DOE/PC/94155--T4

QUARTERLY TECHNICAL PROGRESS

REPORT 5

OCTOBER-DECEMBER 1995

POC-SCALE TESTING
OF AN ADVANCED FINE COAL DEWATERING EQUIPMENT/TECHNIQUE

Prepared for

U.S. Department of Energy
Pittsburgh Energy Technology Center
Pittsburgh, PA 15236

By

J.G. Groppo
B.K. Parekh
Center for Applied Energy Research
University of Kentucky
Lexington, KY 40511

DOE Contract No. DE-AC22-94PC94155

February 1, 1996

TABLE OF CONTENTS

LIST OF FIGURES

EXECUTIVE SUMMARY

Froth flotation technique is an effective and efficient process for recovering of ultra-fine (minus 74 μm) clean coal. Economical dewatering of an ultra-fine clean coal product to a 20% level moisture will be an important step in successful implementation of the advanced cleaning processes. This project is a step in the Department of Energy's program to show that ultra-clean coal could be effectively dewatered to 20% or lower moisture using either conventional or advanced dewatering techniques.

The cost-sharing contract effort is for 36 months beginning September 30, 1994. This report discusses technical progress made during the quarter from October 1-December, 1995.

OBJECTIVES AND SCOPE OF THE PROJECT

The main objective of the proposed program is to evaluate a novel surface modification technique, which utilizes the synergistic effect of metal ions-surfactant combination, for dewatering of ultra-fine clean coal on a proof-of-concept scale of 1 to 2 tph. The novel surface modification technique developed at the UKCAER will be evaluated using vacuum, centrifuge, and hyperbaric filtration equipment. Dewatering tests will be conducted using the fine clean coal froth produced by the column flotation units at the Powell Mountain Coal Company, Mayflower Preparation Plant in St. Charles, Virginia. The POC-scale studies will be conducted on two different types of clean coal, namely, high sulfur and low sulfur clean coal. The Mayflower Plant processes coals from five different seams, thus the dewatering studies results could be generalized for most of the bituminous coals.

APPROACH

The project team consist of the University of Kentucky Center for Applied Energy Research (UKCAER), Powell Mountain Coal Company (PMCC) and Andritz Ruthner Inc.

The UKCAER is the prime contractor of the project which has been divided into nine (9) tasks. The clean coal froth generated by the 'Ken-Flote' columns at the PMCC Mayflower Preparation Plant will be utilized for dewatering studies using hyperbaric, centrifuge and vacuum dewatering techniques.

ACCOMPLISHMENTS DURING THE QUARTER

Laboratory high pressure dewatering of compliance (low sulfur) clean coal slurry showed that using 40 psi (2.8 bar) pressure the filter cake moisture increases from 22% to 27.5%, as the cake thickness increases from 11 mm to 20 mm.

Laboratory vacuum dewatering studies conducted using filter leaf test apparatus for the non-compliance clean coal slurry indicated that a 40 sec cake formation time and 70 sec drying time provided a 10 mm thick filter cake containing 24% moisture. Addition of 10 g/t of an anionic flocculant provided 28% filter cake moisture with 20 mm thick filter cake. For the compliance coal clean coal slurry a 10 mm thick filter cake was obtained containing 35% moisture. The high moisture in the compliance coal slurry was attributed to finer particle size. Increasing cake drying time from 30 sec to 70 sec lowered the filter cake moisture from 29% to 20%.

In the pilot plant studies, for the compliance coal about 0.25 Kg/t of non-ionic surfactant provided a filter cake with 22% moisture. At higher dosage of 1.5 Kg/t of the anionic and non-ionic surfactants provided a filter cake containing 17% and 20%

moisture, respectively. For the non-compliance coal, ~0.5 Kg/t cationic surfactant was not effective in lowering the filter cake moisture to 26.5%. Addition of both anionic and cationic surfactant showed increased solids throughput processed through the vacuum filter.

INTRODUCTION

For cleaning of coal finer than 0.5 mm (28 mesh) processes based on surface chemical technique such as froth flotation and oil agglomeration are the most effective. However, froth flotation process, which is commercially used, produces a product containing 80% moisture. Recently developed column flotation technique, which provides higher recovery of low ash product, also suffers from the same problem of high moisture product. Dewatering of the fine coal to a low (~20%) moisture level using conventional filtration equipment has not been possible. This project offers a novel surface-modification approach to modify coal surface so it could dewater to a low moisture level using conventional and advanced dewatering equipment. The surface modification approach has provided significant reduction in filter cake moisture in laboratory studies at University of Kentucky Center for Applied Energy Research.

The aim of this program is to test the UKCAER-developed novel coal surface modification approach on a pilot scale at the rate of 1-2 tph of solids using vacuum, centrifuge and hyperbaric filtration technique. This proof-of-concept testing is being performed at the Powell Mountain Coal Company Mayflower Plant located in St. Charles, Virginia.

The project involves a teaming arrangement between the University of Kentucky for Applied Energy Research (CAER), the Powell Mountain Coal Company (PMCC), and the Andritz Ruthner Inc. (ARI). The project will extend for a period of 36 months.

APPROACH

A team of scientists and engineers from the Center for Applied Energy Research, Powell Mountain Coal Company, and Andritz Ruthner Inc. has been formed to accomplish the objectives of the program. Each team member brings fine particle dewatering knowledge and experience to the project. The UKCAER, who is the prime contractor, will manage the project and will conduct the major part of the study. The PMCC will provide assistance and facility in conducting the pilot scale tests, and ARI will conduct laboratory dewatering tests and also pilot scale tests using the hyperbaric pressure filtration unit at the PMCC. Figure 1 shows the project organization chart. The project schedule for the first two years of the program is shown in Figure 2.

The CAER collected clean coal froth samples from the Mayflower plant for the laboratory studies. Samples of clean coal slurries were also sent to ARI for studies using their laboratory scale hyperbaric unit. At both organizations, emphasis will be given to identify optimum process and operating conditions using vacuum and pressure techniques to dewater the clean coal slurry to about 20% level moisture. It is believed that the proposed research can achieve low moisture product on a pilot scale to the same extent which has already been achieved in laboratory studies.

The basic components of the process has been tested in laboratory. The purpose of the proposed work here is to evaluate all of the component steps on a consistent basis, and, to the extent possible in laboratory studies, demonstrate the

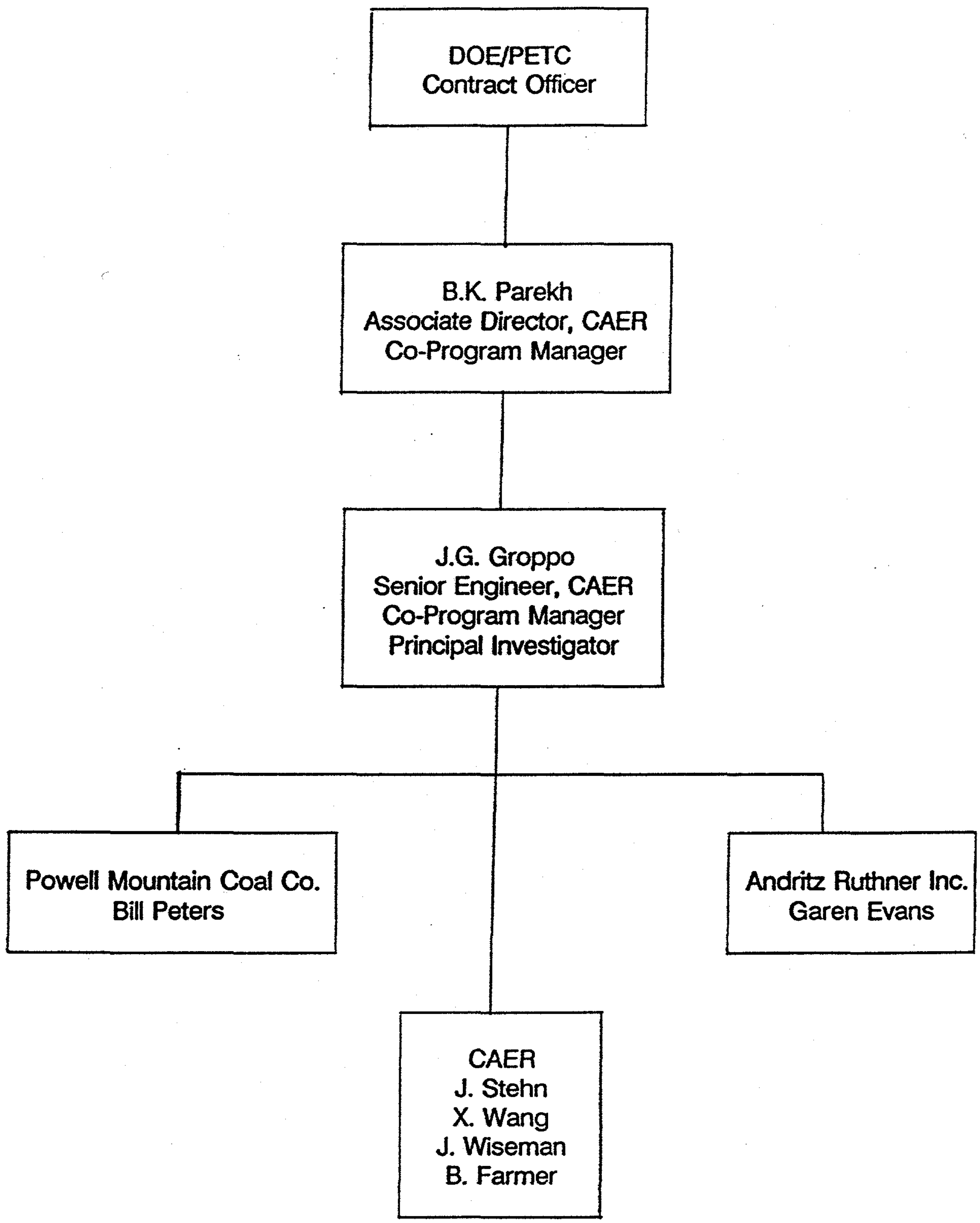

Figure 1. Project management organization chart

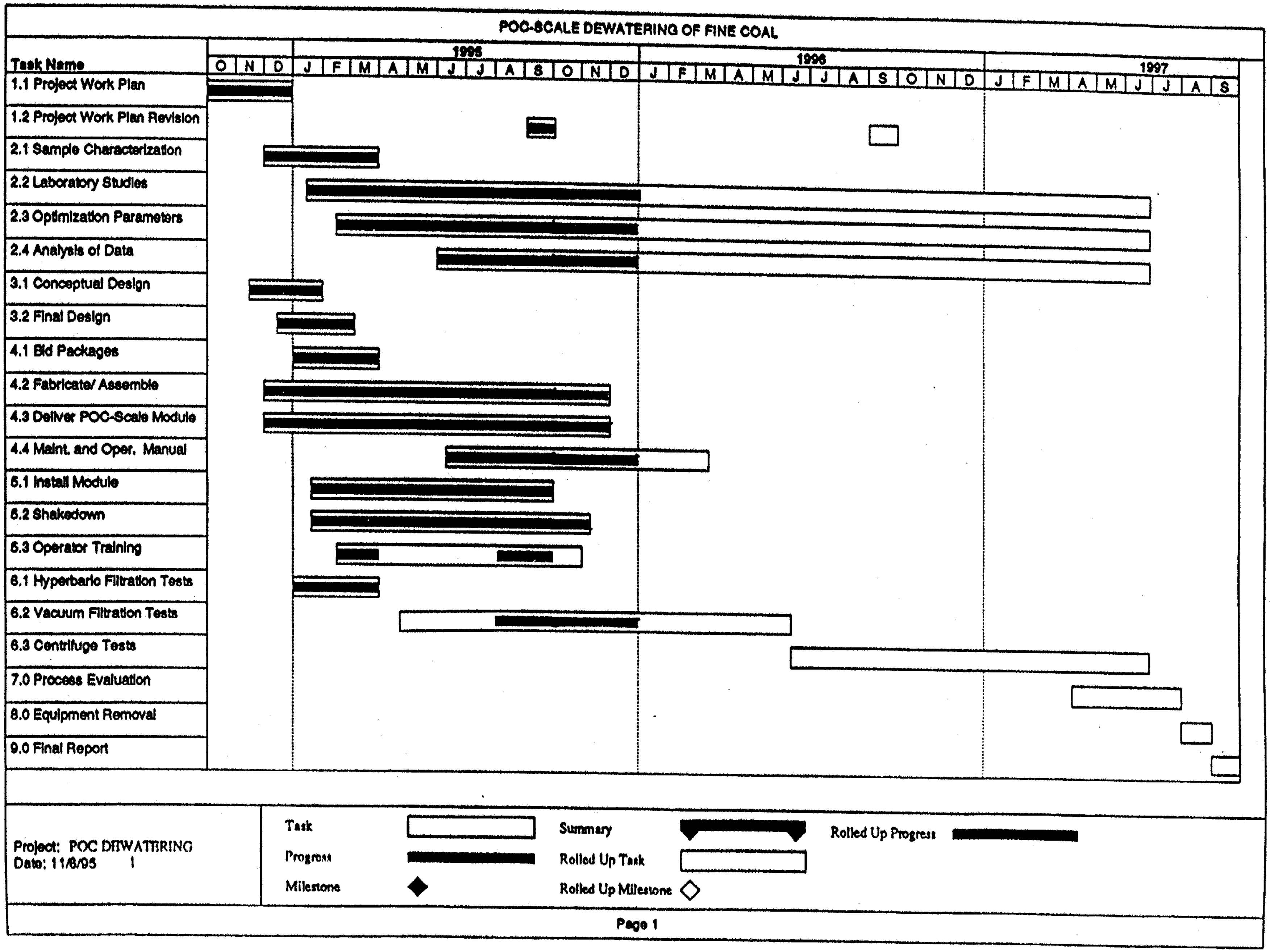

Figure 2. Up-to-date project schedule

feasibility of their integration. The outcome of this program will be to identify a process/technique combination which is able to achieve a 20% or lower moisture in the fine clean coal product and to provide technical and economic evaluation of the integrated concept in sufficient detail for a coal company to decide to install the dewatering process in their plant.

ACCOMPLISHMENTS DURING THE QUARTER

The project has been divided into tasks and subtasks as listed in Table I. Each task and subtask objectives can be inferred from its title. During this quarter (October 1 to December 31, 1995) work was done on Tasks 2, 6 and 9.

Task 2. Sample Analysis and Laboratory Testing:

The laboratory dewatering tests were conducted using both compliance (low sulfur) and non-compliance (high sulfur) clean coal slurries. Figures 3 and 4 show the particle size distribution of high and low sulfur clean coal slurries, respectively. Note, that the D_{50} (median size) of high and low sulfur coal is 37.3 μm and 29.5 μm, respectively. These numbers show that over a period of more than one year the average particle size remained constant.

