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I am submitting herewith a thesis written by Dick Yu-Gen Chen entitled "The Stabilization of Flue Gas Desulfurization Sludge by Addition of Lime and Fly Ash." I recommend that it be accepted in partial fulfillment of the requirements for the degree of Master of Science, with a major in Environmental Engineering.

Dennis W. Weeter, Major Professor

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THE STABILIZATION OF FLUE GAS DESULFURIZATION SLUDGE BY ADDITION OF LIME AND FLY ASH

A Thesis

Presented for the

Master of Science

Degree

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ABSTRACT

One major problem associated with the flue gas desulfurization scrubbers is the disposal of the unstable and potentially polluting sludge that is produced. Certain properties of the sludge must be improved before the sludge is suitable for disposal. A review of the chemical composition and physical properties of the sludge was made to provide information concerning the nature of the problem. The current commercial processes available were also discussed.

The objective of this study is to investigate the effect of the addition of lime and fly ash on improving the strength, which was detected by increasing shear stress of the waste material at different curing times. The formation of new minerals in the treated sludge was detected by X-ray diffractometer. The scaning electron microscope was used to observe the morphology of the treated waste material.

The increase in shear strength of the treated sludge in relation to the curing time shows the possibility of using lime and fly ash as additives to improve the properties of desulfurization sludge.

TABLE OF CONTENTS

CHAPTER		PAGE
I.	INTRODUCTION	1
II.	REVIEW OF LITERATURE	4
III.	EXPERIMENTAL DESIGN AND PROCEDURES	19
	Sample Preparation	19
	Settling Test	23
	Vacuum Filtration Test	25
	Direct Shear Strength Test	25
	X-ray Diffractometry	26
	Scanning Electron Microscopic (SEM) Photo	27
IV.	RESULTS AND DISCUSSION	28
	Batch Settling Study	28
	Buchner Funnel Test	33
	Filter Leaf Test	38
	Shear Strength Test	42
	X-ray Diffractometer Analysis	51
	Scanning Electron Microscopic (SEM) Pictures	62
V.	CONCLUSIONS	74
VI.	RECOMMENDATIONS	76
BIBLIOGRAPHY		77
APPENDICES		81
Appendix A		82
Appendix B		85
VITA		92

LIST OF TABLES

TABLE		PAGE
I.	Unit Operating, Under Construction or Planned As Of February 1975 (20)	8
II.	The Composition of Scrubber Sludge A	20
III.	The Composition of Scrubber Sludge B	21
IV.	Characteristics of Semi-Annual Composite Fly Ash from TVA Widows Creek Steam Plant	22
V.	The Calculated Initial Settling Velocities of Batch Settling Test for Sludge A	30
VI.	The Calculated Initial Settling Velocities of Batch Settling Test for Sludge B	32
VII.	The Results of Leaf Filter Test for Sludge A	41
VIII.	The Results of Shear Strength Test for Treated Scrubber Sludge	44
IX.	The Results of Shear Strength Test for Treated Scrubber Sludge	46
X.	X-ray Diffraction Data on Sludge with 6% Lime and 20% Fly Ash	57
XI.	X-ray Diffraction Data on Sludge with 6% Lime and 40% Fly Ash	58
XII.	X-ray Diffraction Data on Sludge with 6% Lime, 40% Fly Ash, and 15% SiO_2	59
XIII.	Buchner Funnel Test for Sludge A with Initial Solid Concentration at 17%	89
XIV.	Buchner Funnel Test for Sludge A with Initial Solid Concentration at 33%	90
XV.	Buchner Funnel Test for Sludge A (The Total Solid Concentration at 34%)	91

LIST OF FIGURES

FIGURE		PAGE
1.	Lime and Limestone Postcombustion Desulfurization System (22)	10
2.	The Apparatus Used for Settling Sludge Tests Consisting of a One Liter Graduated Cylinder and Stirring Bar	24
3.	Comparison of Batch Settling Characteristics for a Sludge (Sludge A) in Which 11% of Dry Weight Solids is Fly Ash--Three Total Solid Concentrations at 5%, 9%, and 17% . . .	29
4.	The Results of Batch Settling Test Results for Sludge B with the Addition of 0, 10, 30, and 60% of Fly Ash at Initial Solid Concentration of 9%	31
5.	The Results of Batch Settling Test for Sludge B with the Addition of 0, 10, 20, and 30% of Fly Ash at Initial Solid Concentration of 17%	34
6.	The Results of Buchner Funnel Test for Sludge A with the Addition of 0, 10, 20, 30, 50, 70, 90, and 150% Fly Ash at Initial Solid Concentration of 17%	36
7.	The Results of Buchner Funnel Test for Sludge A with the Addition of 5, 10, 20, 30, 50, 70, 90, and 150% Fly Ash at Initial Solid Concentration at 33%	37
8.	The Results of Buchner Funnel Test in Which the Slurry Solid Concentration Was Kept Constant at 34% of Solid . .	39
9.	The Comparison of Strength of Treated Scrubber Sludge at Curing Time of 6 Weeks	50
10.	The Comparison of Strength of Treated Scrubber Sludge at Curing Time of 6 Weeks	52
11.	Comparison of X-ray Diffraction Patterns of Sludge with 6% Lime and 20% Fly Ash	54
12.	Comparison of X-ray Diffraction Patterns of Sludge with 6% Lime and 40% Fly Ash	55
13.	Comparison of X-ray Diffraction Patterns of Sludge with 3.7% Lime, 44.1% Fly Ash, and 9.3% SiO_2	56
14.	Lime Scrubber Sludge Without Fly Ash (5,000 X)	63

FIGURE PAGE

15. Lime Scrubber Sludge With 90% of Fly Ash (2,000 X) 64

16. Crystalline Growth on Admixture of 6% Lime and 20% Fly Ash (2,000 X) 65

17. Crystalline Growth on Admixture of 6% Lime and 20% Fly Ash (10,000 X) 66

18. Crystalline Growth on Admixture of 6% Lime, 40% Fly Ash (2,000 X) 67

19. Crystalline Growth on Admixture of 6% Lime, 40% Fly Ash (5,000 X) 68

20. The EDXRA Spectrum of Protruding Crystal 69

21. The EDXRA Spectrum of Total Sludge Fixed 70

22. The EDXRA Spectrum of Plate-Like Crystal 72

23. The EDXRA Spectrum of Fly Ash 73

24. Sludge With No Additive 83

25. Sludge With 0% Lime and 20% Fly Ash 83

26. Sludge With 0% Lime and 40% Fly Ash 83

27. Sludge With 6% Lime and 20% Fly Ash 83

28. Sludge With 6% Lime and 40% Fly Ash 84

29. Sludge With 0% Lime and 20% Fly Ash 84

30. Sludge With 6% Lime, 40% Fly Ash, and 15% Silica 84

31. Sludge With 6% Lime, 20% Fly Ash, and 8% Silica 84

32. Sludge Without Any Additive 86

33. Sludge With 3% Lime and 0% Fly Ash 86

34. Sludge With 6% Lime and 0% Fly Ash 86

35. Sludge With 0% Lime and 40% Fly Ash 86

36. Sludge With 3% Lime and 10% Fly Ash 87

FIGURE PAGE

37. Sludge With 3% Lime and 20% Fly Ash 87

38. Sludge With 3% Lime and 40% Fly Ash 87

39. Sludge With 3% Lime and 60% Fly Ash 87

40. Sludge With 6% Lime and 40% Fly Ash 88

CHAPTER I

INTRODUCTION

To meet the Federal clean air standards (1), some power industries might need to control sulfur dioxide emissions from their steam plants. This is presently done through a variety of anti-pollution measures including installation of sulfur dioxide, coal washing, and use of low-sulfur coal. Wet scrubbing of the flue gas is one of the most proven means to meet the air quality standards. However, the large amount of waste materials produced by the lime, limestone, and double alkali flue gas desulfurization (FGD) systems could cause a solids disposal problem. Certain properties of the sludge produced by these processes must be improved before the sludge is suitable for disposal.

The chemical composition of sludges produced by the wet scrubbing systems, utilizing slurried lime or limestone as the absorbent, can vary considerably depending upon the scrubber operation, sulfur dioxide removal efficiencies, whether the fly ash is collected before or during scrubber operation, and the difference in the type of absorbent used. The major constituents of the solid waste in the lime scrubbing process are $CaSO_4$, $CaSO_4 \cdot 2H_2O$, $CaSO_3 1/2H_2O$, $CaCO_3$ and $Ca(OH)_2$.

The engineering properties of this material have been explored by many investigators (2,3,4). Reports indicate it has very low strength and strong thixotropic characteristics which make landfill

disposal difficult. A material with thixotropic characteristics will demonstrate a porperty of certain gels which liquify when subjected to vibratory force and then solidify again when left standing for a period of time. It allows the double layer of water on particles to reorient itself through water dipoles which link particles together. In addition to the poor physical characteristics of the waste material, the presence of many soluble materials such as chloride, sulfate, boron, mercury, and selenium in the sludge also create a leaching problem (5) which may contaminate the ground and surface water.

In view of these problems, the treatment of these waste materials before disposal to produce a stable product is necessary. Several commercial and government organizations (6) have conducted studies which introduce additives into this sludge to produce more stable materials.

The Tennessee Valley Authority (TVA) is also involved in intensive research toward treatment of scrubber sludge produced from its own coal-fired generating plants, where wet scrubbing systems are used to reduce the emission of sulfur dioxide. The alternatives for treatment involve either a commercial treatment process or utilization of its own fly ash as an additive to the sulfur dioxide sludge. The present study was carried out in order to provide the additional information about the feasibility of using lime and fly ash to stablize the flue gas desulfurization sludge from TVA's Shawnee pilot scale scrubber. The settling characteristics, filterability of the sludge, and structural properties have been investigated in order to evaluate the optimal technique for handling the sludge.

The increase in shear strength of these admixtures in relation to the treatment time will be developed to search for the optimal combination of additives for sludge treatment.

CHAPTER II

REVIEW OF LITERATURE

The history of man-made air pollution can be traced back to the Industrial Revolution which resulted in the the development of heavy industry and mass production. Pollutants such as particulate matter and SO_2 have been studied and found to be a health hazard to both plants and animals. Combustion of high-sulfur oil and coal is the main source of these pollutants in the atmosphere. The increasing demand for electric energy, most commonly provided by fossil fuel plants, will cause a tremendous amount of particulate matter and sulfur dioxide to be emitted from power plant stacks (7).

A great deal of research has been carried out over the years to study the effects of these air-borne pollutants such as particulate matter and sulfur dioxide. The adverse effects of the former result largely from the toxic nature of a small portion of the total particulate matter emitted. However, large amounts of non-toxic particulate matter can also do injury, by affecting the cleaning mechanism which protects the lungs from invasion by foreign particles. The effects of sulfur dioxide on human health also involve irritation of the respiratory system. Studies carried out under the CHESS (Community Health and Environmental Surveillance System) have shown that ill effects result from extended exposure to even low levels of SO_2 (9). Damages to plant leaves from sulfur dioxide and its associated sulfuric acid aerosol has also been reported (10,11).

In December 1970 Congress passed the Clean Air Act. In April 1971, EPA (Environmental Protection Agency) issued the National Ambient Air Quality Standards (1). In order to comply with these regulations by mid 1975, existing power plants and other industries have been involved in investigating available methods which will result in decreasing sulfur dioxide emissions (6).

Several techniques are currently available to control sulfur dioxide emissions. Some of these techniques are as follows:

(A) Alternatives to Fossil Fuels:

Nuclear power is an alternate way to produce energy. The advantage of a nuclear generating plant is that it is cheaper to use as far as the fuel used is concerned at a large scale facility, because there are no pollutants, namely particulate matter and sulfur dioxides, emitted into the atmosphere. However, there are several prerequisite needs to be considered such as the assurance of safety to operate the plant, the methods required to dispose of the high-level radioactive wastes, and most of all, the reduction of thermal pollution of the natural water supplies (12).

In addition to those drawbacks, the nuclear power industry cannot meet the current electricity demand. Therefore, it is unreasonable not to use the abundant coal resources in the United States.

(B) Coal Desulfurization:

Coal desulfurization has been studied by researchers for many years. The main "pollutants" in coal are sulfur, pyritic sulfur, organic sulfur and inorganic sulfate, and certain trace elements (13).

Coal washing is the least expensive physical method developed to remove the pyritic sulfur. In the washing process the coal is finely ground and then washed with water. Because of the higher density of pyritic sulfur than coal the separation can be achieved. However, the final product can only reduce 40 to 50 percent of sulfur dioxide emissions. Therefore, this process is only suitable for cleaning coal with low sulfur contents.

(C) Coal Gasification:

Utilization of coal gasification processes to produce a high-Btu synthetic natural gas (SNG), which consists primarily of carbon monoxide, hydrogen, and nitrogen from air, with non-pollutants in it for energy production, should become feasible in the near future (14). This process uses a mixture of oxygen and steam which is introduced at the bottom of the gasification unit (gasifier) to heat the coal in the gasifier to produce the SNG. However, the cost of this process will be the major drawback for commercial production.

(D) Use of low sulfur fuel:

Low sulfur fuel can be used to reduce the emission of sulfur dioxide. However, only a few Western states produce coal with low sulfur content, so this is not a practical solution for most of the country. This low-sulfur Western coal also contains less Btu, and is, therefore, less economical to use.

(E) Tall-stack Dispersion:

By using a very tall stack, a power plant can disperse its pollutants over a much wider area, diluting them to such a degree as would meet the air quality standards when the pollutants reached ground

level (15,16). This does nothing, however, to reduce the amount of pollutants emitted into the atmosphere, so the basic problem remains unsolved.

(F) Desulfurization of Stack Gas:

There are many techniques that have been developed to remove SO_2 from stack gas. The processes can be divided into two major categories (17):

(1) Recovery of SO_2 in a useful form, and

(2) Formation of products intended for discard as a solid waste.

In both categories chemicals are used as an absorbent which will react with sulfur dioxide. It is desirable to convert waste gas by product to a useful form with commercial value. However, the average annual operating cost for the recovery process will be higher than for the throw-away process (18). The marketing problem is also a drawback in the United States (19), since a relatively large amount of gypsum is available. For the regenerable process unless there is a considerable economic advantage, the throwaway process still looks more favorable. The disadvantage of throwaway processes is that it generates a huge amount of solid waste which has unstable physical and chemical properties. The disposal of this sludge becomes a major task for the operators who use the throwaway processes as a means to control sulfur dioxide emissions.

Since the objective of this paper is to deal with the treatment of this solid waste it is worthwhile discussing some of the throwaway processes. From Table I the most popular scrubber system currently available is direct lime/limestone scrubbers (20), therefore, the basic

TABLE I

UNIT OPERATING, UNDER CONSTRUCTION OR PLANNED
AS OF FEBRUARY 1975 (20)

Type	Units		Generating Capacity	
	Number	%	MW	%
Lime/Limestone (With Sludge)	67	66.3	25,319	72.3
Double Alkali (With Gypsum)	3	3.0	75	0.2
Sodium Carbonate (Open Loop)	4	4.0	500	1.4
Regenerative	11	10.9	1,376	3.9
Not Selected	16	15.8	7,754	22.2
Total	101	100.0	35,024	100.0

principle and chemistry of the scrubber system is to be discussed as follows.

The concept of lime/limestone scrubber systems is generated from the mechanism of mass-transfer phenomena in chemical engineering. The absorption process of sulfur dioxide from a moving gas stream into an appropriate scrubber liquid stream is very complex. According to Fick's law the rate of transfer of sulfur dioxide depends upon the concentration of SO_2 in both the gas and liquid phases, the temperature, and the contacted area between the gas and liquid phases (21). In addition, the rate of chemical reaction between sulfur dioxide and the absorbent (which is lime/ or limestone in this case) in the scrubber is also involved.

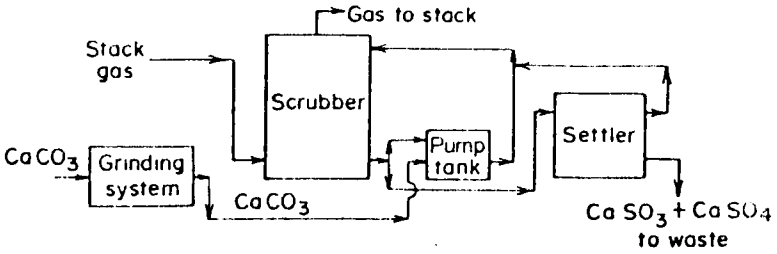
The chemistry of lime/limestone systems is quite complex. Figure 1 summarizes the reaction steps involved in the lime/limestone wet scrubbing system and the scheme of the process.

The gaseous SO_2 is transferred from the flue gas stream into the liquid phase, where it is partially ionized to form bisulfite carbon dioxide which undergoes a reaction similar to that of the SO_2, but at a much lower rate.

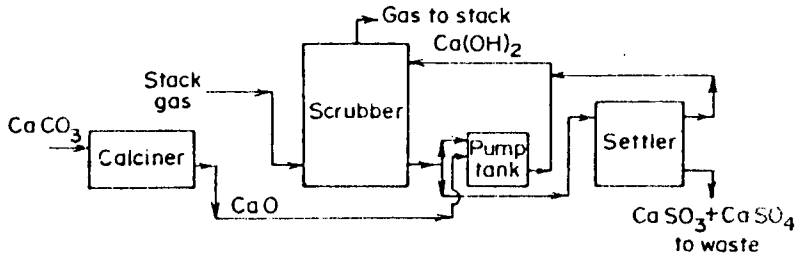
The dissociation of calcium hydroxide provides the calcium ions to react with bisulfite ions to form calcium sulfite. Partial oxidation produces some calcium sulfate. Both calcium sulfate and calcium sulfite have very low solubilities in water, therefore, they are crystallized out of the solution.

The advantage of the lime (CaO) scrubber system over the limestone ($CaCO_3$) system is the higher absorption efficiency. When the lime is added to the system, $Ca(OH)_2$ is produced through hydration of the CaO, thus producing a more soluble compound than limestone. In addition, lime ($Ca(OH)_2$) is more porous than natural $CaCO_3$. Consequently a greater surfact area is available, so more calcium ions are dissolved in the aqueous phase. This becomes a very important factor when the venturi type scrubber is utilized because of the relatively short gas-liquid contact time. In addition, because of the higher reactivity of lime to sulfur dioxide absorption, less absorbent is used in the scrubber. Thus, the volume of sludge produced is reduced. Another advantage in using lime is that it is easier to handle and transport than limestone.

LIMESTONE SCRUBBER SYSTEM



LIME SCRUBBER SYSTEM



CHEMISTRY OF LIME AND LIMESTONE SCRUBBING

(1) $CaO + H_2O \longrightarrow Ca(OH)_2$

(2) $Ca(OH)_2 + CO_2 \longrightarrow CaCo_3 + H_2O$

(3) $CaCO_3 + CO_2 + H_2O \longrightarrow Ca(HCO_3)_2$

(4) $Ca(HCO_3)_2 + SO_2 + H_2O \longrightarrow CaSO_3 \cdot 2H_2O\downarrow + 2CO_2$

(5) $CaSO_3 \cdot 2H_2O + 0.5\,O_2 \longrightarrow CaSO_4 \cdot 2H_2O\downarrow$

Figure 1. Lime and Limestone Postcombustion Desulfurization System (22).

