from Figure 5, the viscosity of the liquefied coal solution is quite high at room temperature; and to achieve viscosities similar to that of aqueous solutions, temperatures in the range of 300 to 600°F would be required. Temperatures in the range of 115 to 125°F were used throughout this study providing solution viscosities around 30 to 60 centipoise.

Feed and Precoat Tank Temperatures

Temperatures were measured in both feed and precoat tanks with calibrated copper-constantan thermocouples and recorded on a Leeds-Northrup Strip Chart Recorder. Heating was accomplished using resistance heaters clamped to the tank periphery and controlled with Variacs. This system provided stable solution temperatures.

Filtration Temperature

Copper-constantan calibrated thermocouples were used to monitor inlet and outlet temperatures across the filter. These temperatures were recorded on the Leeds-Northrup recorder.

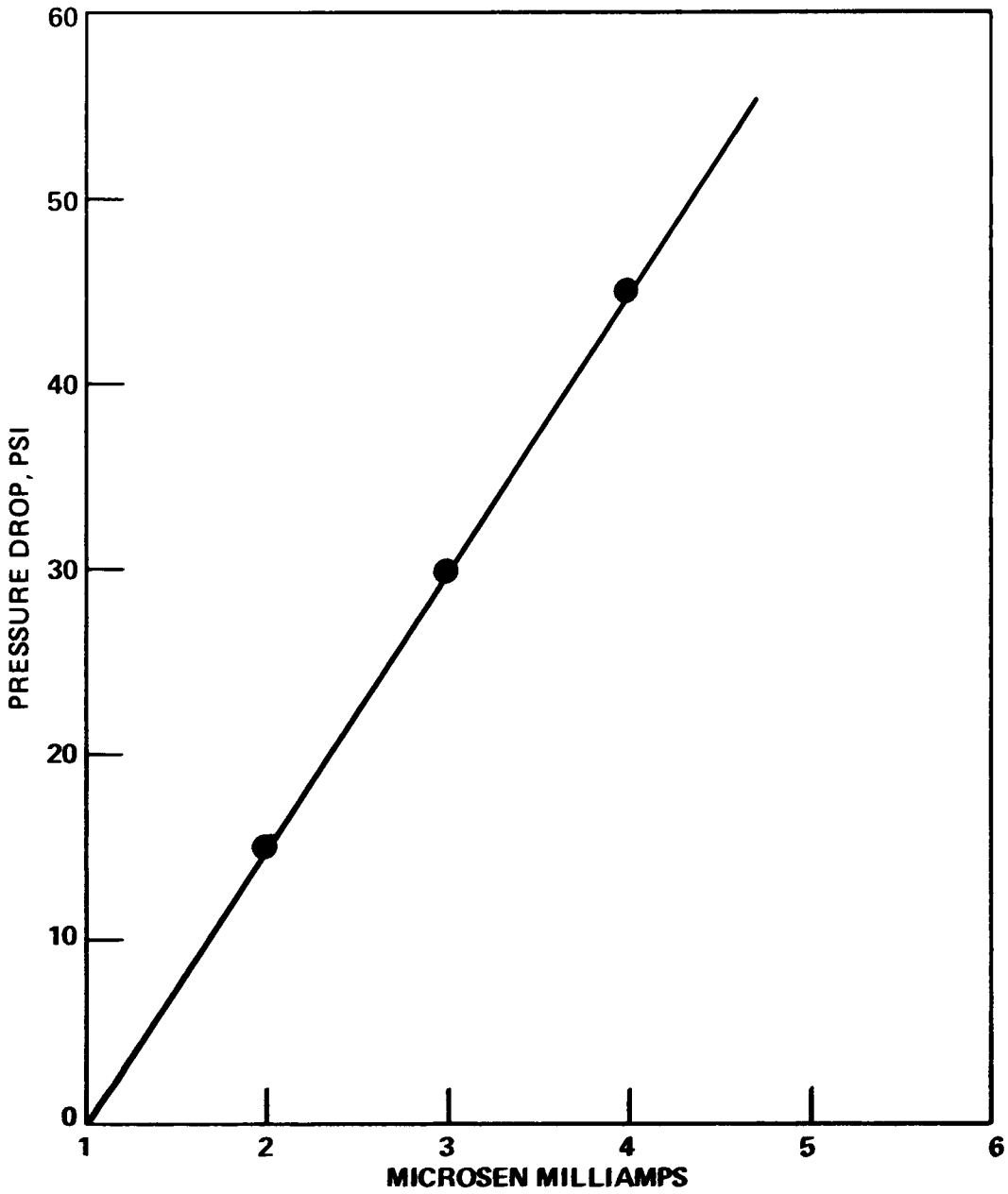


Figure 4. FILTER PRESSURE DROP VERSUS TRANSMITTER MILLIAMPS

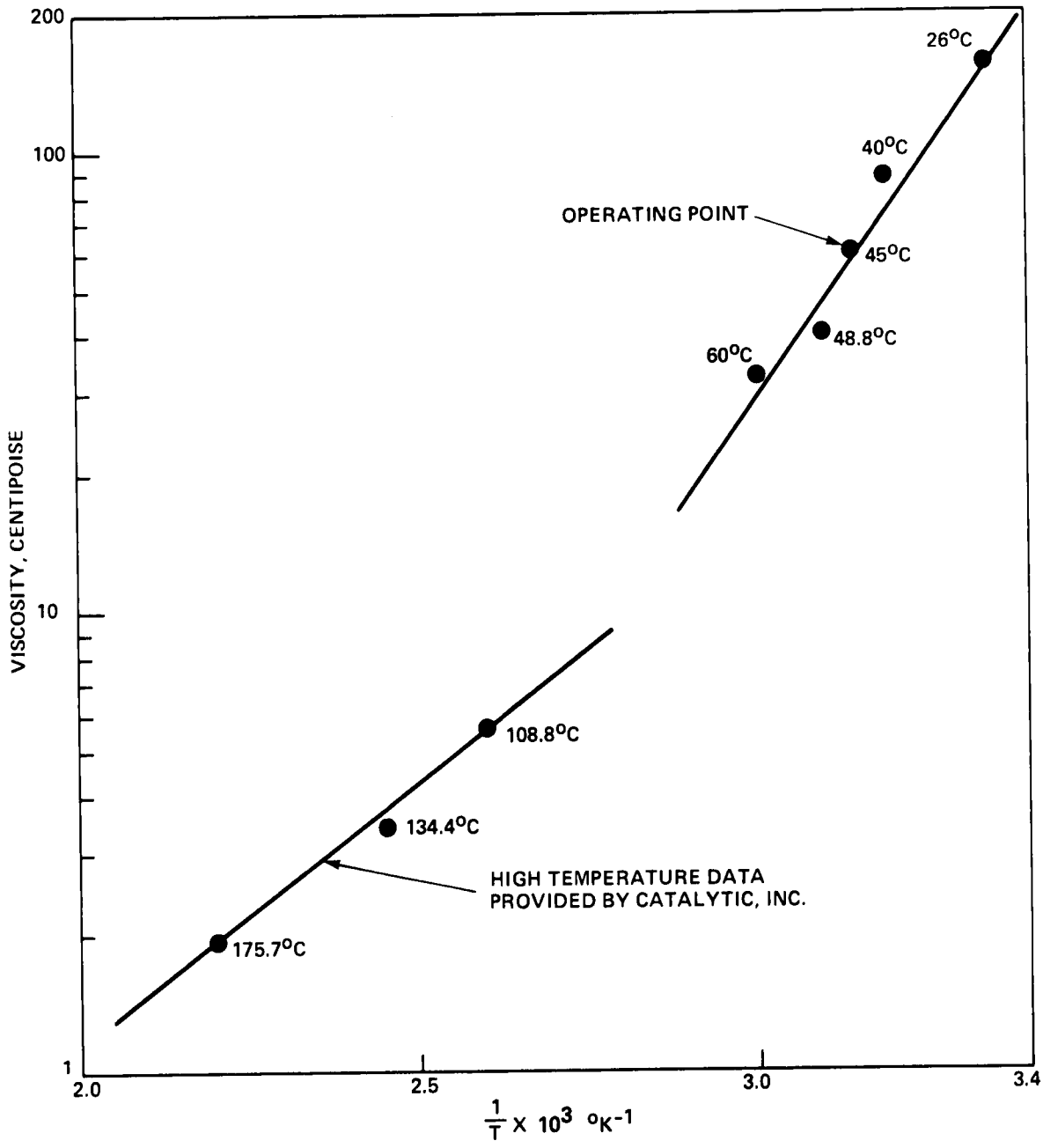


Figure 5. LOG OF VISCOSITY VERSUS RECIPROCAL TEMPERATURE

Auxiliary Heating

To ensure minimal loss of heat when transferring solutions, serpentine line heaters were provided. These line heaters were controlled with Variacs.

E. FLOW MEASUREMENTS

In constant pressure filtration tests, accurate filtrate flow and time measurements provide the basis for determining specific cake resistances and filter medium resistances. These parameters are required for accurate scale-up information. Due to the very low average rates (0.1 to 0.7 gal/hr/ft^2) in laboratory-scale filtration tests and the relatively high solids content (10%) encountered for liquefied coal solutions, no electronic flowmeter or totalizing meter was found suitable. Filtrate volume versus time measurements were obtained by calibrating the filtrate receiver or product tank using demineralized H_2O at the filtration temperature. A sight-glass on the product tank provided the reference for the volume measurements.

F. NITROGEN BLOWBACK SYSTEM

A nitrogen blowback system[13] consisting of a nitrogen header ($\sim$60 psig), a pressure regulator (0 to 60 psig), and block valves were provided to enable blowback of filter vessel contents. Blowback is necessary for removing precoat liquor from filter prior to introduction of feed liquid. If no blowback were provided, filtrate volume versus time measurements would be based on a mixture of clean precoat liquor and dirty feed liquor. Blowback is accomplished by pressuring up the inlet or shell side of filter and physically blowing liquid contents out

another shell side connection. The filter aid used for precoating is held firmly on the filter leaves by partially opening the valve in the filtrate outlet manifold, thus keeping a pressure drop across the leaves at all times during the blowout period. Using a cellulose fiber filter aid ensures adequate retention on the surface of the leaves.

The blowback system is also necessary to remove filter contents at the end of the filtrate cycle as relatively little if any material could be blown through the leaves after the cake space is filled.

CHAPTER III

EXPERIMENTAL PROCEDURES

A. INTRODUCTION

The experimental procedures and techniques employed in this investigation will be outlined in detail in this chapter. Feed material characterization, feed preparation, precoat liquor preparation, blowback procedure, run steps, sampling technique, and cleaning of the apparatus are included. Reference will be made to material included in the foregoing chapter on experimental apparatus as necessary.

B. FEED MATERIAL

Two 55-gallon drums of unfiltered material were obtained from the Solvent Refined coal Process liquefaction pilot plant facility in Wilsonville, Alabama, operated by Catalytic Inc. The pilot plant operating conditions are shown in Table II for Emery coal. Emery feed coal contains typically 7.8% ash, 2% H_2O, and 4.0% sulfur. Around 1.5% ash, 0.43% sulfur, and 6 to 7% of undissolved coal are present after the liquefaction step. Filter feed solids are irregular in shape and 0.5 to 40 μm in size. A large fraction of the particles as shown in Figures 6 and 7 are small-sized and represents a particularly difficult separation problem. A Brookfield viscometer was used to measure low temperature (120°F) unfiltered material viscosity (∿60 centipoise).

C. FEED PREPARATION

The feed solution drum was thoroughly mixed for 30 minutes with a high volume drum pump connected to a recycle line on the constant

TABLE II

SOLVENT REFINED COAL PROCESS PILOT PLANT OPERATING CONDITIONS FOR EMERY COAL

Operating Parameter	Value
Coal Feed Rate	463 lb/hr
Reactor Pressure	2429 psig
Reactor Temperature	817°F
Coal Concentration	32.3%
Space Rate	50.9 lb/hr-ft^3
Feed Gas Rate	10,038 ft^3/hr
Hydrogen Concentration	85%
Hydrogen Consumption	2.3% of MAF† Coal
Conversion	77% of MAF Coal

†Moisture - ash free

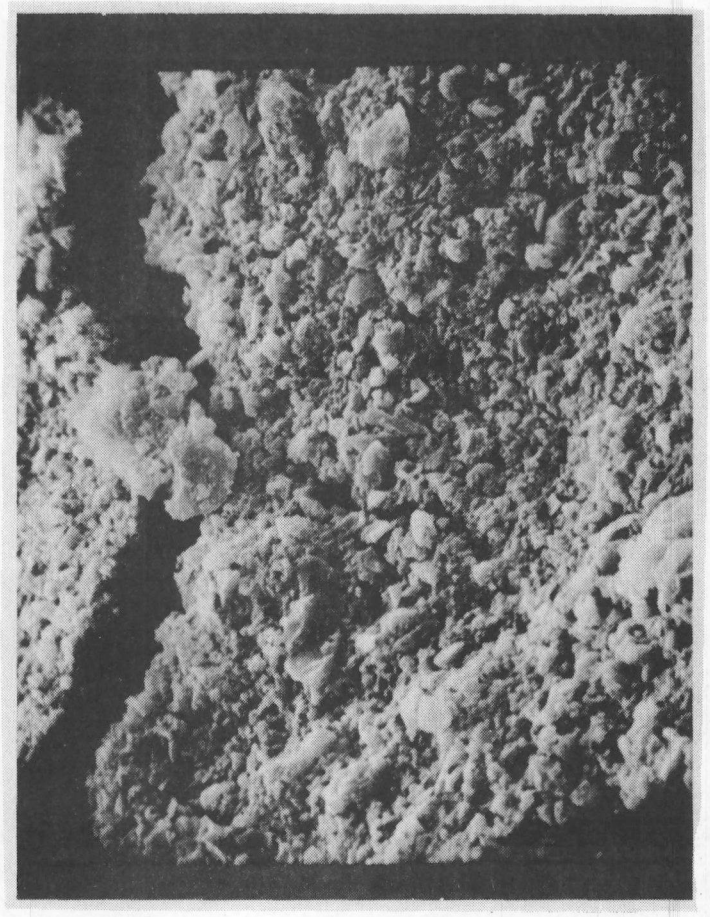


Figure 6. SCANNING ELECTRON MICROGRAPH OF LCF-1 FEED SOLIDS (500X)

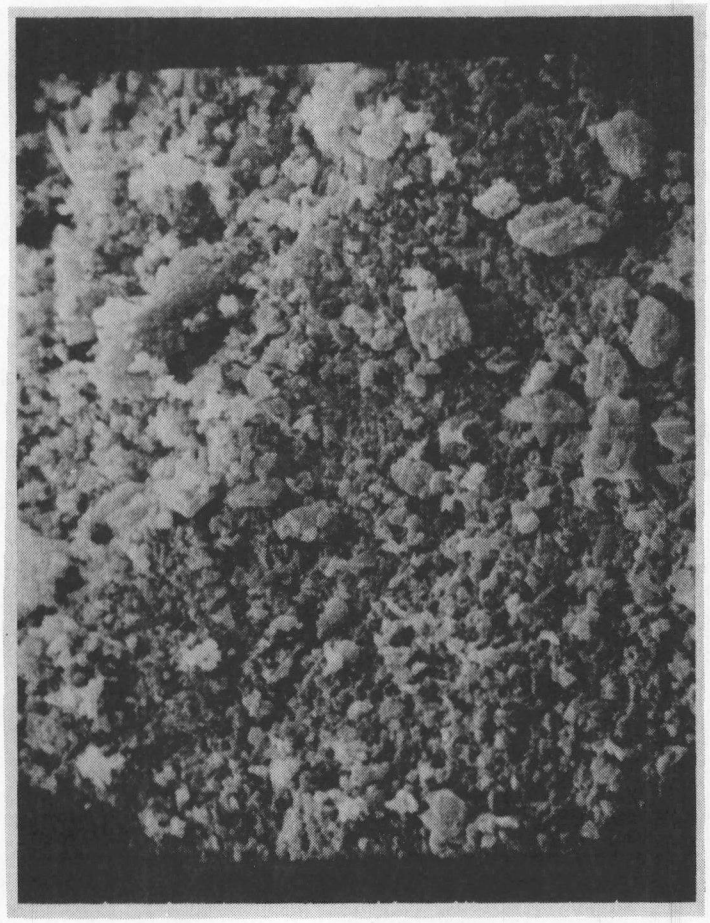


Figure 7. SCANNING ELECTRON MICROGRAPH OF LCF-2 FEED SOLIDS (500X)

pressure filtration system. A feed sample was taken toward the end of the recycle mixing period, and then the drum pump flow was diverted to the feed tank. Around 8 to 9 liters of unfiltered material was charged to the feed tank for all experiments and was kept agitated for uniform suspended solids concentration.

D. PRECOAT LIQUOR PREPARATION

Precoat liquor for all experiments was prepared by filtering 14.4 liters of unfiltered oil through the constant pressure system. Precoat liquor must be relatively free of suspended solids to ensure the filter medium is not fouled by dirty solids. The precoat liquor preparation run was made at 115°F using 0.05 lb/ft^2 ($\sim$1 volume percent) of BW-40 cellulose fiber filter aid deposited as an admix in the unfiltered oil. The average filtration rate for this run was 0.382 $gal/hr/ft^2$. Table III shows typical properties of cellulose fiber filter aid materials.

E. RUN STEPS

For typical cake filtration involving precoat and/or admix, there are several fundamental events during the cycle. Feed and precoat liquor must be prepared as described earlier. Starting with a clean filter, the precoat liquor containing filter aid is introduced into the filter where the filter aid is deposited as a thin cake on the filter leaves. The prefilt or unfiltered oil is then introduced without disturbing the precoat and recirculated for a sufficient time for initial clarity; then recirculation is stopped and the product stream or filtrate directed to the filtrate receiver or product tank. At this point filtration has begun and data can be taken for time versus filtrate volume plots.

TABLE III

PROPERTIES OF CELLULOSE FIBER FILTER AID MATERIALS

Grade of Filter Aid	BW-20	BW-40
Particle Size Length	96 μm	80 μm
Particle Thickness	17 μm	17 μm
Retained on 100 Mesh	45%	18%
Retained on 40 Mesh	15%	<1%
Permeability Rating	2.0*	3.0
Filter Cake Density	10-12 lb/cu ft	11-13 lb/cu ft
Moisture	6%	6%

*Permeability of 1 Darcy unit passes 1 ml per second per cm^2 of a liquid of 1 centipoise viscosity through a cake of 1 cm thickness at 1 atm.