High Pressure Dewatering:

A few of the additional baseline dewatering data using high pressure for the compliance coal are shown in Figures 5, 6 and 7. Using a cake thickness of 11 mm and 40 sec dewatering time (Fig. 5), filter cake moisture containing 22% moisture was obtained using 40 psi (2.8 bar) pressure. Increasing cake thickness to 15 mm (Fig. 6) and 20 mm (Fig. 7) increased moisture to 24.5% and 27.5%, respectively, using 40

Table I. Outline of Work Breakdown Structure

Task 1.	Project Work Planning	
	Subtask 1.1	Project Work Plan
	Subtask 1.2	Project Work Plan Revisions
Task 2.	Samples Analysis and Laboratory Testing	
	Subtask 2.1	Acquisition and Characterization of Samples
	Subtask 2.2	Laboratory Scale Testing
	Subtask 2.3	Optimization of Parameters
	Subtask 2.4	Analysis of Data
Task 3.	Engineering Design	
	Subtask 3.1	Conceptual Design Package
	Subtask 3.2	Final Design Package
	Subtask 3.3	Construction Schedule
Task 4.	Procurement and Fabrication	
	Subtask 4.1	Bid Packages
	Subtask 4.2	Fabricate/Assemble Components
	Subtask 4.3	Deliver POC-Scale Module and Install
	Subtask 4.4	Maintenance and Operating Manual
Task 5.	Installation and Shakedown	
	Subtask 5.1	Install and Tie-in Module
	Subtask 5.2	Startup Procedures/Shakedown
	Subtask 5.3	Operators Training
Task 6.	System Operation	
	Subtask 6.1	Test Coal No. 1
	Subtask 6.2	Test Coal No. 2
Task 7.	Process Evaluation	
Task 8.	Equipment Removal	
Task 9.	Reporting	
	Subtask 9.1	Monthly Reports
	Subtask 9.2	Project Final Report

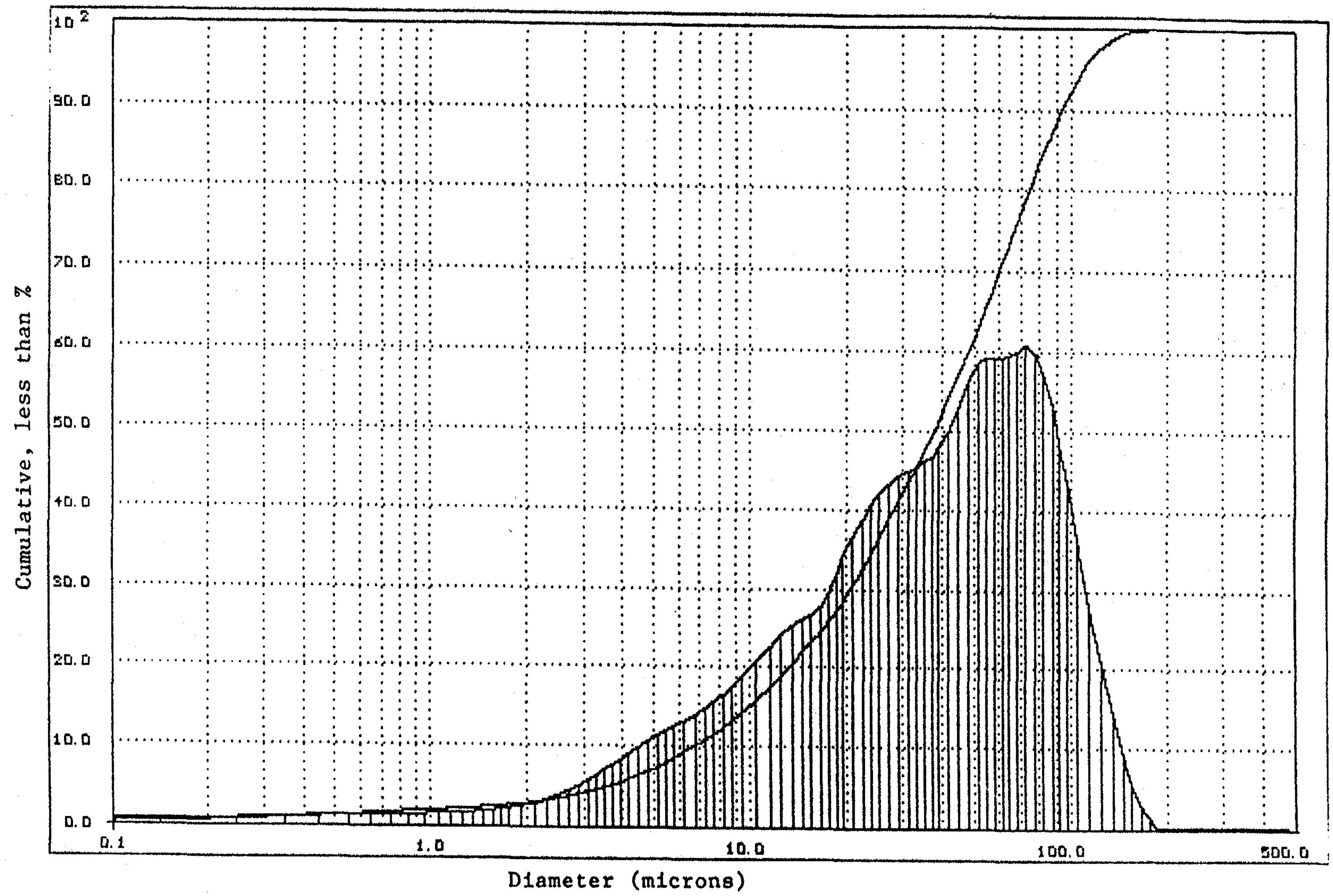

Figure 3. Particle size distribution of high sulfur (non-compliance) clean coal slurry (D_{50} ~ 37.3 μm)

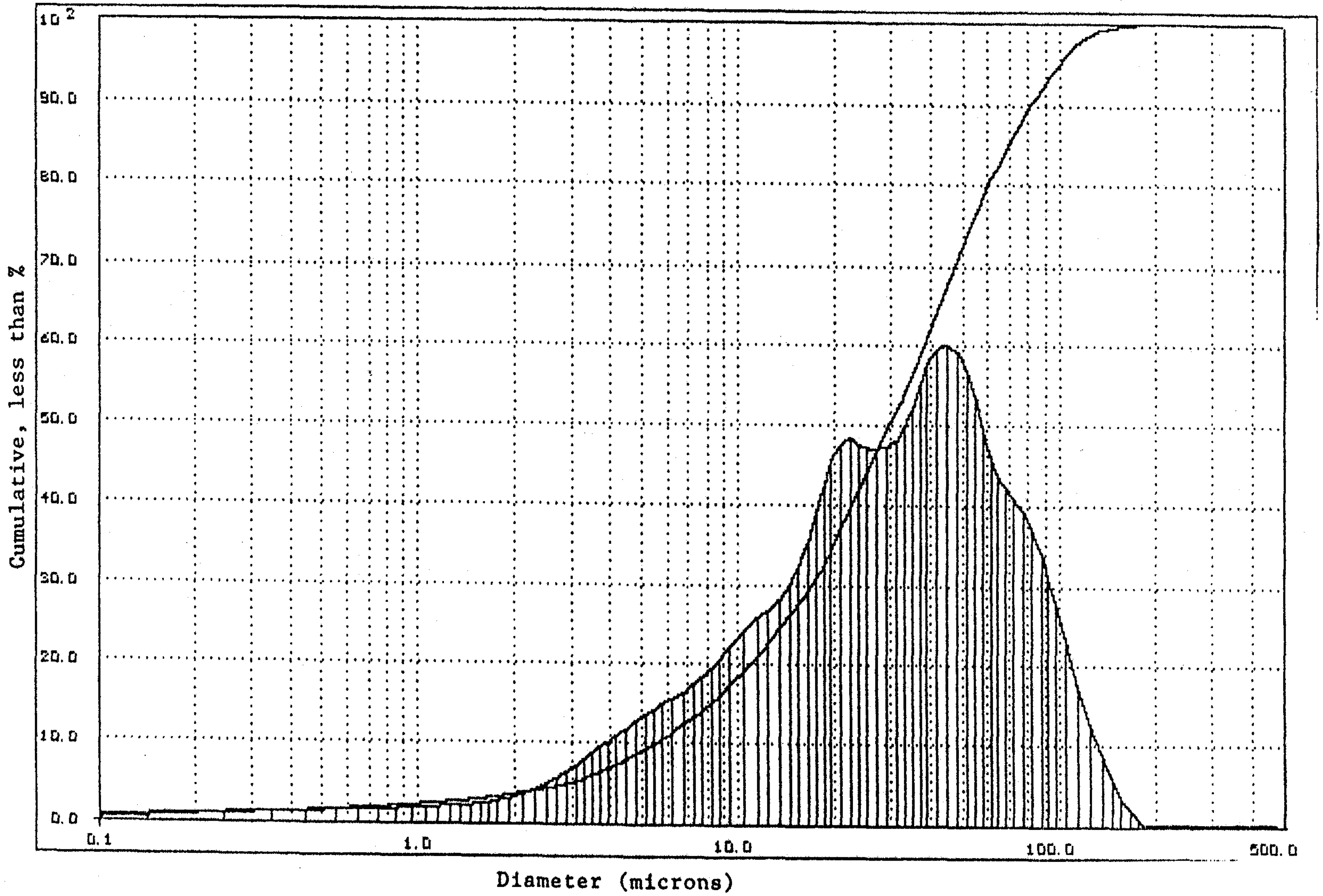

Figure 4. Particle size distribution of low sulfur (compliance) clean coal slurry (D_{50} ~ 29.5 μm)

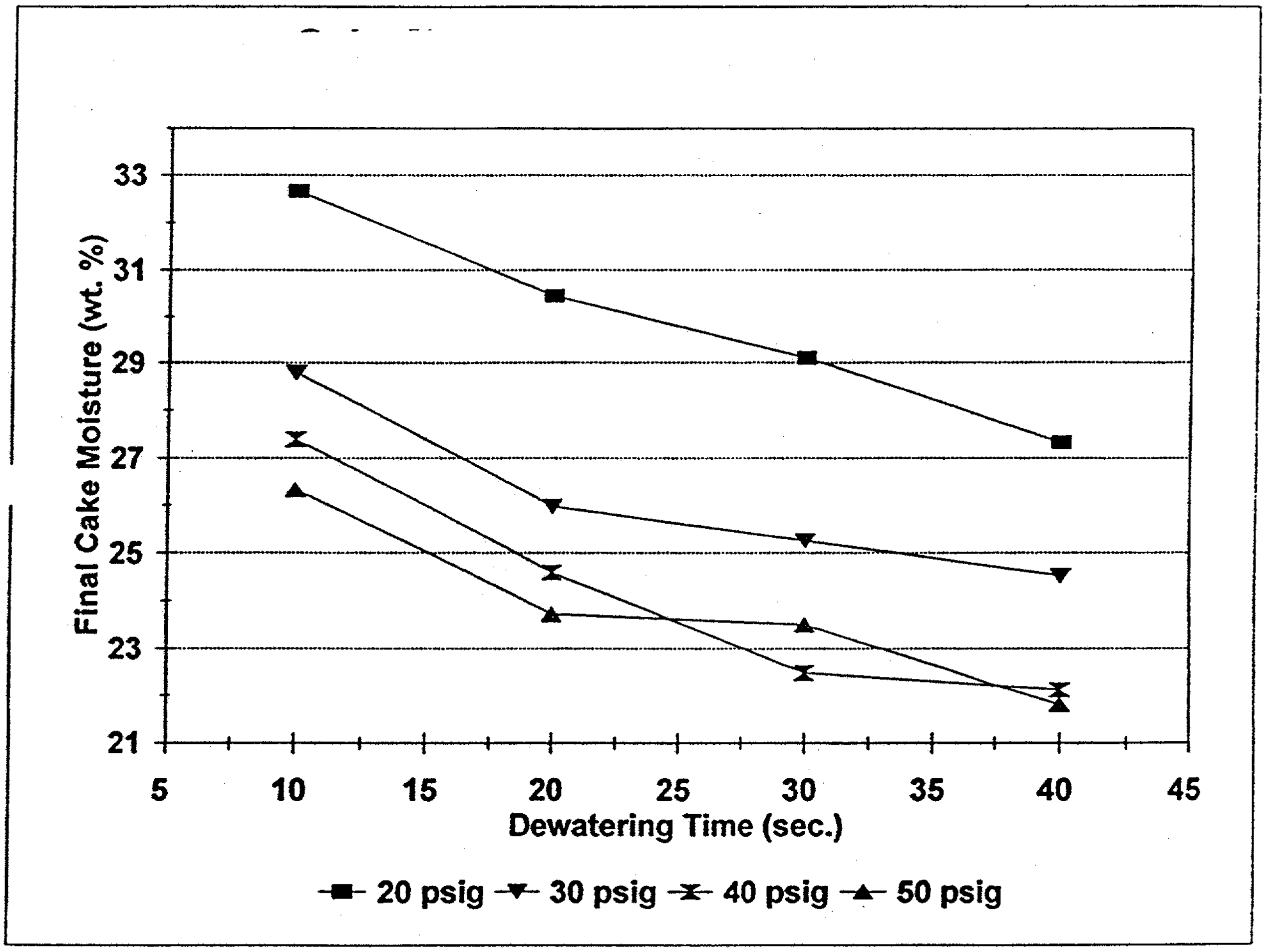

Figure 5. Effect of dewatering time on filter cake moisture using various pressures for 11 mm thick filter cake for the compliance coal slurry