The major drawback for the lime scrubber system is the cost of calcining limestone to lime, which usually adds about $10/ton (of lime) to the raw material cost (17).

From previous discussions, the lime/limestone scrubber system seems to offer a fairly effective way to control the sulfur dioxide emissions. However, the handling problem for a large amount of sludge (22) with poor physical and chemical properties, which will be discussed later, and disposing of it in an environmentally acceptable state, will be another main task for scrubber users.

The chemical constituents and physical properties of lime-limestone sludges have been studied by many investigators (2,3,4). A typical chemical composition of limestone sludge includes about 38 percent limestone, 21 percent calcium sulfite, 12 percent calcium sulfate, and 28 percent fly ash (23). In the lime scrubbing system the contents of calcium carbonate is obviously decreased and the percent of calcium sulfite is increased (5).

Changes in the chemical composition may result from the use of a different absorbent, variations in the sulfur and ash content of the fuel, differences in the operating process, variation in stoichiometric ratio of calcium to SO_2 removed, and SO_2 removal efficiency required. The fly ash content of sludge is quite different when the venturi-type of scrubber is used, compared to when a scrubber with an electrostatic precipitator is used. In the latter system, the ash is collected before the flue gas enters the scrubbing system. The change in fly ash content changes the chemical constituents of the sludge. Because there are many chemical constituents in the mixture of fly ash and scrubber

sludge, the discharge of scrubber liquid and the leaching characteristics of this mixture may also produce surface or ground water pollution problems.

In assessing the potential environmental impact resulting from the disposal of lime and limestone scrubber sludge, Rossoff et al (5) indicated that at least one of the following trace metals in the sludge liquids exceeded the EPA water quality criteria (24): arsenic, boron, cadmium, chronium, lead, mercury, and selenium. The other major soluble species which exceeded the criteria were chloride, sulfate, and total dissolved solids. Attenuation of chemical species by soil is widely accepted in many land disposal practices; however, attenuation of boron, chloride, and selenium is known to be ineffective. Therefore, the concentrations of these three species strongly indicate that environmentally sound techniques such as lined pond or chemical fixation are needed for scrubber sludge disposal (5).

The physical properties of scrubber sludges depend mainly on the percentage of moisture in the sludges and the constituents in the sludges. The major compounds in lime and limestone scrubber sludge as mentioned previously are calcium sulfate, calcium sulfite, calcium carbonate, and fly ash. When a sludge is allowed to settle, the final solid content for sulfate sludge is about 60 to 65 percent, and for sulfite sludge it is about 35 to 45 percent (5). One possibility for decreasing the moisture content of sludges is the vacuum filtration process. This technique can increase the solid content up to 75 to 80 percent for sulfate sludges and to 50 to 55 percent solids for sulfite sludges (5). The reason for the resistance to dewatering of the sulfite

sludge is its thin platelet-like crystal structure. Water is trapped between the crystalline plates and is not easily removed by dewatering techniques, such as vacuum filtration and centrifugation. During the stages of settling, the thin plates settle to a loose bulky gel-like structure. As the free water in the sludge is removed by evaporation or physical dewatering techniques, the sludge solid looks fairly dry and possesses some strength. However, if some vibrational force is applied to the apparently dry sludge cake, it has been found to release all entrapped water and become fluidized. This is characterized as "Thixotropic." This characteristic has been seen even when the sludge has been dewatered up to 70 percent solids (23).

In contrast, the sulfate sludge crystallized in a "blocky" state which will not trap water. Therefore, some attempts have been made to oxidize the sulfite to sulfate (25). This goal was achieved by mixing equal quantities of flue gas and ambient air in the scrubber system, after which 90 percent of the oxidation of sulfite to sulfate was reached. The settling rate of this resulting sulfate sludge was improved about six fold, however, the final compaction was not improved and the thixotropic characteristic of the sludge still existed which lead to the conclusion that the calcium sulfite was not the only cause of the poor compaction and thixotropic characteristic. Calcium carbonate was also involved in the formation of this poor sludge characteristic (24).

From the previous discussions, it is noted that the disposal of scrubber sludges in an environmentally acceptable condition is one of the biggest concerns for scrubber users. The techniques for the sludge

treatment have been developed and tested by Federal and private parties. The concepts behind those stabilization processes are to try to use some additives mixed with sludges. Hopefully, the pozzolanic reaction would occur and produce cemetitious material between particles in the mixture. As a result, the physical structures of sludges would be improved and the soluble chemical species would be bonded to reduce the leaching problems. The currently available stabilization processes will be reviewed in the following sections:

(A) Chicago Fly Ash Company Process (26)

This process was engineered and furnished by Chicago Fly Ash Company which utilizes the formulation developed by Dr. R. L. Berger of the University of Illionis.

The sludge was removed from the settling pond to an open hopper equipped with a drag chain such that the sludges were conveyed to a waiting Redimix truck. Then the desired amount of fly ash and lime were dumped into the truck. The mixture of sludge, lime and fly ash was then mixed throughout before dumping into a disposal basin. The data on the improvement of the sludge was not available. However, this process does not seem to be feasible for a large-scale scrubber system unless there is an efficient way to reduce the capital and operating cost. The estimated annual operating cost in 1973 was $17/ton dry sludge.

(B) Chemfix Process

The Chemfix process involves the reaction of tetrahedral structured silicon atoms with the surrounding polyvalent metal ions through the charged side groups (which in this case are oxygens). This forms a

cross-linked, 3-dimensional polymer matrix which reportedly contains properties very similar to many soils (27).

Laboratory leachate studies on TVA's Shawnee flue gas desulfurization sludge treated with the Chemfix process have shown that most of the heavy metals are tied up in the fixation product, but that sulfate and chloride still present a problem to ground water contamination (28). The cost for this process at a solid content of 50 percent has been reported at $4.50-5.00/ton.

(C) Calcilox Process

Dravo Corporation has developed a sludge stabilizer referred to as Calcilox. The strength-time relationships have been observed on those admixtures (29). Some of the stabilized sludge samples were found to take a longer time to reach maximum strength than others. The curing period was typically 15-30 days, depending on the quantity of additives, solid content of sludge leaving the thickner, curing temperature, and slurry pH.

Several utility companies are using the Dravo fixation technique to treat their sludges (23). Duquesne Light's Phillips Station found some problems in using Calcilox, because of difficulty in determining the amount of additive and length of curing time necessary for different sludges. Dravo reported the process costs about $2.00-4.00/ton of coal burned or $1.50-4.50/ton of raw sludge (28).

(D) Poz-O-Tec R® Process

The Poz-O-Tec R® procedure which was developed by IU Conversion System, Inc. (IUCS) scrubber sludge is based on the reaction between

alumina/silica in the fly ash, the lime compounds, and the sulfur oxide salts to form a cementitious end product which increased the strength of the waste material and encapsulated potentially toxic materials and locked-up soluble ions of many trace elements to prevent a leaching problem. The reports from the IUCS laboratory indicate the reduction of permeability from 10^{-4} to 10^{-6} cm/sec during the first month of curing (30). The cost of the process is about $2.00-4.00/ton of dry fixed solid or $.25-.80/ton of coal burned (23).

Some of these available processes seem to produce promising results, although some problems with the treated sludge remain. However, the cost for commercial treatment will be the major drawback.

(E) Other Alternatives

To find a more economic means to treat sludge to an environmentally sound condition, lime used as stabilizer comes into the picture. The combination of dry fly ash, quicklime and Portland cement has been used to mix with limestone sludges which were previously dewatered to 65 percent solid produced from the Ontario Hydro wet limestone SO_2 pilot scrubber. These mixed sludges were tested to observe their compressive strength in relation to curing time, and their permeability. The results of these tests indicated that the addition of fly ash alone did not provide the cementitious reaction; however, strength increases were shown with a small amount of lime addition.

Utilization of CaO and $Ca(OH)_2$ for the chemical stabilization of soils has gained increasing usage since the late 1940's. Most soils contain large quantities of silica, feldspar, or colloidal clay minerals (31). These compounds are inherently acidic in nature. Some

of the silica and aluminosilicate are in a highly hydrous state on the soil particles surface. When lime (CaO and $Ca(OH)_2$) is mixed with soil the calcium silicate hydrates and aluminate hydrated are produced through the pozzolanic reaction. The ultimate reaction product is calcium silicate (or aluminate) gel which is a cementitious material.

To apply this concept to improve the characteristics of scrubber sludge, a natural pozzolan such as some fly ash must be introduced to produce pozzolanic reactions. Since hydrous silica and alumina are major constituents in fly ash, reactions between lime and these pozzolanic compounds can be expected to occur.

In Minnick's study (32) cementitious gel formations were found to result from the reactions between fly ash and hydrate lime. The X-ray diffractometry was used to identify the species in the curing fly ash-lime mixture, although identification of some specied were difficult due to their non-crystallized form. However, the identification of calcium silicate hydrate, tobermite, and tetra-calcium aluminate, as products of reaction were interpreted by X-ray diffraction patterns. The formation of ettringite and calcium sulfoaluminate hydrate were also reported (32).

The formation of calcium sulfoaluminate hydrate is reported (33) in two forms. When the sulfate level in a lime-fly ash-sulfate system is greater than 5 percent the low sulfate form monosulfoaluminate is produced. Otherwise the high sulfate form, ettringite, will be produced with the gypsum level higher than 15 percent.

Utilization of lime and fly ash as additives for sludge treatment have been reported by Ontario Hydro Stabilization Studies (23).

The addition of sewage sludge, lime, fly ash, cement, and the combination of cement and fly ash to the sludge produced from double alkali scrubbing system, was reported by Scherer (4). The author indicated that the satisfying strength of the waste material can be obtained by adding Portland cement or a cement-fly ash mixture to the waste material.

Mixing the waste material with soil was also investigated (34). The strength of the soil scrubber solids mixture increased by a factor of 20.

CHAPTER III

EXPERIMENTAL DESIGN AND PROCEDURES

Two types of sludge samples were provided by TVA Shawnee pilot plant utilizing a lime scrubbing system as a sulfur dioxide removal emission device. The composition of sludge A is shown on Table II. This sludge was collected from the underflow of a clarifier. Only a very small amount of fly ash is in this sludge because the fly ash collector was used prior to the scrubber as flue gas entered the system. The composition of sludge B is shown on Table III. This was collected from the underflow of the clarifier of a venturi scrubber. It contains about 33 percent fly ash by dry weight of the sludge. This is due to the fact that the system is designed for collecting fly ash and scrubbing SO_2 in the flue gas at the same time.

The fly ash was obtained from the TVA Widows Creek Steam Plant. It is collected from the hoppers of electrostatic precipitators. The chemical constituents and physical properties of this ash are presented in Table IV.

Hydrated lime was bought from a hardware store and it contains 68 percent of CaO.

Sample Preparation

Two sets of treated sludge B samples were prepared for direct shear strength test. One set of samples was prepared in nine 33" x 11.5" x 11.5" plastic containers. The slurry from the TVA Shawnee power plant scrubber was weighed and then the desired amount of additives

TABLE II

THE COMPOSITION OF SCRUBBER SLUDGE A

Parameter	Value
Liquids	
Calcium (Ca), mg/l	876
Magnesium (Mg), mg/l	3019
Sodium (Na), mg/l	4.5
Potassium (K), mg/l	8
Chloride (Cl), mg/l	3722
Sulfite (SO_3), mg/l	212
Sulfate (SO_4) mg/l	7655
pH, unit	6.96
Solids (Dry)	
Calcium (As CaO), % Weight	38.53
Carbonate (CO_2), % Weight	1.48
Sulfite (SO_2), % Weight	37.71
Sulfate (SO_3), % Weight	9.56
Suspended Solids in Slurry, % Weight	4.28
Solid $CaCO_3$ in Slurry, % Weight	0.14
Fly Ash in Slurry, % Weight	0.55

TABLE III

THE COMPOSITION OF SCRUBBER SLUDGE B

Parameter	Value
Liquids	
Calcium (Ca), mg/l	1660
Magnesium (Mg), mg/l	596
Sodium (Na), mg/l	17
Potassium (K), mg/l	43
Chloride (Cl), mg/l	3722
Sulfite (SO_3), mg/l	135
Sulfate (SO_4), mg/l	606
pH, unit	7.95
Solids (Dry)	
Calcium (As CaO), % Weight	25.11
Carbonate (CO_2), % Weight	2.02
Sulfite (SO_2), % Weight	22.3
Sulfate (SO_3), % Weight	1.27
Suspended Solids in Slurry, % Weight	8.2
Solid $CaCO_3$ in Slurry, % Weight	0.37
Fly Ash in Slurry, % Weight	2.55

TABLE IV

CHARACTERISTICS OF SEMI-ANNUAL COMPOSITE FLY ASH FROM TVA WIDOWS CREEK STEAM PLANT

Parameter	Value
Physical Properties	
Specific Gravity	2.31-2.49
Blaine Fineness, cm^2/cm^3	2972-5718
% Passing 325 Sieve (Wet)	50.4-82.5
Loss on Ignition, %	1.9-3.15
Pozz. Index with Cement, %	56.0-81.0
Pozz. Index with Lime, psi	677-1077
Water Requirement, %	96.7-102.5
Multiple Factor, %	33-124
Chemical Properties	
Silica (SiO_2), % 42.08-48.57	42.08-48.57
Calcium Oxide (CaO), %	4.0-6.0
Ferric Oxide (Fe_2O_3), %	16.17-17.72
Alumina (Al_2O_3), %	24.15-29.46
Magnesium Oxide (MgO), %	0.98-1.5
Sulfur Trioxide (SO_3), %	0.7-1.31
Moisture Content, %	0.78-0.99

were mixed into this slurry. The well-mixed slurry was poured into one of the nine plastic containers. The clarified supernatant was drained out as the suspended solids settled to the bottom of the container. The settled solids were allowed to air dry at room temperature. At time intervals of two, four, and six weeks, undisturbed samples were extracted for direct shear testing. Undisturbed samples were obtained by means of a 3.0-inch diameter sampling tube.

The second set of samples was prepared under a controlled environment to keep the moisture content at a constant level. The settled scrubber sludge was mixed with selected percentages of lime, fly ash, and SiO_2 additives. The samples were prepared in a mold of three inches in diameter and 2.125 inches in height. The sludge samples, with or without additive at the weight of 16 ounces were compacted when the moisture content of the admixture reached 30 percent. Compaction effort was varied to obtain a constant unit dry weight. Prepared samples were sealed in Saran Wrap and waxed to prevent the moisture content from changing during curing. At time intervals of one, two, and four weeks, specimens were subjected to direct shear testing.

Settling Test

The possibility of using fly ash to improve the settling rate of the sludge was investigated. The settling characteristic of a sludge was tested in a 1000 ml graduated cylinder (Figure 2). A stirrer equipped with a 1-rpm motor was used to prevent the suspended sludge particles from adhering to the cylinder wall. The sludge was mixed, with or without additives, then the mixture was poured into a 1000-ml

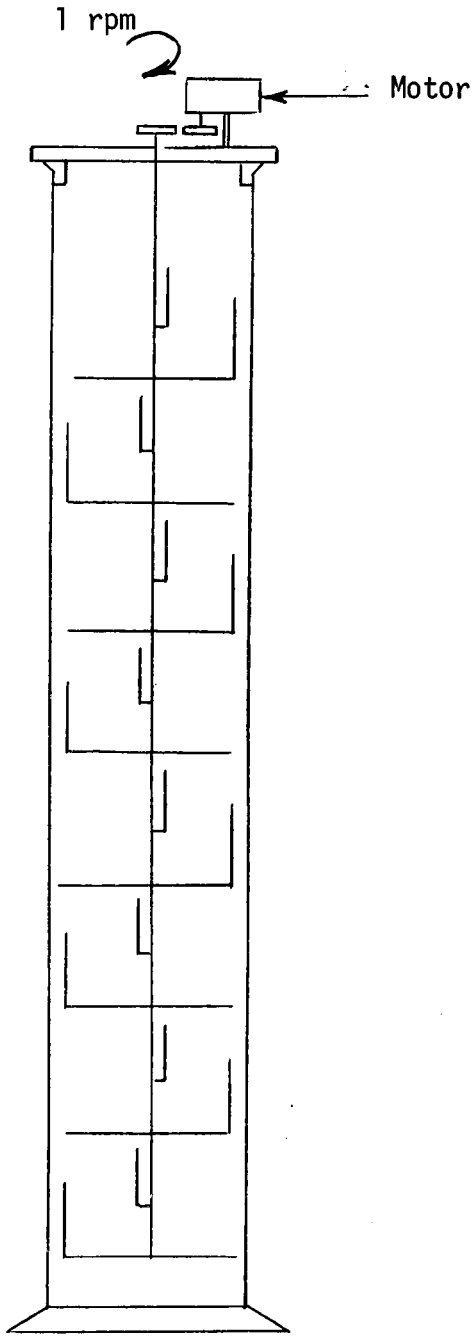


Figure 2. The Apparatus Used for Settling Sludge Tests Consisting of a One Liter Graduated Cylinder and Stirring Bar.

cylinder. After starting the motor, the interfacial height and clarified supernatant was recorded at different time periods.

A series of runs was performed on sludge A at solid concentrations of 5 percent, 9 percent, and 17 percent. Other runs were performed on sludge B, with initial solid concentrations of 17 percent and 9 percent, with the addition of fly ash at concentrations of 0, 10, 20, 30, and 60 percent.

Vacuum Filtration Test

To evaluate the improvement of filterbility and filtration yield by adding fly ash into the sludge, the vacuum filtration test was carried out. The specific resistance and compressibility of the sludge A were measured by the Buchner funnel and a leaf filter test. A 150 ml of sludge sample, with or without additives, was poured into a Buchner funnel which was connected to a constant vacuum reservoir (15 inches of Hg). The funnel was 3.7 inches in diameter and 18 inches in depth. Number one Whatman filter paper was used.

Leaf filter tests were conducted using a 0.1-square-foot Emico filter with a Polymax #B (Polypropylene) filter media. (Polymax #B (Polypropylene) Style No. 224-001-01 National Filter Media, New Haven, Connecticut). The detail procedure for both test were referenced from Environmental Engineering Unit Operation and Unit Processes Laboratory Manual (35).

Direct Shear Strength Test

Direct shear test was used to measure the strength change with time of the treated sludge. Because of the paste-like nature of this

waste material, it is very difficult to mold the specimen using the standard method for testing. Therefore, the direct shear test was chosed to measure the strength. The strength of each admixture is measured by the Karol-Warner direct shear machine. Direct shear tests were conducted at a constant strain rate of 0.05 inches per minute. Normal stresses applied to test specimens were 2 pounds per square inch and 6.1 pounds per square inch.

The stress-strain curve is developed for each admixture at several different curing times. Changes in the shear stress (ordinate) as the different normal load (abscissa) applied are also plotted for each specimen at the different curing times. The cohesion of the tested specimen is expressed by the intercept of the line at the ordinate, and the angle of friction is expressed by the slope of this line. The comparison of the difference in cohesion and the angle of friction will indicate the changes in the strength characteristic.