Actual run steps are described below and include feed preparation and sampling, precoat liquor preparation, heating to filtration temperature, precoating filter leaves, blowout of precoat liquor, introduction of prefilt and recirculation, filtration and product collection, product sampling, blowout of prefilt liquor, and filter leaves cleaning.

Feed Preparation

Feed material was prepared as mentioned above by thoroughly mixing drum contents, sampling and filling feed tank with around 8 to 9 liters of unfiltered oil. After the feed tank was filled with the desired amount of unfiltered oil, the feed tank agitator was turned on and heating was initiated and controlled to around 115 to 120°F.

Precoat Liquor Preparation

Precoat liquor from previously filtered oil was charged to the precoat tank, and the required amount of either BW-20 or BW-40 cellulose fiber filter aid was added and kept suspended by agitation. If experimental conditions from the factorial design required body mix of filter aid, around 1% by weight was added to the dirty liquor in the feed tank. Heating was initiated and maintained at the filtration temperature (115 to 120°F). Line heaters were turned on to maintain temperatures for the subsequent processing steps.

Precoating Filter Leaves

Precoating was accomplished by pumping the precoat liquor containing filter aid into the shell side of filter until precoat liquor overflowed the filter. By overflowing the filter, all air was vented. At this point, the valve in the filtrate outlet manifold was opened and

the vent line closed to allow precoat liquor to flow through the filter leaves. Typical precoating rates[13] are given in the literature as 1 gal/min/ft^2 for aqueous materials with low viscosities with proportionately lower rates for viscous feed materials. Previously suspended filter aid material was deposited on the filter leaves as a thin cake (0.03 to 0.06 inch) and provided the actual septum for subsequent cake filtration. Precoating was continued for 10 to 15 minutes at approximately 10 psi. until all filter aid was deposited. If no filter aid were deposited, the septum would become blinded quite rapidly with dirty solids in the prefilt. Careful attention must be given to ensuring clean precoat liquor and filter leaves.

Blowback of Precoat Liquor

Blowout of precoat liquor left in the filter at the end of the precoating cycle was necessary because filtrate volume versus time measurements should be based on prefilt or feed solution rather than a mixture. Blowback was accomplished by pressuring up the inlet or shell side of the filter and forcing out precoat liquor through a drain connection. At the same time, the valve in the filtrate outlet manifold was held partially open to keep a pressure drop across the filter leaves, ensuring that filter aid deposited in the precoat cycle does not drop off. At the end of the blowout cycle, a small positive pressure was maintained in the filter.

Introduction of Prefilt and Data Acquisition

Prefilt liquid was introduced into the filter and allowed to overflow out the vent line and return to the feed tank in order to purge all air and noncondensables. After all air was purged, the filtrate

outlet valve was opened, the vent line closed, and the solution recycled to feed tank. Constant pressure conditions were then met by setting the pressure control system. After suitable time for stabilizing temperatures and pressures (∿10 min), the filtrate flow was diverted to the product collection tank and the filtration cycle initiated. Filtrate volume and collection time measurements were made until the end of the cycle after which both inlet and outlet valves were closed to maintain pressure in the system for blowout of the heel.

Product Sampling and Prefilt Blowback

Product material was homogenized by recycling from the bottom of the product collection tank back to the product tank with a seal-less magnetic drive pump. A sample line was provided to collect the well mixed material from the recycled line.

Feed material left in the filter at the end of the cycle was blown back to the feed tank for the next run and a slight nitrogen pressure set up across the filter to dry out leaves. The filtrate outlet line from filter leaves was opened and routed to the precoat tank during the drying period.

Filter Leaves Cleaning

The filter must be disassembled, and the filter leaves cleaned for the next run. Filter leaf cleaning is of paramount importance because leaves that are blinded due to lack of proper care can greatly restrict flow by cutting down effective filtration area, reducing cycle times considerably. Several cleaning solutions were tried for removing the Solvent Refined Coal solution from inside the filter leaves and for removing solids from the filtration surface. Initially, trichloroethylene

at room temperature was used to clean the media and was determined to be ineffective. Methylene chloride was also tried. At the suggestion of persons at the SRC pilot plant in Wilsonville, Alabama, hot toluene (150°F) was successfully used to clean the filter leaves. Filter leaves cleaning and precoat liquor preparation were two very troublesome areas in this study.

F. EXPERIMENTAL ANALYSIS

The most frequently employed technique for investigating the filterability of solutions is obtained by laboratory-scale constant pressure filtration systems[7]. Data obtained with the laboratory pressure filter dictate the proper grade and amount of cellulose fiber filter aid to give maximum liquefied coal filtration efficiency and aid in selecting filter size in relation to filtration cycle length and overall flow and clarity.

Several factor variables can be investigated in a constant pressure filtration test. Basic factor variables include pressure, amount of cellulose fiber filter aid for precoat and/or body mix, type and grade of filter aid for precoat and/or body mix, and temperature. Response variables include filtration rate, filtrate clarity, and filter cake character.

A fractional factorial experiment design[19] was used to examine pressure and the amount and grade of cellulose fiber filter aid to use for precoat and body mix in the filtration of the Solvent Refined Coal process unfiltered oil. A two-level design was used with high and low levels of four variables; namely, pressure, amount of filter aid for precoat, use of body mix, and the grade of cellulose fiber for precoat.

A half replicate experimental design was chosen to screen original factor variables, and the chief response variable was the filtered liquor flow rate, with filtrate clarity a secondary, although important, consideration. The experiment was set up assuming a constant feed solids concentration, density, and feed solution viscosity. Optimized experiments were based on factor variable screening runs.

CHAPTER IV

EXPERIMENTAL RESULTS AND DISCUSSION OF RESULTS

A. INTRODUCTION

Constant pressure filtration tests were made to determine the filtration parameters for removing ash and unconverted coal material from a Solvent Refined Coal liquefaction intermediate. Filtrate volume versus time measurements are shown graphically. Using a form of the Poiseuille equation for flow through cake channels, the average specific cake resistance and filter medium resistance were determined for each experimental run. A plot of $\frac{\Theta}{V/A}$ versus V/A yielded the average specific cake resistance as the slope and the filter medium resistance as the intercept. The determination of the filter cake resistance and pressure dependency is also discussed. This is necessary in projecting filtration rates for other operating conditions. Initial experiments were planned utilizing a fractional factorial procedure with four variables: pressure, grade of cellulose fiber filter aid, use of precoat and/or body mix, and amount of filter aid for precoat. Filtrate flow rate and ash content were the response variables studied. Comparisons regarding flow rates, ash contents, and filter aids were made with reported work by others.

B. EXPERIMENT DESIGN

A fractional factorial design was chosen to study the effect of four variables on filtration rate and filtrate clarity for the solvent Refined Coal liquefaction intermediate material. The variables were given high and low levels for design purposes.

Fractional Factorial Design

The initial filtration experiments were set up utilizing a two-level fractional factorial[19] design. Four factors were studied in an eight-experiment design as shown in Tables IV and V. Pressure was adjusted to a low level of 25 psig and a high level of 50 psig. Two grades of cellulose fiber filter aids were studied. The amount of filter aid for precoating purposes was varied from a low level of 0.025 lbm/ft^2 to 0.050 lbm/ft^2; normal filter aid usage rates for precoating are 0.02 to 0.15 lbm/ft^2, as reported by others[13]. Body mix addition of filter aid was studied to determine whether it could be beneficial to filtrate flow.

C. RUN RESULTS

Experimental conditions for the fractional factorial design runs are shown in Table VI. Average filtrate flow rates and ash contents are shown in Table VII. Run procedures are listed in a previous chapter.

Run LCF-1

In run LCF-1 the pressure was controlled to 25 psig. Sixteen grams (0.05 lbm/ft^2) of BW-40 cellulose fiber filter aid was added to the precoat liquid for precoating filter. The clean precoat liquid was derived from previously filtered material. Body mix of filter aid was not used in this run. The run data are tabulated in Table VIII, and Figures 8 and 9 show plots of time versus filtrate volume and $\frac{\Theta}{V/A}$ versus V/A, respectively. The average filtrate flow rate was 0.3063 gal/hr/ft^2 in this run and compares favorably with work done by investigators at ORNL[14, 26] with similar material using Johns-Manville asbestos-diatomaceous earth filter aid. For this run 1.4 liters of filtrate was

TABLE IV

FRACTIONAL FACTORIAL EXPERIMENT DESIGN*

Factor Variable 1	Factor Variable 2	Factor Variable 3	Factor Variable 4
-	-	-	-
+	-	-	+
-	+	-	+
+	+	-	-
-	-	+	+
+	-	+	-
-	+	+	-
+	+	+	+

* - Factor low level

+ Factor high level

TABLE V

FRACTIONAL FACTORIAL EXPERIMENT DESIGN
RUN CONDITIONS

Run Number	Pressure, psig	Precoat, lbm/ft^2	Body Mix†, %	Filter Aid
1	25	0.025	0	BW-20
2	50	0.025	0	BW-40
3	25	0.050	0	BW-40
4	50	0.050	0	BW-20
5	25	0.025	1	BW-40
6	50	0.025	1	BW-20
7	25	0.050	1	BW-20
8	50	0.050	1	BW-40

†Body mix of filter aid added at rate of 1 gram/100 grams feed turbidity.

TABLE VI

EXPERIMENTAL RUN CONDITIONS FOR THE CONSTANT PRESSURE FILTRATION SYSTEM

Run Number	Pressure, psig	Precoat, lbm/ft^2	Body Mix, %	Filter Aid
LCF-1	25	0.050	0	BW-40
LCF-2	25	0.025	0	BW-20
LCF-3	45	0.050	0	BW-20
LCF-4	50	0.025	0	BW-40
LCF-5	25	0.050	1	BW-20
LCF-6	25	0.025	1	BW-40
LCF-7	50	0.050	1	BW-40
LCF-8	50	0.025	1	BW-20
LCF-3*	40	0.050	0	BW-20
		Optimization Runs		
OPT-1	40	0.10	0	BW-20
OPT-2	40	0.20	0	BW-20
OPT-3	40	0.40	0	BW-20
OPT-4	40	0.30	0	BW-20

*Repeat

TABLE VII

FILTRATION RATE AND ASH CONTENT FOR
SRC FILTERED OIL

Run Number	Filtration Rate, gal/hr/ft^2	Ash Content, %
LCF-1	0.3063	0.02
LCF-2	0.1467	0.08
LCF-3	0.7465	0.25
LCF-4	0.2450	0.12
LCF-5	0.1422	0.09
LCF-6	--	--
LCF-3*	1.148	0.05
OPT-1	1.590	0.04
OPT-2	3.281	0.04
OPT-3	2.901	0.03
OPT-4	1.54	0.02

*LCF-3 repeat

TABLE VIII

RUN DATA (LCF-1)

Filtration Time Θ, min	Filtrate Volume V, liters	$\frac{\Theta}{(V/A)}$	$\frac{V}{A}$
0	0	0	0
3	0.275	7.52	0.398
6	0.375	11.04	0.543
9	0.475	13.07	0.688
12	0.500	16.56	0.724
15	0.530	19.52	0.768
20	0.625	22.08	0.905
25	0.685	25.18	0.992
30	0.732	28.27	1.060
60	1.000	41.40	1.449
90	1.150	54.00	1.666
105	1.400	51.75	2.028

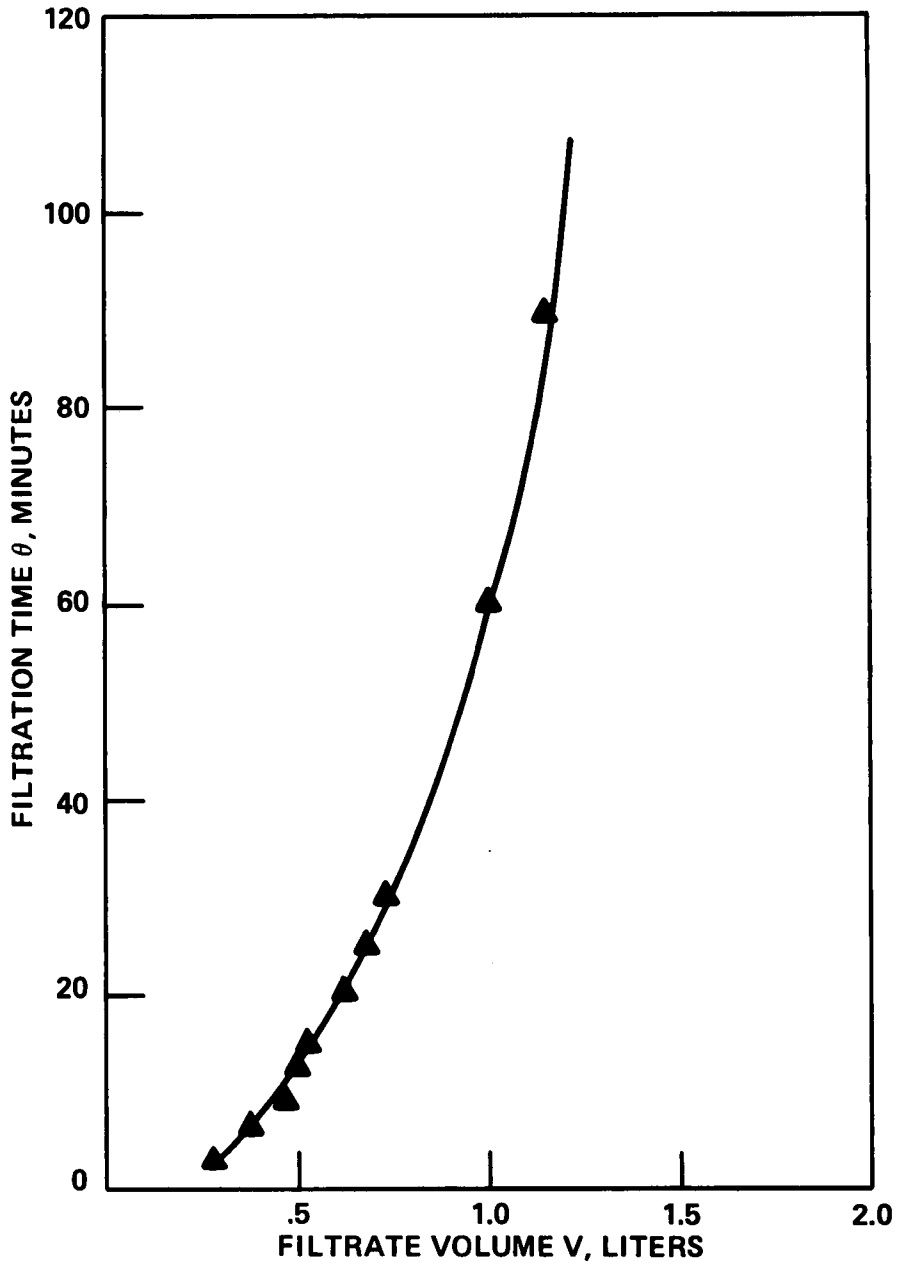


Figure 8. FILTRATION TIME VERSUS FILTRATE VOLUME FOR LCF-1

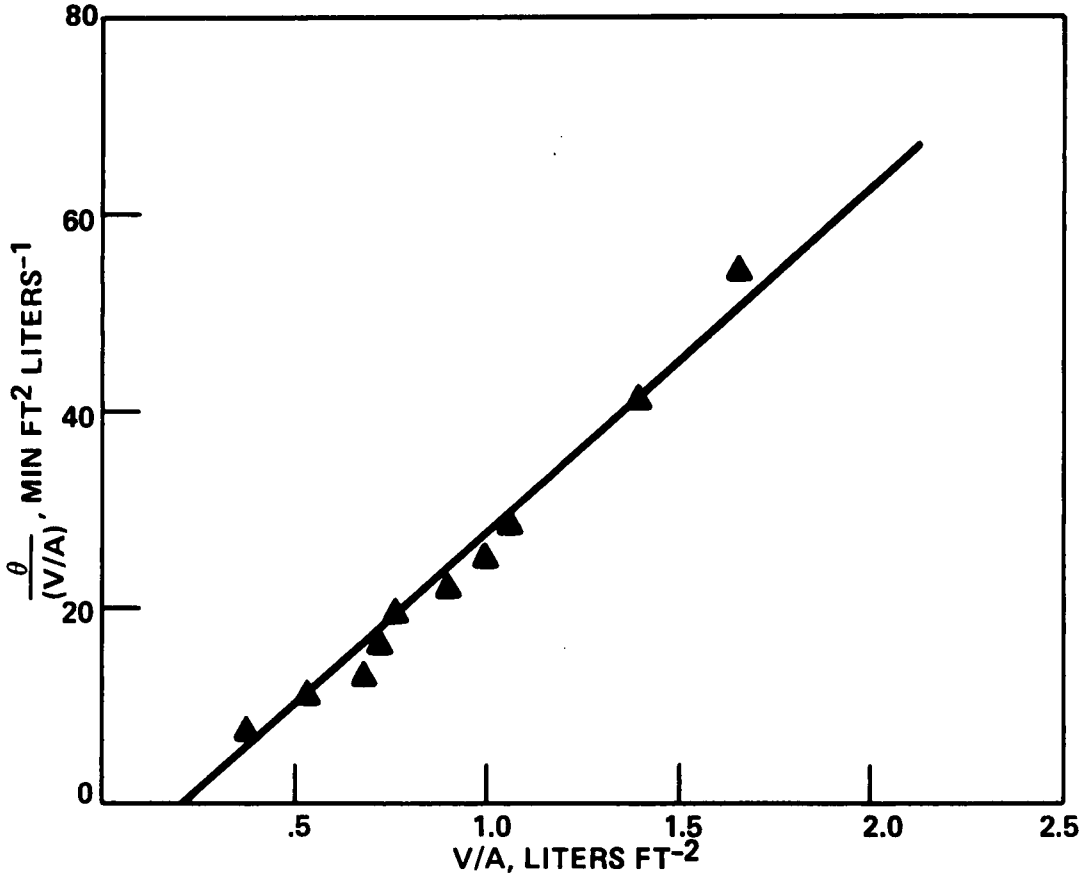


Figure 9. $\frac{\theta}{(V/A)}$ VERSUS V/A FOR LCF-1

collected with an ash content of 0.02%. Ash levels[8, 20, 26] below 0.15% are considered to be adequate for boiler fuels, based on EPA[8] guidelines. The average specific cake resistance was calculated to be 5.86 as shown in Table IX. (The calculation procedure and units are explained in Appendix A.) Complete sample analyses for both feed solution and product solution are listed in Appendix B. Pyridine insolubles content, a measure of unconverted coal present in the feed and product solutions, showed a sizable reduction from 5.82% down to 0.04%.