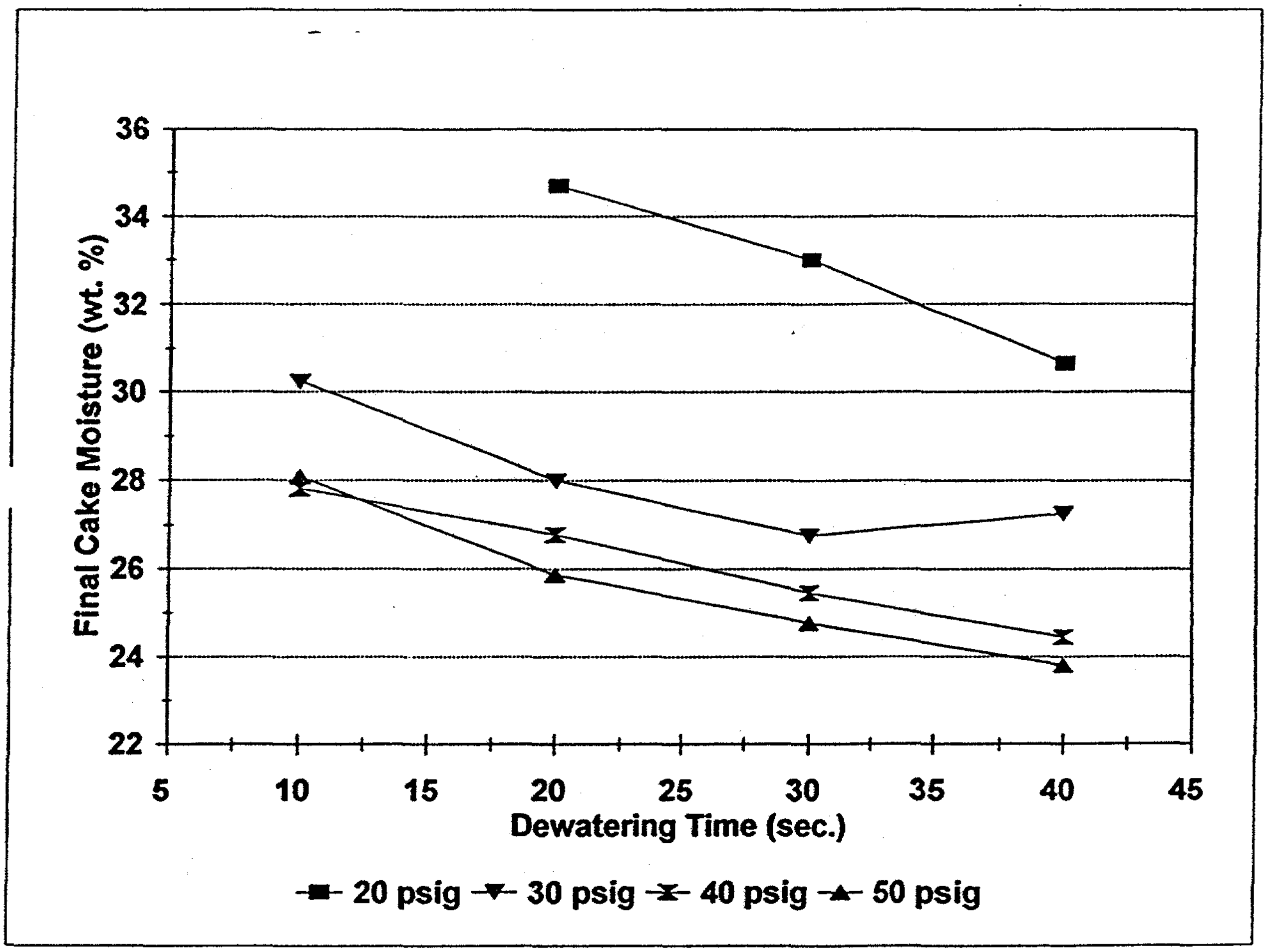

Figure 6. Effect of dewatering time on filter cake moisture using various pressure for a 15 mm thick filter cake for the compliance coal

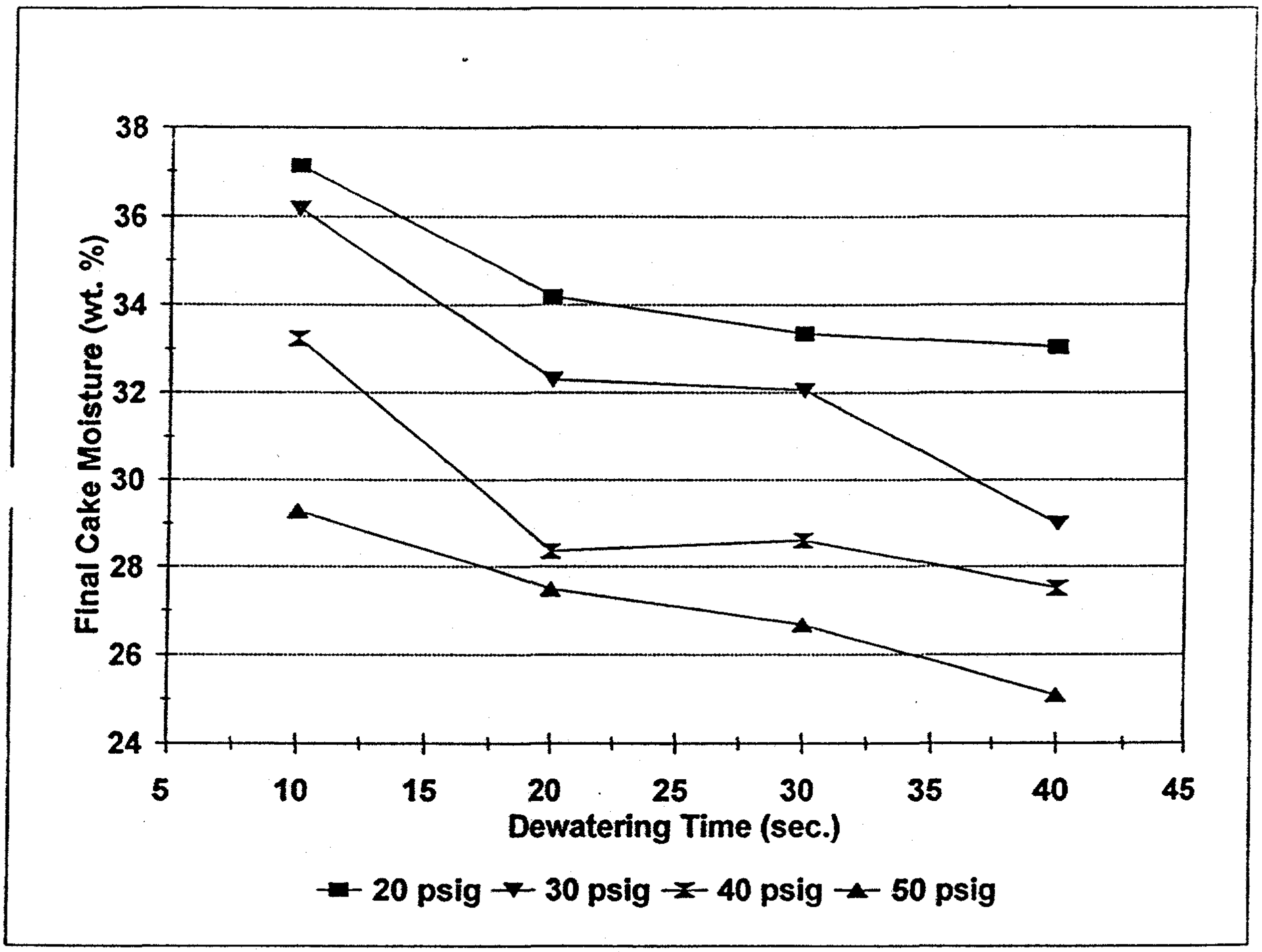

Figure 7. Effect of dewatering time on filter cake moisture using various pressures for a 20 mm thick filter cake for the compliance coal

sec dewatering time and 40 psi (2.8 bar) pressure. These results were in close agreement to that obtained previously.

Vacuum Dewatering:

The vacuum dewatering tests were conducted using a simple filter-leaf assembly shown in Figure 8. This setup simulates the actual plant conditions for vacuum filtration; especially the formation of filter cake without any segregation of large and small particles. Figure 9 shows the effect of cake thickness and cake formation time on filter cake moisture. Note, that as the cake thickness increases the cake formation time increases, however, the moisture content of filter cake after 4 mm cake thickness remains constant.

Figure 10 shows the effect of drying time, which is the time for moisture removal after the cake is formed, on cake moisture. As expected, the moisture reduces from 27.5% to 24.2% as the drying time is increased from 30 sec to 70 sec. These data (Figures 9 and 10) indicate that using a cake thickness of 10 mm with 40 sec formation time, and 70 sec drying time will be ideal for obtaining a low (~24%) moisture filter cake.

In the pilot plant testing at the Powell Mountain Coal Company, the total dewatering time (cake formation and drying time) is about one minute. Figure 11 simulates the 60 sec dewatering time in presence of various dosages of an anionic flocculant (sodium 2-ethylhexyl sulfate). Note, that increasing flocculant dosage from 5 g/t to 20 g/t did not show any significant reduction in moisture content of filter cake which remains constant around 28%, however, the cake thickness increases from 10 mm to 20 mm as the flocculant dosages is increased from 5 g/t to 10 g/t. These data

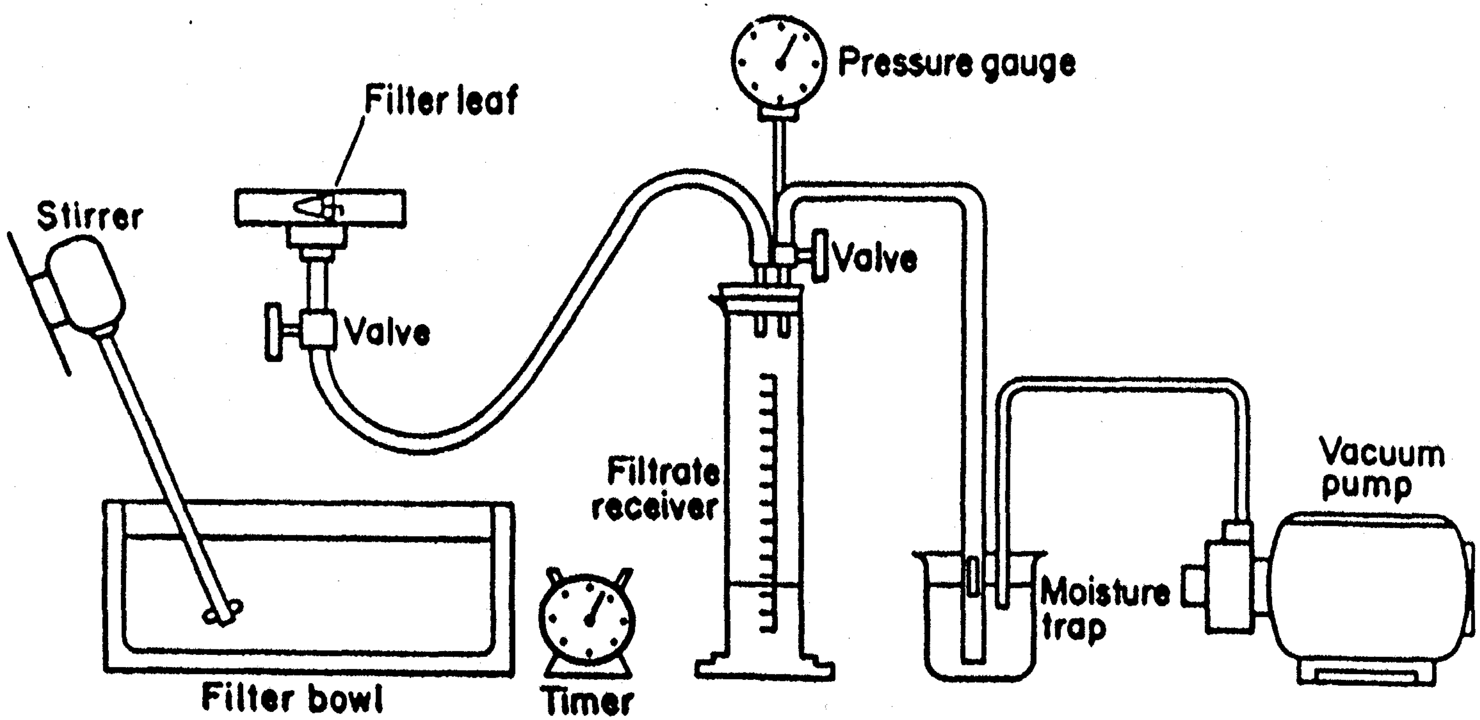

Figure 8. Simple filter-leaf test apparatus setup

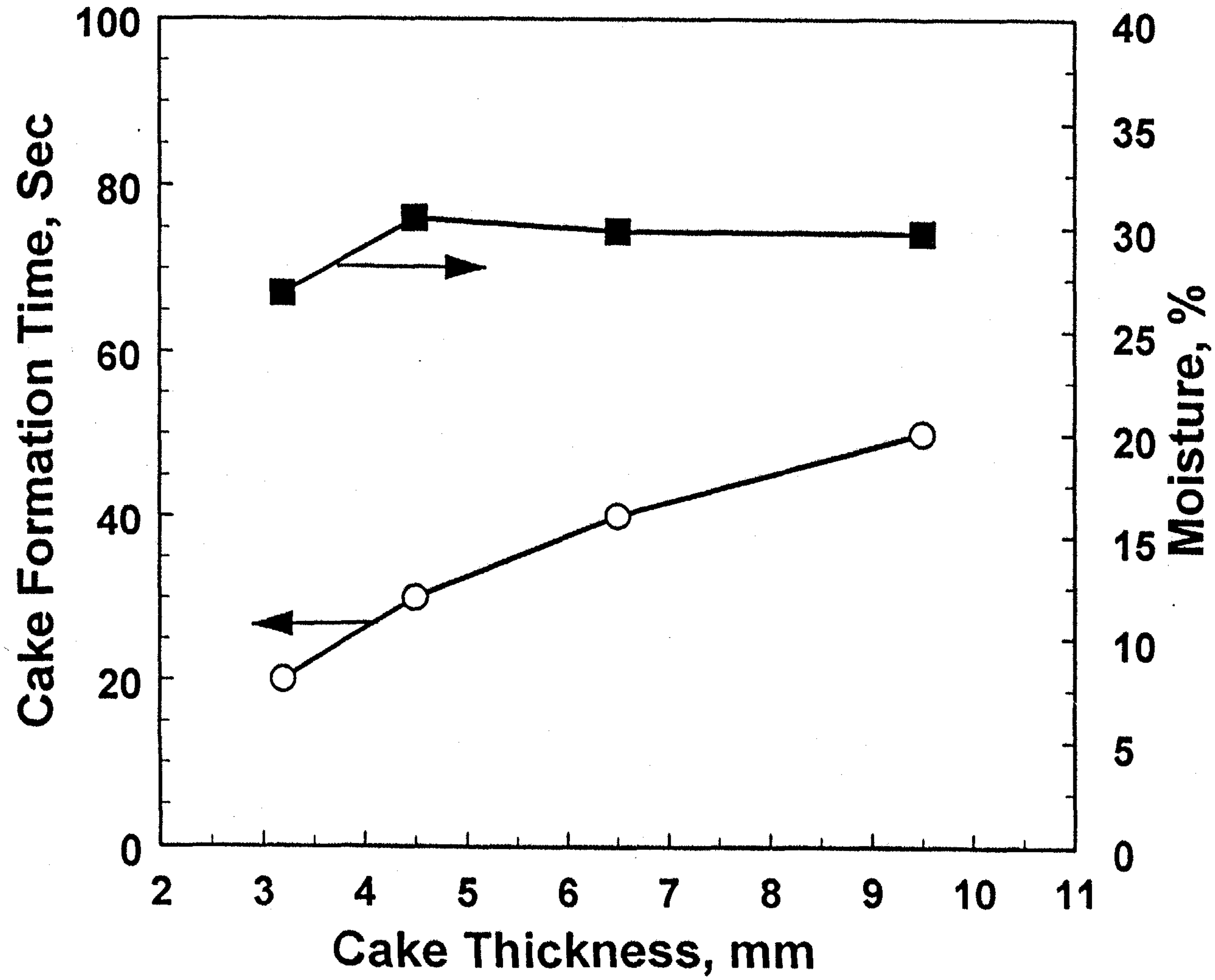

Figure 9. Effect of cake thickness and cake formation time on filter cake moisture using vacuum filtration for the non-compliance coal