X-ray Diffractometry

The identification of species in the treated sludge was performed by X-ray diffractometry. After the direct shear strength of a specimen was measured by the direct shear machine, a piece of this treated material was collected and dried at a temperature of 38°C. The dried sample was ground and screened through a 325-mesh sieve. The powdered sample was placed on a sample holder, then a Norelco 12045 diffractometer with a copper target tube was used to X-ray the sample. The

diffraction charts for each sample were run from 5° through 60°. The ASTM data card was used to interpret the X-ray patterns.

Scanning Electron Microscopic (SEM) Photo

The morphology of the sludge mixture was observed by scanning electron microscope. Samples of 10-week-old treated waste material were cut into a size of 1/8" x 1/8" x 1/8". Care was applied as the cutting proceded to keep the surfact intact so the growth of any crystalline could be seen under the SEM. The specimen was glued on an aluminum stool and some silver paint was applied aroung the bottom of the sample to provide better contact between sample and aluminum stool. Then a vacuum (less than 10^{-5} torr) was applied to dry the sample.

The dried samples were placed under the SEM. The image of the sample particles was shown on a screen at any desired magnification up to 20,000 times and pictures were taken.

The energy dispersive X-ray analyzer (EDXRA) unit was also used to take the EDXRA spectrum, which reveals the constituent elements in the sludge sample. Where an interesting spot on a sample particle was shown on the screen, such as the crystalline growth, the fly ash particles (sphere), the sulfite crystal (plate-like), the EDXRA spectrum were also taken to detect the constituent of this particulate spot.

CHAPTER IV

RESULTS AND DISCUSSION

Batch Settling Study

For the batch settling experiment, interfacial height of the slurry is plotted against time to demonstrate the settling characteristic of each slurry. Figure 3 is a plot of the settling characteristic of sludge A, which only has 11 percent fly ash incorporated into the dry weight solids, at three different solid concentrations in slurry. Initial settling velocities were calculated from the initial slope calculated in Figure 3. The results are summarized in Table V which indicates the settling velocity increases as the solid concentration in slurry decreases.

For sludge B in which 33 percent of fly ash was incorporated into the dry weight solids, the batch settling rates of scrubber slurry were studied at two solid concentrations of 9 and 17 percent. These are the slurry concentrations at the scrubber effluent and clarifier underflow of Shawnee venturi scrubbing facility.

The settling characteristics of sludge B with initial solid concentrations of 9 percent, then with the addition of fly ash at 0. 10, 20, 30, and 60 percent by dry weight of sludge is observed in Figure 4. The calculated initial settling velocity was tabulated in Table VI, which indicated that no significant improvement in settling rate resulted from the addition of fly ash.

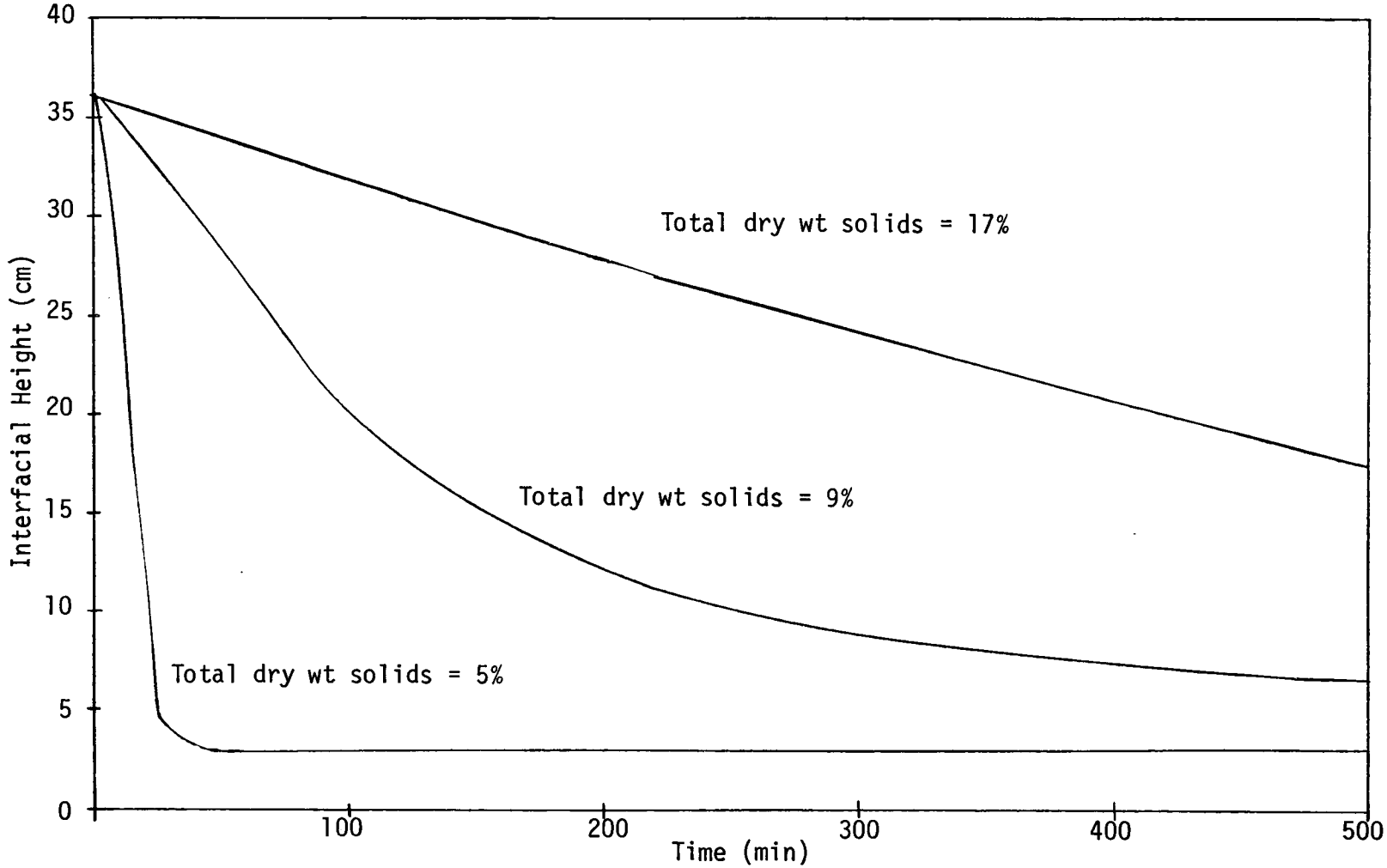


Figure 3. Comparison of batch settling characteristics for a sludge (sludge A) in which 11% of dry weight solids is fly ash--three total solid concentrations at 5%, 9%, and 17%.

TABLE V

THE CALCULATED INITIAL SETTLING VELOCITIES OF BATCH SETTLING TEST FOR SLUDGE A

Sludge Concentration (% by weight)	Initial Settling Velocity (cm/hr)
17	0.24
9	9.93
4	76.97

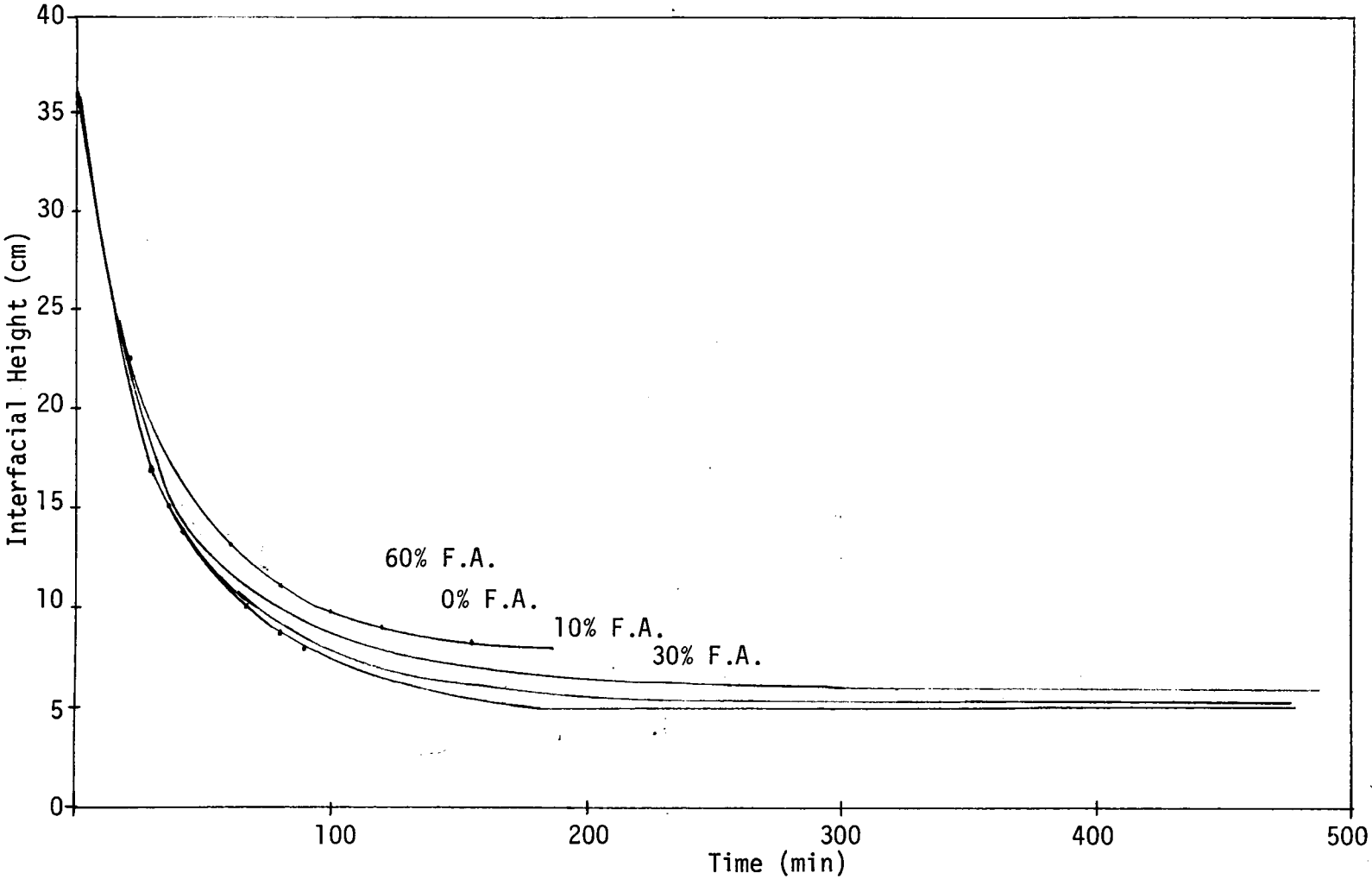


Figure 4. The results of batch settling test results for sludge B with the addition of 0, 10, 30, and 60% of fly ash at initial solid concentration of 9%.

TABLE VI

THE CALCULATED INITIAL SETTLING VELOCITIES
OF BATCH SETTLING TEST FOR SLUDGE B

Fly Ash Added % By Dry Wt. of Sludge	Slurry Initial Concentration at 9% Solid Vi (cm/hr)	Initial Slurry Concentration at 17% Solid Initial Settling Velocity V_i, (cm/hr)
0	43.77	6.1
10	42.5	7.3
20	40.4	8.8
30	39.64	7.2
60	36.1	7.4

The results of the batch settling experiment for the addition of fly ash at 0, 10, 20, 30, and 60 percent sludge dry weight into the slurry B with initial solid concentration of 17 percent is seen in Figure 5. The calculated initial settling velocities are listed in Table VI. The settling rate had a little improvement when 20 percent of fly ash was added into the slurry which was 17 percent solid. However, this improvement is not significant for engineering design.

In summary, there was no significant improvement in the settling rate brought about by the addition of fly ash. However, the comparison between Table V and Table VI indicated the sludge which incorporated 31 percent of fly ash (sludge B) into a dry weight solid has a higher settling rate than the sludge with 11 percent (sludge A) fly ash content.

Buchner Funnel Test

The Buchner funnel tests are performed on samples prepared from sludge A with an initial solid concentration of 17 percent and 33 percent. The filtrate volumes, V, collected in time, t, were plotted as t/V versus V. If the value of the slope of the line passed through the data points is b, then the specific resistance, r, can be expressed as:

$$r = \frac{2PA^2b}{\mu w} \qquad (1)$$

where P is the applied vacuum, A the area of filter medium, μ the viscosity of the filtrate, and w the weight of cake solid per unit volume of filtrate.

There are several factors which will affect the measurement from the Buchner funnel test. The compressibility of the cake is one

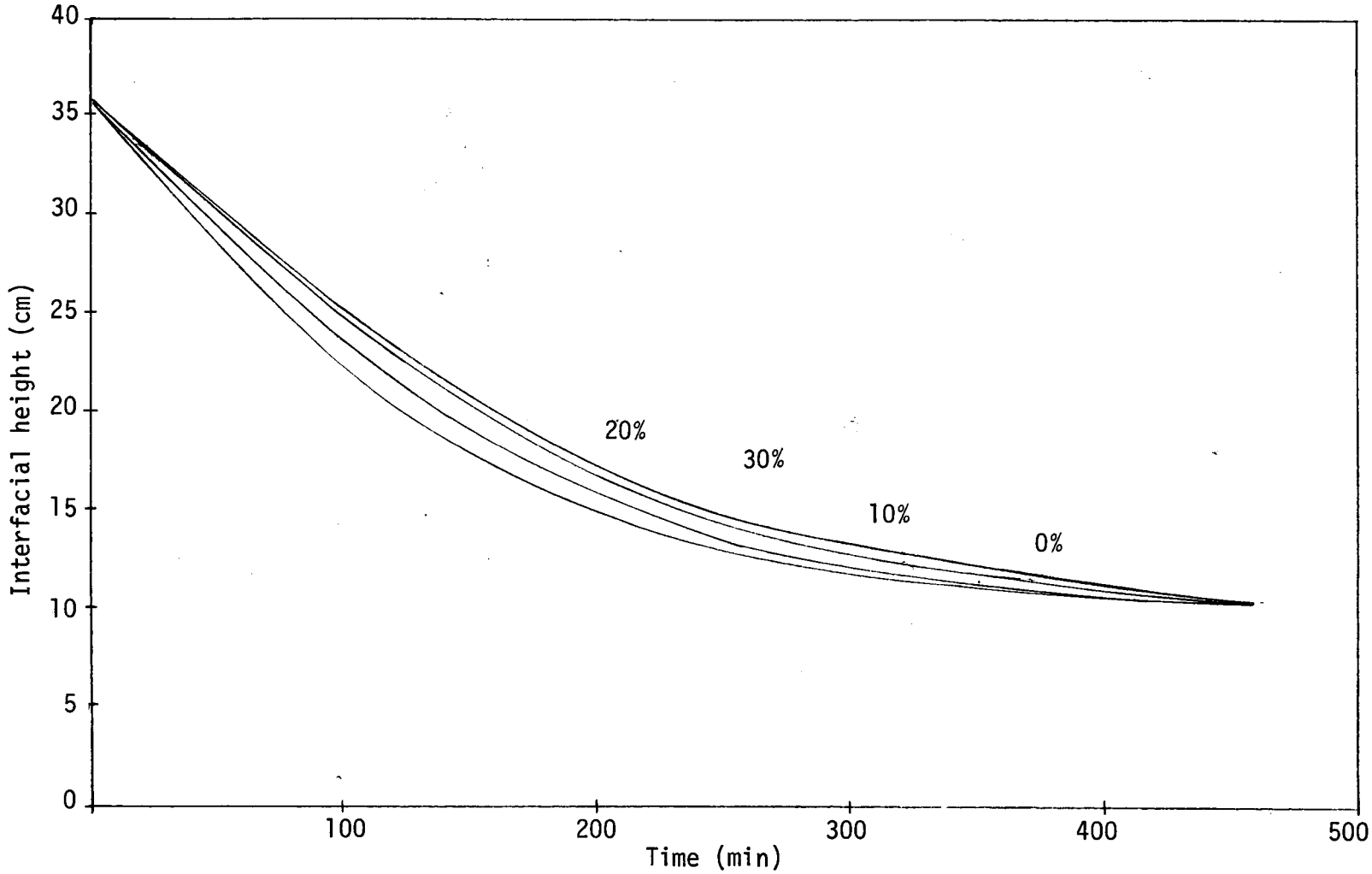


Figure 5. The results of batch settling test for sludge B with the addition of 0, 10, 20, and 30% of fly ash at initial solid concentration of 17%.

of the main concerns. If a sludge is compressible--as is the case in most sludges--then the sludge cake tends to pack in a more dense manner, which increases the pressure and results in greater specific resistance. Temperature also affects the specific resistance of sludge by varying the viscosity of the filtrate. Therefore, care must be exercised if the design is to be based on data collected from the Buchner funnel test.

The results of the Buchner funnel test for Sludge A with the addition of 10, 20, 30, 50, 70, 90, and 150 percent fly ash at initial solid concentration of 17 percent are presented in Figure 6 which indicated the sludge with the original 11 percent fly ash content (sludge A) demonstrates a very high resistance to dewatering. As the amount of fly ash added into sludge increases, the resistance to dewatering decreases. The optimal filterability of the sludge with an initial solid concentration of 17 percent was reached when the addition of fly ash was 90 percent by dry weight of sludge. However, if the addition of fly ash continues to increase, the resistance of sludge to dewatering increases again.

For the sludge with initial concentration of 33 percent (sludge B), the addition of 50 percent of fly ash seems to have the lowest resistance to dewater (Figure 7).