Run LCF-2

In this run the pressure was adjusted to 25 psig. However, for this run 8 grams of BW-20 cellulose fiber filter aid was added to the precoat liquor for a coverage rate of 0.025 lbm/ft^2. Body mix was not used. Time versus filtrate volume data is tabulated in Table X and shown graphically in Figures 10 and 11. The average filtrate flow rate was 0.1467 gal/hr/ft^2 and shows a substantial reduction with the reduced filter aid usage. The filter cake was not as well formed as in run LCF-1. Unconverted coal and ash levels were reduced to 0.17% and 0.08%, respectively. The average specific cake resistance was 13.98, and the filter medium resistance was essentially zero.

Run LCF-3

For this run, the pressure was increased to around 50 psig. In this run some difficulty was experienced in adjusting system feed pressure to the 50-psig level. There was considerable cycling of pressure throughout the first part of the run, and the pressure could only be controlled to 45 psig. As in run LCF-1, the filter cake was well formed and was discharged easily. The run data are shown in Table XI. Plots

TABLE IX

EVALUATION OF FILTRATION
EQUATION PARAMETERS

Run Number	Pressure, psig	Slope, $\frac{\mu\alpha W}{2\Delta P}$	Specific Cake Resistance, $\alpha^{\dagger}$	Intercept, $\frac{\mu r}{\Delta P}$	Filter Medium Resistance, $r^{\dagger}$
LCF-1	25	34.67	5.86 (28.1)	--	--
LCF-2	25	85.71	13.98 (28.1)	--	--
LCF-3	45	5.00	1.21 (37.1)	7.8	5.8
LCF-4	50	88.89	25.63 (38.9)	--	--
LCF-5	25	281.25	31.30 (28.1)	--	--
LCF-6	--	--	--	--	--
LCF-3*	40	3.44	5.73	3.00	4.28
OPT-1	40	1.09	1.63	7.3	9.73
OPT-2	40	0.517	0.957	3.10	4.13
OPT-3	40	0.290	2.09	3.80	5.06
OPT-4	40	2.27	1.84	4.87	4.99

*LCF-3 repeat

†Units are shown in Appendix A. Numbers in parentheses represent cake resistance determinations from compressibility considerations.

TABLE X

RUN DATA (LCF-2)

Filtration Time Θ, min	Filtrate Volume V, liters	$\frac{\Theta}{(V/A)}$	$\frac{V}{A}$
0	0	0	0
30	0.575	36	0.833
60	0.775	53.41	1.123
90	0.900	69.00	1.304
120	0.975	84.92	1.413
150	1.060	97.64	1.536
180	1.150	108.00	1.666

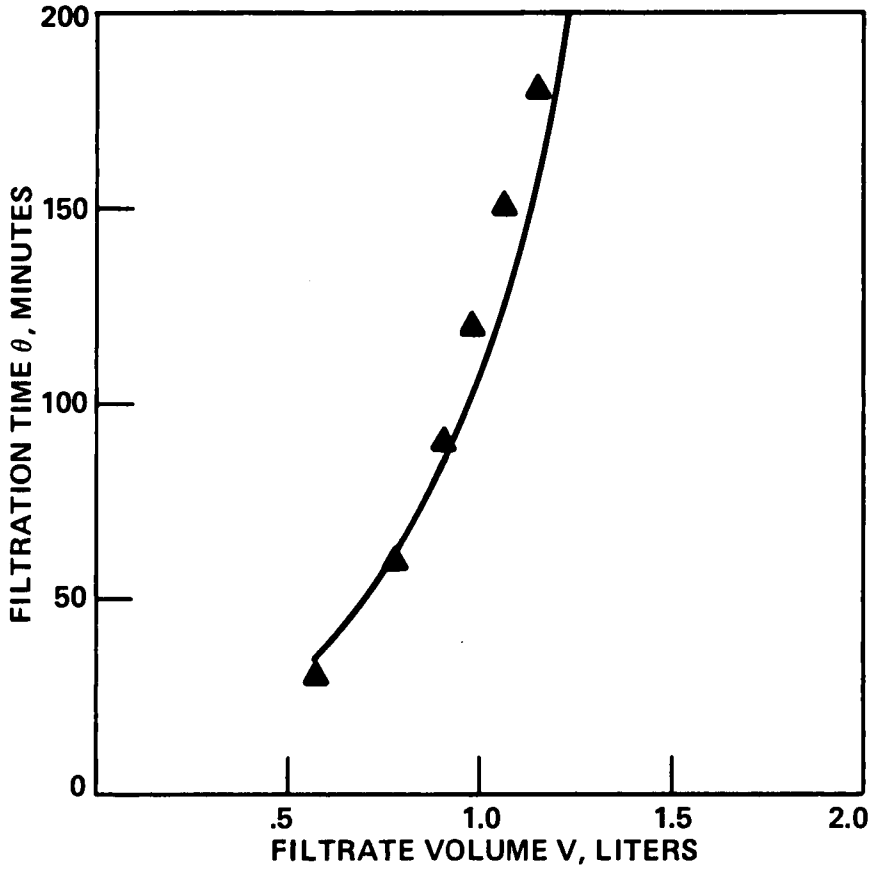


Figure 10. FILTRATION TIME VERSUS FILTRATE VOLUME FOR LCF-2

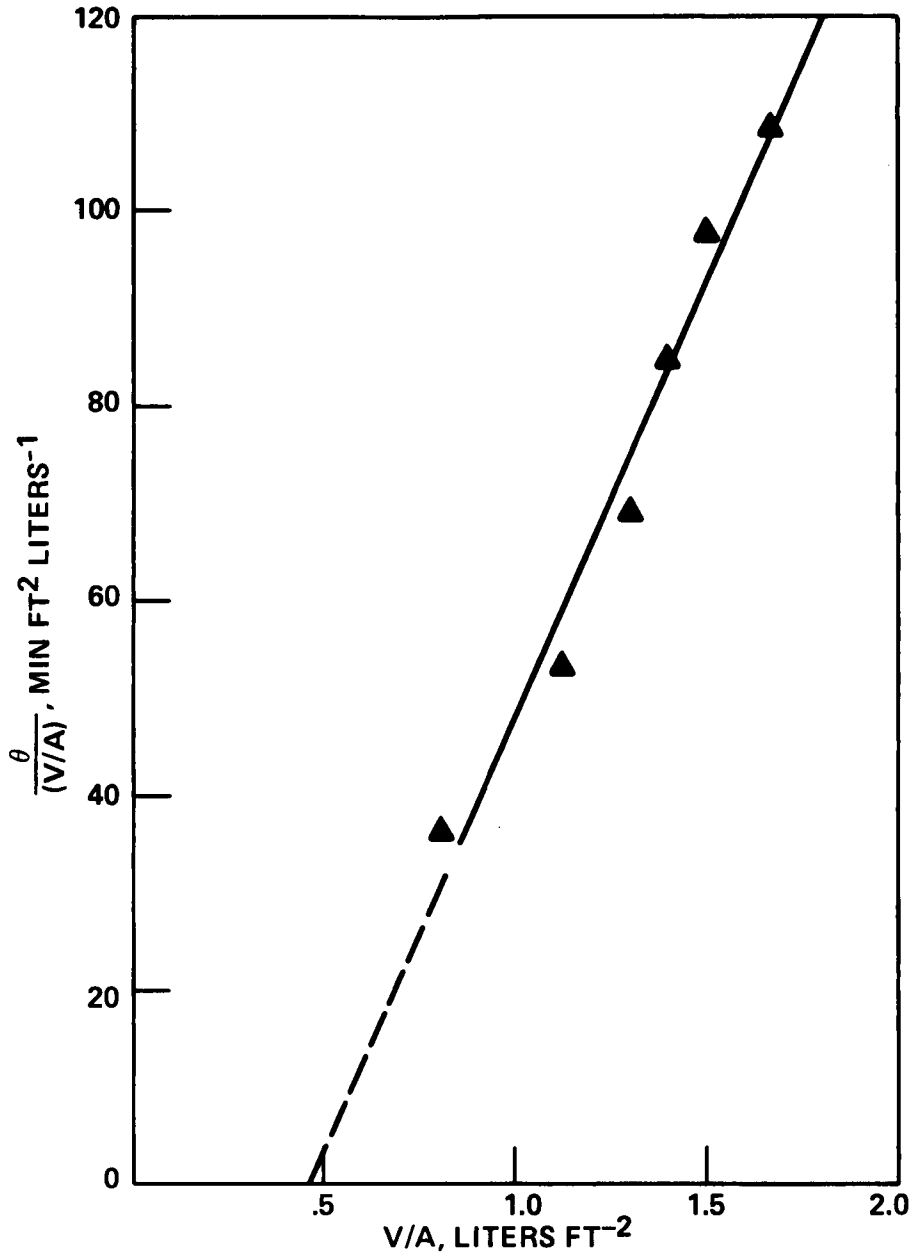


Figure 11. $\frac{\theta}{(V/A)}$ VERSUS V/A FOR LCF-2

TABLE XI

RUN DATA (LCF-3)

Filtration Time Θ, min	Filtrate Volume V, liters	$\frac{\Theta}{(V/A)}$	$\frac{V}{A}$
0	0	0	0
20	0.975	14.15	1.413
25	1.060	16.27	1.536
30	1.300	15.92	1.884
35	1.400	17.25	2.028
40	1.500	18.40	2.173
45	1.610	19.28	2.333
60	1.950	21.23	2.826

of time versus filtrate volume and the parameters $\frac{\Theta}{V/A}$ versus V/A are shown in Figures 12 and 13, respectively. The ash level was much higher for this run, as shown in Table VII, page 47. A flow rate of 0.7465 gal/hr/ft^2 was obtained in this run and probably represents the effect of higher pressure (45 psig) and high precoat level (0.050 lbm/ft^2). The effect of using BW-20 filter aid might have had some effect. The average cake resistance was calculated to be 1.21 and the filter medium resistance 5.85.

Run LCF-4

Run LCF-4 yielded a filtrate flow of 0.245 gal/hr/ft^2 at a pressure drop of 50 psig. Eight grams of BW-40 filter aid was used for precoat. Body mix was not used. Poor filter cake formation was experienced in this run as was the case in LCF-2 and may be due to inadequate precoating. Ash content and pyridine insolubles levels were 0.12% and 0.79%, respectively. Run data and time versus filtrate volume are shown in Table XII and Figures 14 and 15. The cake resistance and filter medium resistance were 25.63 and zero, respectively.

Run LCF-5

Run LCF-5 was the first run utilizing body mix of filter aid as well as precoat. In this run a precoat of 16 grams of BW-20 filter aid was applied to the filter leaves and around 25 grams of BW-200 filter aid was added per 100 grams of dirty solids in the feed tank solution. Cellulose fiber BW-200 is a much smaller sized filter aid than either BW-20 or BW-40. The flow was essentially zero after about 40 minutes of filtration at 25 psig and 120°F. Also during the filtering period the gear pump mechanical seal was leaking badly. It was discovered that

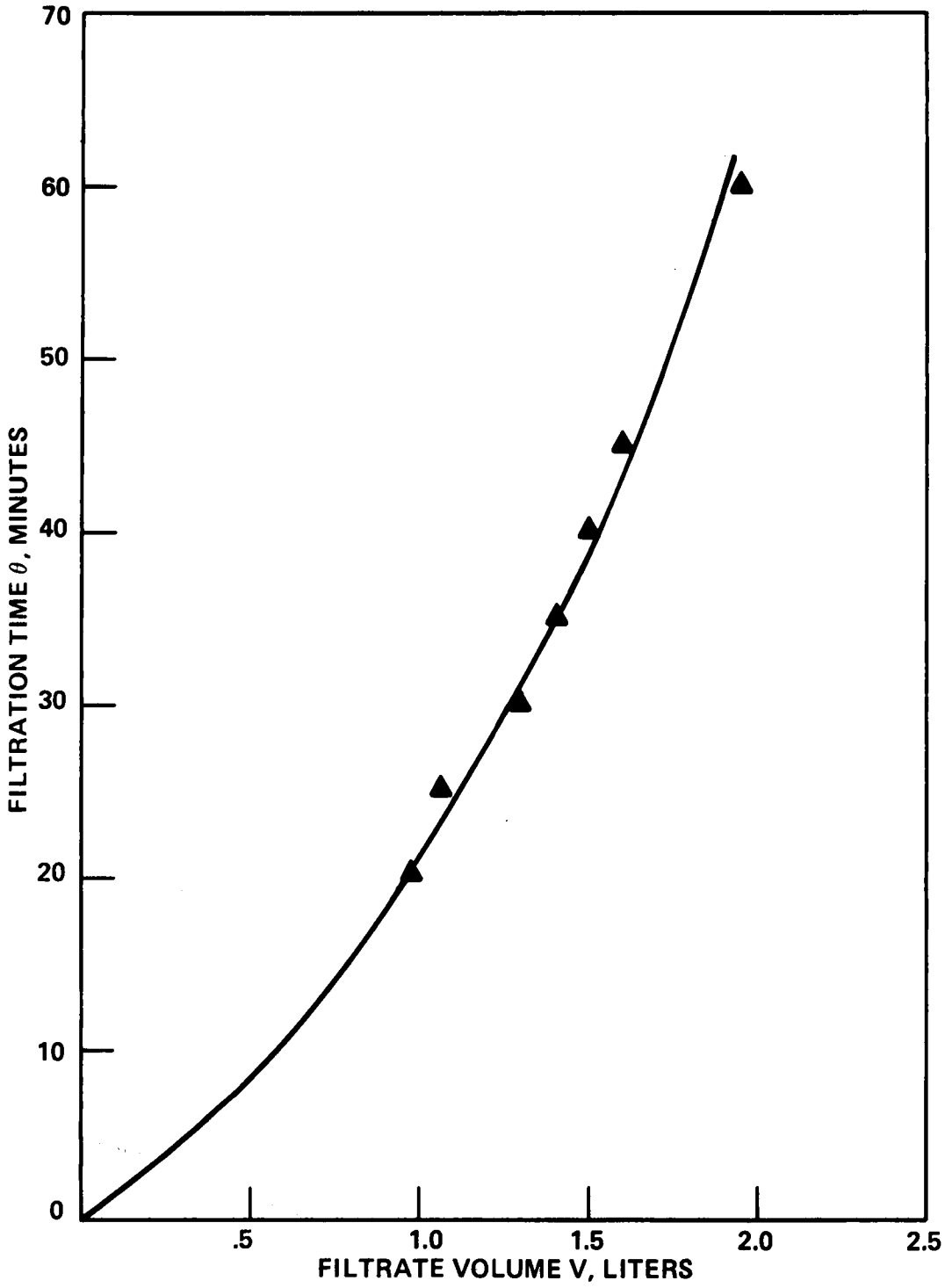


Figure 12. FILTRATION TIME VERSUS FILTRATE VOLUME FOR LCF-3

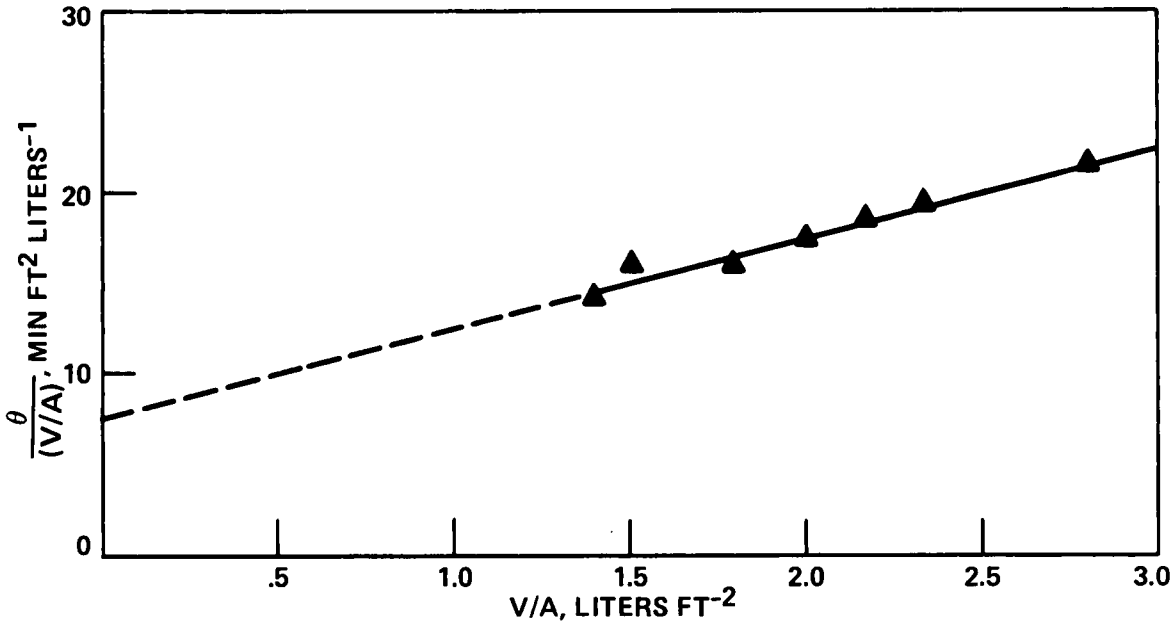


Figure 13. $\frac{\theta}{(V/A)}$ VERSUS V/A FOR LCF-3

TABLE XII

RUN DATA (LCF-4)

Filtration Time Θ, min	Filtrate Volume V, liters	$\frac{\Theta}{(V/A)}$	$\frac{V}{A}$
0	0	0	0
5	0.350	9.86	0.507
10	0.400	17.27	0.579
15	0.420	24.67	0.608
30	0.550	37.64	0.797
45	0.675	46.01	0.978
60	0.750	55.24	1.086
75	0.800	64.71	1.159