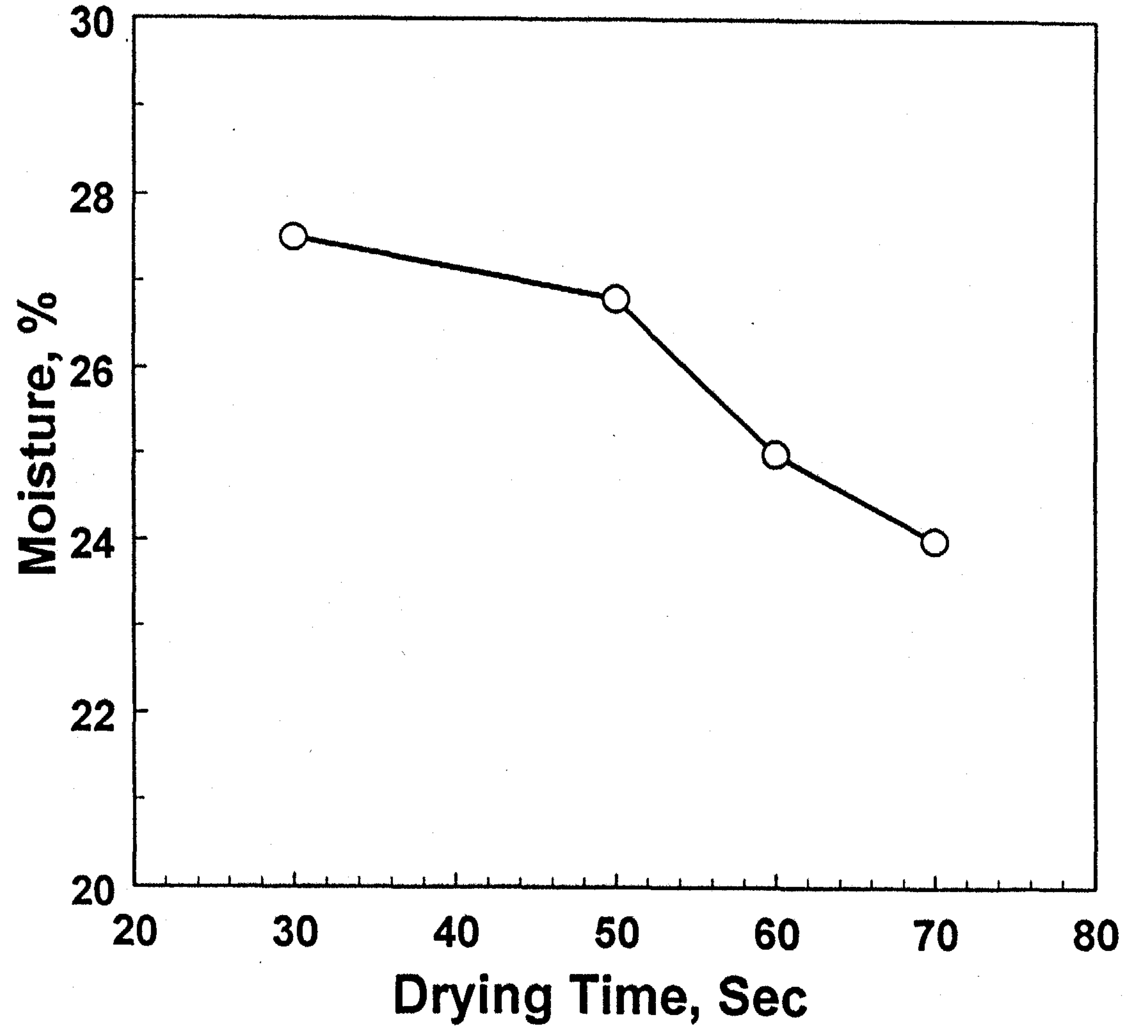

Figure 10. Effect of drying time on filter cake moisture using vacuum filtration for the non-compliance coal

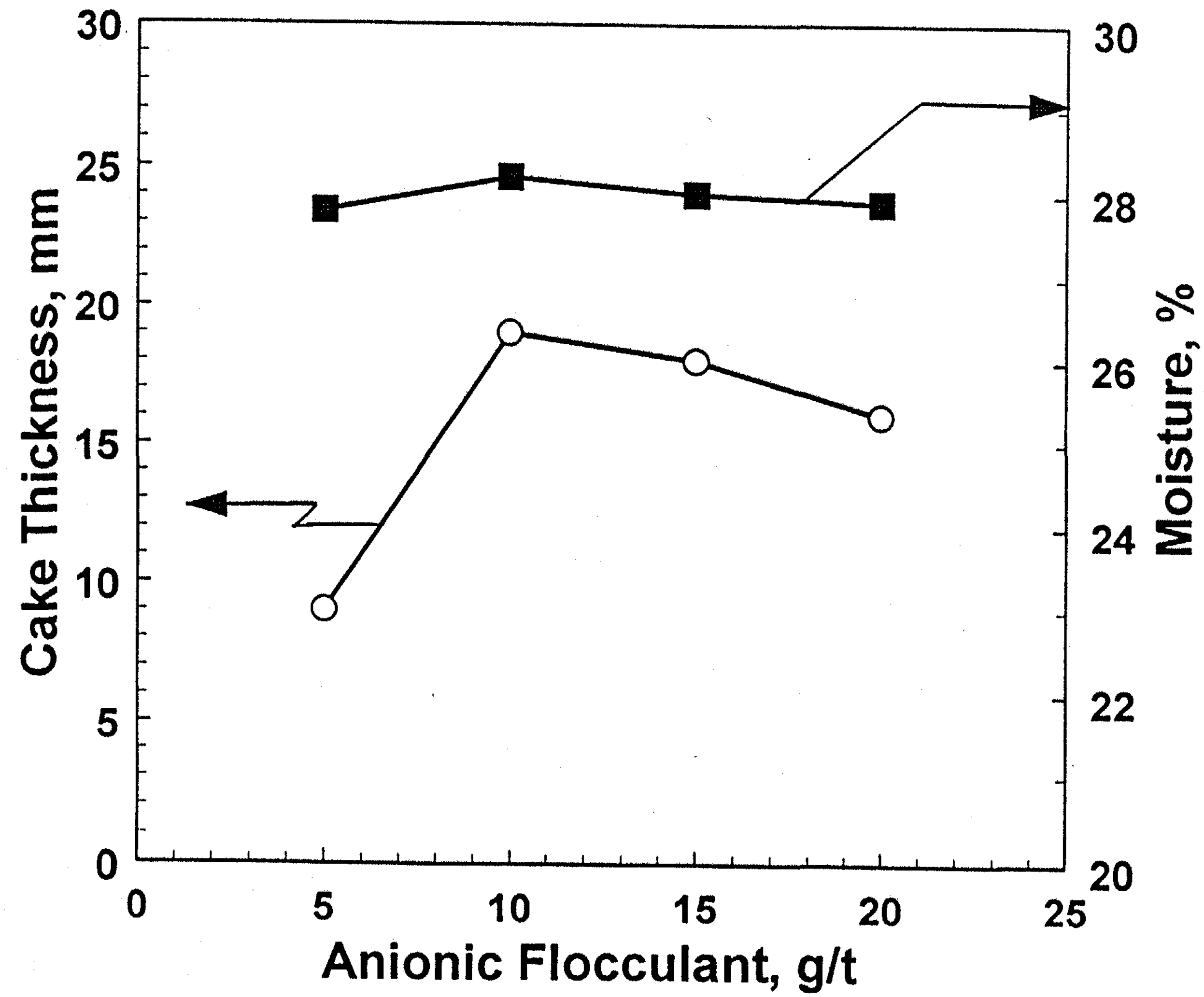

Figure 11. Effect of anionic flocculant dosage and cake thickness on filter cake moisture content for the non-compliance coal (cake formation time 30 sec; drying time 30 sec)

indicates that anionic flocculant will lower the filter cake moisture by absolute 2% over than obtained without flocculant and the amount of solids throughput will double using 10 g/t of the flocculant.

Figure 12 shows the effect of addition of the anionic flocculant with 15 sec cake formation time and 45 sec drying time. As expected, with larger drying time the filter cake moisture averaged 24%. However, the cake thickness was about 11 mm.

Figures 13 and 14 depicts the effect of cake thickness and cake formation time on filter cake moisture for the low sulfur (compliance) coal slurry, respectively. Note, that the moisture, as expected, increases with cake thickness. However, 35% filter cake moisture of 10 mm thick cake was 5% higher than the non-compliance coal. This could be due to fine particle size of the non-compliance coal slurry. Increasing cake formation time from 20 sec to 50 sec increased filter cake moisture from 28% to 30%.

Figure 15 shows the effect of drying time on filter cake moisture using a 30 sec cake formation time. Note, increasing drying time from 30 secs to 70 secs lowered the filter cake moisture from 29% to 20%.

Task 6. System Operation:

Pilot-scale vacuum filtration testing was conducted to determine the effect of surfactant type and dosage on cake moisture and throughput. The surfactants utilized in this phase of the investigation are summarized in Table II. In each test, the drum speed was held constant at 1 rpm and the slurry feed rate was controlled to maintain the filter tub level to allow for maximum drum submergence. Baseline testing was

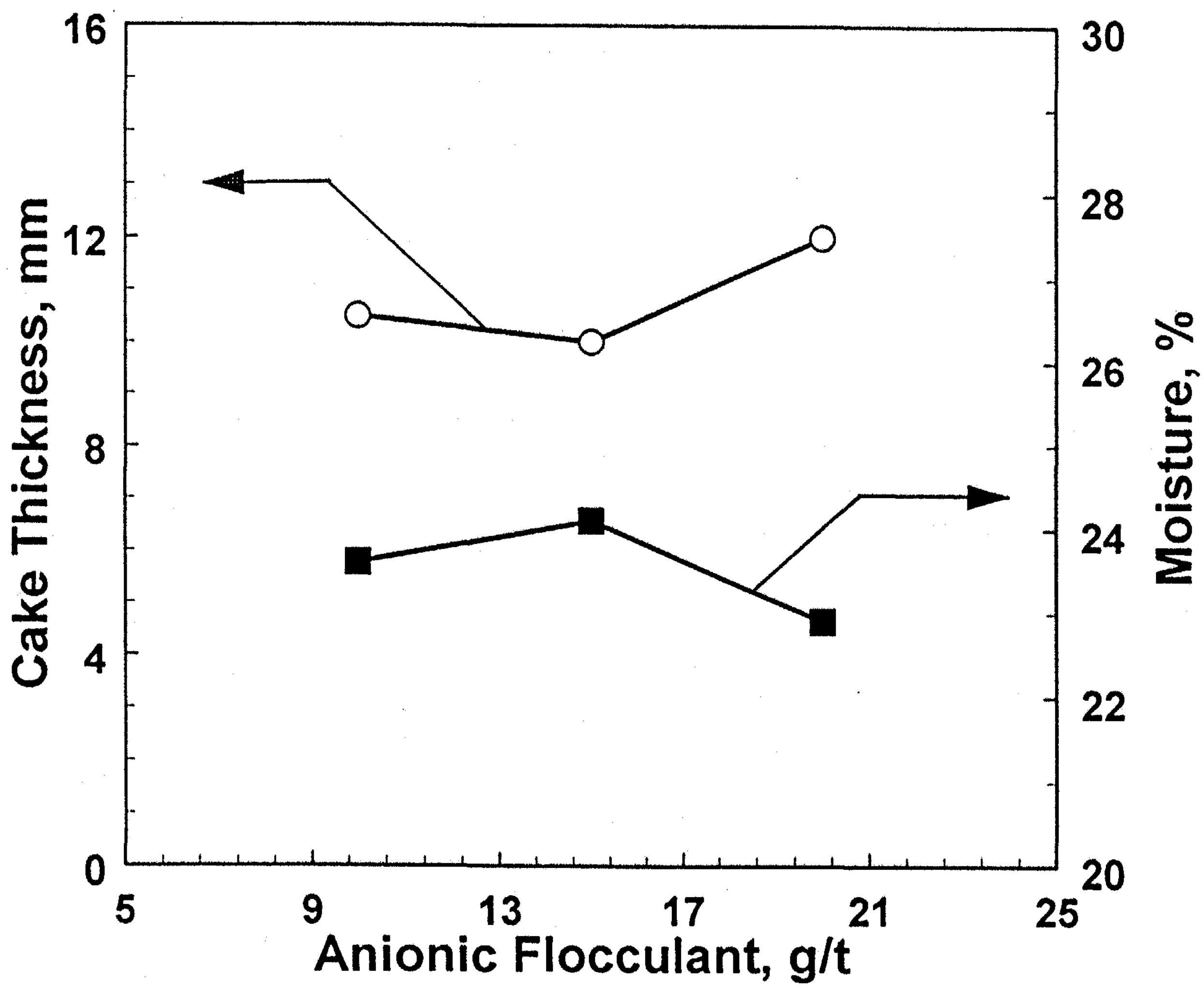

Figure 12. Effect of anionic flocculant on dewatering of non-compliance coal slurry (cake formation time 15 sec and drying time 45 sec)