Upon using the quation (1), care must be exercised because there are two variables in one equation, if the applied pressure P and surface area of filter media, and the viscosity of the filtrate were kept constant. The two variables are b, the slope of t/V versus V, and w, the weight of cake solid per unit volume filtrate. The

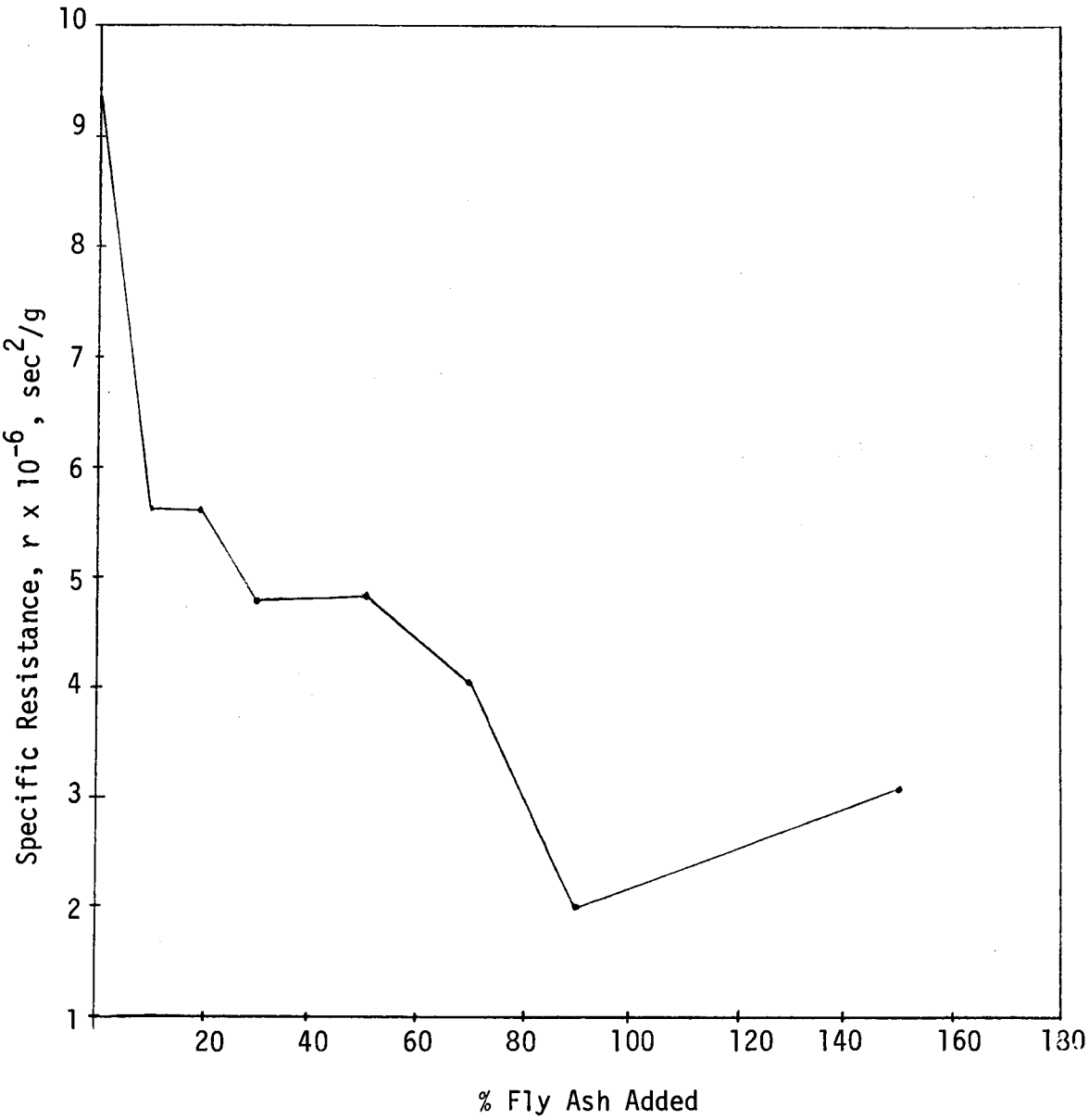


Figure 6. The results of Buchner Funnel Test for sludge A with the addition of 0, 10, 20, 30, 50, 70, 90, and 150% fly ash at initial solid concentration of 17%.

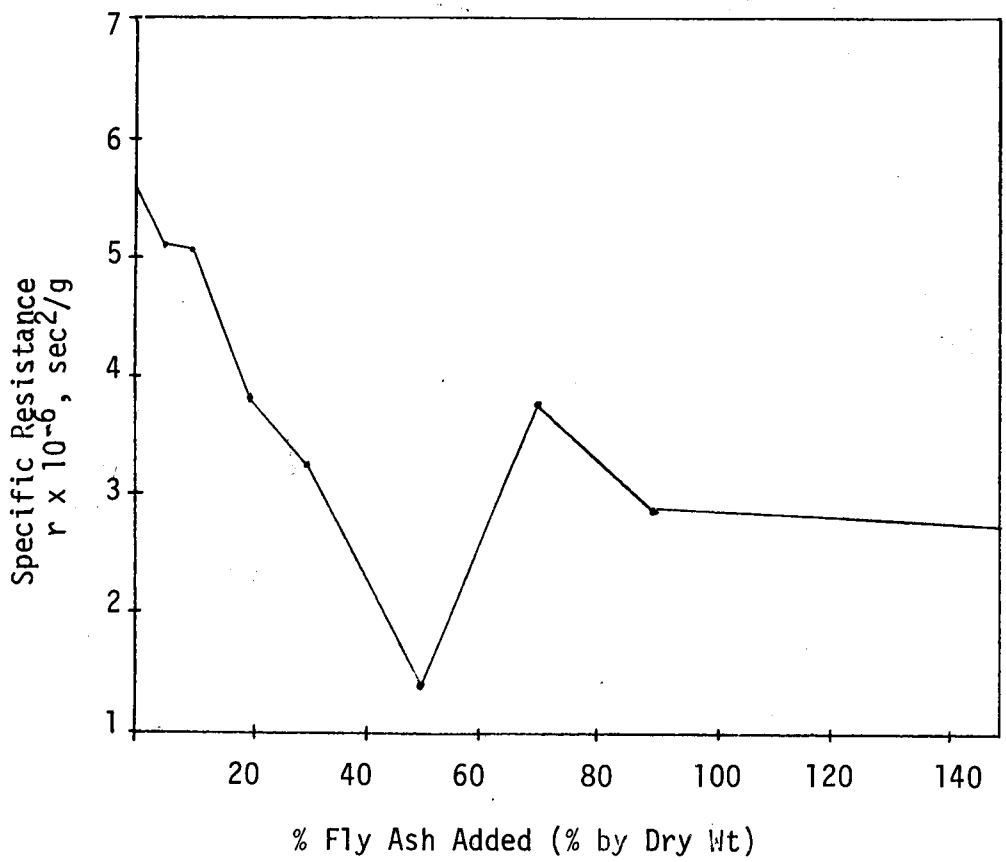


Figure 7. The results of Buchner Funnel Test for sludge A with the addition of 5, 10, 20, 30, 50, 70, 90, and 150% fly ash at initial solid concentration at 33%.

addition of fly ash into slurry will vary the weight of solid cake and the amount of filtrate produced from the Buchner funnel tests. Therefore, the Buchner funnel test was carried out where fly ash was added and the solid concentrations of the slurry was kept at 34 percent. The results (Figure 8) show no significant difference in filterability of the slurry if its solid concentration remains constant, although the percentage of fly ash varies in this 34 percent of solid. This result demonstrates the change in slurry solid concentration as the result of adding fly ash will not affect the specific resistance sufficiently. Therefore, any improvement in filterability of scrubber slurries by addition of fly ash into the slurry is not a result of increasing solid concentration in waste slurries, but as a result of increasing drainage channel for liquid to release the trapped water by embedding the fly ash between the sludge particles. This increase in drainage channel could be seen in scanning electron microscopic pictures which will be discussed later.

Finally, the specific resistance depends on the initial concentration of slurry, the higher the solid concentration, the lower the specific resistance to dewatering. The addition of fly ash will help the filterability. However, the improvement was not very significant.

Filter Leaf Test

Rotary-drum vacuum filtration is a widely used method for dewatering sludges. In the process for design a rotary-drum vacuum filtration system the results from leaf test can provide fairly reliable information.

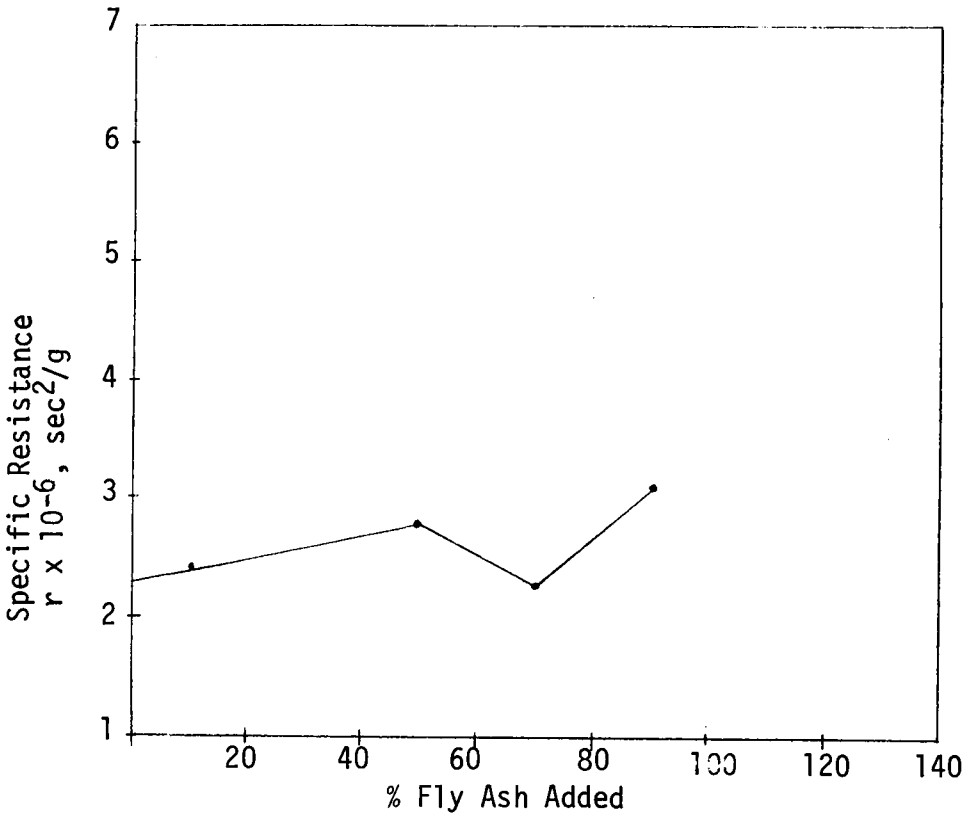


Figure 8. The results of Buchner Funnel Test in which the slurry solid concentration was kept constant at 34% of solid.

The rate of vacuum filtration is usually expressed as yield in pounds of dry solids produced per square foot of filter area per hour. When the leaf test is performed to simulate an actual vacuum filter, the yield of a vacuum filter with the same filter-media, vacuum level, cake form time (the time the filter media is submerged in the slurry), and cake drying time (the time the filter cake is exposed to the air) can be computed as:

$$Y = \frac{W}{A\ t_c} \qquad (2)$$

where W is the dry weight of cake, A the area of filter media, t_c cycles per unit of time (35).

In addition to filter yield, the final moisture content of the filter cake and solid in filtrate are also important factors in judging the performance of the vacuum filtration. However, in this case, the final moisture content of the cake seems to have more effect in vacuum filter design than does the solids in filtrate which will be pumped back to the scrubber system for reuse.

The leaf test was performed on the slurry which contained 17 percent of solid in which 11 percent of fly ash was incorporated. The results of the addition of fly ash into this slurry are tabulated in Table VII. The filter yield increases continuously with the increase in the percent of fly ash added into the slurry. As a result, the moisture contents in the cake are decreased.

Finally, the main objective of this test is to provide some basic design information on filtration equipment. For example, it

TABLE VII

THE RESULTS OF LEAF FILTER TEST FOR SLUDGE A

% Fly Ash Added	Filter Yield (lb/ft-hr)	Moisture Content of Cake* (%)	Solid Content of Cake (%)	Initial Solid Content in Slurry (%)
0	37.12	110	47.6	17.3
10	40.6	109	47.8	18.4
20	45.9	103	49.4	20.6
30	52.6	98.5	50	21.2
50	65.4	91	52	23.7
70	72	86	54	26.1
90	72	86	54	27.8

*Moisture content = $\frac{\text{weight of water in sample}}{\text{weight of dry solid}}$

would be helpful in predicting the size of the vacuum filter needed if the sludge generating rate and settling characteristic of slurry could be obtained.

Shear Strength Test

The relationship between curing time and strength gain for sludges with different additives can be seen in the results of the shear strength tests which were performed on each sample at different times. Increases in strength are evaluated by comparing the cohesion and the angle of internal frictions of the different samples.

The stress versus strain curves were developed under two different normal loads of 2 and 6.1 psi. The peak strengths for each specimen at a certain normal load were used to develop the stress versus normal load relationship, which had been described as a straight line and can be represented by the Mohr-Coulomb equation:

$$S = C + \sigma \tan \phi$$

in which S is the sheer stress on the plane of failure at failure. The empirical constant, C and ϕ, represent, respectively, the intercept at shear strength axis and the slope of the straight line in the shear strength versus normal stress graph. This empirical constant is called the cohesion and the angle of internal friction of the tested material. The comparison of the cohesion and angle of internal friction between samples will indicate the change in strength of the material.

Because of the cohesionless properties of the waste material, making it impossible to mold the sample for compression testing, the direct shear strength test seems to offer more useful results.

The results of the direct shear test for samples were tabulated and a summary of the age, peak cohesion, the angle of internal friction, moisture content, and dry density of tested samples is given in Table VIII and Table IX. The shear stress versus normal stress relationships are plotted in Figure 24 to Figure 40 in the appendix.

The thixotropic characteristic was demonstrated in the samples with no additives in them. The thixotropic characteristic of a material (in this case, the scrubber sludge) can be demonstrated by allowing the waste material to air dry to the point at which it forms an apparently solid state, then subjecting it to a mechanical vibration. The solid state material will transform into sol. This phenomenon can be explained by the character of the double layer of water attached to the particle of waste material, mainly clacium sulfite, which is held together to form a gel-like structure by the oriented molecular of water attached to it. When this gel-like structure is subject to vibration, the orientation of the water molecular is broken. The free liquid water is released between particles and a sol is formed. The gel-like structure can be reformed for a considerable length of time when the water molecular reorient themselves between particles. The characteristic of a material to regain its shear strenth with time after the material is remolded is defined as the thixotropic characteristic.

The addition of 20 percent to 40 percent fly ash alone will increase the cohesion of scrubber sludge-fly ash admixture a little, but has no effect on the angle of internal friction ϕ. This indicates the addition of fly ash alone will not help to build up the strength

TABLE VIII

THE RESULTS OF SHEAR STRENGTH TEST FOR TREATED SCRUBBER SLUDGE

(The moisture of samples content kept constant)

Flyash Added (%)	Hydrate Lime Added %	$Si0_2$ Added %	Curing Time (week)	Dry Density* (lb/ft^3)	Moisture** Content W (%)	Cohession (psi)	Angle of Interal Friction (degree)
0	0	0	1	57	32	3	43.2
			2	57	32	3.5	45.5
			4	57	32	4.1	47.5
20	0	0	1	61	32	2.8	38.5
			2	61	32	2.85	50.5
			4	61	32	2.8	38.5
40	0	0	1	63	32	6	36.5
			2	63	32	4.35	44.6
			4	63	32	6.65	39.5
20	6	0	1	59	33	3.85	35.5
			2	59	33	13.7	43
			4	59	33	16.9	60
40	6	0	1	60	30	9.27	43
			2	60	30	19.8	14.5
			4	60	30	20.6	12

TABLE VIII (Continued)

Flyash Added (%)	Hydrate Lime Added %	$Si0_2$ Added %	Curing Time (week)	Dry Density* (lb/ft^3)	Moisture** Content W (%)	Cohession (psi)	Angle of Interal Friction (degree)
20	0	8	1	62	32	4.7	47
			2	62	32	7.6	30.5
			4	62	32	6.2	37.5
20	6	8	1	59	31	2.7	59.5
			2	59	31	2.7	59.5
			4	--	--	---	----
40	6	15	1	61	32	14.1	3.5
			2	61	32	14.7	7.5
			4	61	32	20.6	24.2

*Dry density dry = $\frac{\text{Dry weight of sample}}{\text{Volume of sample}}$

**Moisture content W = $\frac{\text{Weight of water in sample}}{\text{Weight of dry solid}}$

TABLE IX

THE RESULTS OF SHEAR STRENGTH TEST FOR TREATED SCRUBBER SLUDGE

(The moisture content was not controlled)

Flyash Added (%)	Hydrate Lime Added (%)	Curing Time (week)	Dry Density* (lb/ft^3)	Moisture** Content W (%)	Cohession (psi)	Angle of Interal Friction (degree)
0	0	2	48	78	3.16	18.4
		4	48	27	1.95	45
		6	48	17	3.8	6.3
40	0	4	49	84	0.16	26.6
		6	50	55	4.1	36.9
0	3	2	45	100	0.6	21.8
		4	45	65	4.8	18.4
		6	45	41	4.5	18.4
10	3	2	44	106	0.5	24.8
		4	44	63	4.65	26.6
		6	44	43	2.5	45
20	3	2	43	107	0.35	29.7
		4	43	76	4.35	14
		6	43	48	4.25	29.7
40	3	2	45	88	0.55	21.8
		4	45	69	3.7	15.9
		6	45	44	6.5	21.8

TABLE IX (Continued)

Flyash Added (%)	Hydrate Lime Added (%)	Curing Time (week)	Dry Density* (lb/ft^3)	Moisture** Content W (%)	Cohession (psi)	Angle of Interal Friction (degree)
60	3	2	47	86	1.55	8.1
		4	46	76	5.55	7.1
		6	46	41	8.15	24.0
0	6	2	41	127	0.2	21.8
		4	42	70	2.75	45
		6	42	41	3.25	53
40	6	2	50	90	0.15	26.6
		4	50	62	3.4	26.6
		6	50	37	3.4	51.3

*Dry density dry = $\frac{\text{Dry weight of sample}}{\text{Volume of sample}}$

**Moisture content W = $\frac{\text{Weight of water in sample}}{\text{Weight of dry solid}}$

of the scrubber waste material. There is no time-strength gain relationship as a result of the thixotropic characteristics of the waste material.

The addition of 6 percent lime with 20 percent fly ash to the scrubber sludge produces significant strength gain with time. This indicates the cementitious material is forming between particles, bonding the waste material together to produce higher cohesion between particles. However, the power effect on angle ϕ may be due to the lower coefficient of uniformity, which causes the higher void ratio in compacted samples. Greater effect on ϕ might be achieved by increasing particle uniformity through better gradation of material.

With the addition of 6 percent lime and 40 percent fly ash into sludge, the admixture also indicated a significant strength increase. This indicates that an increase in the amount of fly ash in the admixture, with the same amount of lime added, will increase the strength in the waste material. As a result of the increase in cohesion, the ϕ value actually decreased, but this is not of major concern with treated material. A definite time-strength gain relationship can be obtained by this lime-fly ash combination.

The addition of silica (SiO_2) with the lime and fly ash into the sludge does not seem to help out more strength than when the lime and fly ash were added without the silica. The results from the addition of 15 percent silica into the admixture of 40 percent fly ash--6 percent lime--sludge shows a result very similar to the one without the addition of silica. The addition of 8 percent silica into the admixture of 20 percent fly ash--6 percent lime--sludge shows little strength increase

in the 2-week-old sample comparing to the admixture without the silica. The data at fourth week for this sample were missing, therefore, no conclusion can be made. However, some strength increase could be expected.

The plot of shear stress versus normal stress for all the samples with constant moisture content at curing time of fourth week is shown on Figure 9. The cohesion of the sludge treated with lime and fly ash increased about three times. However, the angle of internal frictions was not significantly affected by the treatment process.

For those admixtures in which moisture content was not kept constant, the specimens without any additives showed a small strength increase with time. The difference in strength seems to lie more in ϕ than in cohesion. The decrease in moisture content seems to cause an increase in strength for the first four weeks. After the fourth week, however, additional moisture loss seems to result in decreased strength.

No strength gain was observed when lime alone, at 3 and 6 percent, was added to the sludge. The results from the admixture with 40 percent fly ash as the only additive also shows no strength gain with time.

Fly ash was added at 10, 20, and 40 percent along with 3 percent lime into the waste slurry and the slurry was allowed to settle, then subjected to air drying at room temperature. This test also showed little strength increase other than beyond the effects of drying.