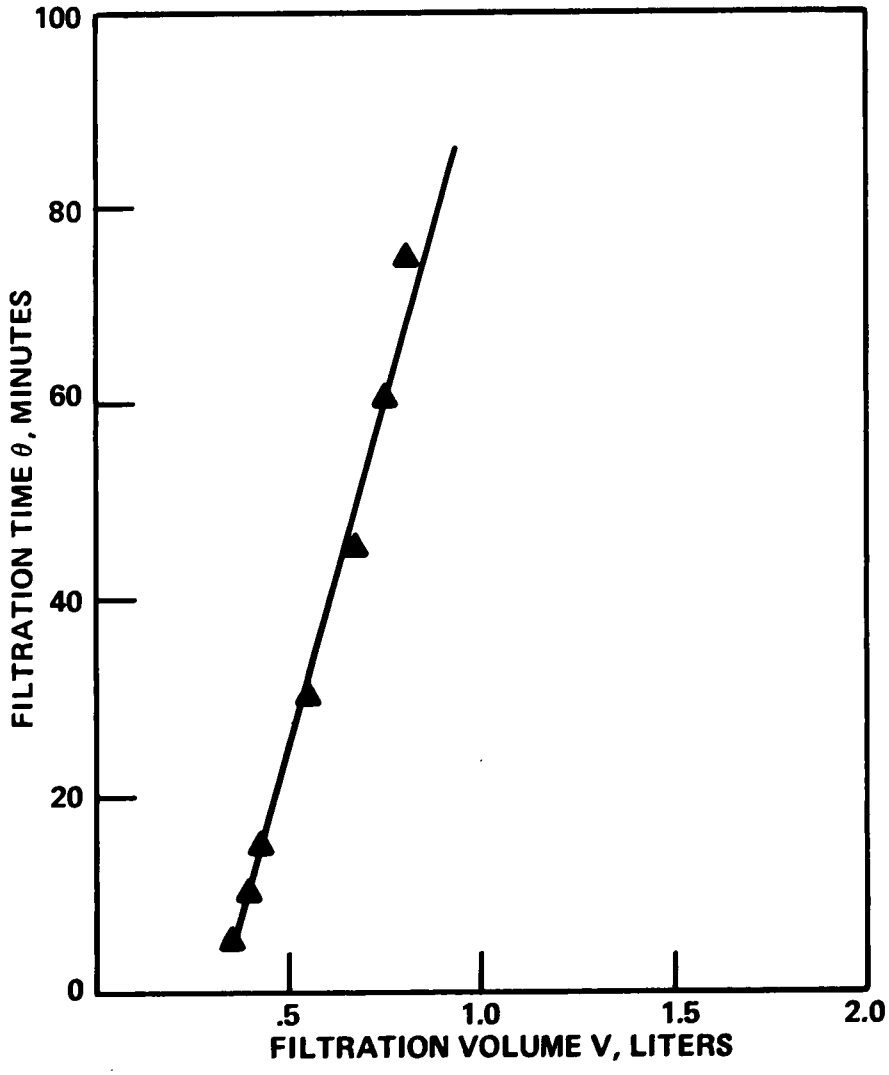


Figure 14. FILTRATION TIME VERSUS FILTRATE VOLUME FOR LCF-4

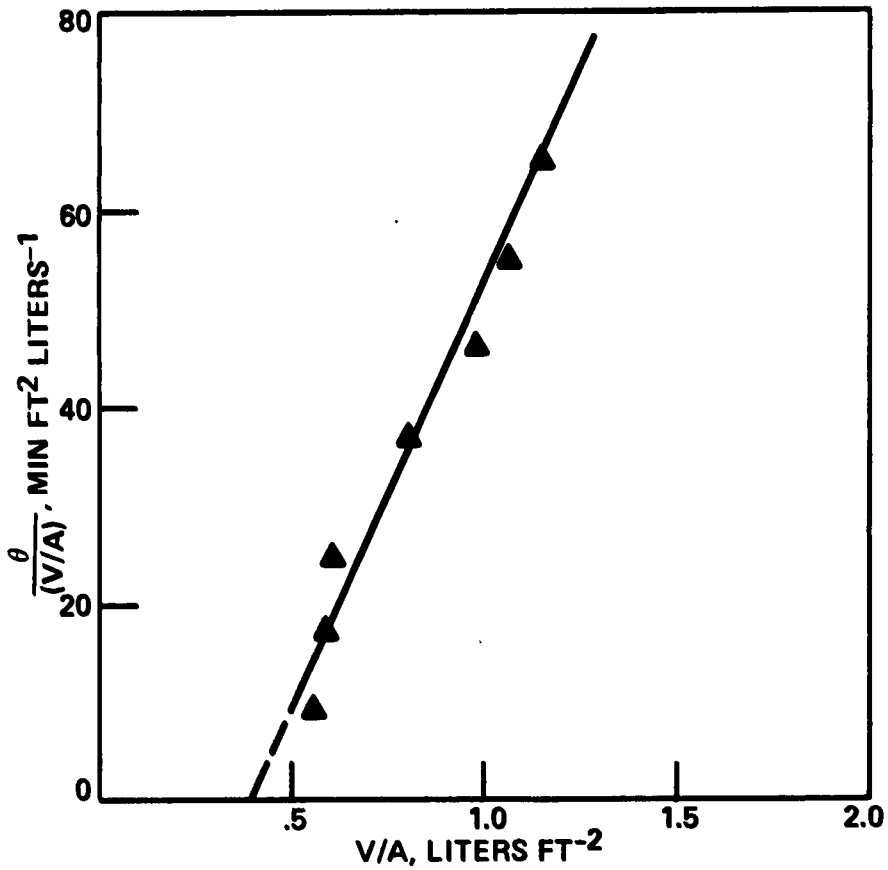


Figure 15. $\frac{\theta}{(V/A)}$ VERSUS V/A FOR LCF-4

this rate of body mix (25%) was much too high and that levels of body mix around 1 to 5% should be used. Higher body mix rates can cause an actual increase in filtration resistance.[9, 36] It is also assumed to use the same filter aid for both precoat and body mix. When the amount of filter aid added is so small that the particles do not interact and form a coherent structure, resistance may be adversely affected. Run LCF-5 was repeated with a 1% body mix rate with the same results. Upon disassembling the filter, the leaves were apparently plugged inside the filtrate channels. Again the leaves were cleaned thoroughly and reassembled. This time the run was completed with an extremely low (0.07 gal/hr/ft^2) filtrate rate. The filter cake was not well formed. At this point the use of body mix itself came under question, as previous runs had not shown this phenomenon.

Run LCF-3 was repeated to see if the system would respond as before and to try to isolate problems with run LCF-5. As in the two earlier attempts, the filtrate flow was essentially zero. At this point it seemed obvious that either the filter leaves had become blinded or that the precoat liquor was dirty. The filter leaves were washed with hot methylene chloride and scrubbed with 20% HNO_3 acid. A final rinse using perchloroethylene was given to the filter leaves. A third attempt was made for run LCF-5, and the run data are shown in Table XIII. The filtrate flow was 0.1422 gal/hr/ft^2, and the filter cake was somewhat uniform. However, it does appear that body mix of cellulose fiber filter aid was not beneficial; and further research revealed that Gordon Smith, a leader in the solid-liquid separation field, states that cellulose fibers[33] may result in poor-flow-pressure drop relationships when used as body mix. Filtrate volume versus time plots are shown for LCF-5

TABLE XIII

RUN DATA (LCF-5)

Filtration Time Θ, min	Filtrate Volume V, liters	$\frac{\Theta}{(V/A)}$	$\frac{V}{A}$
0	0	0	0
10	0.45	15.33	0.652
20	0.48	28.77	0.695
30	0.52	39.84	0.753
45	0.57	54.47	0.826
60	0.60	69.04	0.869
105	0.65	111.46	0.942

in Figures 16 and 17. Ash content and pyridine insolubles were 0.09% and 1%, respectively.

Run LCF-6

This run exhibited a very low flow rate such that no data could be taken. The pressure was adjusted to 25 psig. Eight grams of BW-40 was used for both precoat and body mix. It appeared that again the body mix offered no additional flow increase over precoating as in run LCF-5 and, in fact, was seriously detrimental; therefore, body mix runs LCF-7 and LCF-8 were eliminated from the experimental design. (One advantage of fractional factorial experiment design is that the exploration of one factor can be abandoned in midstream without reducing the validity of the remaining experiments.)

Run LCF-3 Repeat

Run LCF-3 was repeated since such low flow rates were experienced in runs LCF-5 and LCF-6. The flow rate was a quite reasonable 1.14 gal/hr/ft^2 and further supported the contention that the previous difficulties were due to the body mix effect, and not a system problem. The volume versus time data for this repeat run are shown in Table XIV. The plots of filtrate volume versus time and $\frac{\Theta}{(V/A)}$ versus V/A are shown in Figures 18 and 19. The ash content was 0.05%, and the pyridine insolubles content was 0.12%. Cake resistance and filter medium resistance were 5.73 and 4.28, respectively, which compares favorably with these determinations for the first run. Feed material for this run was from the second drum of the two originally supplied by Catalytic, Inc., and the variations in results from the original LCF-3 can easily be explained as due to this.

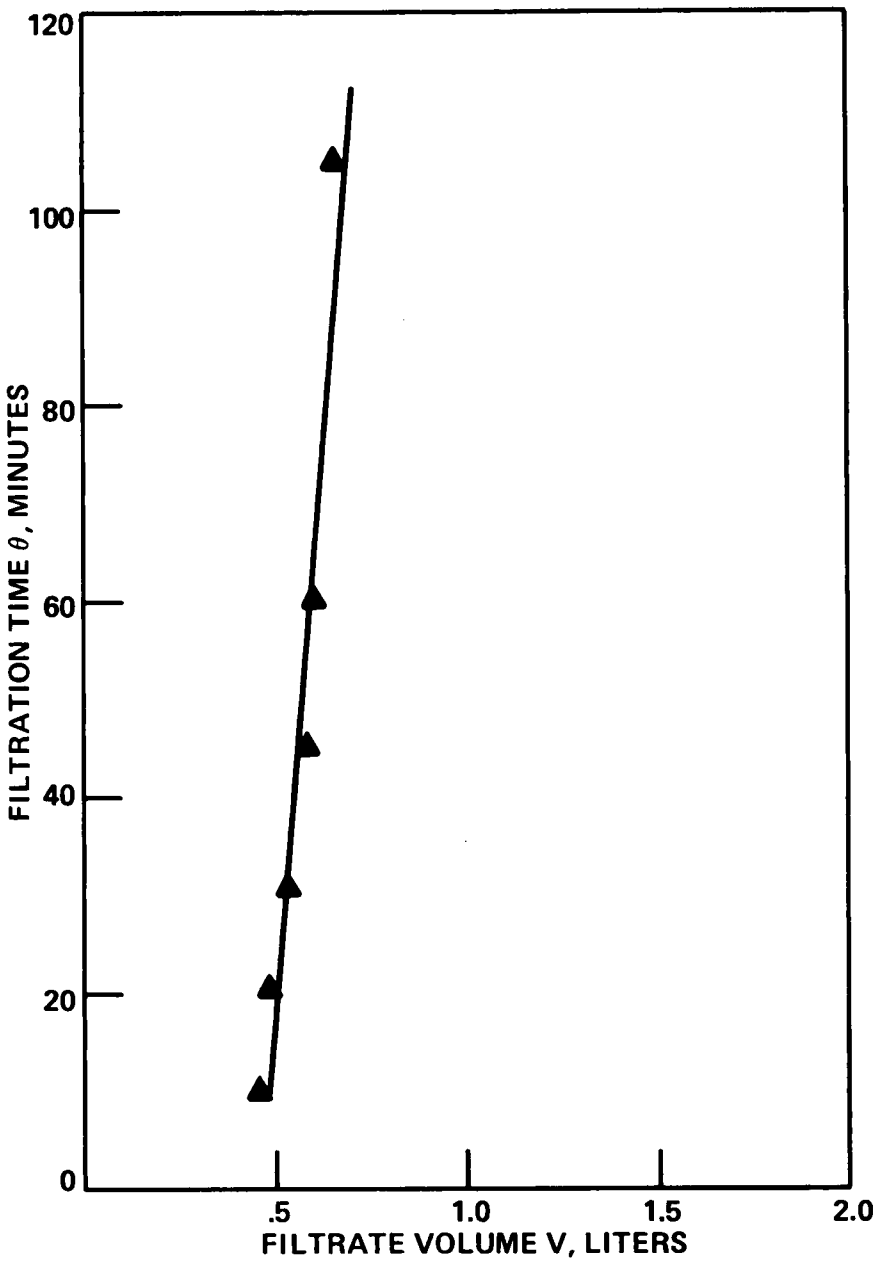


Figure 16. FILTRATION TIME VERSUS FILTRATE VOLUME FOR LCF-5

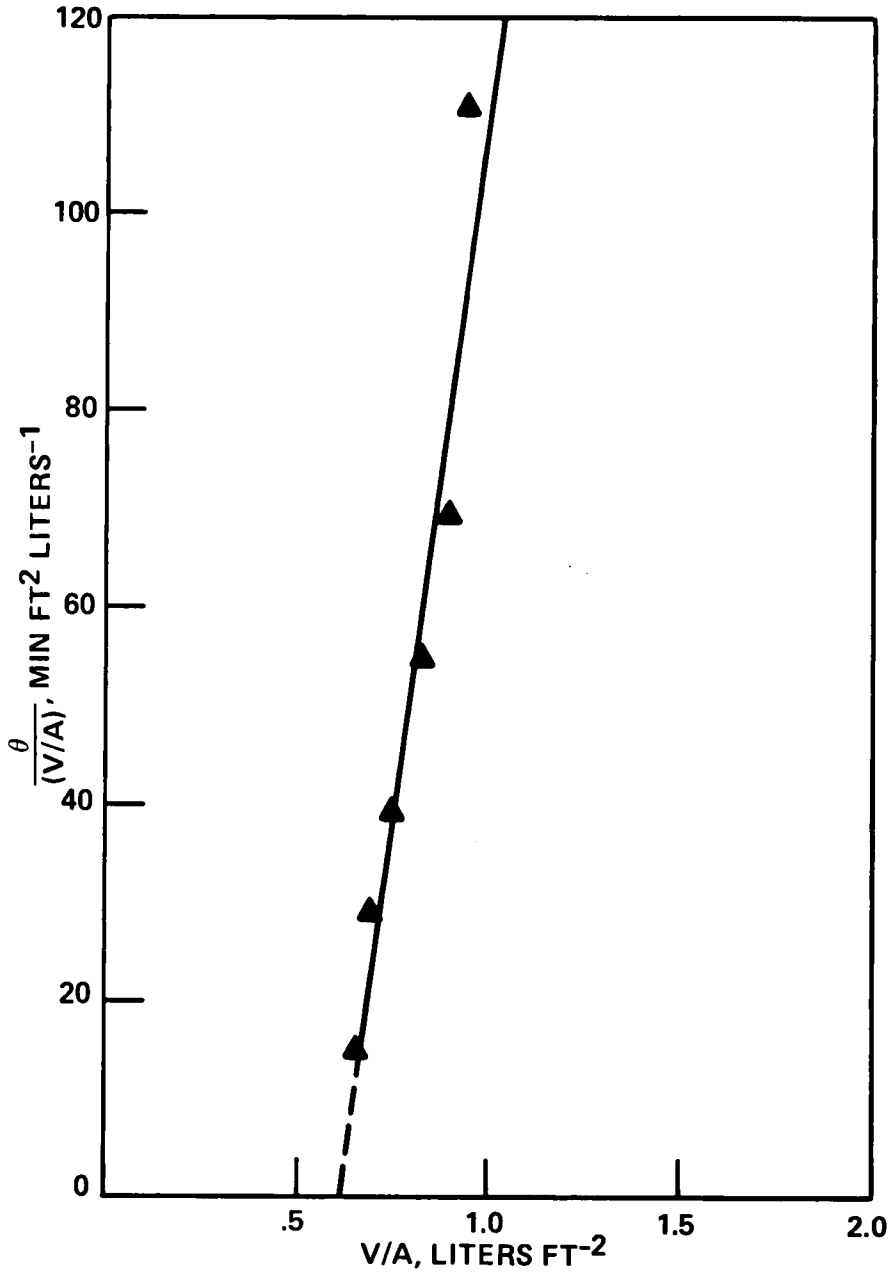


Figure 17. $\frac{\theta}{(V/A)}$ VERSUS V/A FOR LCF-5

TABLE XIV

RUN DATA (LCF-3 REPEAT)

Filtration Time Θ, min	Filtrate Volume V, liters	$\frac{\Theta}{(V/A)}$	$\frac{V}{A}$
0	0	0	0
20	1.373	10.055	1.989
30	1.831	11.307	2.653
40	2.112	13.071	3.060
45	2.250	13.803	3.260