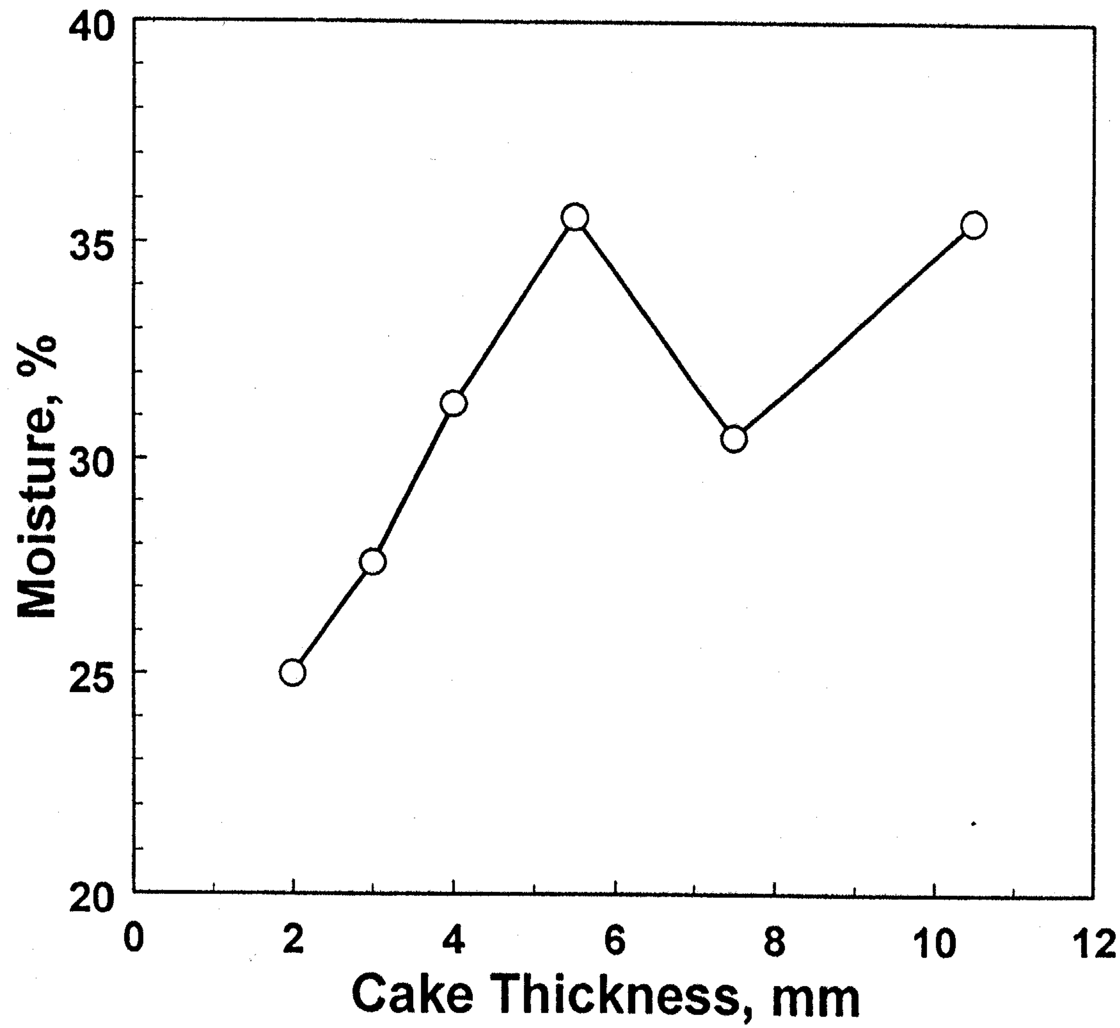

Figure 13. Effect of cake thickness on filter cake moisture for the compliance coal slurry

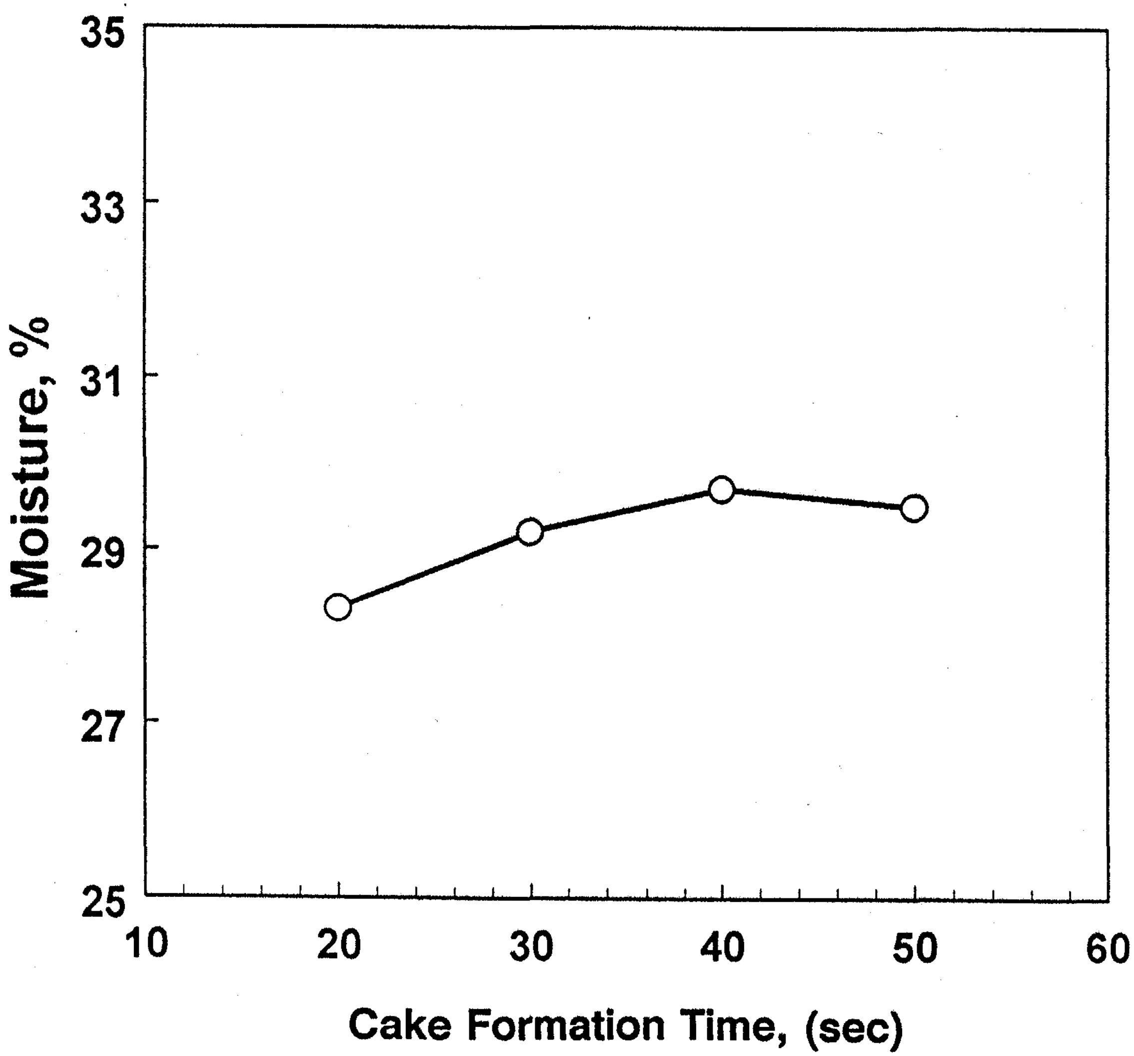

Figure 14. Effect of drying time on filter cake moisture of the compliance coal slurry

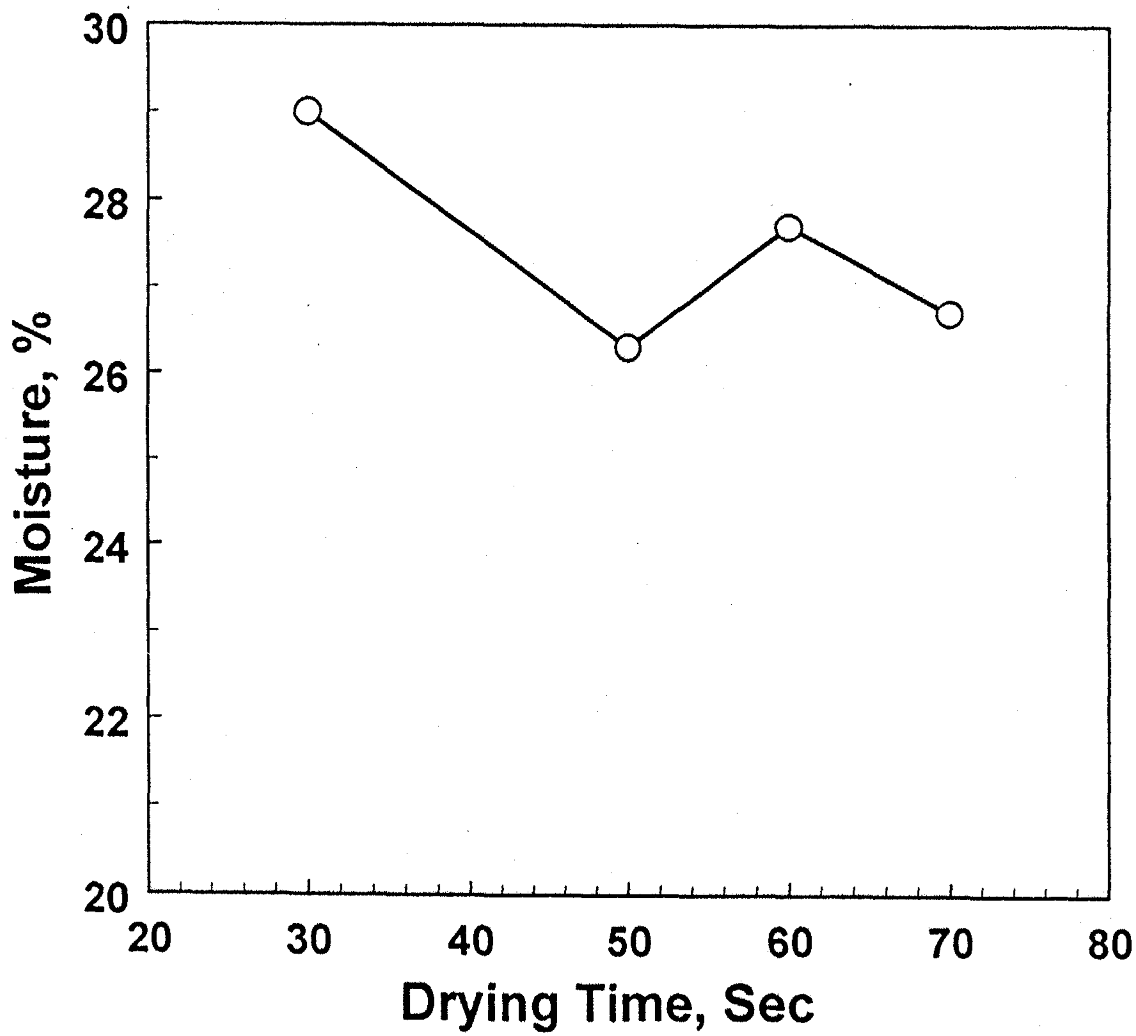

Figure 15. Effect of cake formation time on filter cake moisture of the compliance coal slurry

Table II. List of Surfactants Used in Filtration Studies

	Sodium 2-Ethylhexyl Sulfate	Octyl Phenoxy Polyethoxy Ethanol	1-Hexadecyl Pyridinium Chloride
Type	Anionic	Nonionic	Cationic
Abbreviation	S2ES	X114	CPCl
Commercial Name	NAS 08	TRITON-X-114	Cetyl Pyridinium Chloride
Active Ingredient (weight %)	40	100	100
Formula	$C_4H_9(C_2H_5)CH_2SO_4Na$	C_8H_{17}-C_6H_4-$(OCH_2CH_2)_{7\text{-}8}OH$	$C_{16}H_{33}C_5H_5NCl$
Molecular Weight	232	536	340
Manufacturer	Niacet Corporation Niagara Falls, NY	Rohm and Haas Philadelphia, PA	Sigma Chemical Co. St. Louis, MO
Critical Micelle Conc. (mg/l)	2500	120	246

conducted before and after each series of tests utilizing surfactant to ensure that all residual surfactant was removed from the system.

The effect of surfactant dosage on cake moisture for the high sulfur coal is shown in Figure 16. Baseline tests produced a filter cake containing 29.7% moisture. As expected, increasing surfactant dosage decreased cake moisture. At the lowest dosage of surfactant tested (0.25 Kg/t), the cationic surfactant (cetyl pyridiniumchloride or CPCl) produced the lowest cake moisture (27%) of the three surfactants tested. Further increasing the dosage of CPCl to 1.5 Kg/t reduced the cake moisture to 24.5%. The anionic surfactant (sodium 2-ethylhexyl sulfate or S2ES) effectively reduced cake moisture to 25.2% at a dosage of 1 Kg/t which was further reduced to

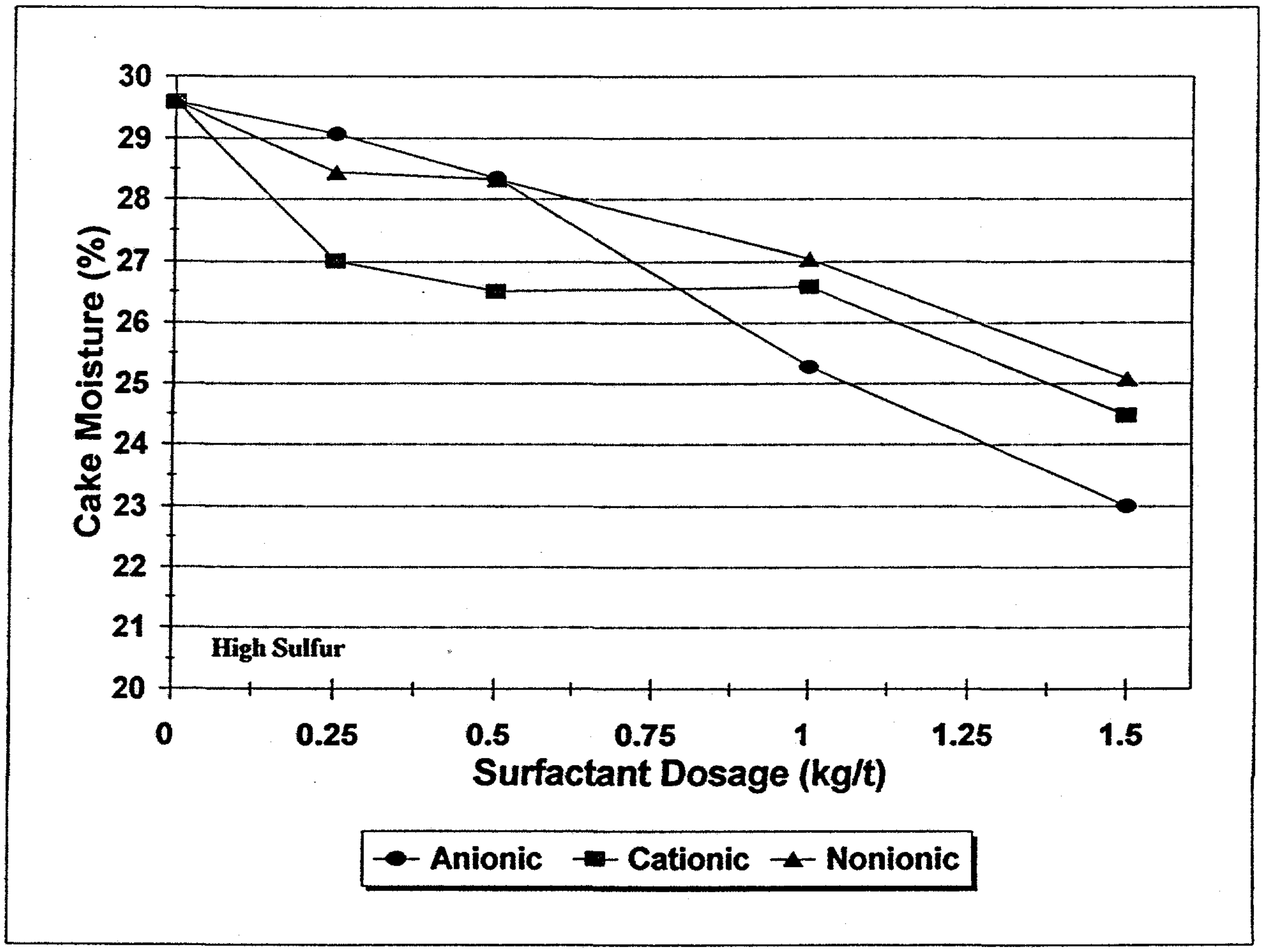

Figure 16. Effect of anionic, cationic and nonionic surfactant dosage on cake moisture for POC testing with high sulfur coal

23.0% at a dosage of 1.5 Kg/t. The nonionic surfactant (octyl phenoxy polyethoxy ethanol or X114) was the least effective surfactant for dewatering of the high sulfur coal slurry.