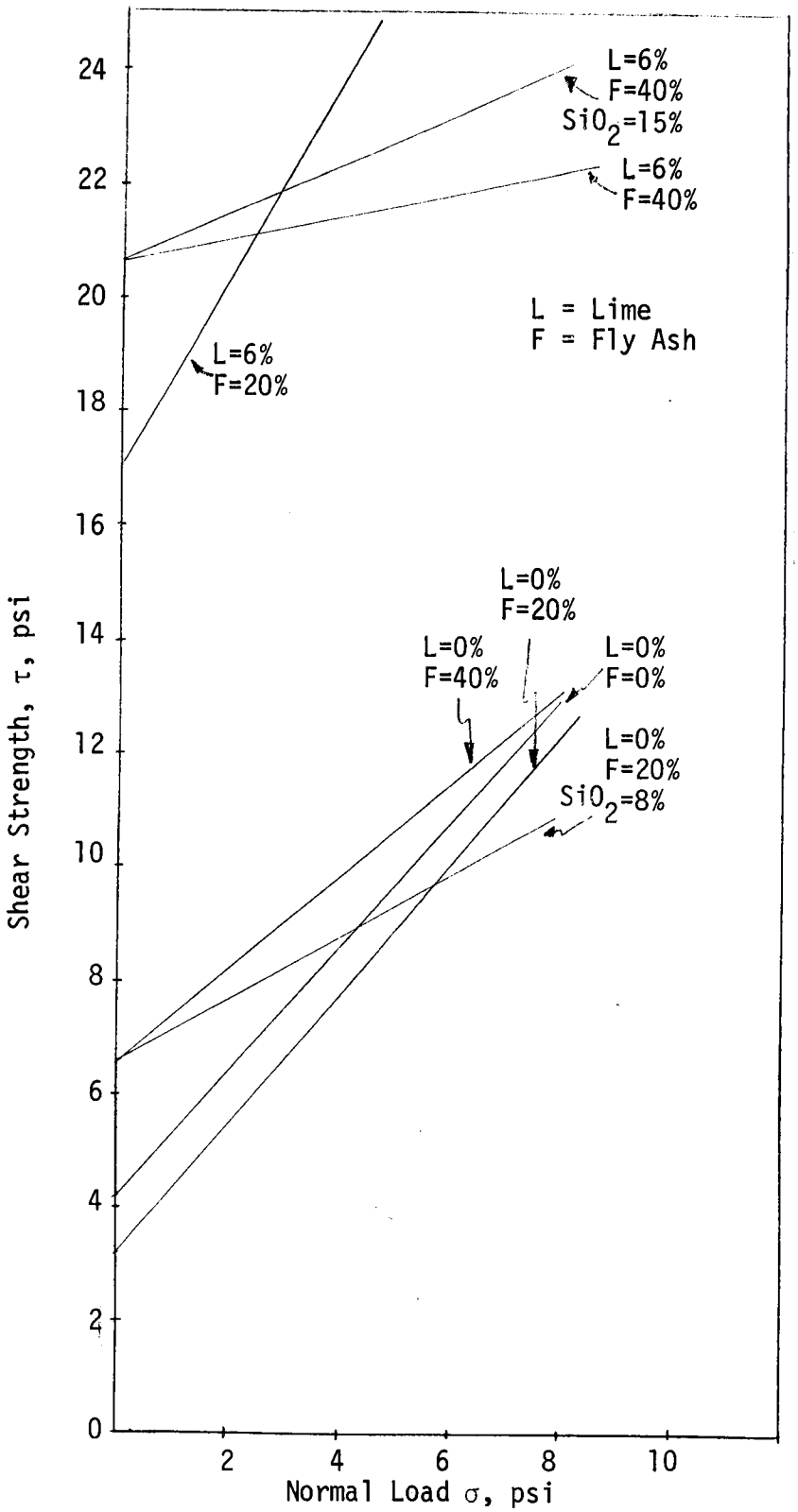


Figure 9. The comparison of strength of treated scrubber sludge at curing time of 6 weeks.

Even when the percentage of lime added into slurry was increased to 6 percent along with the 40 percent fly ash, the strength increase was very small.

The only group of samples showing strength gain with time was the admixture of 3 percent lime--60 percent fly ash--scrubber sludge mixture.

The shear strength versus normal stress graph for all the samples in this nonconstant moisture content group at sixth week of curing time is shown on Figure 10. The cohesion and angle ϕ are not much different compared to the control specimen which contains no additives. The only strength gain in this group is in the mixture with 60 percent fly ash and 3 percent lime in it.

In summary, the results of the addition of lime and fly ash with sludge from lime scrubbing system have shown a definite strength increase. It seems the degree of increase depends on the amount of fly ash added, the more the fly ash added, the better the pozzolanic reaction produced, and the higher the strength increase. However, the optimal combination of lime and fly ash to the scrubber sludge system would be site specific and would depend on the characteristics of sludge.

X-Ray Diffractometer Analysis

The results from X-ray diffractometer for all the samples were examined and an effort was made to identify all the species in those mixtures. However, it is very difficult to identify all the species in the X-ray diffraction patterns by ASTM searching manual because of

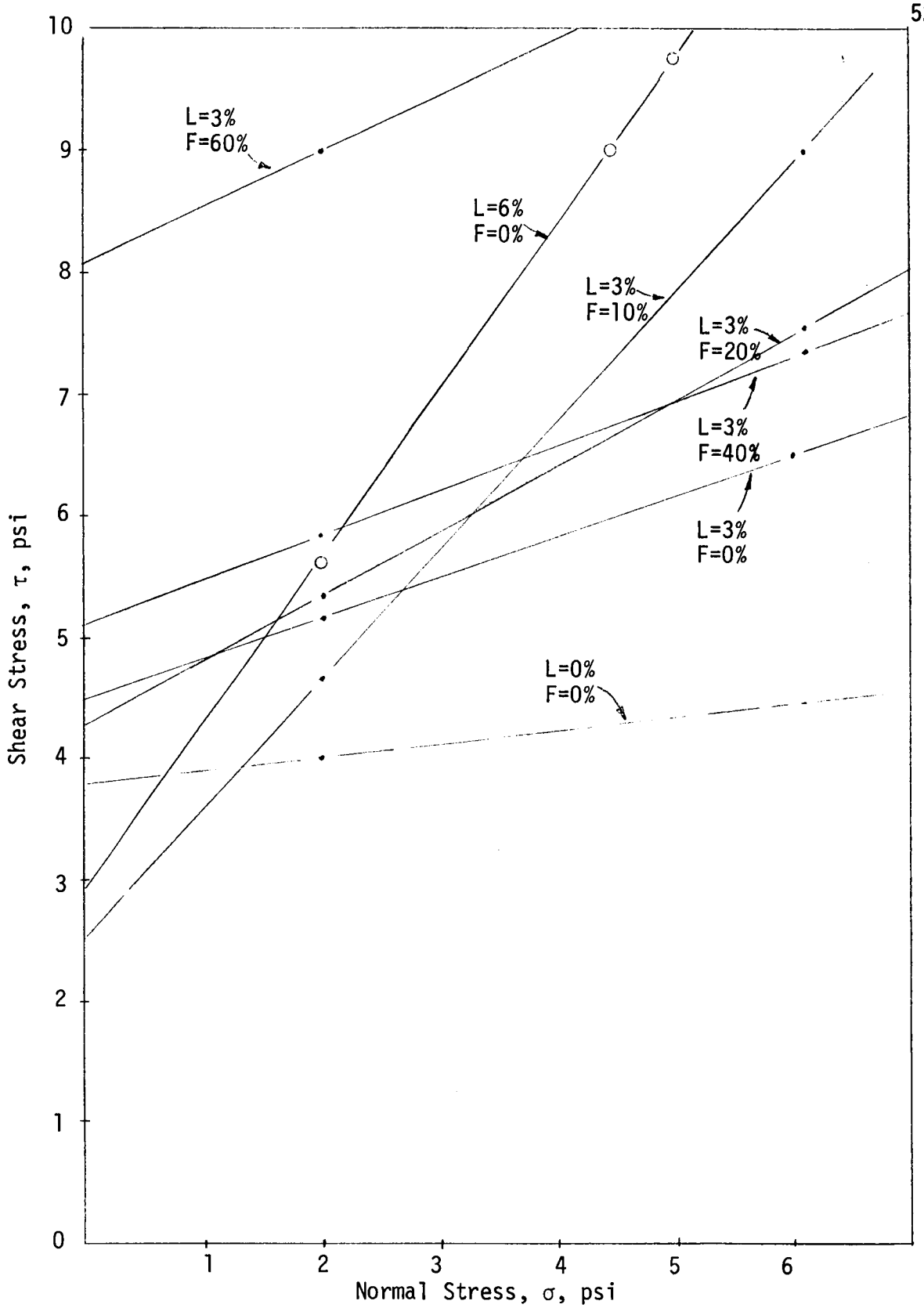


Figure 10. The comparison of strength of treated scrubber sludge at curing time of 6 weeks.

the extremely complex mixture here involved. Therefore, efforts were focused on identifying the formation of certain minerals such as tobermorite (calcium silicate hydrated family) and ettringite (calcium aluminum sulfate hydrate) which are believed by several investigators (32,36) to be cementitious materials.

The major constituents of the sludge-lime-fly ash mixtures, such as calcium sulfate, calcium sulfite, and calcium hydroxide are also examined to observe any changes in those constituents.

The X-ray diffraction patterns of samples with strength increase were examined at the curing times of one week and eight weeks.

The tobermorite group has its most intense peak at d-spacing, which is the distance in angstroms (A°) between two lattice planes, of 14 A°, its second intense peak at 3.07 A°, and several other peaks at 5.53 A°, 2.98 A°, 1.83 A° with a relative intensity of 80 percent. These peaks can be seen in the X-ray diffraction patterns of the 8-week-old specimens which had shown a definite strength increase. The X-ray diffraction patterns were shown on Figure 11, 12, and 13. The new mineral formation in tobermorite family can be observed at the 8-week-old specimen. The results of X-ray diffraction pattern for interested mineral was also tabulated in Table X, XI, and XII. The peaks for each mineral shown in the X-ray pattern were listed and compared to the peaks indicated by ASTM card. The dash line on the tables denoted no peak appeared at that particular d-spacing which is shown at ASTM card for a particular mineral. It indicates no such mineral in the specimen.

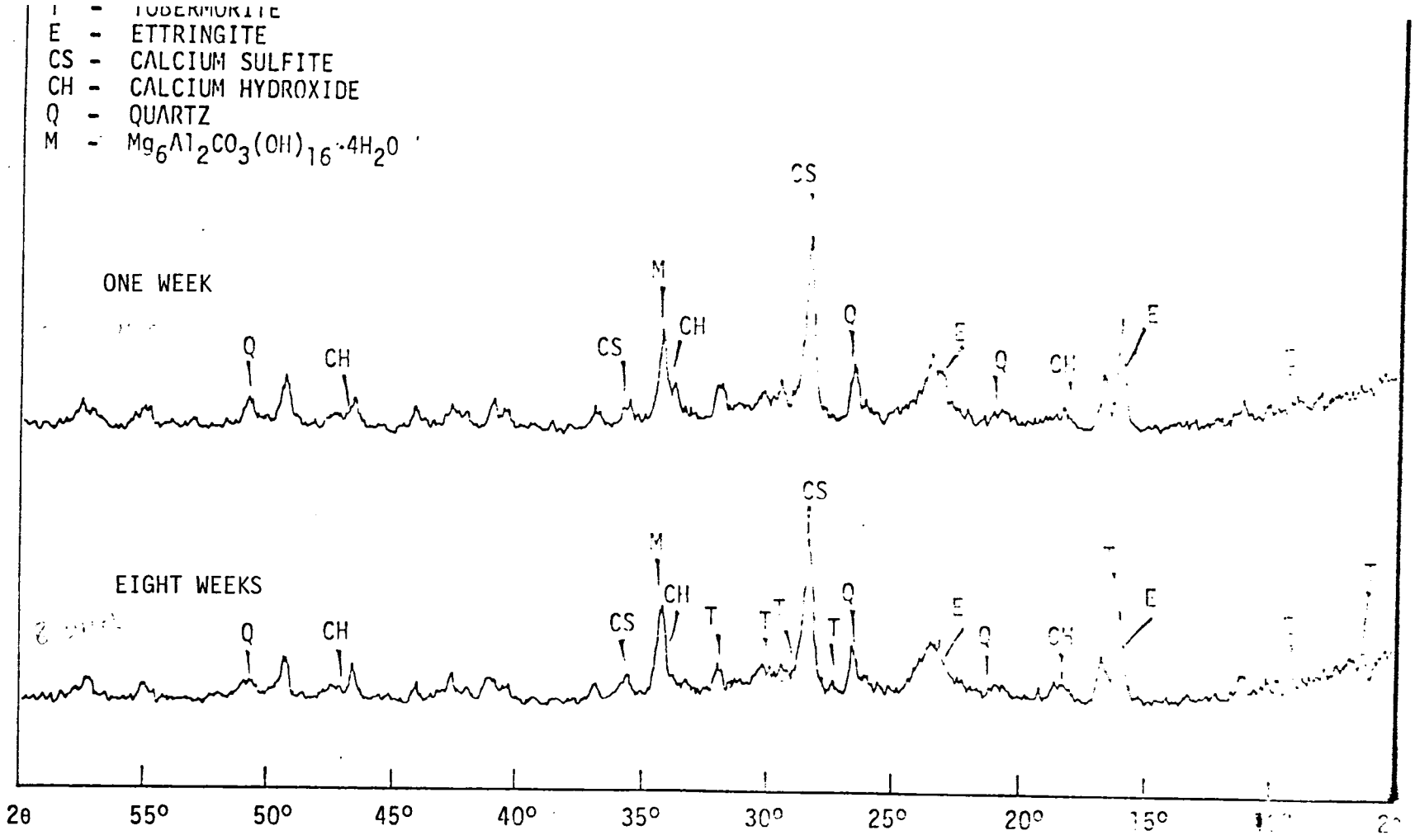


Figure 11. Comparison of X-Ray Diffraction Patterns of Sludge with 6% Lime and 20% Fly Ash.

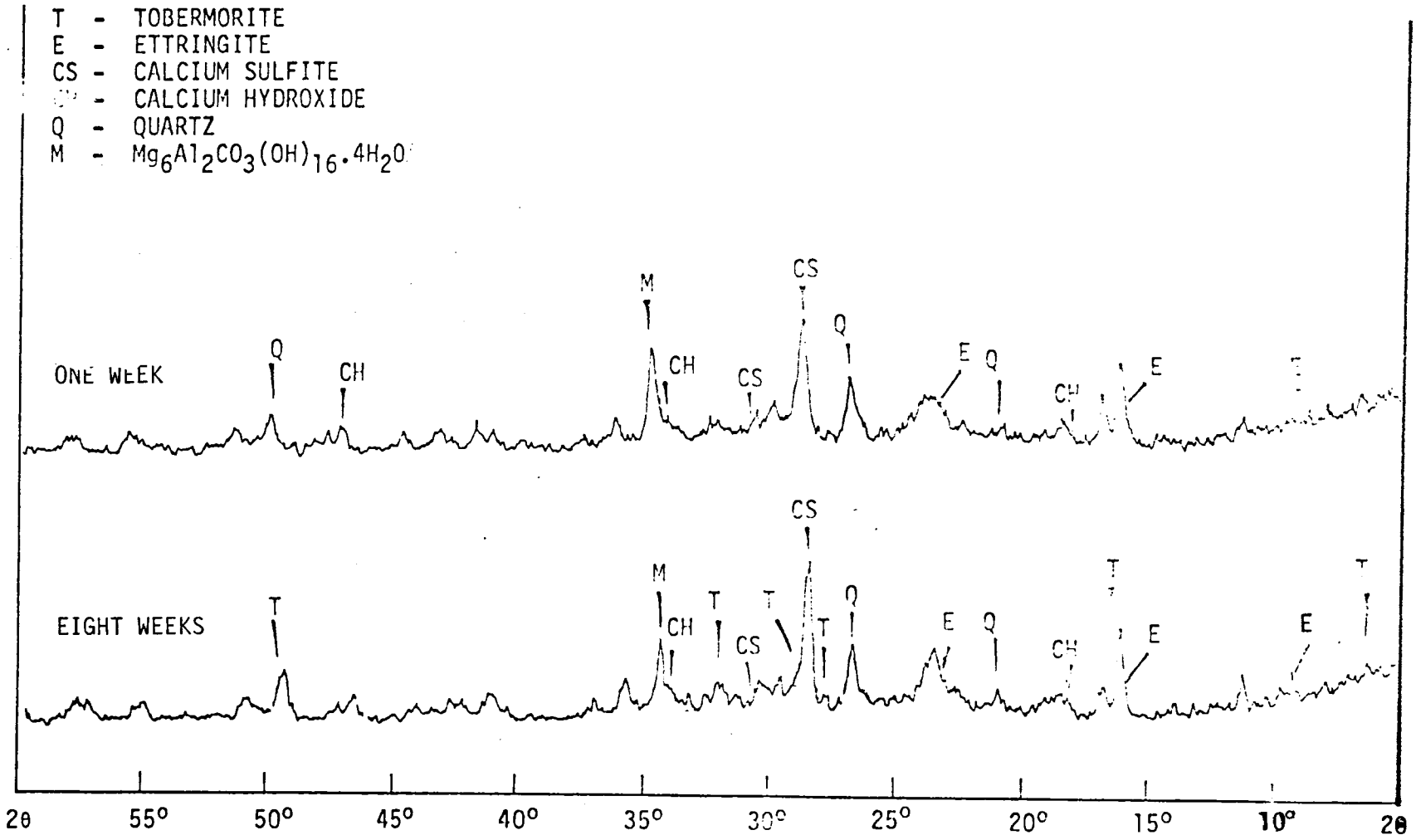


Figure 12. Comparison of X-Ray Diffraction Patterns of Sludge with 6% Lime and 40% Fly Ash.