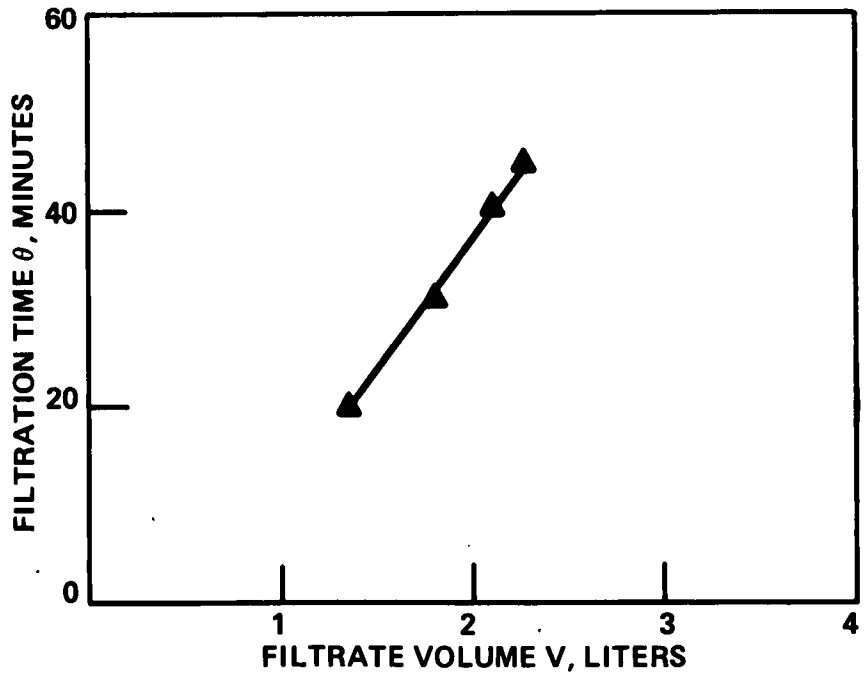


Figure 18. FILTRATION TIME VERSUS FILTRATE VOLUME FOR LCF-3 REPEAT

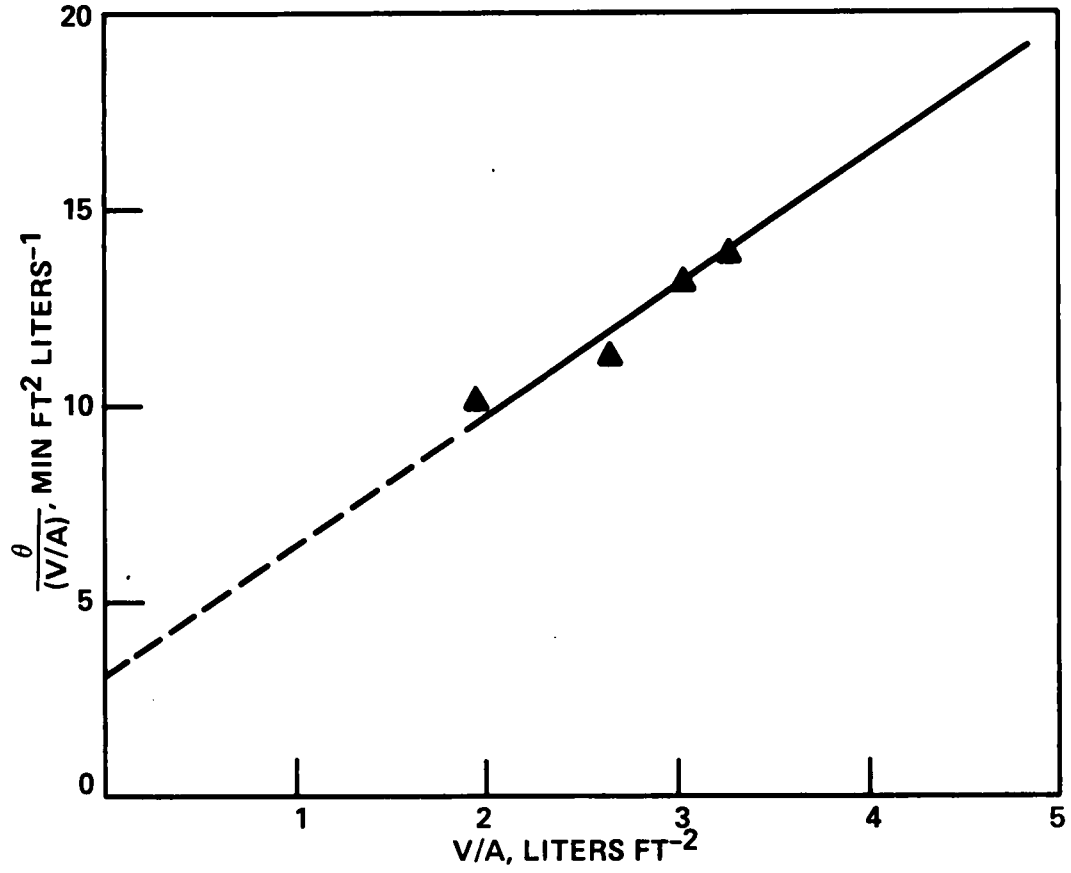


Figure 19. $\frac{\theta}{(V/A)}$ VERSUS V/A FOR LCF-3 REPEAT

Discussion of Fractional Factorial Runs Results

The factorial design run data yielded useful information. Table XV shows the procedure[19] for analyzing the factorial runs results. Since the body mix runs were deleted, the data represent a subset of the original design, and some very general conclusions were reached. Since in most runs the product solution ash contents were satisfactory, indicating that this should not be a problem with cellulose fiber filtration methods, the filtrate flow rate was chosen as the main response variable. The procedure suggested that the precoat level or amount of filter aid for precoating purposes had the greatest influence on the flow, pressure level the second greatest contributing factor. Filter aid particle size had very little, if any, effect on the filtrate flow rate. The variation of the flow with precoat level was 0.3306 lbm/ft^2 per $gal/hr/ft^2$. The rates obtained for runs LCF-1 - 5 were somewhat higher than data reported by the Oak Ridge National Laboratory[26] with Solvent Refined Coal materials at similar pressures and for higher temperatures using higher levels of diatomaceous earth-asbestos precoat. For instance, for filtration at 100°C and 25 psig, filtrate flows of 0.2 to 0.5 $gal/hr/ft^2$ were obtained for deposited cakes in the range of 1/8 to 1/4 inch. At these temperatures, the viscosity of the feed solution would be around 1 to 5 CP. The viscosity of the SRC feed material for runs LCF-1 - 5 was around 60 CP and the flows should be increased proportionately for the higher temperatures which apply to the ORNL data.

Cake and filter medium resistance determinations are shown in Table IX, page 52. A plot of log α versus log ΔP is shown in Figure 20 for runs LCF-2 and LCF-4, although this is not strictly a valid procedure for determining pressure effects because these runs were not made using

TABLE XV

FRACTIONAL FACTORIAL EXPERIMENT ANALYSIS

Run Number	Flow Rate, gal/hr/ft^2	Pressure Factor Level	Precoat Factor Level	Body Mix Factor Level	Filter Aid Factor Level
LCF-2	0.1467	-	-	-	-
LCF-4	0.2450	+	-	-	+
LCF-1	0.3063	-	+	-	+
LCF-3	0.7465	+	+	-	-
Σ+	1.4445	0.9915	1.0528	0	0.5513
Σ-	0	0.4530	0.3917	1.4445	0.8932
Difference	1.4445	0.5385	0.6611	-1.4445	-0.3419
Effect	0.3611	0.2693	0.3306	-0.7223	-0.1710

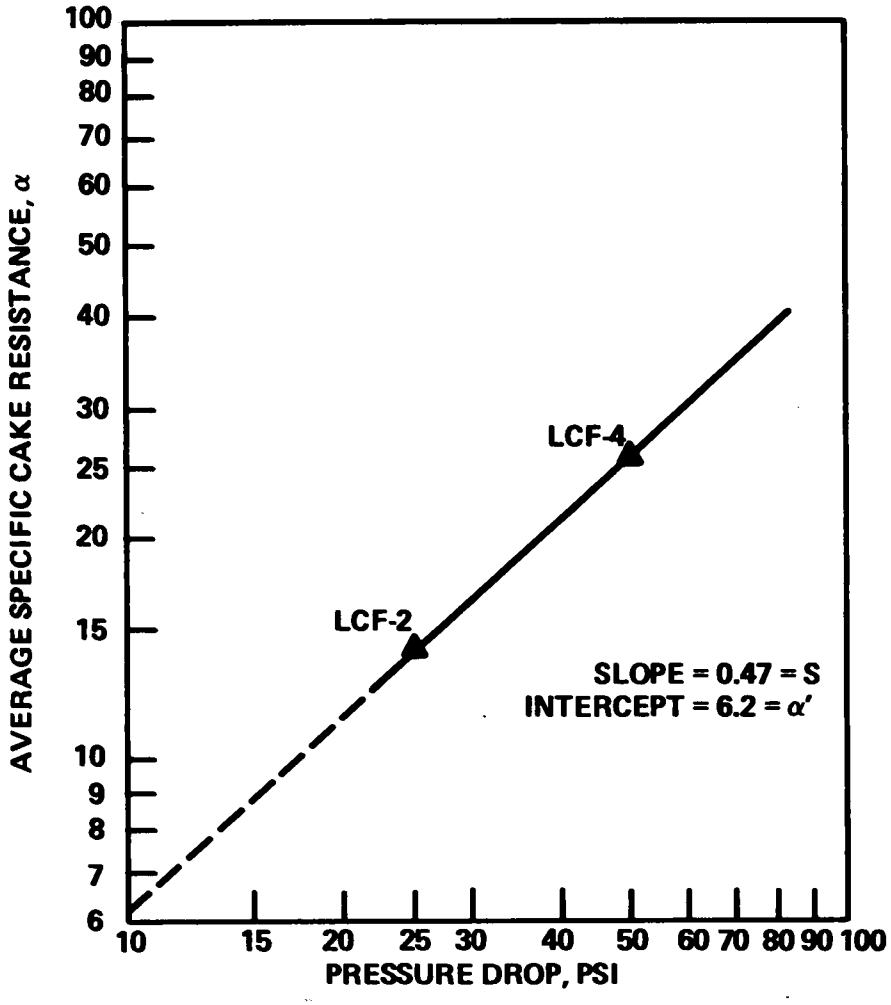


Figure 20. LOG OF AVERAGE SPECIFIC CAKE RESISTANCE VERSUS LOG PRESSURE DROP

the same grade of filter aid. However, the grade of filter aid in the factorial analysis had little effect. The slope or cake compressibility was determined to be 0.47, and the intercept or particle size constant α' was 6.2. There was fairly good agreement between cake resistance determinations from the compressibility factor relationship and calculations as outlined in Appendix A. The cake and filter medium resistance determined by the slope and intercept of the filtrate volume versus time plots should be adequate for similar feed materials, however. Figure 21 shows a comparison of filtrate volume versus time for runs LCF-1 through LCF-5. Runs LCF-1 and LCF-3 clearly show the effect of higher flows for increased precoat usage. Figure 22 shows the lower slopes for LCF-1 and LCF-3 which reduce specific cake resistances.

D. OPTIMIZATION RUNS

The fractional factorial runs results showed a sizable influence on average filtration rate for higher filter aid usage rates. Additionally, the use of BW-20 rather than BW-40 cellulose fiber filter aid was marginally indicated; and higher pressure levels were also favored. Four runs were made at 40 psig to demonstrate the effect of precoat usage level on the average filtration rate for the Solvent Refined Coal liquefaction intermediate. These runs were made with the second of the two feed drums of material that was sent from Catalytic, Inc., in Wilsonville, Alabama. As shown in Appendix B, the viscosity at 120°F and feed solids concentration were around 30 CP and 2.5%, respectively, for the optimization runs. These values differed by a factor of two from those obtained on analysis of the first drum, even though both drums were obtained from a run made during a similar time period in the liquefaction facility.

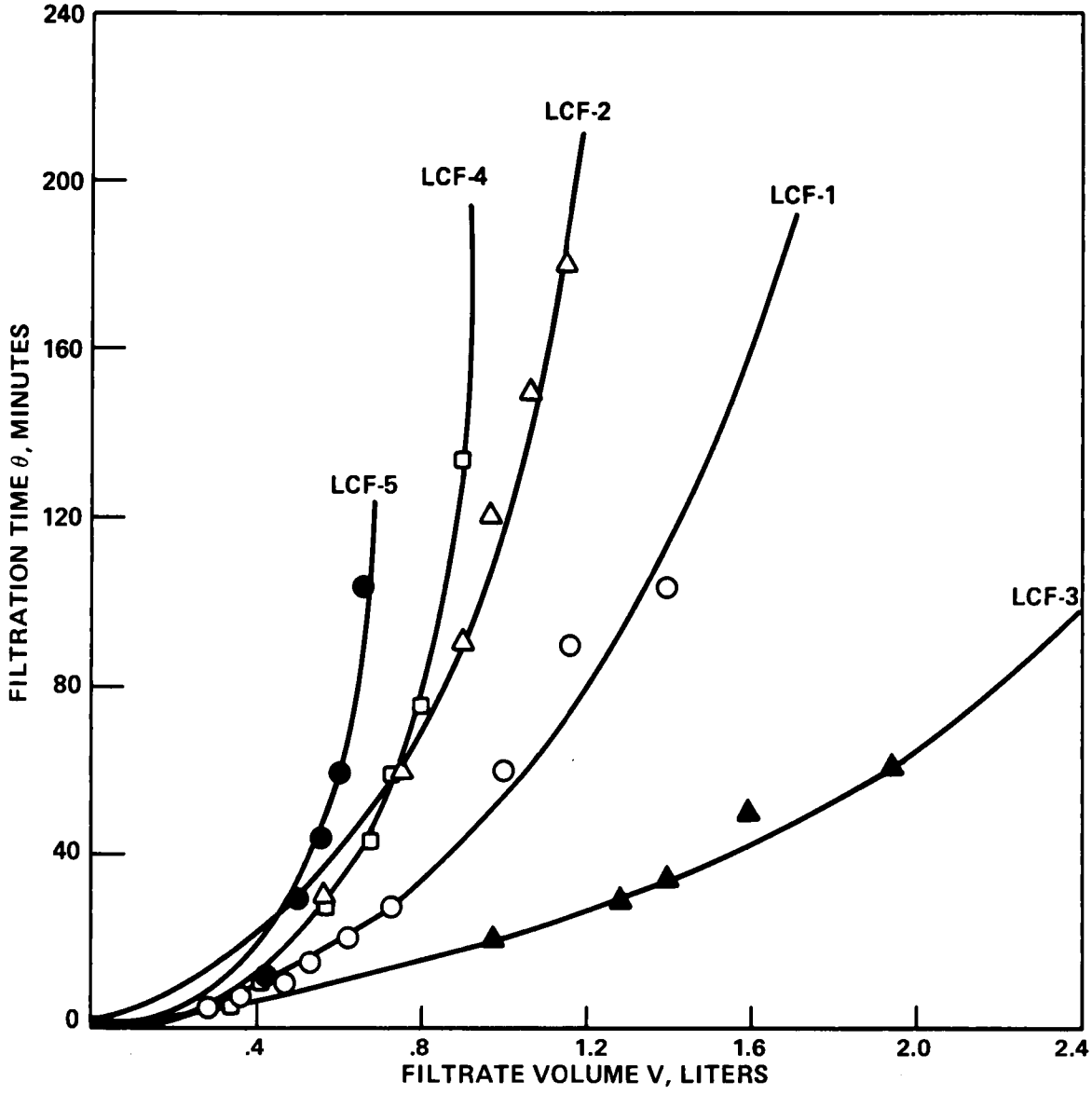


Figure 21. FILTRATION TIME VERSUS FILTRATE VOLUME FOR LCF-1, 2, 3, 4, 5

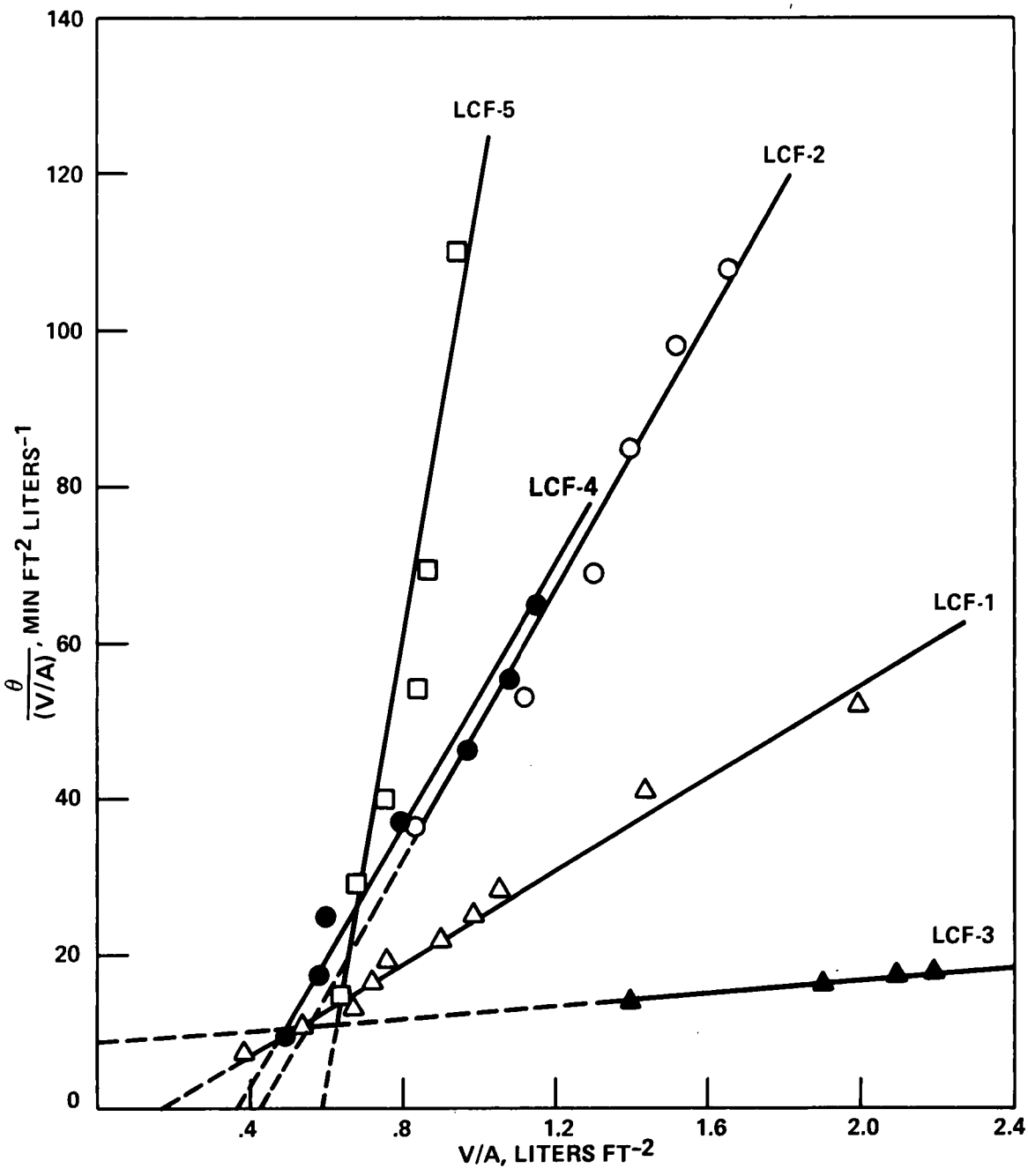


Figure 22. $\frac{\theta}{(V/A)}$ VERSUS V/A FOR LCF-1, 2, 3, 4, 5

Run OPT-1

This run was the first in a series for determining optimum filter aid usage for maximum filtrate flow. Thirty-two grams of BW-20 filter aid was used for a coverage rate of 0.1 lbm/ft^2. The pressure was adjusted to 40 psig. Clean precoat liquid was obtained from Catalytic, Inc., and represented "filtered liquor" from their pilot scale pressure filter. Run data are shown in Table XVI. The average filtration rate was 1.59 gal/hr/ft^2 and represented more than a two-fold increase over the rate obtained in LCF-3 (0.7465 gal/hr/ft^2) and somewhat higher than the rate (1.14 gal/hr/ft^2) obtained in LCF-3 repeat. The run LCF-3 repeat was also made with feed solution from the second drum. Figure 23 shows the volume versus time relationship. Approximately 1.5 liters was collected in only 20 minutes; Figure 24 exhibits the flat slope obtained with the increased precoat usage. The cake compressibility obtained by calculation in this run was 1.630 as shown in Table IX. The filter medium resistance was 9.730. Sample analyses are shown in Appendix B.