The effect of surfactant dosage on throughput for the high sulfur coal is summarized in Figure 17. In general, there was little change in the throughput, except at the highest dosage. Surfactants X114 and CPCl produced similar throughput (8 to 13 $lb/ft^2/hr$) at dosages below 1 Kg/t. At the highest dosage of 1.5 Kg/t, the throughput obtained with S2ES increased to 17.5 $lb/ft^2/h$. Similar results were achieved with CPCl, however the overall throughput was higher (16 to 25.5 $lb/ft^2/hr$). The higher throughput obtained with CPCl was not necessarily due to the surfactant. Note that the baseline results obtained when CPCl was higher than compared to the other surfactants. This was due to higher feed solids during the tests with CPCl which resulted in higher throughput, however, there was no difference in the cake moisture.

For surfactant dosage tests using the compliance coal slurry, similar cake moisture results were obtained (Figure 18). Increasing surfactant dosage to 0.5 Kg/t reduced moisture from a baseline of 27% moisture to as low as 22 to 23% moisture. Higher dosages of cationic surfactant provided no further moisture reduction, while increasing the dosage of nonionic and anionic surfactant reduced the cake moisture to 17 and 19.2% moisture respectively.

The throughput obtained with surfactants on the compliance coal is summarized in Figure 19. For anionic and nonionic surfactants, there was no change in throughput when a low dosage of <0.5 Kg/t was used. At higher dosages of nonionic surfactant, no changes in throughput occurred. However, when higher

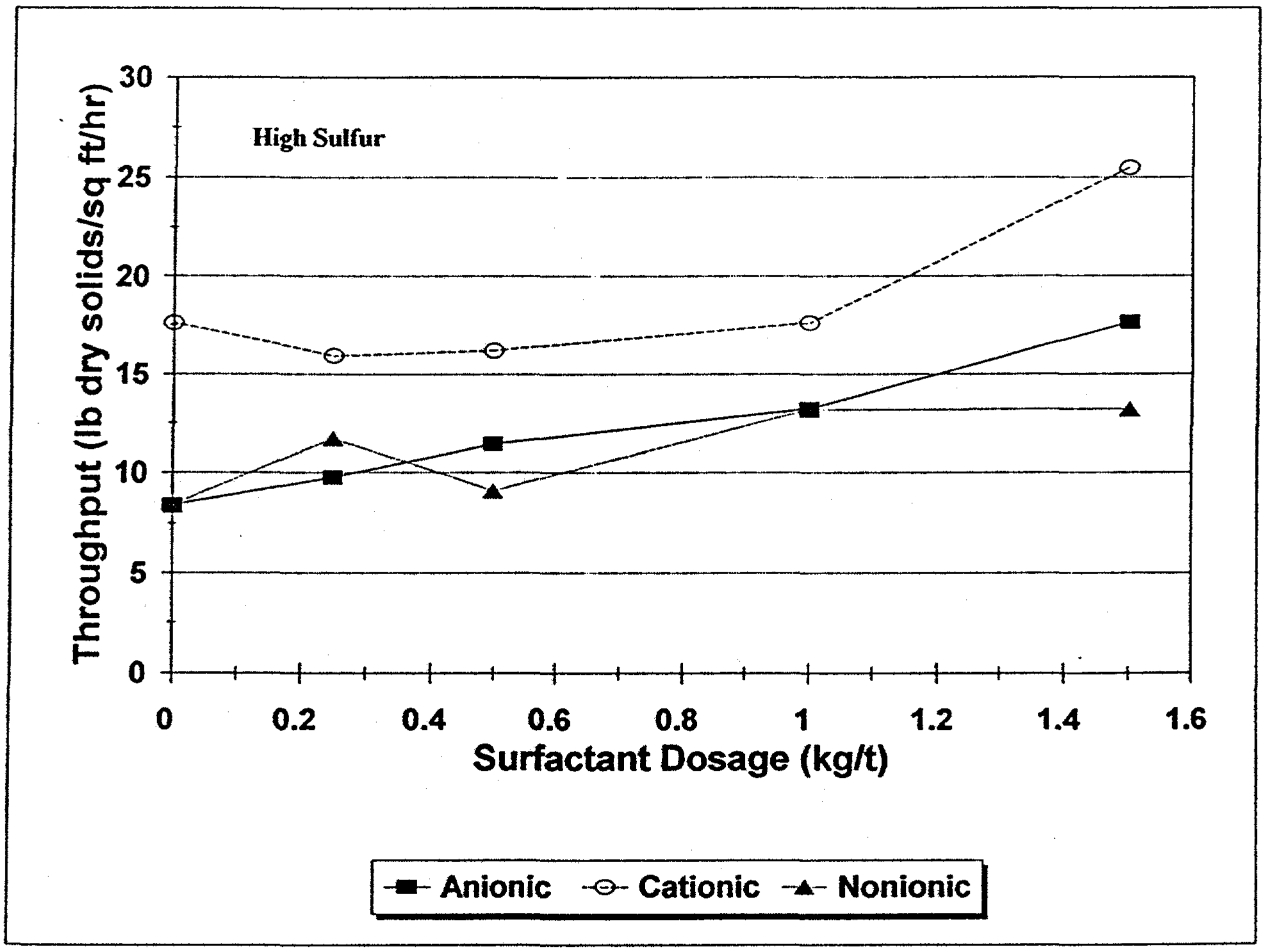

Figure 17. Effect of anionic, cationic and nonionic surfactant dosage on throughput for POC testing with high sulfur coal

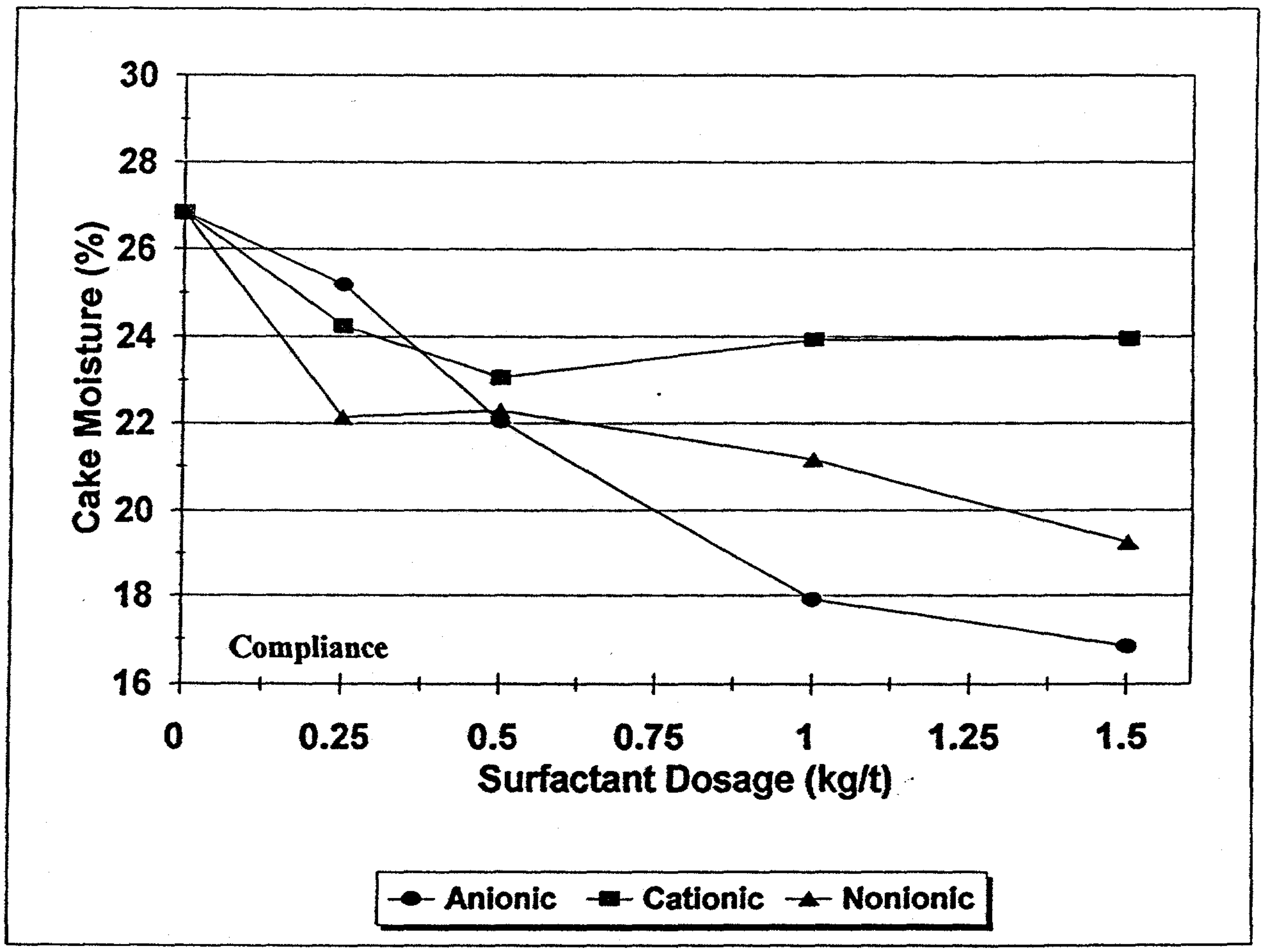

Figure 18. Effect of anionic, cationic and nonionic surfactant dosage on cake moisture for POC testing with compliance coal

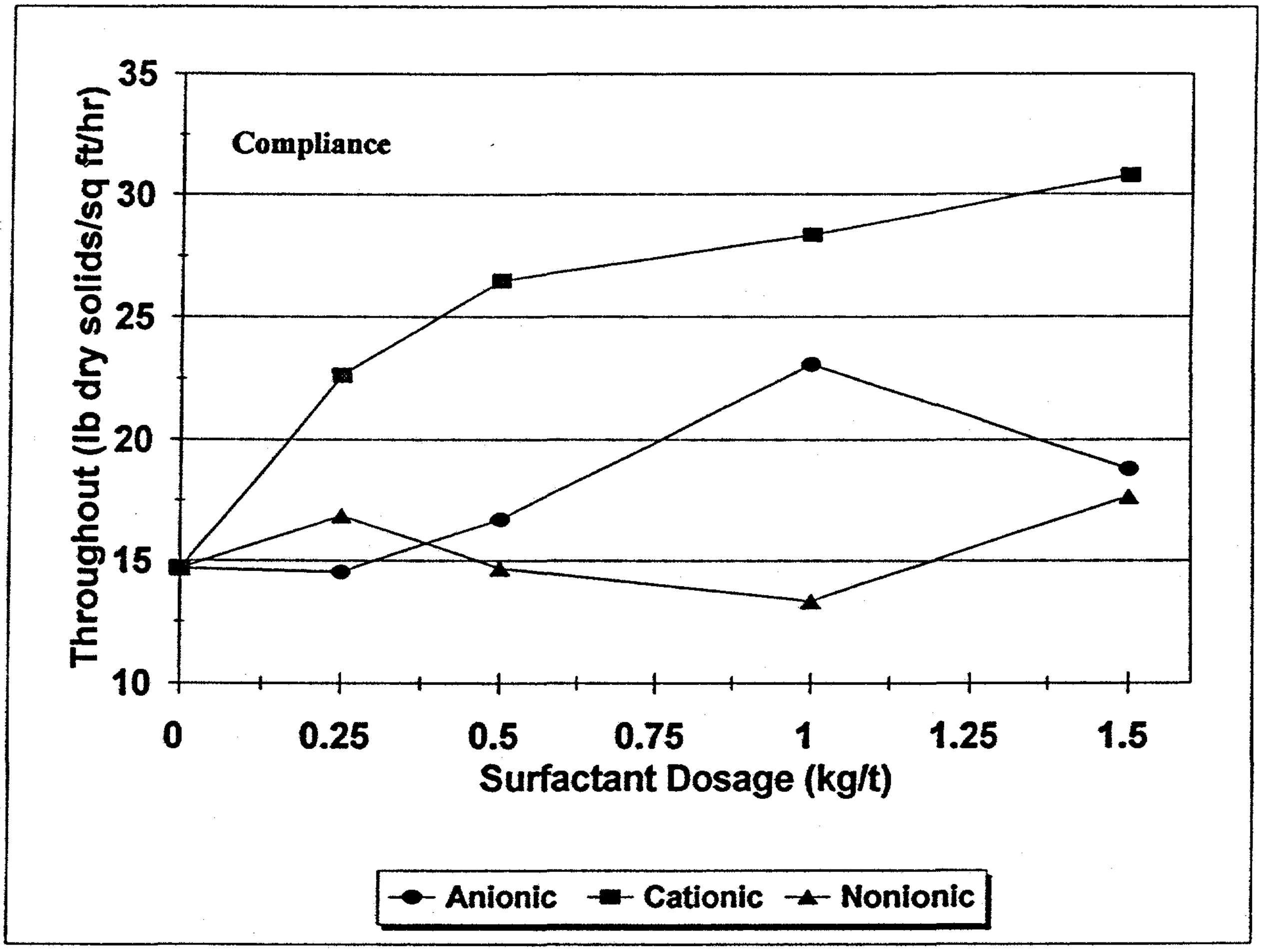

Figure 19. Effect of anionic, cationic and nonionic surfactant dosage on throughput for POC testing with compliance coal.

dosages of anionic surfactant were used, the throughput increased from 17 $lb/ft^2/hr$ to 23 $lb/ft^2/hr$ at a dosage of 1 Kg/t. For the cationic surfactant, increasing the dosage produced higher throughput. As the surfactant dosage was increased, throughput also increased. A dosage of 1.5 Kg/t increased throughput to 31 $lb/ft^2/hr$ from 15 $lb/ft^2/hr$ when no surfactant was used.