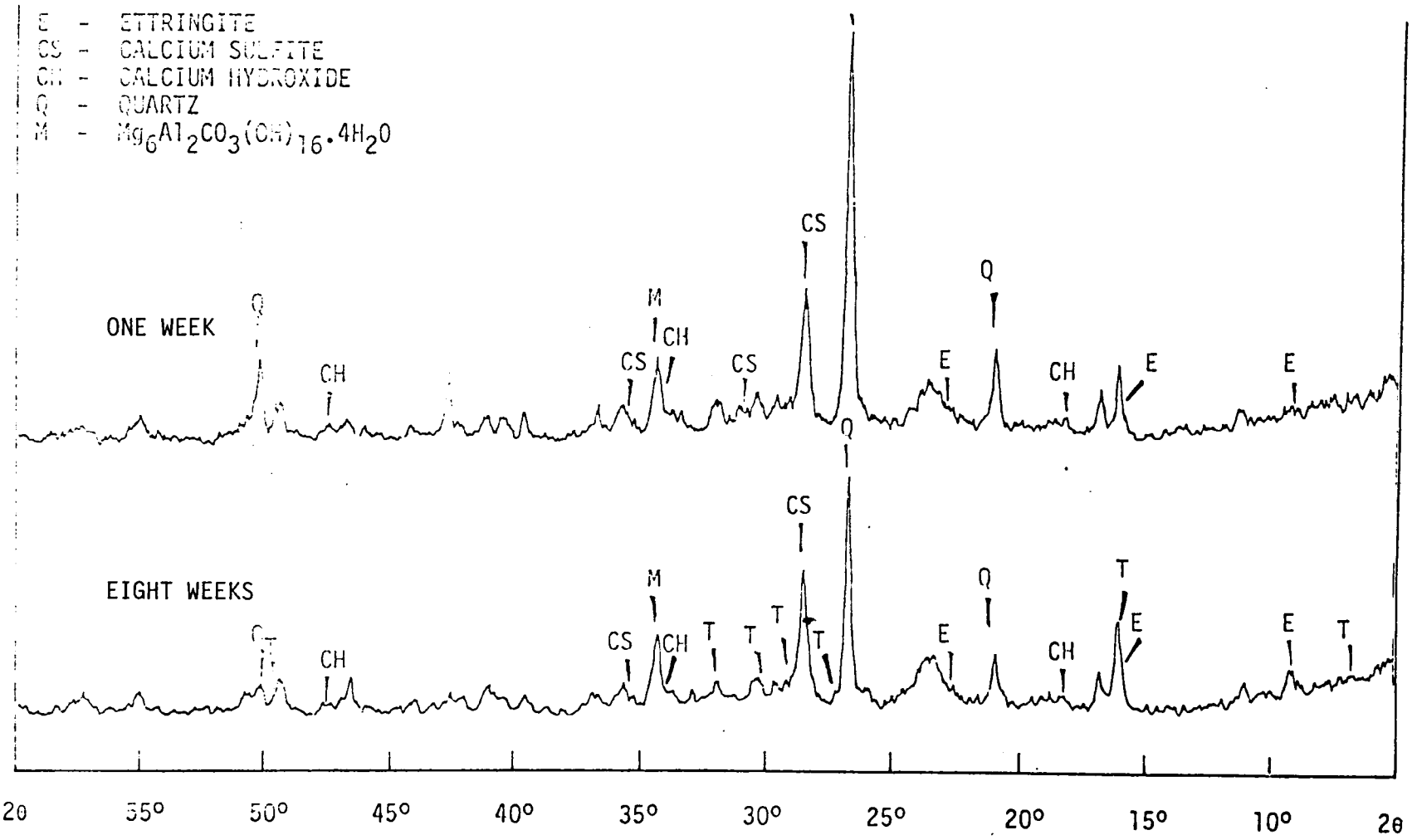


Figure 13. Comparison of X-Ray Diffraction Patterns of Sludge with 3.7% Lime, 44.1% Fly Ash, and 9.3% SiO_2.

TABLE X

X-RAY DIFFRACTION DATA ON SLUDGE
WITH 6% LIME AND 20% FLY ASH

Compound	Peak in ASTM Card (D-Spacing, A)	Peak in X-Ray Pattern of 1-Week-Old Sample (D-Spacing, A)	Peak in X-Ray Pattern of 8-Week-Old Sample (D-Spacing, A)
Quartz	3.34*	3.34	3.34
	4.26	4.26	4.26
	1.82	1.82	1.82
Tobermorite	14*	-	14
	3.07	3.07	3.07
	5.53	5.53	5.53
	2.98	-	2.98
	2.81	2.81	2.81
	1.83	-	1.83
	3.25	-	3.25
Ettringite	9.67*	9.67	9.67
	5.6	5.6	5.6
	3.88	3.88	3.88
$CaSO_3$-1/2H_2O	3.12*	3.12	3.12
	2.53	2.53	2.53
	2.9	2.9	-
$Ca(OH)_2$	2.63*	2.63	2.63
	4.9	4.9	4.9
	1.93	1.93	1.93

*Primary Peak

TABLE XI

X-RAY DIFFRACTION DATA ON SLUDGE
WITH 6% LIME AND 40% FLY ASH

Compound	Peak in ASTM Card (D-Spacing, A)	Peak in X-Ray Pattern of 1-Week-Old Sample (D-Spacing, A)	Peak in X-Ray Pattern of 8-Week-Old Sample (D-Spacing, A)
Quartz	3.34*	3.34	3.34
	4.26	4.26	4.26
	1.82	1.82	1.82
Tobermorite	14*	-	14
	3.07	3.07	3.07
	5.53	5.53	5.53
	2.98	2.98	2.98
	2.81	-	2.81
	1.83	1.83	1.83
	3.25	3.25	3.25
Ettringite	9.67*	9.67	9.67
	5.6	5.6	5.6
	3.88	3.88	3.88
$CaSO_3$-1/2H_2O	3.12*	3.12	3.12
	2.53	2.53	2.53
	2.9	2.9	2.9
$Ca(OH)_2$	2.63*	2.63	2.63
	4.9	4.9	4.9
	1.93	1.93	1.93

*Primary Peak

TABLE XII

X-RAY DIFFRACTION DATA ON SLUDGE WITH 6% LIME, 40% FLY ASH, AND 15% SiO_2

Compound	Peak in ASTM Card (D-Spacing, A)	Peak in X-Ray Pattern of 1-Week-Old Sample (D-Spacing, A)	Peak in X-Ray Pattern of 8-Week-Old Sample (D-Spacing, A)
Quartz	3.34*	3.34	3.34
	4.26	4.26	4.26
	1.82	1.82	1.82
Tobermorite	14*	-	14
	3.07	3.07	3.07
	5.53	5.53	5.53
	2.98	-	2.98
	2.81	2.81	2.81
	1.83	-	1.83
	3.25	-	3.25
Ettringite	9.67*	9.67	9.67
	5.6	5.6	5.6
	3.88	3.88	3.88
$CaSO_3$-1/2H_2O	3.12*	3.12	3.12
	2.53	2.53	2.53
	2.9	2.9	-
$Ca(OH)_2$	2.63*	2.63	2.63
	4.9	4.9	4.9
	1.93	1.93	1.93

*Primary Peak

The formation of calcium sulfoaluminate by hydrate which was found by other investigators (34) in lime-fly ash-sulfate systems will produce an end product of ettringite when the sulfate content in sludge mixture is greater than 15 percent. Otherwise, the low sulfate sulfoaluminate form will be produced. In the X-ray diffraction patterns of all the cured samples with the additions of lime and fly ash, the formation of calcium sulfoaluminate hydrate (ettringite) was observed even in the 1-week-old sample. This indicated the formation of ettringite is a rapid process.

The calcium sulfite hemihydrate ($CaSO_3 \cdot 1/2\ H_2O$) can be identified very easily by its primary peak at d-spacing of 3.12 A°. When comparing all the X-ray diffraction patterns for each admixture at the two curing times, the calcium sulfite seems unchanged or only slightly changed, in the intensity of its primary peak. This indicates the calcium sulfite species is not involved in the stabilization process chemically.

The peaks for calcium hydroxide seem to change only a little during the curing time of eight weeks. The most intense peak for calcium hydroxy is at d-spacing of 2.63 A°, which is overlayed by the peak of some other psecies with same d-spacing. The addition of lime to the sludge-fly ash mixtures was at a fairly low percentage (6 percent) which normally will not be shown at the X-ray diffraction pattern (less than 15 percent). The primary peak of calcium hydroxide can be explained as the consumption of calcium hydroxide in the pozzolanic reaction.

The definite decrease in intensity of the quartz (SiO_2) peak (3.34 A°) of the 8-week-old samples with the addition of 8 percent and 15 percent of silica into lime-fly ash-sludge mixture was observed. It indicated the silica was involved in the reaction. However, the results of the reaction does not provide more strength to the admixture of sludge-lime-fly ash - silica system than the admixture without the addition of silica.

Fly ash was added at 20 and 40 percent along with a 6 percent lime into sludge then the moisture was kept at 30 percent. The shear strength gain with lime of these samples was discussed previously. The X-ray pattern at eight weeks shows new mineral formation in tobermorites family (Figures 11 and 12). A slight decrease in calcium sulfite could also be observed.

Two samples were treated with the addition of silica, at 8 percent and 15 percent, respectively, along with the lime and fly ash and then the moisture content was kept at 30 percent. The eight week samples also indicated the formation of minerals in tobermorites family by X-ray diffraction pattern (Figure 13).

In summary, the X-ray diffraction patterns are shown in Figures 10 to 13. Some of the peaks are not identifiable due to the extremely complex mixture system. For instance, the board peak at d-spacing of 3.8 A° may consist of more than eight minerals. However, the X-ray patterns still serve their purpose by explaining some products of pozzolantic reaction.

Scanning Electron Microscopic (SEM) Pictures

The SEM pictures for all the samples with strength increase indicated by direct shear strength measurement were taken when the samples had been cured for ten weeks.

The pictures taken of sludge without any fly ash are shown in Figure 14. The plate-like calcium sulfite packed into a loss structure can be seen in the pictures. Water can be trapped between these plate-like calcium sulfite crystals. After 90 percent of fly ash was added (Figure 15) the drainage capacity was improved to release water.

The crystalline growth of the admixture with 6 percent lime and 20 percent fly ash can be seen in Figures 16, 17, 18, and 19. A more clear overall picture can be seen in Figure 17. The element constituents of one of the worm-like crystalline is analyzed by the energy dispersive X-ray analyzer (EDXRA) unit of SEM. The EDXRA spectrum (Figure 20) indicates that the silica and calcium are two major elements in this crystalline, therefore, calcium silicate hydrate crystal growth can be assumed.

The sample with more fly ash added (6 percent lime, 40 percent fly ash) shows more crystalline growth in Figures 18 and 19. The gel formation on the treated sludge can be seen in Figure 17. The samples with the addition of silica show a growth pattern similar to those shown by the lime-fly ash-sludge systems.

The EDXRA spectra (Figure 21) for the samples were taken along with the pictures. The element constituents on the EDXRA agree fairly well with the predicated constituents for each sample. The major element

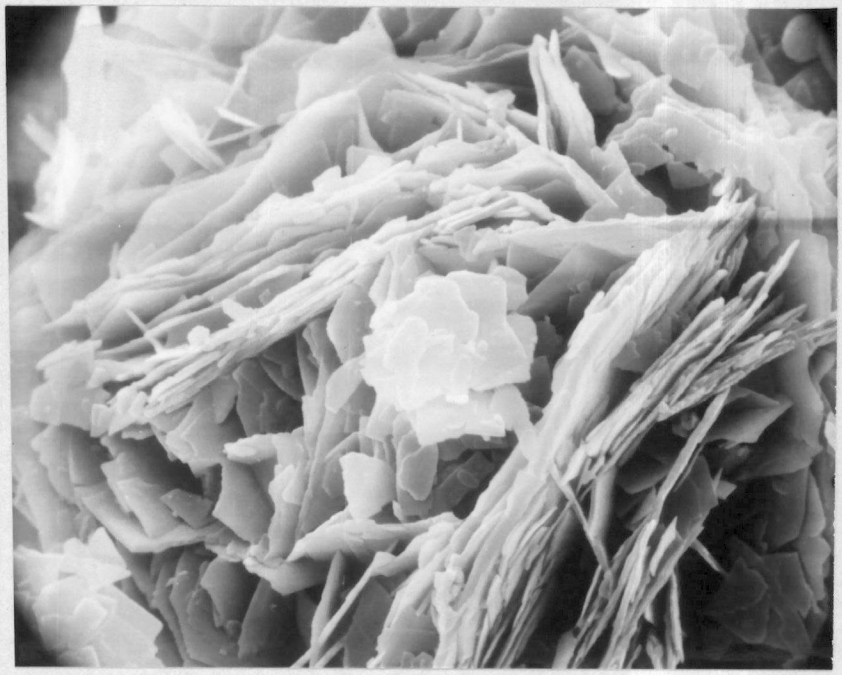


Figure 14. Lime Scrubber Sludge Without Fly Ash (5,000 X).

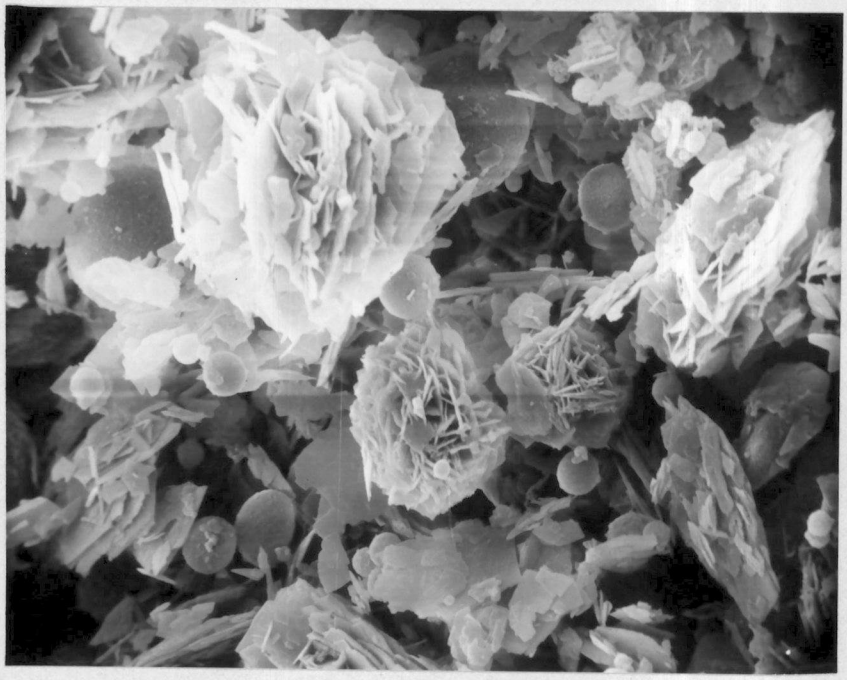


Figure 15. Lime Scrubber Sludge With 90% of Fly Ash (2,000 X).

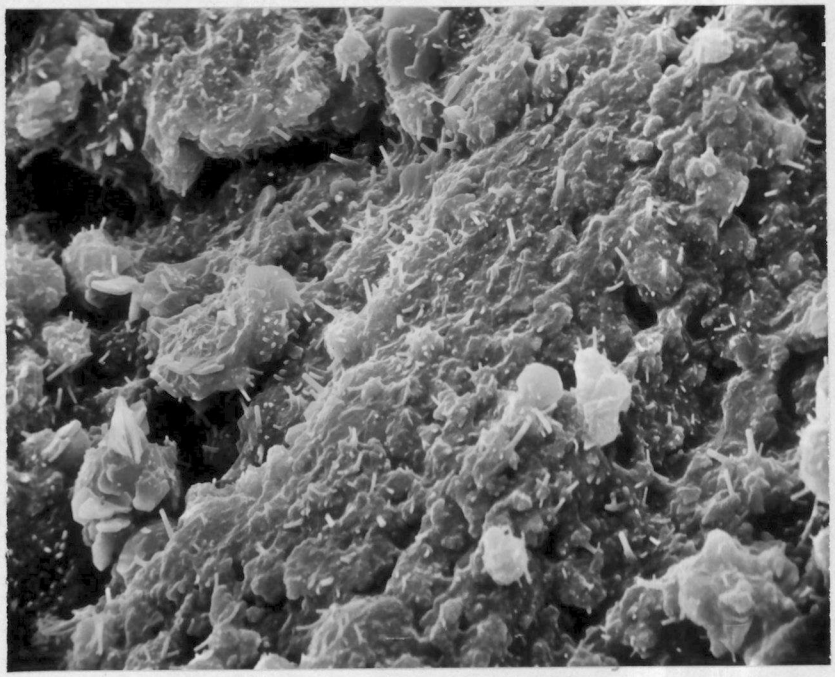


Figure 16. Crystalline Growth on Admixture of 6% Lime and 20% Fly Ash (2,000 X).

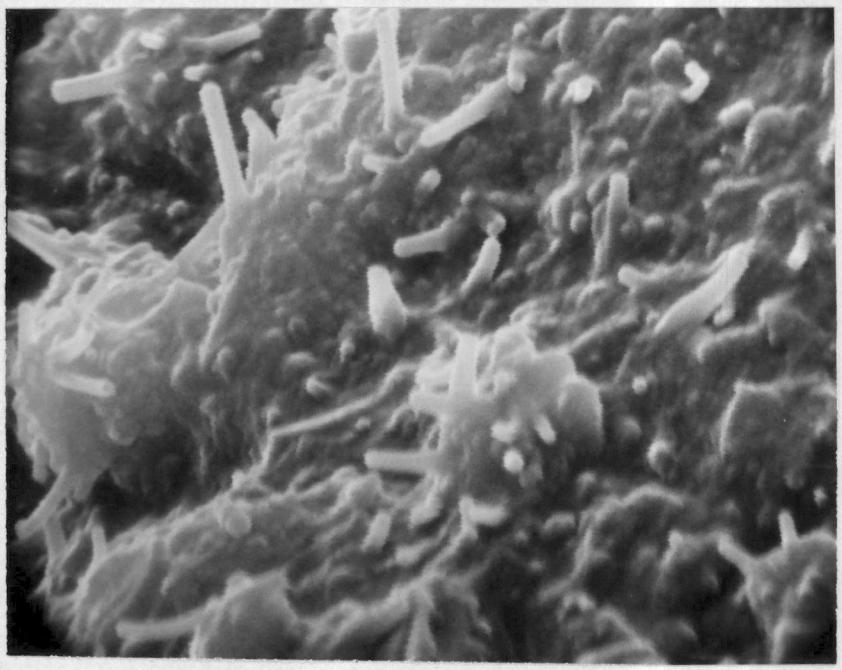


Figure 17. Crystalline Growth on Admixture of 6% Lime and 20% Fly Ash (10,000 X).

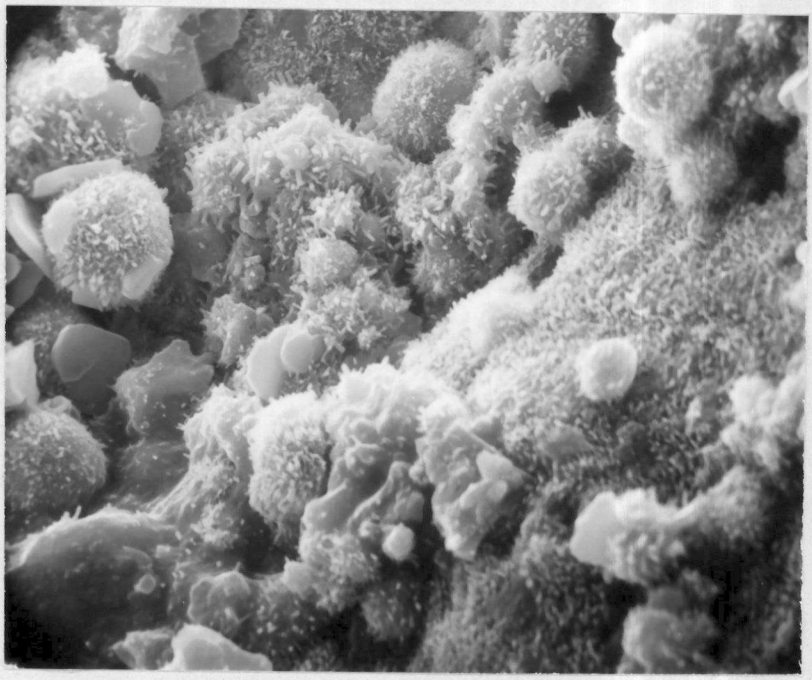


Figure 18. Crystalline Growth on Admixture of 6% Lime, 40% Fly Ash (2,000 X).