Run OPT-2

Filter aid for the precoating operation was increased to 0.2 lbm/ft^2 in this run. All other run conditions were the same. An average filtrate flow rate of 3.28 gal/hr/ft^2 was obtained. Cake and filter medium resistances were 0.957 and 4.130, respectively. Ash and pyridine insolubles content for the product solution were 0.04% and <0.01%, respectively. Run data are shown in Table XVII, and Figures 25 and 26 show filtrate volume versus time relationships. In this run the flow rate was two times larger than run OPT-1. Again, precoat usage greatly influenced the average filtrate flow rate.

TABLE XVI

RUN DATA (OPT-1)

Filtration Time Θ, min	Filtrate Volume V, liters	$\frac{\Theta}{(V/A)}$	$\frac{V}{A}$
0	0	0	0
1	0.0923	7.518	0.133
2	0.184	7.518	0.266
3	0.276	7.500	0.400
4	0.369	7.490	0.534
5	0.461	7.485	0.668
6	0.553	7.490	0.801
7	0.646	7.478	0.936
8	0.738	7.483	1.069
9	0.830	7.487	1.202
10.5	0.923	7.853	1.337
12	1.015	8.157	1.471
13	1.107	8.104	1.604
17	1.376	8.525	1.994
20.5	1.546	9.151	2.240
26.0	1.800	9.969	2.608

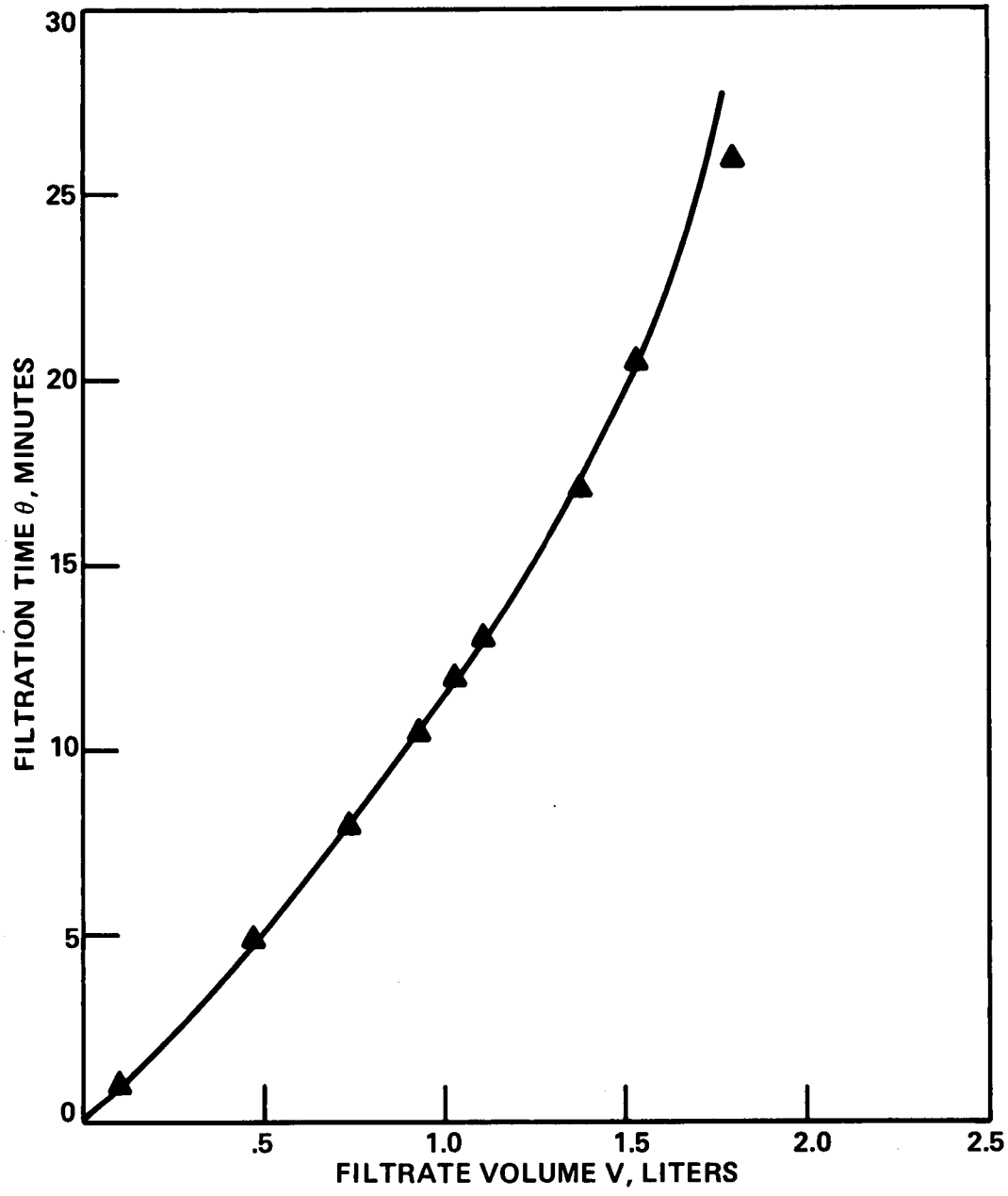


Figure 23. FILTRATION TIME VERSUS FILTRATE VOLUME FOR OPT-1

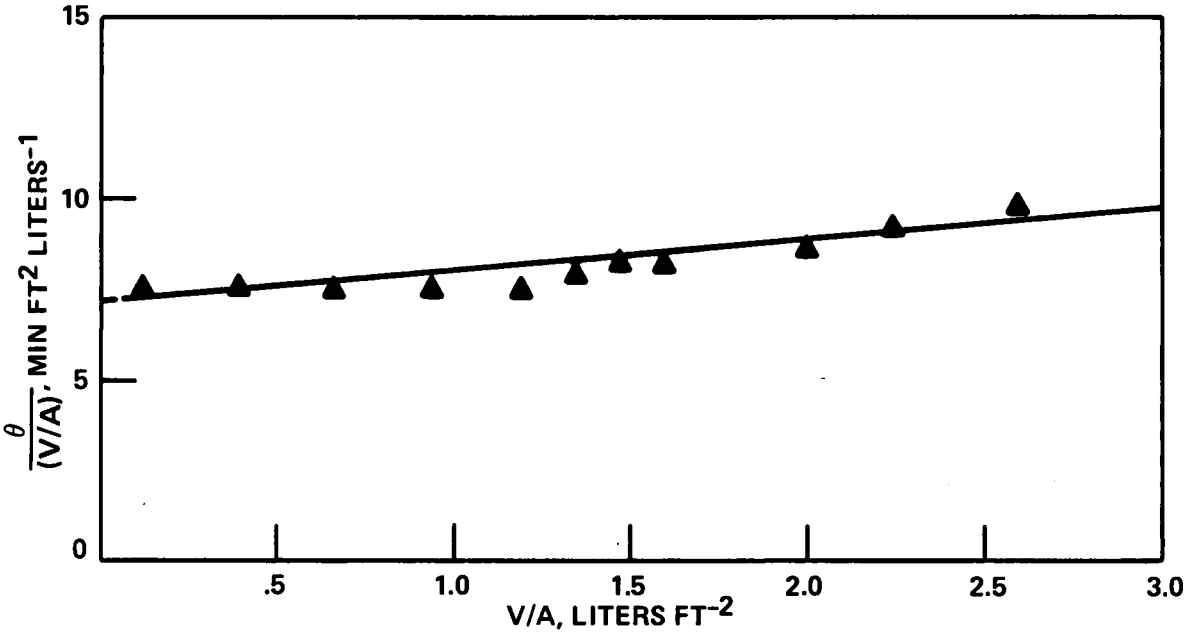


Figure 24. $\frac{\theta}{(V/A)}$ VERSUS V/A FOR OPT-1

TABLE XVII

RUN DATA (OPT-2)

Filtration Time Θ, min	Filtrate Volume V, liters	$\frac{\Theta}{(V/A)}$	$\frac{V}{A}$
0	0	0	0
1	0.2	3.46	0.289
3	0.4	5.181	0.579
4	0.8	3.451	1.159
5	1.0	3.450	1.449
9	1.4	4.437	2.028
10	1.6	4.314	2.318
12	1.8	4.601	2.608
14	2.0	4.830	2.898

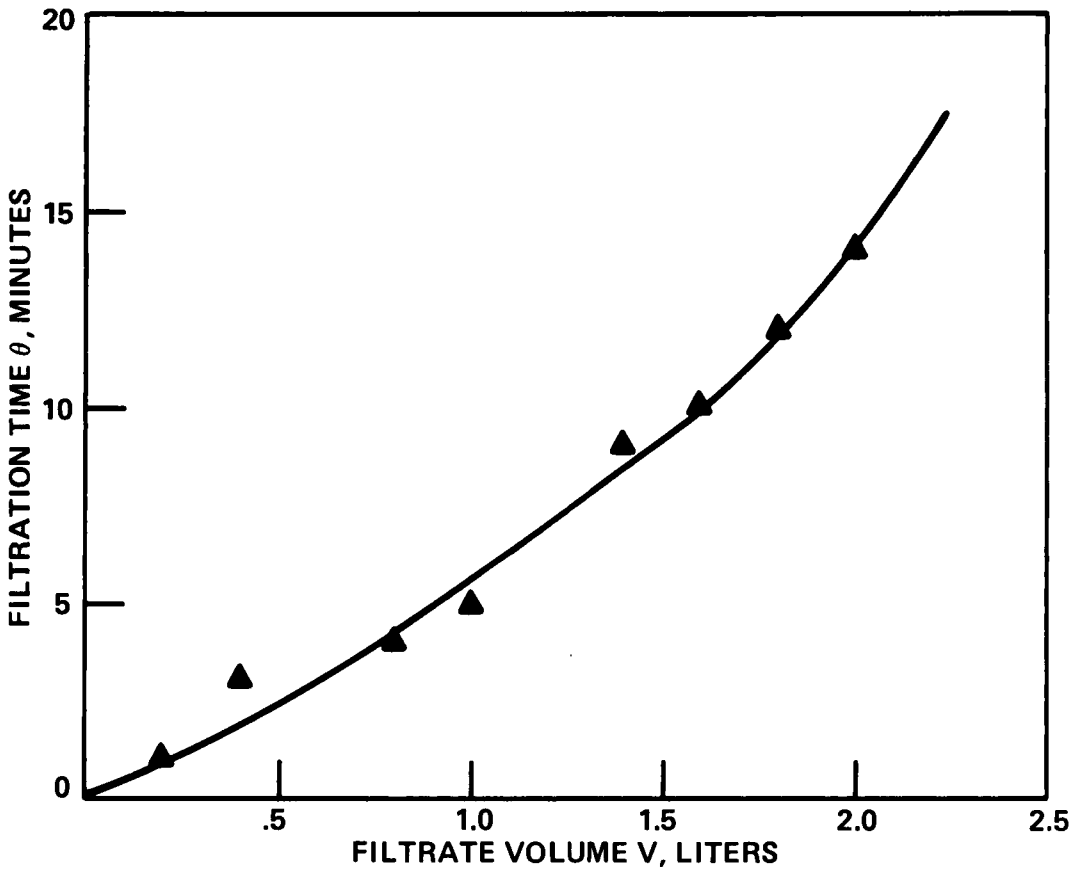


Figure 25. FILTRATION TIME VERSUS FILTRATE VOLUME FOR OPT-2

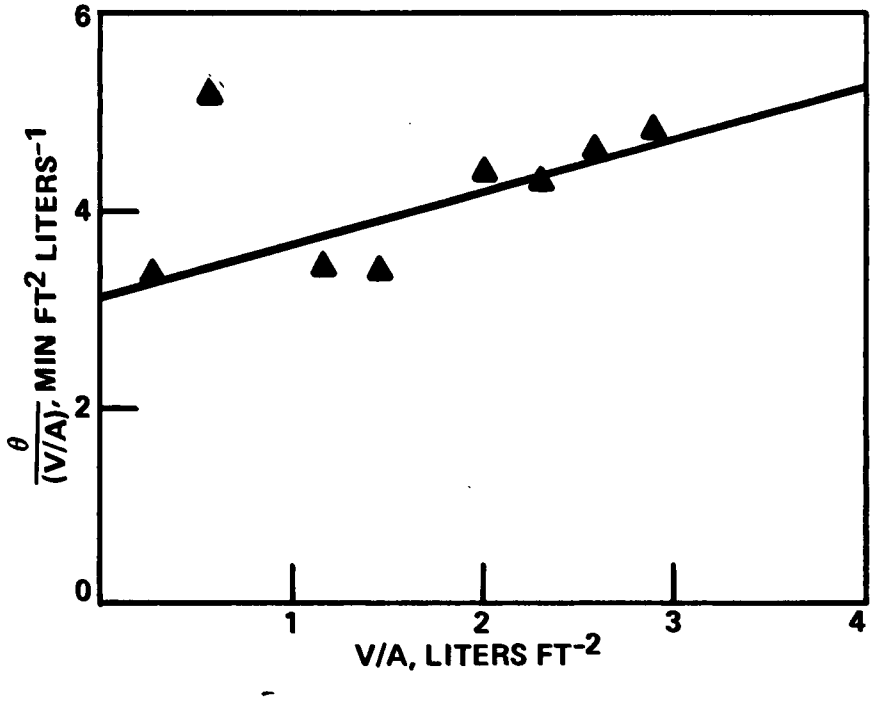


Figure 26. $\frac{\theta}{(V/A)}$ VERSUS V/A FOR OPT-2

Run OPT-3

In this run a precoat level of 0.4 lbm/ft^2 was used. The pressure was maintained at 40 psig and temperature at 120°F. An average filtration flow rate of 2.9 gal/hr/ft^2 was observed. This rate is somewhat lower than the rate obtained in run OPT-2 (3.28 gal/hr/ft^2) and demonstrates a lowering of filtrate flow with the use of too much filter aid for precoat. This phenomenon is widely documented.[8, 10, 13] Cake and filter medium resistances are shown in Table IX, page 52. Appendix B shows the sample analyses. The feed solution solids content was only 0.54% as compared to ∿2% for runs OPT-1 and OPT-2. Apparently, the agitation was not sufficient to thoroughly homogenize the SRC solution in the feed drum. Run data are listed in Table XVIII. Figures 27 and 28 show time versus filtration volume relationships. Figure 28 exhibits a very flat slope which results in a low cake resistance determination. Ash and pyridine insolubles content were 0.04% and <0.01%, respectively.

Run OPT-4

A precoat level of 0.3 lbm/ft^2 was used in this run. Other run conditions were not changed. An average filtrate flow rate of 1.54 gal/hr/ft^2 was obtained. Cake and filter medium resistances were 1.84 and 4.99, respectively. The product solution contained only 0.02% ash and 0.01% pyridine insolubles. Run data are shown in Table XIX. Figure 29 shows the volume versus time relationship and Figure 30 shows the $\frac{\Theta}{(V/A)}$ versus V/A plot. Appendix B shows all sample analyses.

Discussion of Optimization Runs Results

Figure 31 shows a plot of time versus filtrate volume for LCF-3 Repeat, OPT-1, OPT-2, OPT-3, and OPT-4; and Figure 32 shows the

TABLE XVIII

RUN DATA (OPT-3)

Filtration Time Θ, min	Filtrate Volume V, liters	$\frac{\Theta}{(V/A)}$	$\frac{V}{A}$
0	0	0	0
1.5	0.20	5.19	0.289
2.5	0.40	4.317	0.579
3.5	0.60	4.027	0.869
5.5	1.00	3.795	1.449
8.0	1.40	3.944	2.028
9.5	1.60	4.098	2.318
13.0	2.00	4.485	2.898
16.0	2.40	4.60	3.478
20.0	2.80	4.929	4.057
24.0	3.20	5.175	4.637
28.5	3.60	5.462	5.217