A comparison of the results obtained with cationic surfactant with both coals is shown in Figure 20. A surfactant dosage resulted in lower cake moisture. Lower moistures were obtained for the compliance coal, primarily due to lower feed solids, however it is apparent that a dosage of 0.5 Kg/t was sufficient to reduce cake moisture for both feed coals. At this dosage, the compliance slurry reduced cake moisture from a baseline of 27% to 23% while for the high sulfur coal, cake moisture was reduced from 29.6% to 26.5%. The resulting throughputs from these tests are summarized in Figure 21. When the cationic surfactant was used, solids throughput increased with increasing surfactant dosage and actually doubled from 15 to 31 $lb/ft^2/hr$ by increasing the surfactant dosage from 0 to 1.5 Kg/t. No significant change in the solids throughput occurred for the high sulfur coal, except at the highest dosage tested (1.5 Kg/t) where a throughput of 25.5 $lb/ft^2/hr$ was obtained.

For the nonionic surfactant, cake moisture with the compliance coal was reduced from 27 to 22% moisture at a dosage of only 0.25 Kg/t (Figure 22). Increasing the dosage to 1.5 Kg/t reduced cake moisture to as low as 19%. For the high sulfur coal, the reduction in cake moisture was not as significant. Increasing the dosage from 0 to 0.25 Kg/t reduced moisture from 29.7 to 28.2%. At a high dosage

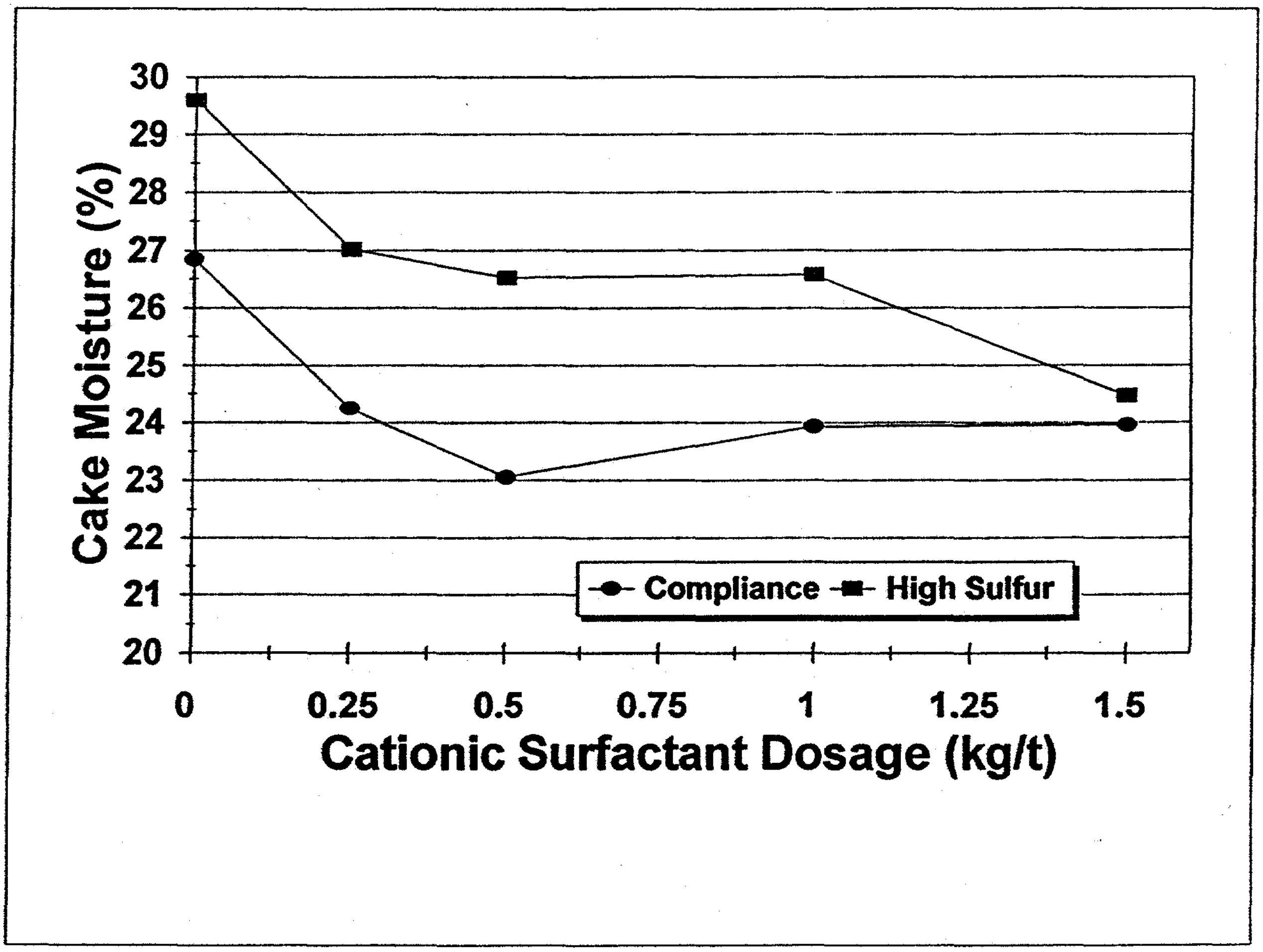

Figure 20. Effect of cationic surfactant dosage on cake moisture for POC testing with high sulfur and compliance coal

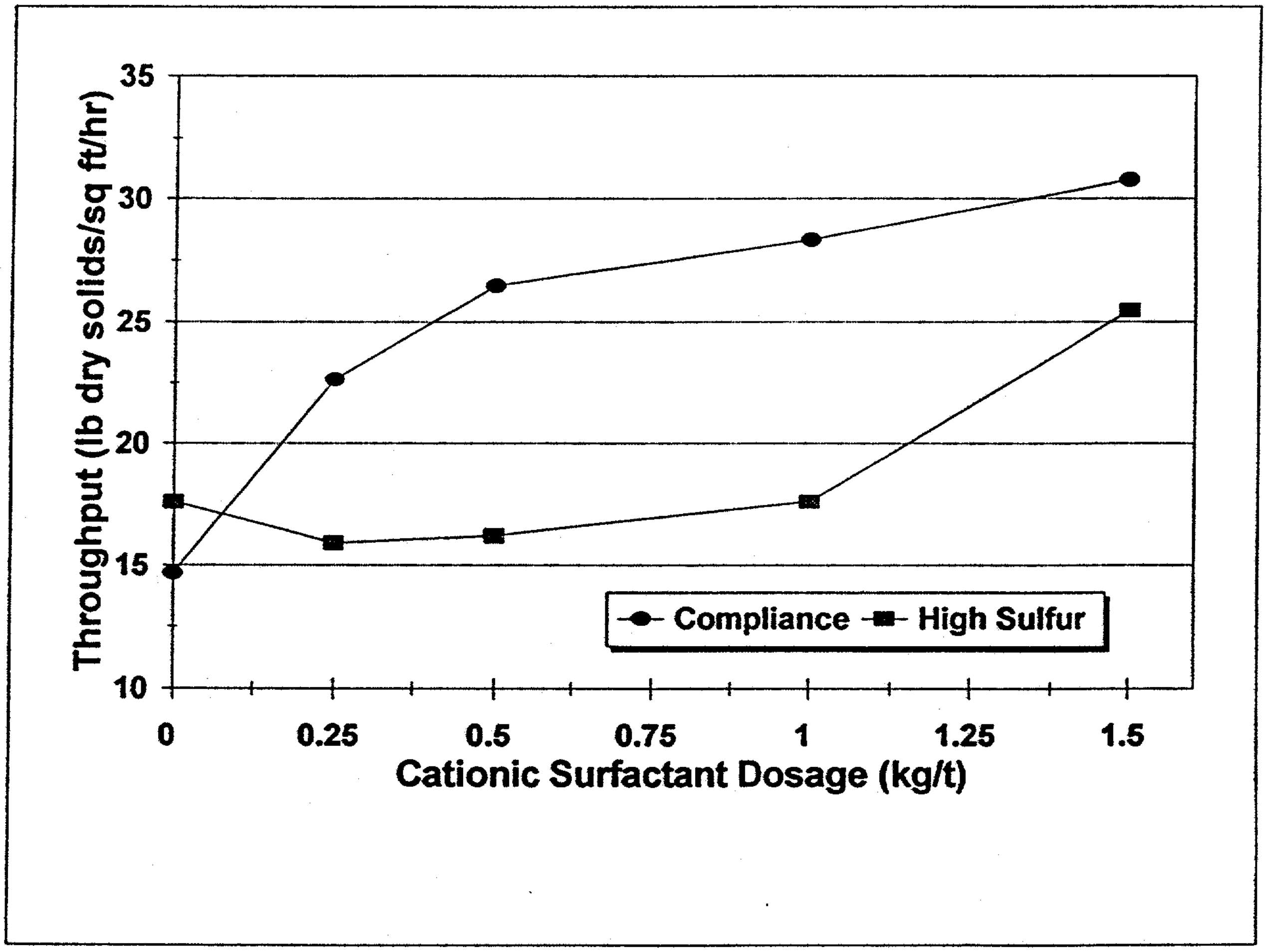

Figure 21. Effect of cationic surfactant dosage on throughput for POC testing with high sulfur and compliance coal

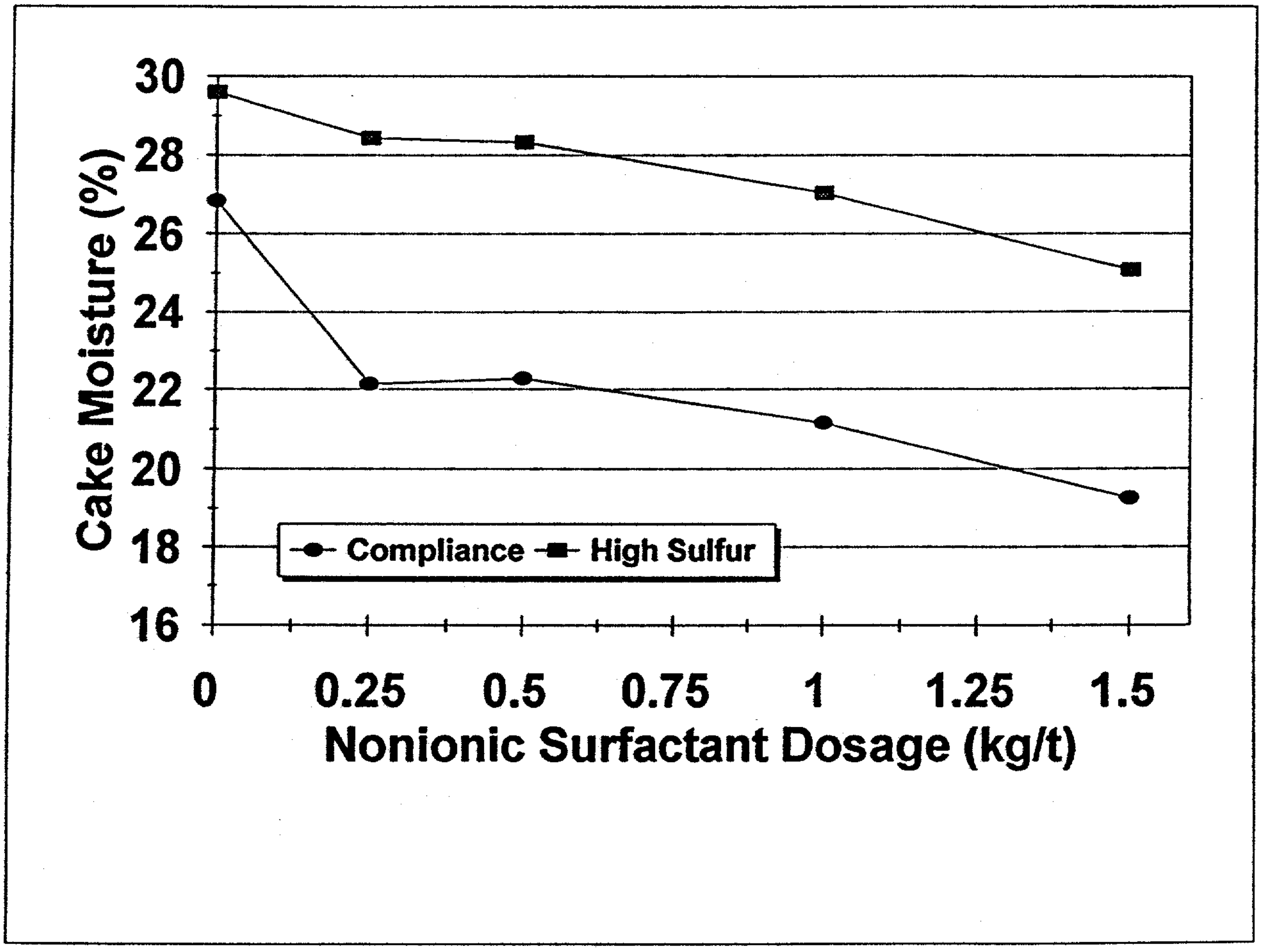

Figure 22. Effect of nonionic surfactant dosage on cake moisture for POC testing with high sulfur and compliance coal

of 1.5 Kg/t, moisture was reduced to 25%. These results show that the nonionic surfactant was more effective for reducing moisture with the compliance coal.

The throughput obtained with the complaint coal was essentially unchanged by increasing nonionic surfactant dosage as shown in Figure 23. The throughput was 13 to 16 $lb/ft^2/hr$ throughout the dosage range tested. For the high sulfur coal, the throughput increased with increasing dosage from a baseline throughput of 8.2 $lb/ft^2/hr$ to 17.9 $lb/ft^2/hr$ at a dosage of 1.5 Kg/t.