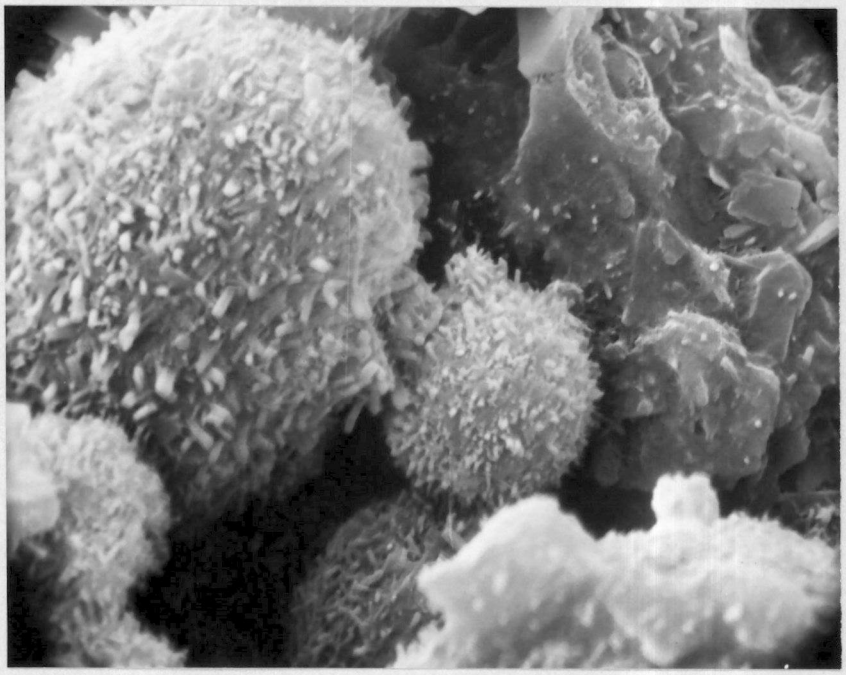


Figure 19. Crystalline Growth on Admixture of 6% Lime, 40% Fly Ash (5,000 X).

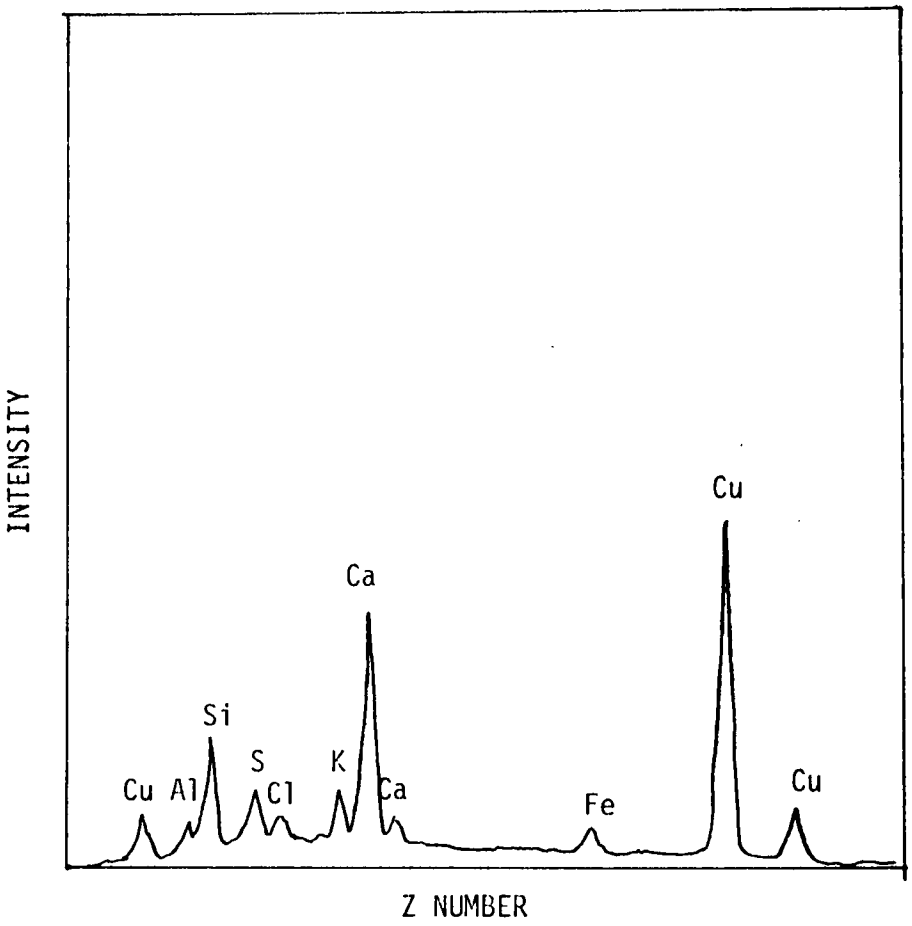


Figure 20. The EDXRA Spectrum of Protruding Crystal.

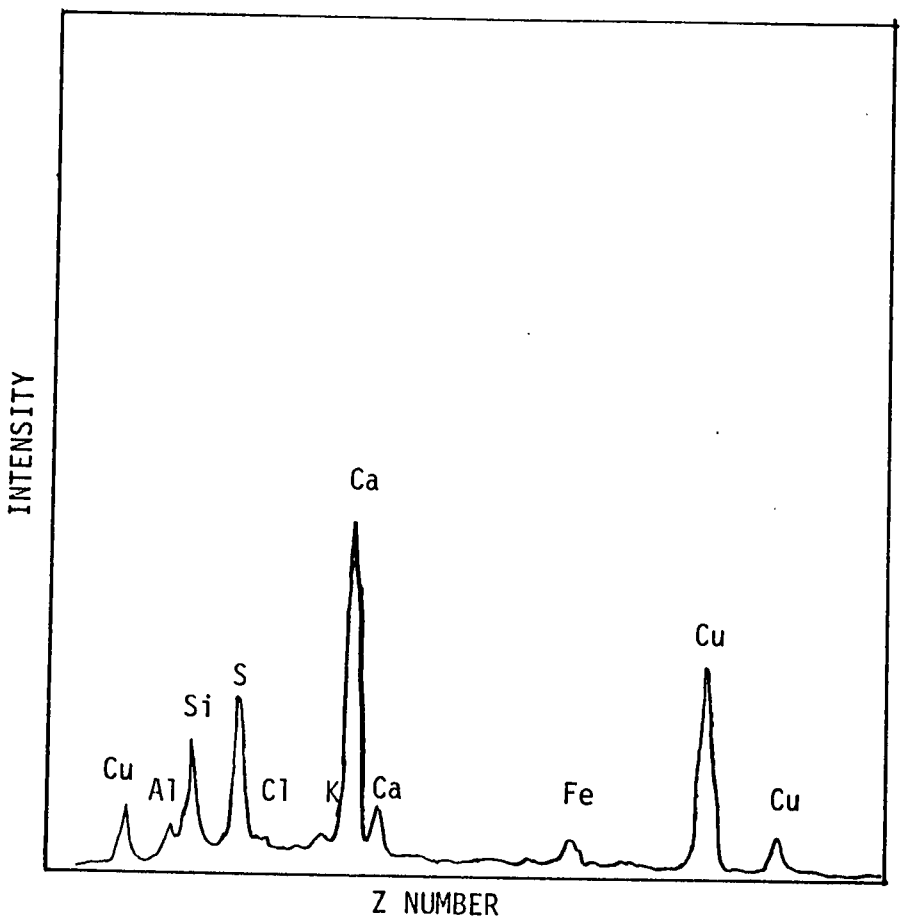


Figure 21. The EDXRA Spectrum of Total Sludge Fixed.

constituents in all the 10-week treated sludge specimens are calcium, silicon, sulfur, and some iron, aluminum, potassium, and chlorine. The copper peaks shown in the EDXRA is from the copper coating on the sample. The EDXRA spectrum for plate-like crystalline was also taken and the major elements shown were calcium and sulfur (Figure 22).

The EDXRA spectrum on fly ash (Figure 23) shows the element constituents are silicon, aluminum, iron, calcium, and potassium, agree with the constituents in the fly ash.

In summary, the SEM pictures give a very good demonstration of crystalline growth in the treated sludge. The strength increase of treated material could be produced from the gel formation and crystalline growth between particles.

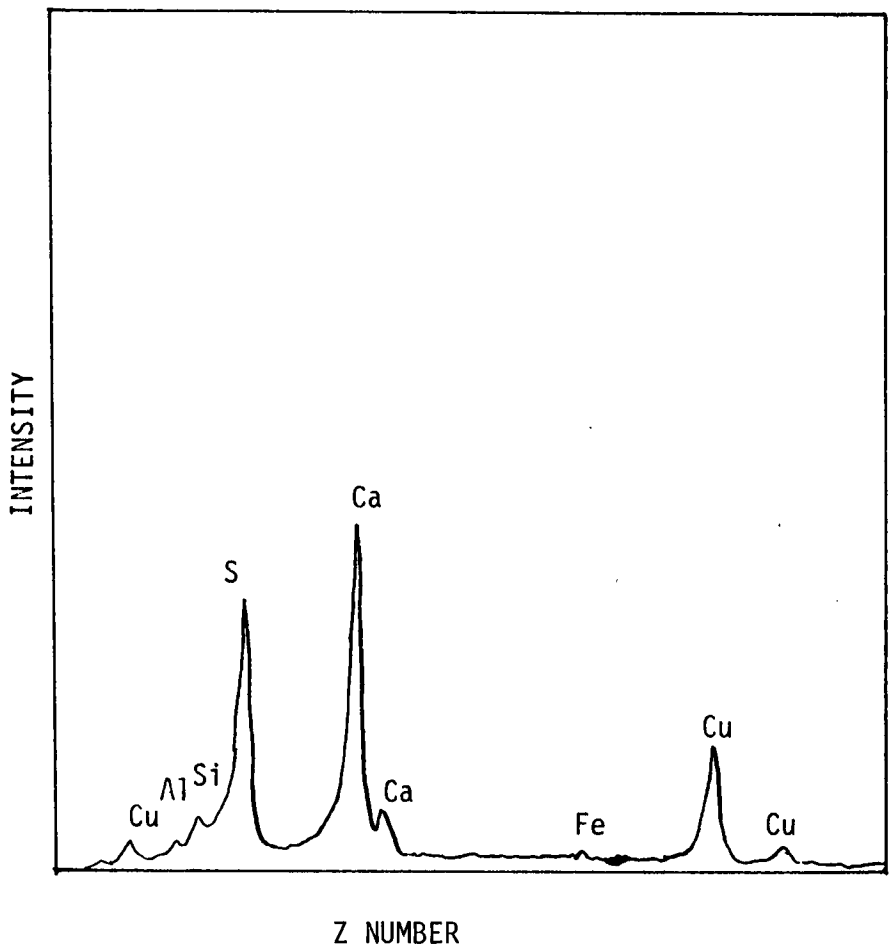


Figure 22. The EDXRA Spectrum of Plate-Like Crystal.

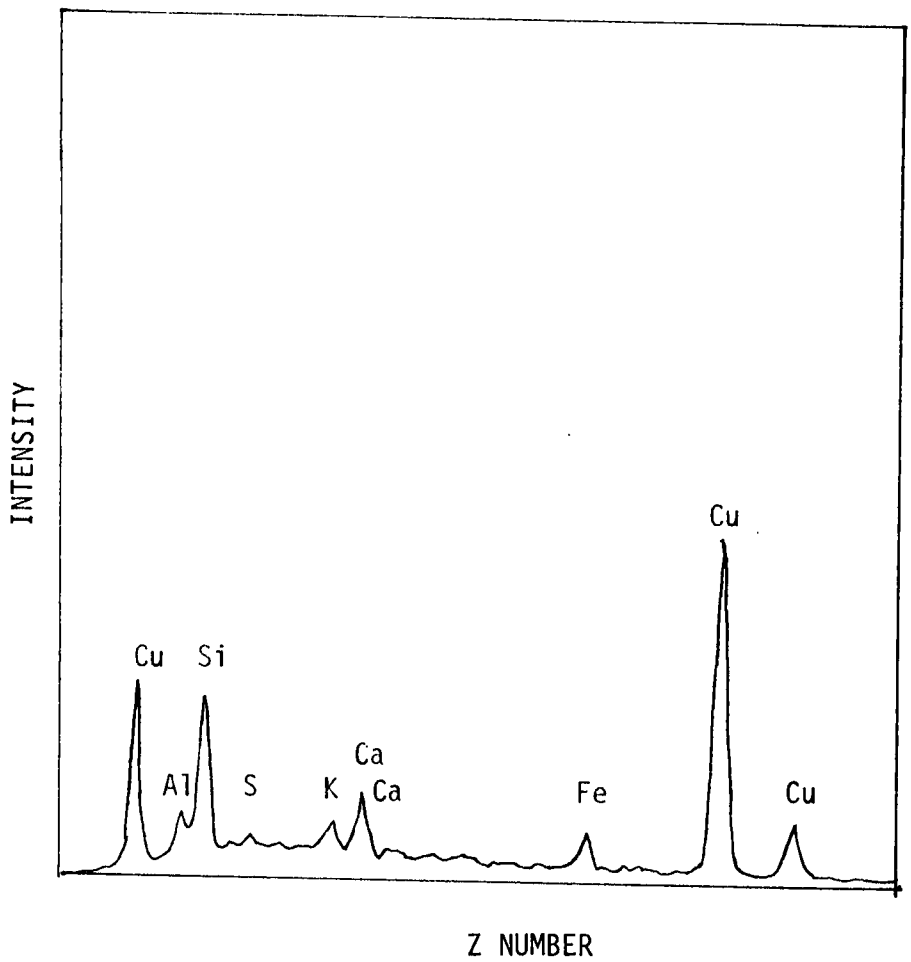


Figure 23. The EDXRA Spectrum of Fly Ash.

CHAPTER V

CONCLUSIONS

Based on the results of the foregoing studies on the stabilization of waste material from lime scrubbing systems, the following conclusions can be made:

1. The settling rate depends upon the solid concentration of slurry. The lower the solid concentration the higher the settling rate.

2. The addition of fly ash does not increase the settling velocity of slurry which has 9 percent of solids in which 33 percent of fly ash was incorporated. As the slurry concentration increases to 17 percent, the addition of fly ash into the sludge increases the settling velocity somewhat, however, the increase is not significant enough to affect the engineering design.

3. The specific resistance of the sludge could be decreased by addition of fly ash up to 90 percent by dry weight of slurry. Since the stabilization of scrubber sludge required addition of fly ash, it is helpful to increase the fly ash contents in slurry to help the dewatering of the slurry by vacuum filtration.

4. The addition of fly ash alone into the sludge of a lime scrubbing system will not show a strength increase.

5. The addition of fly ash at 20 and 40 percent along with 6 percent lime into the waste material will result in an increase in shear strength with time when the moisture content of this admixture is kept constant at 30 percent.

6. The addition of silica (SiO_2) into the fly ash lime-scrubber sludge system produces a strength increase very sililar to the strength increase produced without the addition of silica.

7. The gel and crystalline formation can be seen in SEM pictures. From X-ray diffraction patterns of 8-week-old specimens with shear strength increases, the formation of new minerals in tobermorite's family was detected. Therefore, crystallines are assumed to be the mineral in tobermorite. The EDXRA spectrum also provided the information that the major constituent elements of this crystalline are calcium and silica which are the major constituent elements in tobermorite (calcium silicate hydrate).

8. The formation of ettringite was also detected in X-ray diffraction patterns of the treated sludge with the addition of lime and fly ash.

CHAPTER VI

RECOMMENDATIONS

The future studies for treating the waste material for field application are recommended as follows:

1. The optimal combination of fly ash and lime for each different sludge produced from scrubbing systems could be found by the testing program designed for this experiment. However, the optimal moisture content and the density for the treated scrubber sludge must be determined by further studies.

2. The field application of this lime-fly ash technique seems to need more studies. Specifically, methods should be sought to mix these additives (lime and fly ash) into the sludge uniformly, and to control moisture content during the curing process.

3. The leachate characteristic of this treated material needs to be investigated to prevent the contamination of ground water if this material is be subjected to landfill disposal or used for roal base construction.

4. The effects of freeze and thaw on this treated material should also be investigated.

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BIBLIOGRAPHY

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APPENDICES

APPENDIX A

Figures 24 through 31 represent the shear stress versus the normal loads for sludge B added to a different amount of lime and fly ash (constant moisture content).

Key:

● 1 Week

○ 2 Weeks

□ 4 Weeks

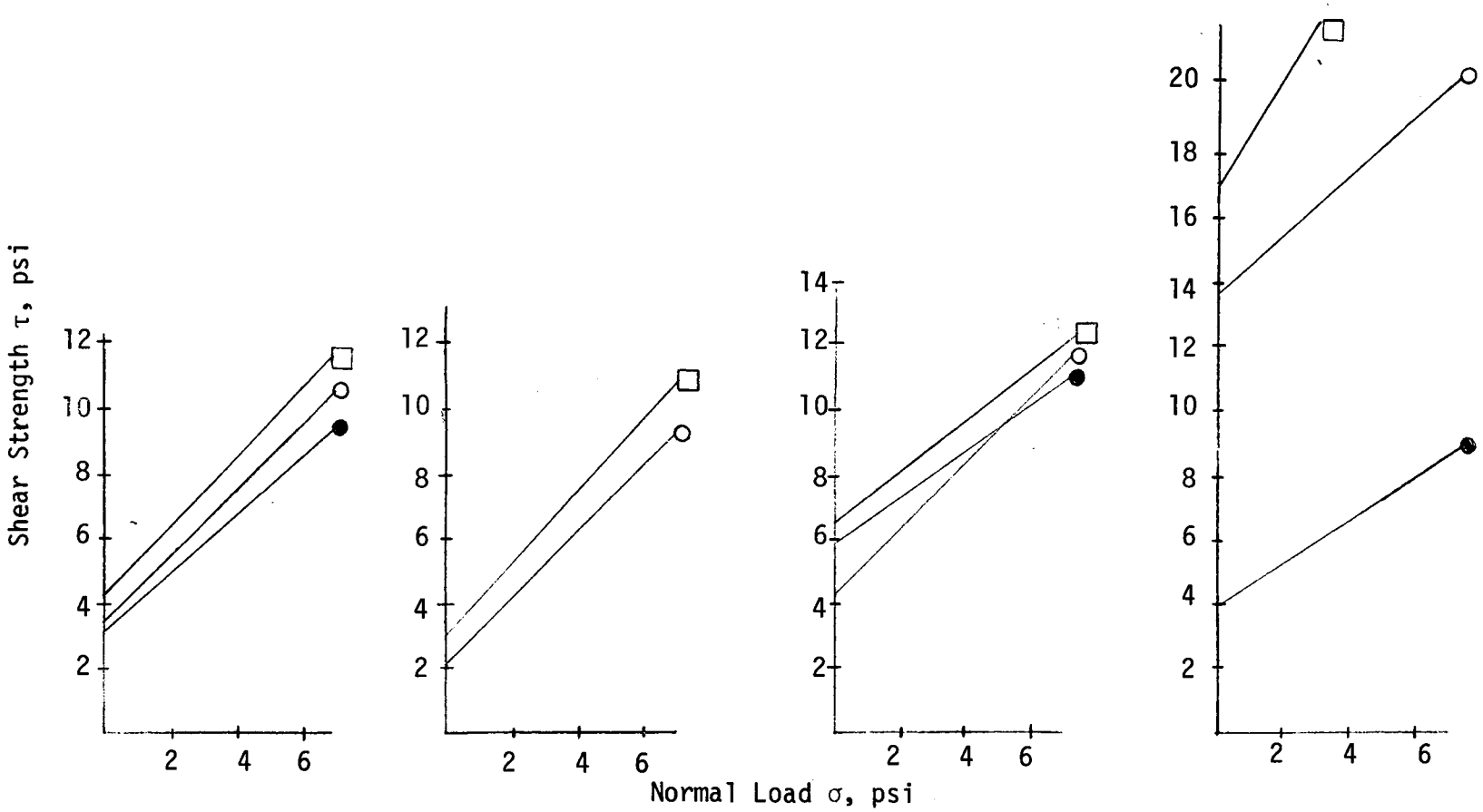


Figure 24. Sludge With No Additive.