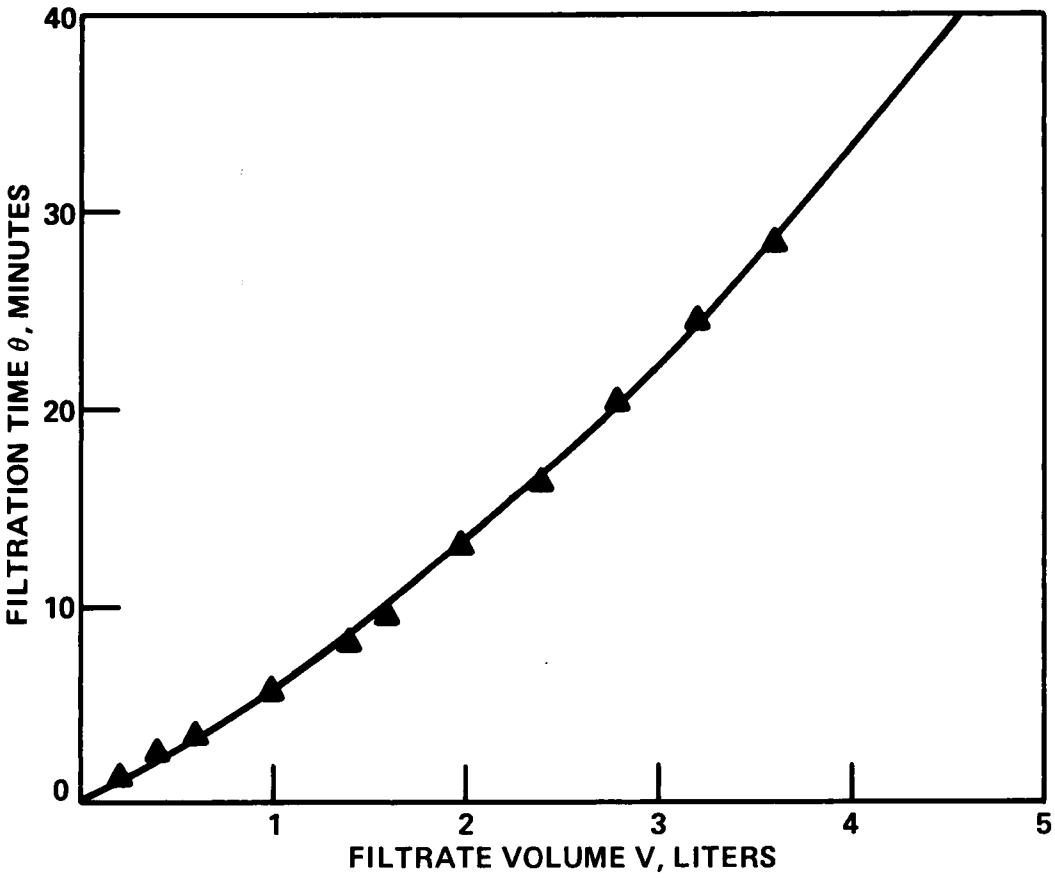


Figure 27. FILTRATION TIME VERSUS FILTRATE VOLUME FOR OPT-3

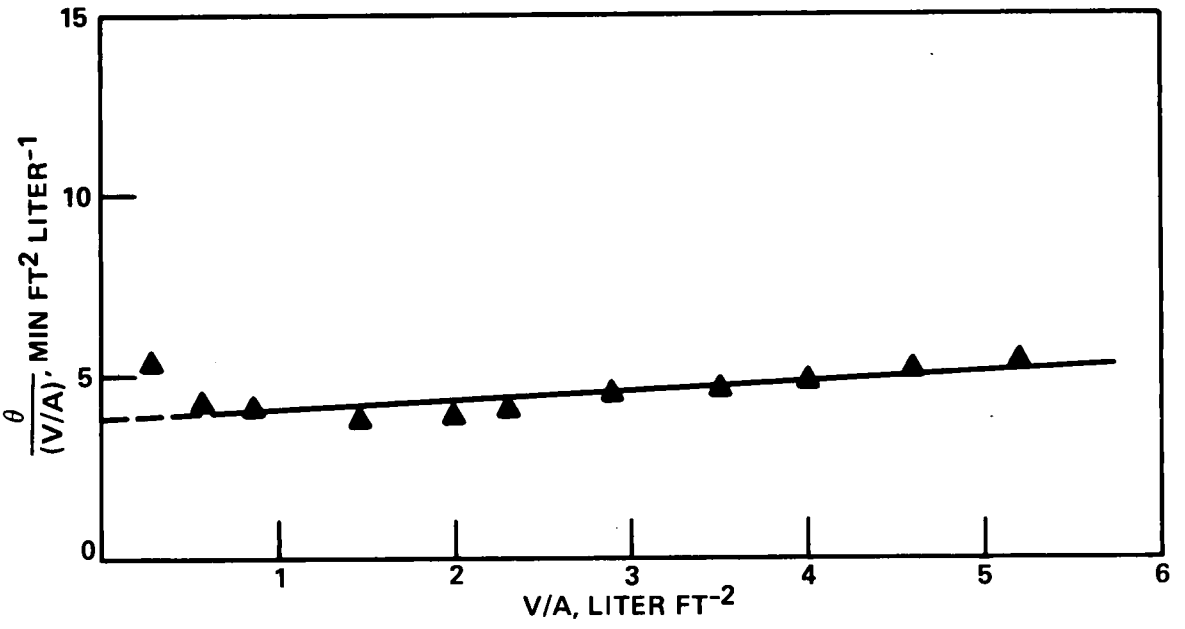


Figure 28. $\frac{\theta}{(V/A)}$ VERSUS V/A FOR OPT-3

TABLE XIX

RUN DATA (OPT-4)

Filtration Time Θ, min	Filtrate Volume V, liters	$\frac{\Theta}{(V/A)}$	$\frac{V}{A}$
0	0	0	0
0.7	0.1	5	0.14
1.6	0.2	5.71	0.28
2.5	0.3	5.81	0.43
3.4	0.4	5.96	0.57
4.5	0.5	6.25	0.72
5.7	0.6	6.62	0.86
6.9	0.7	6.83	1.01
8.5	0.8	7.39	1.15
11.5	1.0	7.98	1.44
15.0	1.2	8.67	1.73
21.3	1.5	9.81	2.17
23.4	1.6	10.12	2.31
25.3	1.7	10.28	2.46

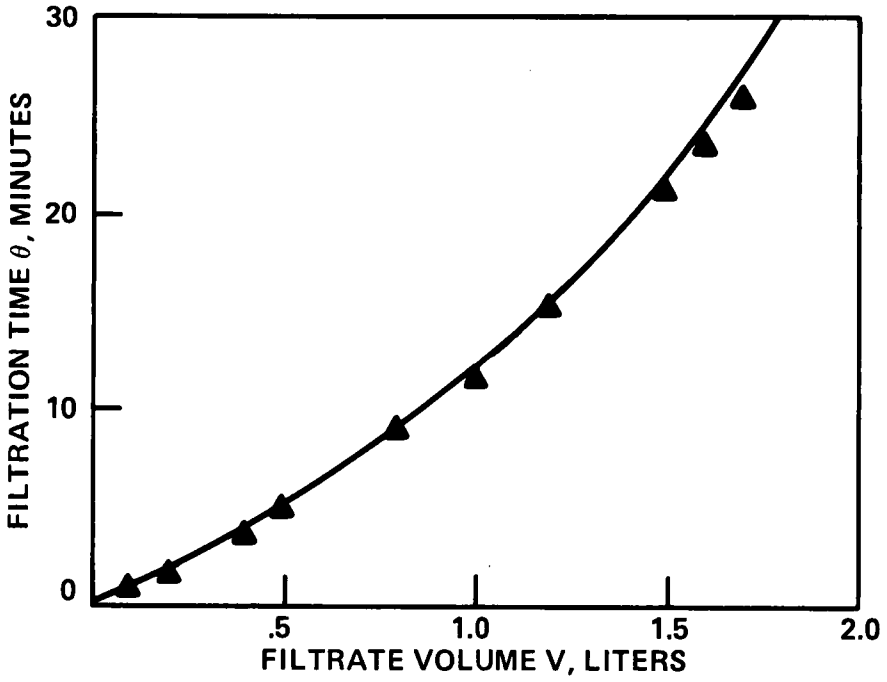


Figure 29. FILTRATION TIME VERSUS FILTRATE VOLUME FOR OPT-4

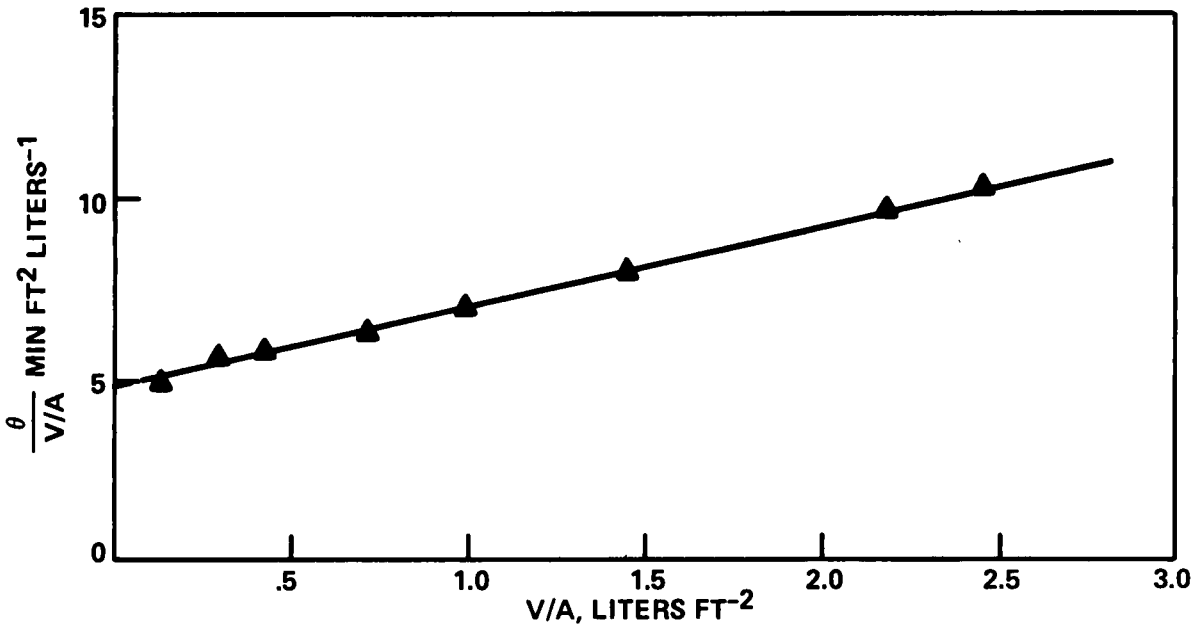


Figure 30. $\frac{\theta}{(V/A)}$ VERSUS V/A FOR OPT-4

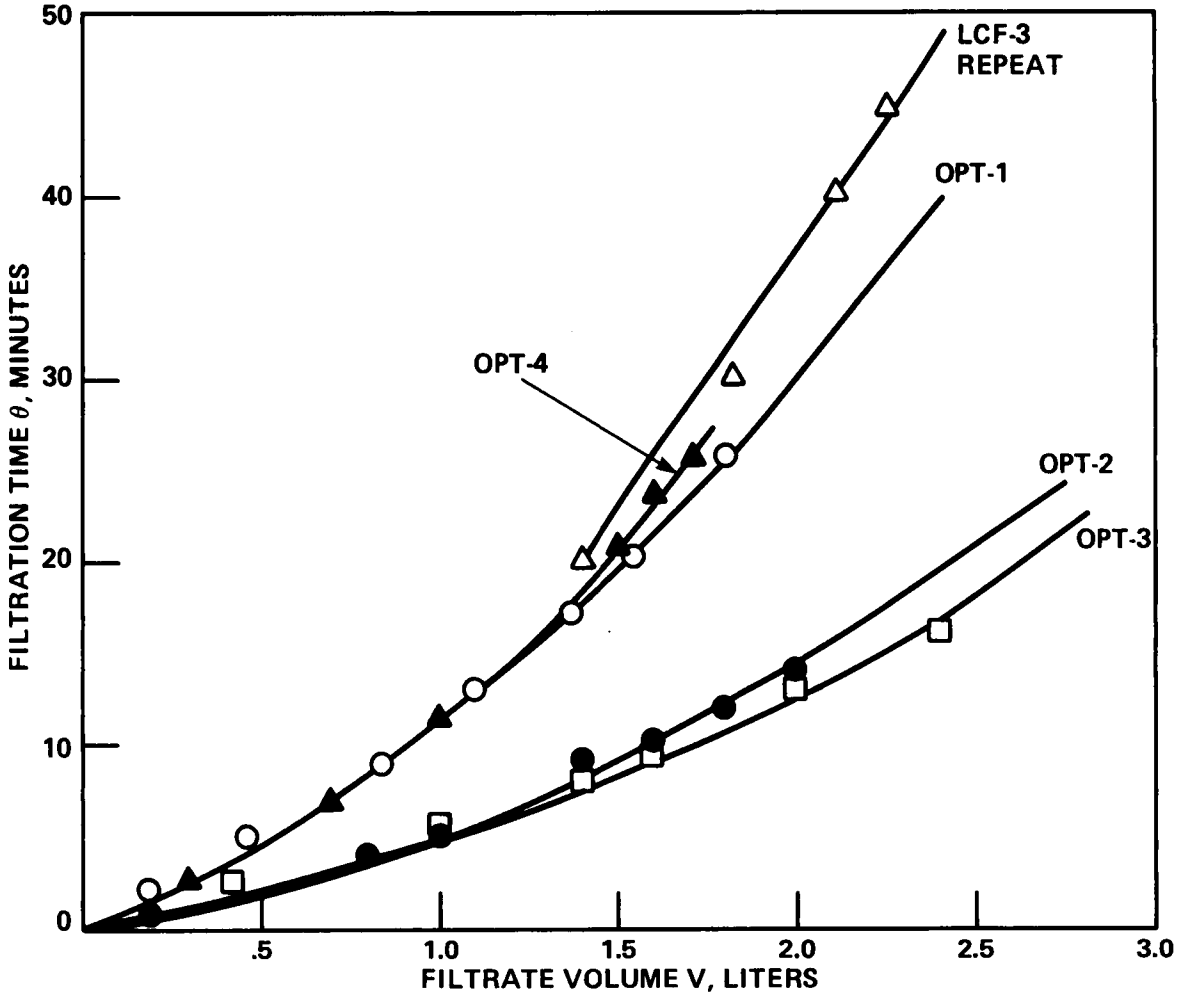


Figure 31. FILTRATION TIME VERSUS FILTRATE VOLUME FOR LCF-3 REPEAT, OPT-1, 2, 3, 4

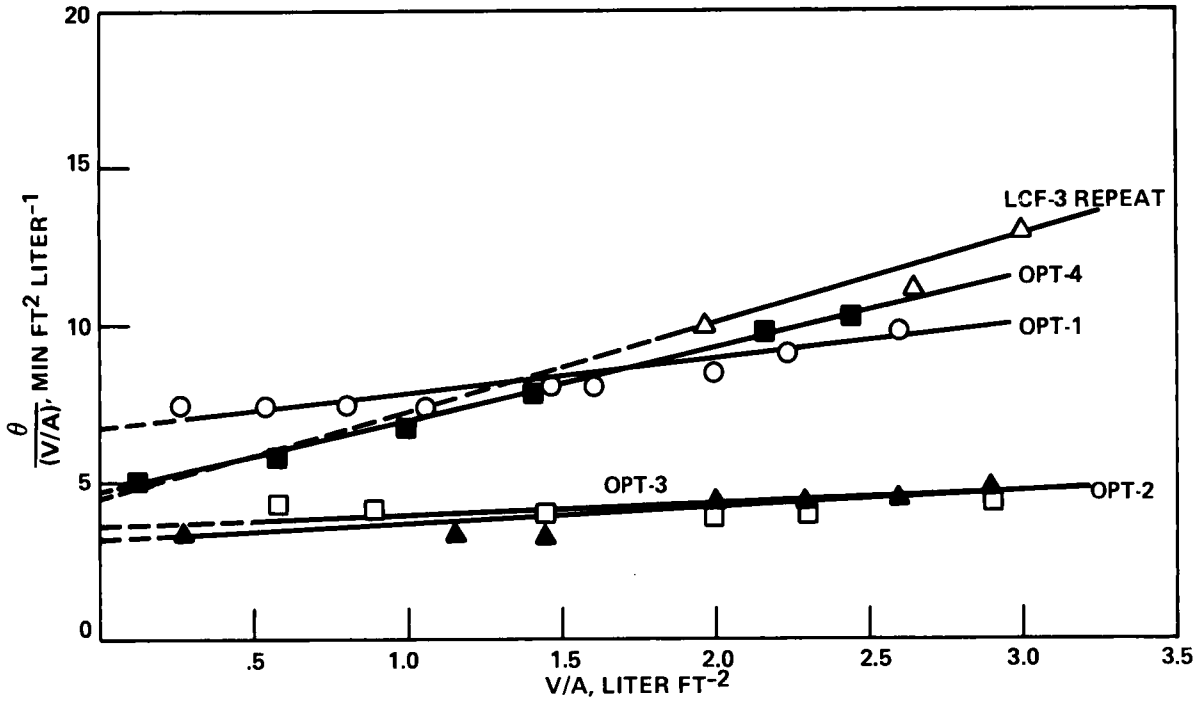


Figure 32. $\frac{\theta}{(V/A)}$ VERSUS V/A FOR LCF-3 REPEAT, OPT-1, 2, 3, 4

parameters $\frac{\Theta}{(V/A)}$ versus V/A for these runs. Figure 33 shows the average filtrate flow versus precoat usage level as plotted on three different consistent bases. In each case, the appearance of the plot is essentially of the same character. While these curves do not show a clear optimum, the behavior of the system beyond a precoat level of 0.2 lbm/ft^2 does become less consistent. Typical practice in the field would favor a level around 0.2 lbm/ft^2 for production usage in order to assure a more consistent operation at high filtrate flows.

E. PREDICTION OF FLOWS FOR OTHER OPERATING CONDITIONS

To accurately scale-up filtration data, the specific cake resistance, the filter medium resistance, and the solution viscosity must be known. Cake resistance is a function of the applied pressure. For incompressible cakes the resistance is proportional to the applied pressure and for compressible cakes the resistance is independent of pressure. The viscosity must be known as a function of temperature. Scale-up to large filtration equipment for Solvent Refined Coal liquids from Emery coal is possible using the average flow rate data for the various processing conditions shown previously in this chapter. For other coal-derived liquids, the parameters α, α', r, and s as determined in this chapter enable predictions of filtration rates by use of the filtration equation (3).