The anionic surfactant was effective for reducing cake moisture for both the compliance and high sulfur coals, however, the dosage required was higher for the cationic and nonionic surfactants. As shown in Figure 24, cake moisture was reduced for the compliance coal from a baseline of 27% moisture to 18% moisture at a dosage of 1 Kg/t. Further increasing the dosage to 1.5 Kg/t reduced the cake moisture to 17%. For the high sulfur coal, baseline moisture of 29.8% was reduced to 25.5% with 1 Kg/t anionic surfactant and further increasing the dosage to 1.5 Kg/t reduced the moisture to 23%.

The resulting throughput with anionic surfactant is summarized in Figure 25. For both the compliance and high sulfur coals, increasing surfactant dosage produced higher throughput. For the compliance coal, increasing the dosage from 0 to 1.5 Kg/t more than doubled throughput from 8.3 to 17.9 $lb/ft^2/hr$. For the high sulfur coal, the baseline throughput with no surfactant was 14.3 $lb/ft^2/hr$; this was increased to 23 $lb/ft^2/hr$ with 1 Kg/t anionic surfactant.

The results obtained from POC testing clearly show that increasing surfactant dosage reduced cake moisture and in most cases increased throughput. The results

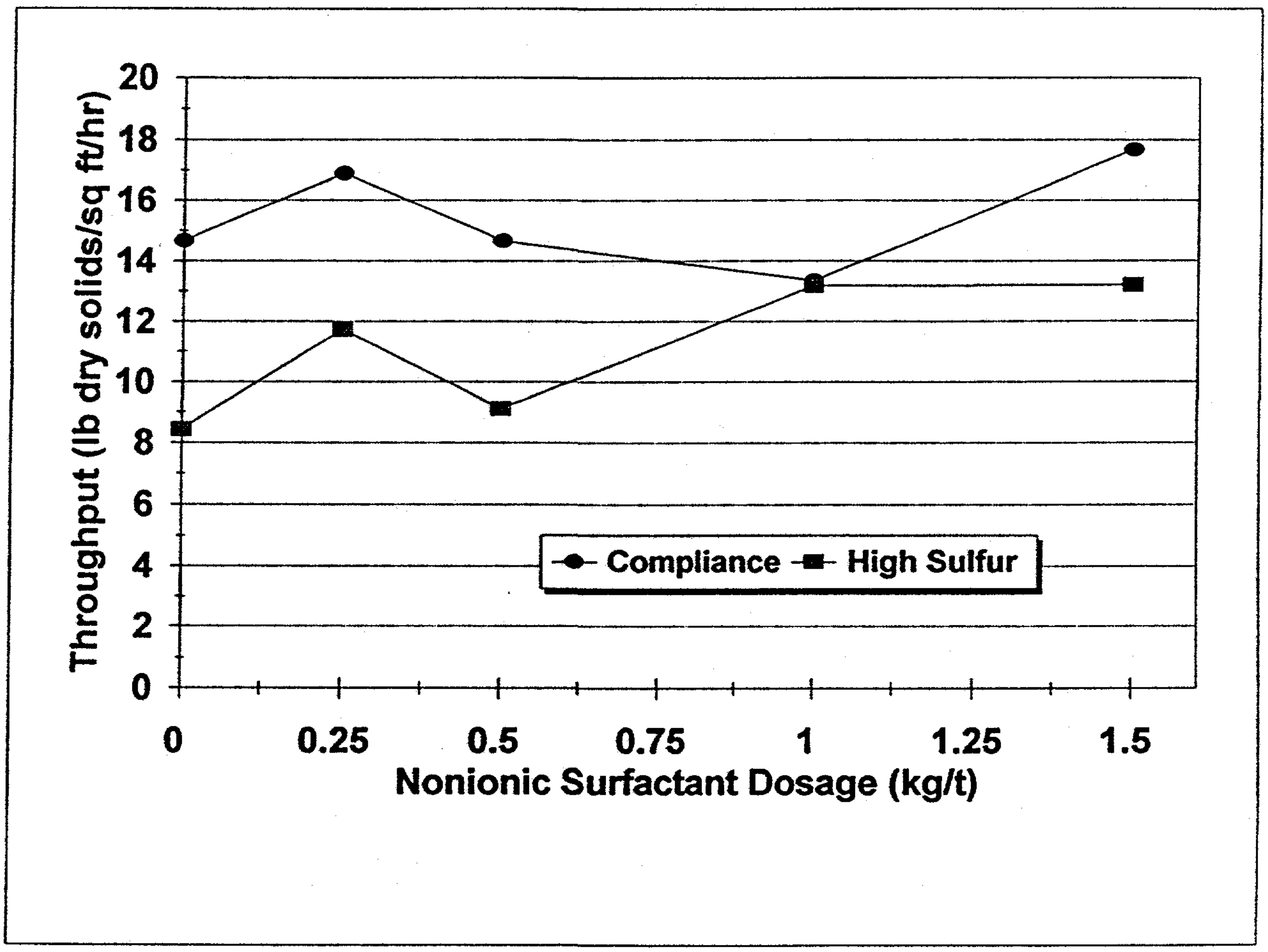

Figure 23. Effect of nonionic surfactant dosage on throughput for POC testing with high sulfur and compliance coal

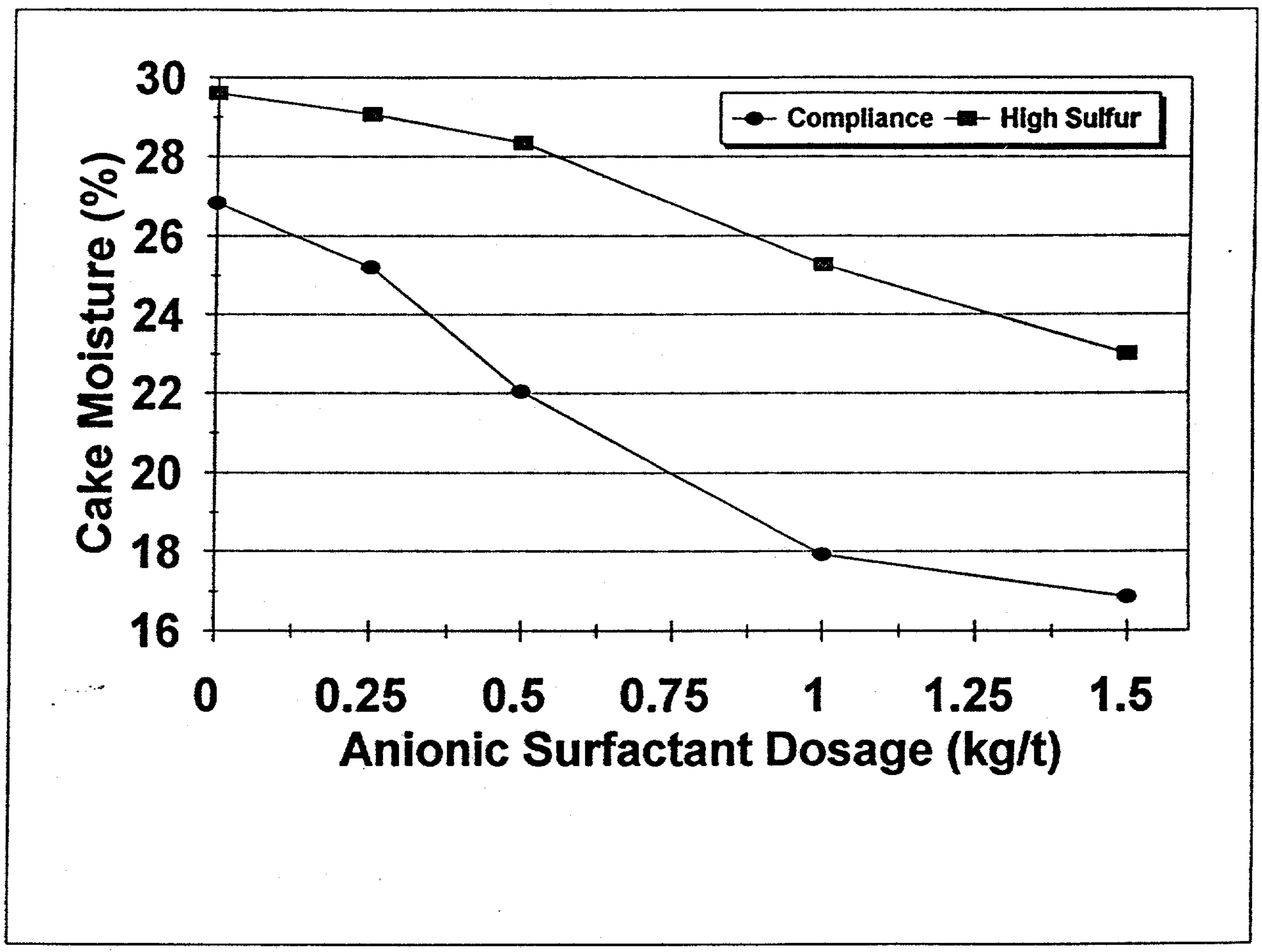

Figure 24. Effect of anionic surfactant dosage on cake moisture for POC testing with high sulfur and compliance coal

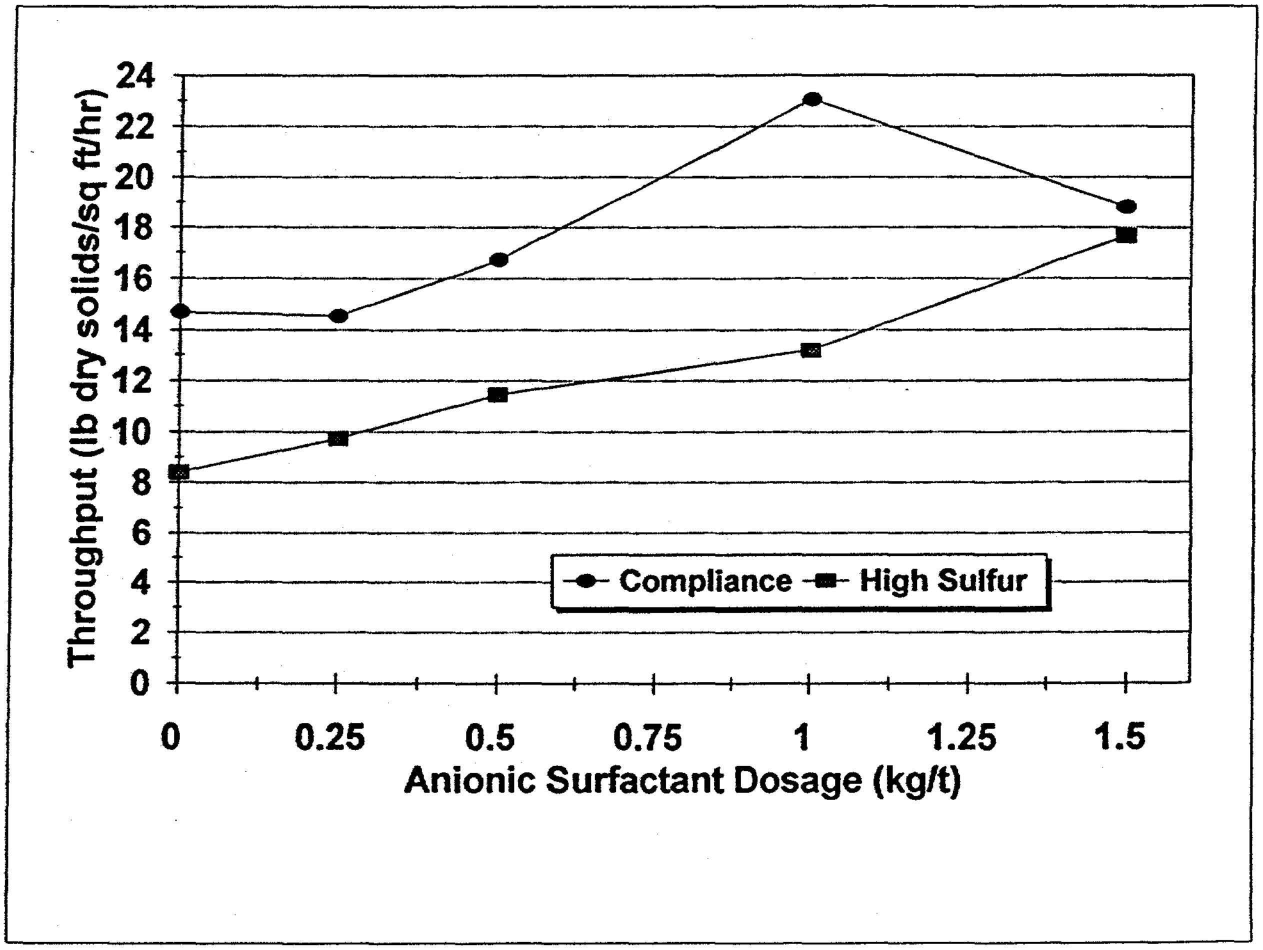

Figure 25. Effect of anionic surfactant dosage on throughput for POC testing with high sulfur and compliance coal

obtained were different for the two types of coal tested, due in part to the differences in the feed solids of the slurries tested. For the compliance coal at the lowest surfactant dosage tested (0.25 Kg/t), nonionic surfactant produced the lowest cake moisture (22%). At a dosage of 0.5 Kg/t, all three surfactants preformed similarly on the compliance coal (22 to 23%) moisture. At higher dosages (1 to 1.5 Kg/t) the anionic surfactant was the most effective (17 to 18% moisture) followed by the nonionic surfactant (19 to 21% moisture). The cationic surfactant was the least effective at higher dosage producing cake moistures of 24%.

For the high sulfur coal at low dosage (<0.5 Kg/t), the cationic surfactant was the most effective for moisture reduction producing cakes as low as 26.5% moisture. At higher dosage, the anionic surfactant was the most effective.

To summarize the throughput for the compliance coal, the cationic surfactant clearly showed increased throughput with increasing dosage. The anionic surfactant increased throughput, but no increase was evident until higher dosages (>0.5 Kg/t) were utilized. Nonionic surfactant had no effect on throughput with the compliance coal.

With the high sulfur coal, no significant increases in throughput were observed with any surfactant at dosages below 0.5 Kg/t. At higher dosages, both cationic and anionic surfactants increased throughput, however the increase was not as significant as was observed with the compliance coal. The nonionic surfactant did not affect throughput with either the compliance or high sulfur coal slurry.

ACTIVITIES FOR NEXT QUARTER

Additional laboratory vacuum dewatering tests will be performed using various types of dewatering reagents. Vacuum drum filter pilot plant tests will be conducted using various flocculants and metal ions.