Effect of Temperature

The filtrate rate should vary inversely with solution viscosity. The temperature effect can be approximated by

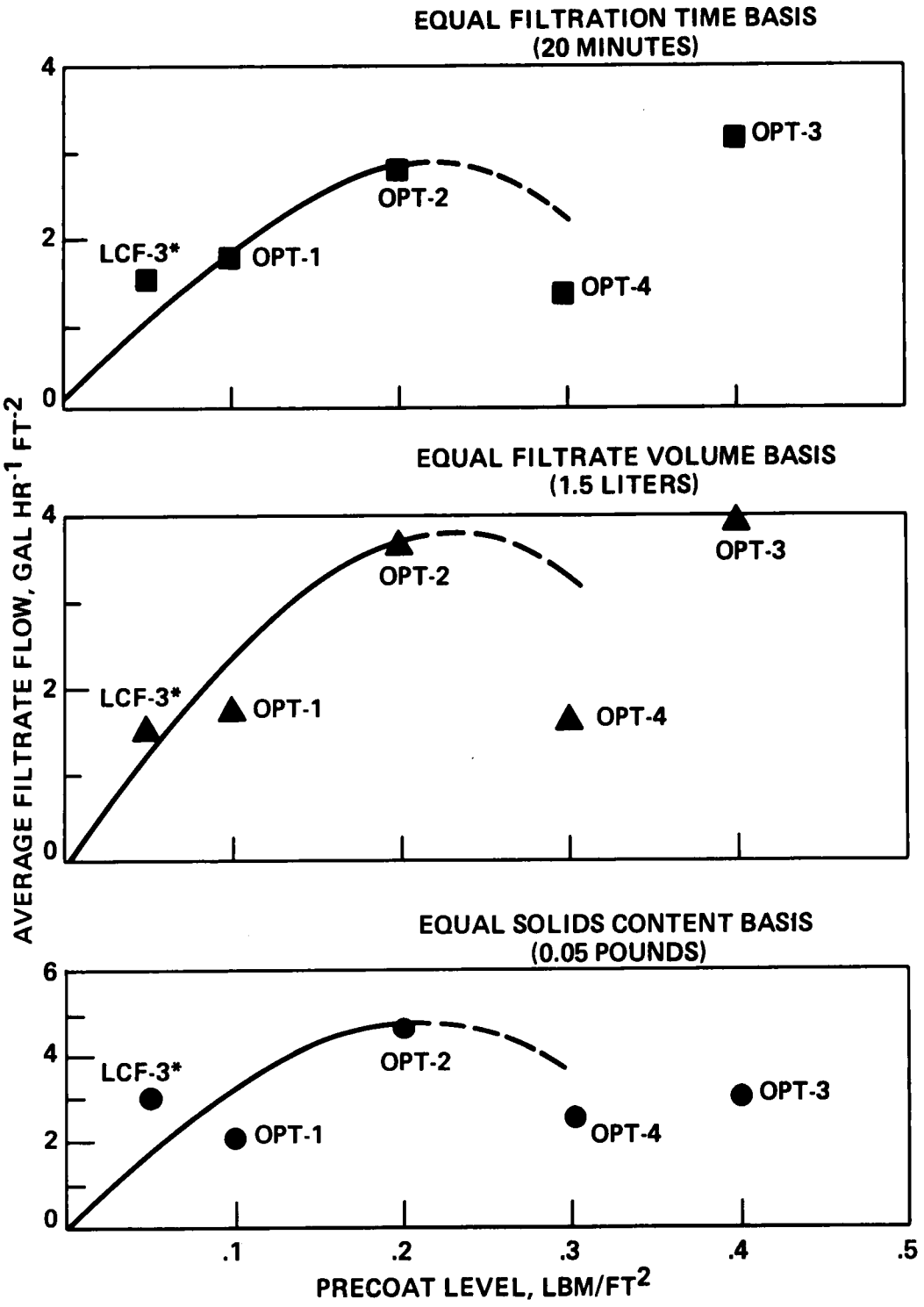


Figure 33. AVERAGE FILTRATE FLOW VERSUS PRECOAT LEVEL

$$\frac{G_2}{G_1} = \frac{\mu_{t_1}}{\mu_{t_2}} \tag{6}$$

where μ = solution viscosity, CP

t_1 = test temperature, °F

t_2 = required temperature, °F

G_1 = known flow rate, gal/hr/ft^2

G_2 = predicted flow rate, gal/hr/ft^2.

For run OPT-2 (∿2.5% feed solids), if plant conditions assumed a temperature of 400°F (1.5 CP) and a pressure of 40 psig, a filtrate flow of

$$G_2 = G_1 \frac{\mu_{120°F}}{\mu_{400°F}}$$

$$= (3.28 \frac{\text{gal}}{\text{hr ft}^2})(\frac{30 \text{ CP}}{1.5 \text{ CP}}) = 65.6 \text{ gal/hr/ft}^2$$

could be expected. This rate compares with 3 to 5 gal/hr/ft^2 for the work[14] done at the Oak Ridge National Laboratory with a SRC material that contained 3.19% solids. Asbestos-diatomaceous earth filter aid was used at a level of 8.76 lbm/hr/ft^2 in their work with no attempt at optimizing usage. Cellulose fiber filter aid usage for run OPT-2 was 0.2 lbm/ft^2 (∿0.857 lbm/hr/ft^2).

Effect of Pressure

For estimating flows at pressures above the 25 to 50 psig level used in this study, an estimate must be made of the specific cake resistance pressure dependency and the filter medium resistance. An effort was made to do this as shown earlier in the chapter for runs LCF-2 and LCF-4 and the limitations noted. This involved using

equation (2) for determining the filter cake compressibility factor s. A plot of the logarithm of the calculated cake resistances versus the logarithm of the pressures for these two runs gave the particle size constant α' as 6.2 and the compressibility factor s as 0.47. Comparing this estimate of the average specific cake resistance with that calculated from the $\frac{\Theta}{(V/A)}$ versus V/A plots yielded fairly good agreement for runs LCF-1 and LCF-3 with better agreement for runs LCF-4 and LCF-5. The specific cake resistance calculated in run LCF-4 was 25.63 and the estimate from the compressibility determination was 38.9. The filter medium resistance was essentially zero. Using this information and the filtration equation (3) for prediction of the flow at 150 psig yields

$$\frac{\Theta}{V/A} = \frac{\mu\alpha W}{2\Delta P}\frac{V}{A} + \frac{\mu r}{\Delta P}$$

$$\frac{\Theta}{V/A} = \left[\frac{(60)(6.2)(150)^{0.47}(5.78)}{(2)(150)}\right]\left(\frac{0.8}{0.69}\right) + 0$$

$$\frac{\Theta}{V/A} = 87.57\ \frac{\text{min ft}^2}{\text{liter}}$$

$$\frac{V/A}{\Theta} = 0.18\ \text{gal/hr/ft}^2$$

This rate is higher than that calculated for run LCF-4 (0.10 gal/hr/ft^2) using the cake resistance approximation at 50 psig and indicates the affect on flow due to increased pressure.

Discussion

Design conditions[4] of 700°F and 225 psig have provided filtrate flows of up to 45 gal/hr/ft^2 for the Solvent Refined Coal liquefaction intermediate material in pilot tests performed by workers at the

Wilsonville, Alabama SRC plant. These tests were made utilizing a pre-coat level of 0.2 lbm/ft^2 with fly ash as filter aid. No body mix of filter aid was used. A smaller pilot setup was used to remove coal dust from a tar/oil solution. The unit had a filtration rate of 15 gal/hr/ft^2 at design conditions of 400°F and 150 psig. The percentage of solids was reduced from 2.5 in the feed to 0.05 in the filtrate. These reported values are lower than the scaled results obtained in most of the runs in this study. In addition the combustibility of cellulose fibers might aid process economics by reducing disposal costs. SRC material can be filtered readily with cellulose fiber filter aids and provide flows as good as reported values for less severe operating conditions.

CHAPTER V

CONCLUSIONS AND RECOMMENDATIONS

The use of cellulose fibers as precoats in the filtration of a Solvent Refined Coal intermediate resulted in the following conclusions and recommendations.

A. CONCLUSIONS

1. Cellulose fibers can be successfully used to clarify coal-derived oils to ash levels below 0.15%. Ash levels below 0.15% are satisfactory for boiler fuels.

2. Precoat levels as low as 0.025 to 0.050 lbm of precoat/ft^2 of filter surface are successful in providing good clarity filtrates.

3. An optimum filtrate flow rate for SRC liquefaction intermediates exists near 0.2 lbm of precoat/ft^2 of filter surface. Scaled filtrate flows considerably in excess of those obtained in other studies were obtained using cellulose fiber filter aids.

4. The filtration cost using cellulose fibers as shown in Appendix C is roughly 58% of that estimated by others.[8] Production costs were around $0.60/barrel of filtrate.

5. Cellulose fibers provide an added cost incentive because the fibers can be burned. The cake consisting of filter aid as well as the ash and unconverted coal can be disposed of accordingly.

B. RECOMMENDATIONS

1. More complete run data are needed to ascertain the specific cake resistance pressure dependency for more accurate scale-up of filtrate flow rates.

2. Experimental runs with precoats of sized coal, fly ash, and diatomaceous earth-asbestos mixtures should be made to examine the process economics as compared to cellulose filter aids. Perhaps under optimized conditions, similar results to those obtained in this study could be obtained for other filter aid materials.

3. Based on the results obtained in this study, a full-scale test should be run in the SRC liquefaction facility in Wilsonville, Alabama.

4. The viscosity curve (Figure 5, page 28) shows that extrapolation of the data from Catalytic, Inc., would predict a lower viscosity than the one measured for this study. This may indicate that some polymerization of the material has taken place. While this would make these filtration results more conservative, future studies should obtain some measure of this aging effect through a more complete periodic analysis of the feed material including measurements such as benzene insolubles.

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APPENDIXES

APPENDIX A

SAMPLE CALCULATION OF FILTRATION EQUATION PARAMETERS

APPENDIX A

SAMPLE CALCULATION OF FILTRATION EQUATION PARAMETERS

The integrated form of the filtration equation for constant pressure was given in Chapter I as equation (3).

$$\frac{\Theta}{V/A} = \frac{\mu\alpha W}{2\Delta P}(V/A) + \frac{\mu r}{\Delta P}$$

where $\frac{\mu\alpha W}{2\Delta P}$ and $\frac{\mu r}{\Delta P}$ are the slope and intercept, respectively. The specific cake resistance was related to the pressure by equation (2).

$$\alpha = \alpha' (\Delta P)^s$$

where α' is the particle size constant and s is the compressibility factor.

Sample Calculation of α and r

In Figure 13, page 59, the slope for run LCF-3 was 5, and the intercept was 7.8. Therefore,

$$\frac{\mu\alpha W}{2\Delta P} = 5 \frac{\text{min ft}^4}{\text{liters}^2} \qquad \text{(A.1)}$$

and

$$\frac{\mu r}{\Delta P} = 7.8 \frac{\text{min ft}^2}{\text{liter}} . \qquad \text{(A.2)}$$

The specific cake resistance α is calculated from equation (4).

$$\alpha = \text{slope}\ \frac{2\Delta P}{\mu W}$$

where $\Delta P = 45$ psig

$\mu = 60$ CP

$W = 6.22\ \frac{\text{lbm}}{\text{ft}^3}$.

For run LCF-3

$$\alpha = \frac{\left(\frac{5\ \text{min}\ \text{ft}^4}{\text{liters}^2}\right)(2)\left(45\ \frac{\text{lbf}}{\text{in.}^2}\right)}{(60\ \text{CP})\left(6.22\ \frac{\text{lbm}}{\text{ft}^3}\right)}$$

$$= 1.21\ \frac{\text{lbf min ft}^7}{\text{in.}^2\ \text{liters}^2\ \text{lbm CP}}\ .$$

The filter medium resistance r is calculated from equation (5).

$$r = \text{intercept}\ \frac{\Delta P}{\mu}\ ,$$

where $\Delta P = 45$ psig

$\mu = 60$ CP.

For run LCF-3

$$r = \frac{\left(7.8\ \frac{\text{min ft}^2}{\text{liter}}\right)\left(45\ \frac{\text{lbf}}{\text{in.}^2}\right)}{60\ \text{CP}}$$

$$= 5.85\ \frac{\text{lbf min ft}^2}{\text{in.}^2\ \text{liter CP}}\ .$$

APPENDIX B

SAMPLE ANALYSES

TABLE XX

SAMPLE ANALYSES (FEED DRUM NO. 1)

	LCF-1		LCF-2		LCF-3		LCF-4		LCF-5	
	Feed	Product	Feed	Product	Feed	Product	Feed	Product	Feed	Product
Pyridine Insolubles, %	5.82	0.04	6.10	0.17	7.50	1.18	7.14	0.79	8.95	1.00
Ash, %	1.37	0.02	1.36	0.08	1.58	0.25	1.29	0.12	1.97	0.09
Volatile Matter, %	86.1	90.6	85.3	90.7	84.2	90.4	86.2	90.5	82.6	89.2
Heat Content, Btu/lb	16197	16255	16392	16145	16260	16138	16349	16690	15999	16694
Molecular Weight	274	274	271	274	270	275	270	277	239	242
Density, g/ml	1.1017	1.0839	1.1012	1.0716	1.1071	1.0772	1.1066	1.0746	1.1120	1.0769
N, %	0.93	0.85	0.95	0.84	0.81	0.87	0.93	0.91	0.95	0.89
S, %	0.49	0.33	0.43	0.36	0.43	0.38	0.41	0.34	0.38	0.43
Fe, %	0.071	<0.01	0.054	<0.01	0.085	0.02	0.080	<0.01	--	--
C, %	84.45	85.58	85.76	84.89	85.85	83.17	87.24	87.81	87.3	84.3
H, %	7.12	7.63	7.15	7.46	7.21	6.86	7.26	7.53	7.4	7.1

TABLE XXI

SAMPLE ANALYSES (FEED DRUM NO. 2)

Analysis	LCF-3*		OPT-1		OPT-2		OPT-3		OPT-4	
	Feed	Product	Feed	Product	Feed	Product	Feed	Product	Feed	Product
Pyridine Insolubles, %	2.08	0.12	1.80	<0.01	1.68	<0.01	0.50	0.007	3.18	0.01
Ash, %	0.41	0.05	0.35	0.04	0.43	0.04	0.04	0.03	0.50	0.02
Volatile Matter, %	89.2	90.5	88.35	90.14	88.6	90.5	89.21	90.24	84.70	90.13
Heat Content, But/lb	15936	15645	16754	16852	16792	16898	16889	16920	16590	16935
Molecular Weight	230	239	268	270	258	247	248	262.30	261	248
Density, g/ml	1.1006	1.1029	1.072	1.06	1.062	1.060	1.06	1.06	1.0835	1.0647
N, %	0.90	0.75	0.88	0.92	0.84	0.81	0.88	0.84	0.96	0.89
S, %	0.65	0.31	0.54	0.42	0.37	0.36	0.38	0.36	0.64	0.63
Fe, %	--	--	0.0224	0.0031	0.0398	0.0022	0.0251	0.0068	0.0547	0.0011
C, %	83.8	82.1	88.18	88.27	84.7	85.5	88.0	88.7	84.34	86.45
H, %	7.0	6.8	7.43	7.63	8.49	7.45	7.57	7.73	6.88	7.45

*LCF-3 repeat

APPENDIX C

ECONOMIC CONSIDERATIONS FOR A 23,000 BARREL/DAY COAL LIQUEFACTION FACILITY

APPENDIX C

ECONOMIC CONSIDERATIONS FOR A 23,000-BARREL/DAY COAL LIQUEFACTION FACILITY

The total cost for filtration in a coal liquefaction facility will be comprised of both filter cost and filter aid cost. Both of these costs are directly related to the overall filtration rate. Experimental data from the fractional factorial runs and the optimization runs indicated the filtration rate was strongly influenced by precoat usage. Pressure, use of body mix, and type of filter aid were secondary considerations. Cost data[22, 26, 27] from work done at the Oak Ridge National Laboratory indicated that for a commercially available rotary precoat filter (700 ft^2) modified for high temperature and high pressure operation the purchase cost would be approximately $388,000. The total installed cost of the filtration section of the plant was assumed to be 4 times the cost of the filters themselves. A fixed charge of 24% per year was assumed on the capital investment. The filter aid cost is the precoat level times the area requirement times the unit cost of the filter aid. Therefore, the total cost equation is

$$\frac{\text{Cost}}{\text{yr}} = 388{,}000\ (\frac{A}{700})(4)(0.24) + AMC \qquad (C.1)$$

where A = area of filter, ft^2

M = total precoat level, $lbm/ft^2/hr$

C = unit cost of filter aid, $/lbm.

The requirement was determined from the size of plant needed (F gal/hr) and the filtration flow rate (G gal/hr/ft^2). The cost equation becomes

$$\frac{\text{Cost}}{\text{yr}} = \left(\frac{F}{G}\right)(532) + \left(\frac{F}{G}\right)(M)(C) \qquad (C.2)$$

The number of times the filter must be cleaned and precoated per year based on filtration cycle length must also be taken into account. Therefore, let

$$M = DP \qquad (C.3)$$

where D is the number of filter cleaning cycles per year, and P is the precoat level per cycle.

The final cost equation becomes

$$\frac{\text{Cost}}{\text{yr}} = \left(\frac{F}{G}\right)(532) + \left(\frac{F}{G}\right)(D)(P)(C) \ . \qquad (C.4)$$

The optimum precoat usage level was determined to be around 0.2 lbm/ft^2 (0.857 lbm/hr/ft^2) in run OPT-2. This rate has also been chosen by other investigators[4, 26] to be near the economic optimum case, and cost estimates have generally been derived from this level of precoat usage. The flow rate for this precoat level in run OPT-2 was around 3.3 gal/hr/ft^2 and the filter feed material contained about 2.5% solids. An average flow rate of 66 gal/hr/ft^2 could be expected at 400°F operation.

To determine the filter surface area required in a commercial plant, the filtration rate was reduced to allow downtime for precoating, cleaning, and maintenance. For this analysis a 50% reduction in flow rate was assumed. A plant designed for 23,000 barrels/day of filtrate

(F = 40,300 gal/hr) at a reduced filtration rate of 33 gal/hr/ft^2 would require 1221 ft^2 of filter area. Filter aid usage rate was assumed at 0.857 lbm/hr/ft^2 as in run OPT 2. Now, from the cost equation (C.4),

$$\frac{\text{Cost}}{\text{yr}} = \underset{\text{(Filter Cost)}}{1221\ (532)} + \underset{\text{(Precoat Cost)}}{(1221)(33943)(0.2)(0.25)}$$

$$\text{Cost} = (649{,}572 + 2{,}072{,}220)\ \$/\text{yr}$$

$$\text{Cost} = \$2.721 \times 10^6/\text{yr}$$

The cost is roughly 58% of a capital cost[8] estimate (1975) reported by the Oak Ridge National Laboratory for a rotary precoat filter for coal-derived oils. They assumed a reduced filtrate flow rate of 6 gal/hr/ft^2, an optimum precoat usage rate of 0.28 lbm/hr/ft^2, and a cost of \$500/ft^2 for the filter. If operating costs are assumed to be 70% of capital cost as in the ORNL report[8] for the optimum flow rate run (OPT-2), total capital and operating filtration costs would be \$4.625 x 10^6/yr and represents \$0.60/barrel of oil produced. This cost per barrel of oil produced is roughly 60% of costs reported by others.[8, 27]

VITA

Harold T. Conner, Jr. was born in Fulton, Kentucky, on January 5, 1946, and graduated from Weakley County Training School in Martin, Tennessee, in June 1963. He entered the University of Tennessee Martin in September 1963, and spent two years in pre-engineering. In August 1966, he enrolled in the University of Tennessee Knoxville and graduated in December 1968, with a Bachelor of Science degree in Chemical Engineering. During his undergraduate career, he worked seven quarters with the Nuclear Division of Union Carbide Corporation at the Oak Ridge Gaseous Diffusion Plant as a participant in The University of Tennessee Cooperative Engineering Program.

In 1968, he returned to the Oak Ridge Gaseous Diffusion Plant as a full-time employee working in the Materials and Systems Development Department, Gaseous Diffusion Development Division. During his employment, he began graduate study by taking courses in the Oak Ridge Resident Graduate Program.

He married the former Joyce Ann Graham in November 1970, and they have two sons, Harold III and Michael Brian.

He is an associate member of the American Institute of Chemical Engineers.