Figure 25. Sludge With 0% Lime and 20% Fly Ash.

Figure 26. Sludge With 0% Lime and 40% Fly Ash.

Figure 27. Sludge With 6% Lime and 20% Fly Ash.

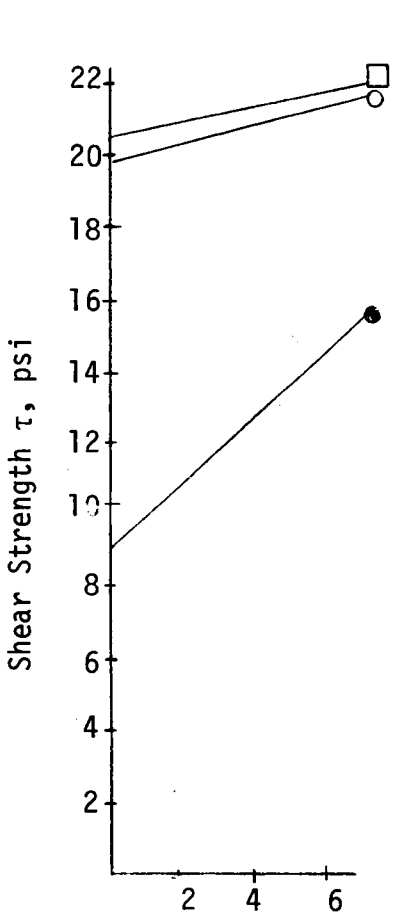


Figure 28. Sludge With 6% Lime and 40% Fly Ash.

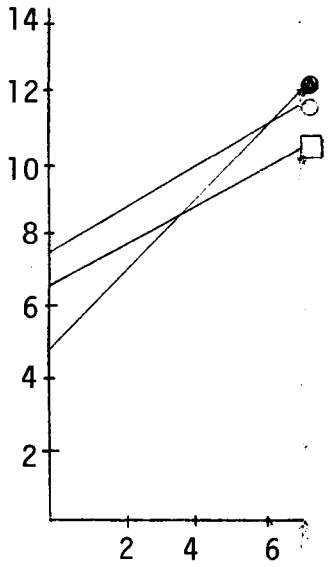


Figure 29. Sludge With 0% Lime and 20% Fly Ash.

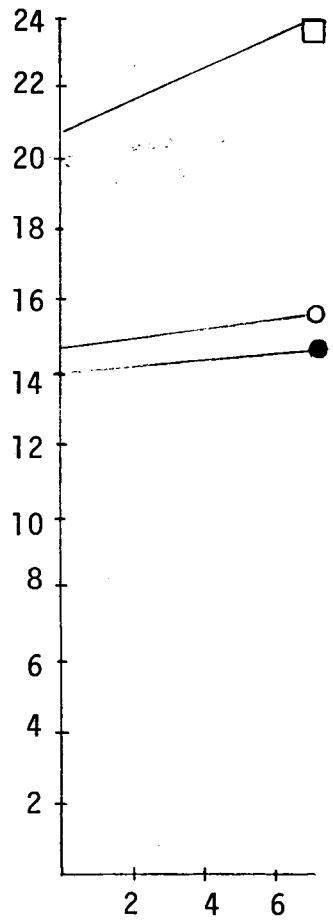


Figure 30. Sludge With 6% Lime, 40% Fly Ash, and 15% Silica.

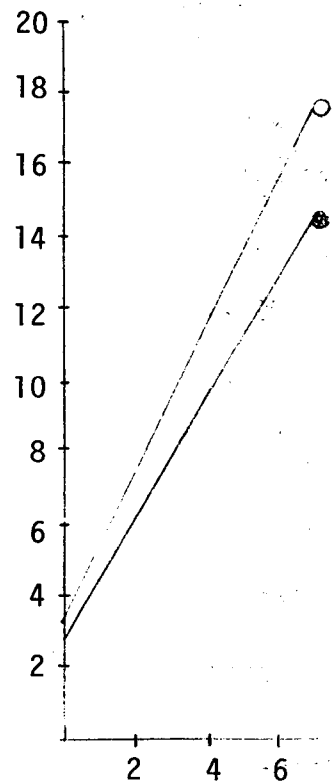


Figure 31. Sludge With 6% Lime, 20% Fly Ash, and 8% Silica.

Normal Load σ, psi

APPENDIX B

Figures 32 through 40 represent the shear stress versus the normal loads for sludge B added to a different amount of lime and fly ash (moisture content not controlled).

Key:

○ 2 Weeks

□ 4 Weeks

■ 6 Weeks

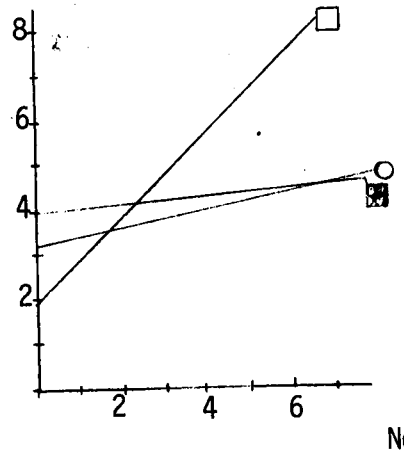


Figure 32. Sludge Without Any Additive.

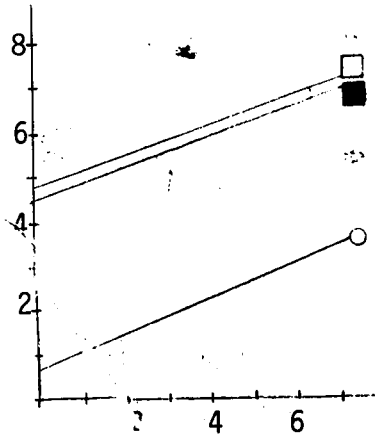


Figure 33. Sludge With 3% Lime and 0% Fly Ash.

Shear Stress τ, psi

Normal Load σ, psi

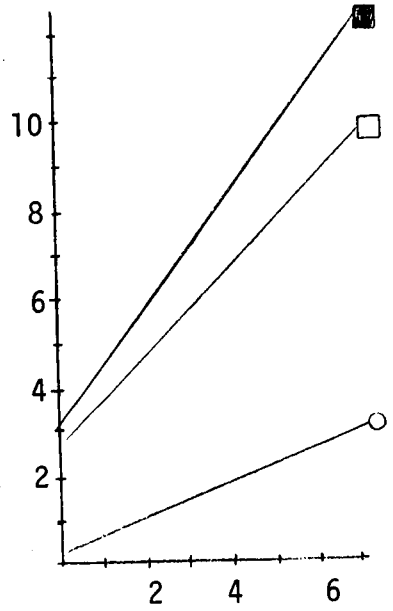


Figure 34. Sludge With 6% Lime and 0% Fly Ash.

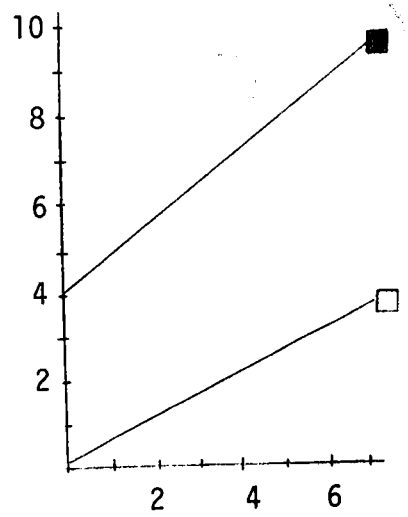


Figure 35. Sludge With 0% Lime and 40% Fly Ash.

Normal Load σ, psi

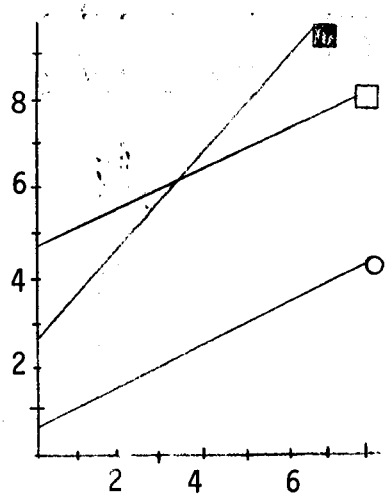


Figure 36. Sludge With 3% Lime and 10% Fly Ash.

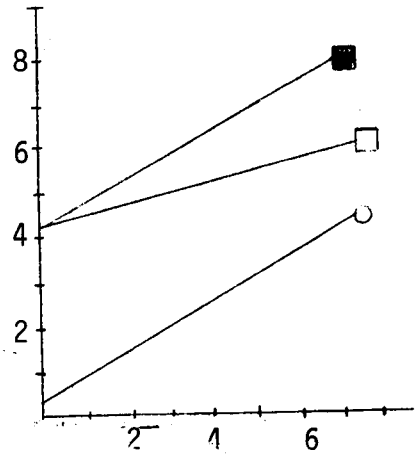


Figure 37. Sludge With 3% Lime and 20% Fly Ash.

Shear Stress τ, psi

Normal Load σ, psi

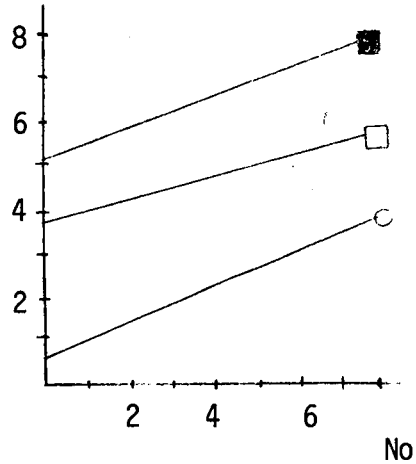


Figure 38. Sludge With 3% Lime and 40% Fly Ash.

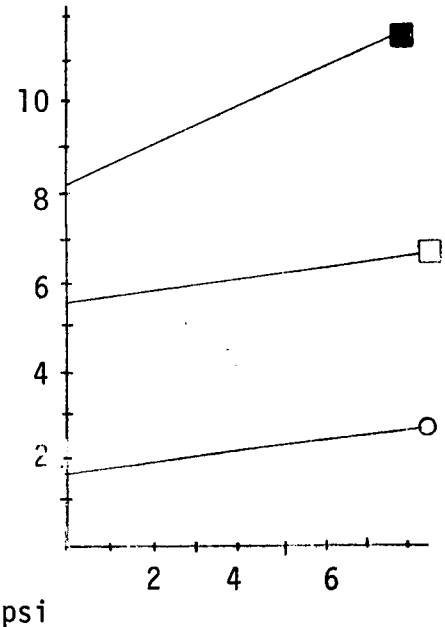


Figure 39. Sludge With 3% Lime and 60% Fly Ash.

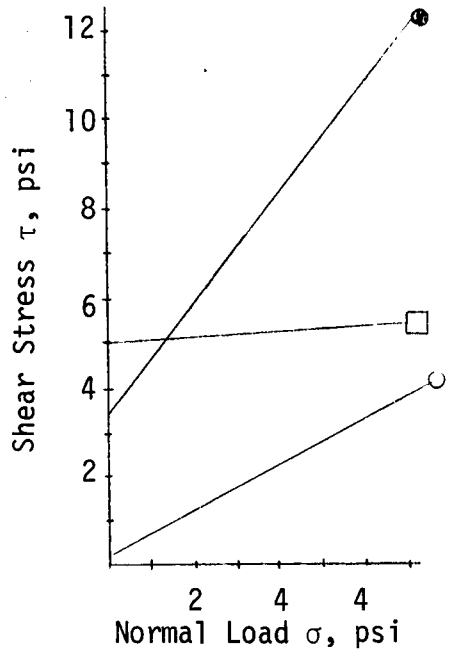


Figure 40. Sludge With 6% Lime and 40% Fly Ash.

TABLE XIII

BUCHNER FUNNEL TEST FOR SLUDGE A WITH INITIAL SOLID CONCENTRATION AT 17%

	% Fly Ash Added															
	0		10		20		30		50		70		90		150	
Time (sec)	Fil. (ml)	t/v (sec/ml)	Fil. (ml)	t/v (sec/ml)	Fil. (ml)	t/v (sec/ml)	Fil. (ml)	t/v (sec/ml)	Fil. (ml)	t/v (sec/ml)	Fil. (ml)	t/v (sec/ml)	Fil. (ml)	t/v (sec/ml)	Fil. (ml)	t/v (sec/ml)
0	3	0	25	0	3	0	2	0	3	0	3	0	3	0	3	0
5	12	.42	12	.42	13	.38	16	.31	23	.26	24	.23	20	.33	13	.38
10	23	.43	31	.32	31	.32	34	.29	39	.27	33	.33	36	.32	27	.37
15	32.5	.46	42	.36	42.5	.35	46	.33	47	.34	47	.34	47	.36	37	.41
20	40	.5	52	.38	52	.38	55	.36	56	.37	56	.38	56	.39	45	.44
25	47	.53	61	.41	61	.41	64.5	.39	65	.40	65	.40	67	.40	53	.47
30	53.5	.56	70	.43	68	.44	73	.41	72	.43	70	.45	74	.43	59	.51
35	59.5	.59	77	.45	76	.46	80.5	.43	78.5	.46	78	.47	82	.45	66.5	.53
40	65	.62	83.5	.48	82	.49	-	-	85	.48	84	.49	87	.49	72	.56
45	70	.64	90	.5	88	.51	93.5	.48	91	.51	90	.52	90	.53	77	.58
50	75.5	.66	96.5	.52	94.5	.53	97	.52	96	.53	-	-	92	.57	81	.62
55	80	.69	100	.55	98	.56	[illegible]	.56	98	.58	94	.60	93	.63	83.5	.66
60	85	.71	102.5	.59	100	.6	100	.6	100	.62	95	.65	94	.67	84.8	.71
65	89	.73	104	.63	102	.64	101	.64	102	.65	96	.7	-	-	85.5	.76
70	93.5	.75	105	.67	102.5	.68	101.8	.69	102.5	.7	-	-	-	-	86	.81
80	100	.8	106	.74	103.8	.77	102.3	.78	103	.80	96.5	.86	-	-	86	.93
90	104	.86	106.5	.85	104	.87	-	-	-	-	96.7	.96	-	-	-	-
b*	.00475		.0031		.0034		.0031		.0037		.0036		.00193		.00423	
w*	.256		.279		.308		.329		.386		.452		.494		.694	
r*	9.41×10^6		5.63×10^6		5.59×10^6		4.77×10^6		4.856×10^6		4.035×10^6		1.98×10^6		3.087×10^6	

*$r = \frac{2PA^2b}{\mu w}$; b (sec/ml^2), w ($g/c.c.$), r (sec^2/g).

TABLE XIV

BUCHNER FUNNEL TEST FOR SLUDGE A WITH INITIAL SOLID CONCENTRATION AT 33%

	% Fly Ash Added																			
	0		5		10		20		30		50		70		90		150	
Time (sec)	Fil. (ml)	t/v (sec/ml)	Fil. (ml)	t/v (sec/ml)	Fil. (ml)	t/v (sec/ml)	Fil. (ml)	t/v (sec/ml)	Fil. (ml)	t/v (sec/ml)	Fil. (ml)	t/v (sec/ml)	Fil. (ml)	t/v (sec/ml)	Fil. (ml)	t/v (sec/ml)	Fil. (ml)	t/v (sec/ml)
0	2.5	0	2.5	0	2	0	2	0	2	0	2.5	0	2.5	0	2.5	0	2.5	0
5	7.5	.66	9	.55	6	.83	6	.83	9	.55	9	.56	11	.45	8	.63	7	.71
10	14	.71	19	.53	13.4	.74	-	-	17	.59	12	.83	18	.55	16	.63	13	.77
15	20.5	.73	27	.55	20	.75	20	.75	25	.6	24	.63	24	.63	22	.68	17	.88
20	25	.8	32	.63	25	.80	25	.80	31	.65	29	.70	28	.71	27	.74	21	.95
25	30	.83	37	.68	30	.83	30	.83	36	.69	34	.74	33	.76	30	.83	23.5	1.06
30	33.5	.9	42	.71	34	.88	34.5	.87	40.5	.74	37.5	.80	36	.83	33	.91	26	1.15
35	37	.95	46	.76	37	.95	37.5	.93	44	.80	41	.85	39	.90	36	.97	27.5	1.27
40	41	.95	49	.82	41	.98	42	.95	47	.85	43	.93	41	.98	38	1.05	29	1.38
45	44	1.02	51.5	.87	45	1	45	1	49	.92	45	1	42.7	1.05	39	1.15	30	1.5
50	47	1.06	54	.93	47.5	1.05	47	1.06	50.5	.99	46	1.09	44	1.14	40	1.25	31	1.63
55	50	1.1	55	1	50	1.1	49.5	1.11	51.5	1.06	47	1.17	-	-	41	1.34	31.5	1.75
60	53	1.13	57	1.05	52	1.15	5.15	1.16	52	1.15	48	1.25	45	1.33	41	1.46	32	1.88
70	56	1.25	57	1.22	54	1.30	53.5	1.31	53	1.32	-	-	46	-	42	1.67	32.5	2.15
80	58	1.38	-	-	55	1.36	55	1.36	53.5	1.40	49	1.63	46.5	1.61	42	1.90	33	2.42
90	60	1.50	-	-	57	1.57	55.5	1.44	54	1.48	49	1.73	47	1.91	42.7	2.11	-	-
b*	0.01072		0.0105		0.0107		.0096		.0089		.0047		.0149		.0134		.0208	
w*	.967		1.036		1.070		1.270		1.375		1.664		1.998		2.389		3.908	
r*	5.6×10^6		5.1×10^6		5.05×10^6		3.84×10^6		3.27×10^6		1.43×10^6		3.78×10^6		2.86×10^6		2.696×10^6	

*$r = \frac{2PA^2b}{\mu w}$; b (sec/ml^2), w (g/c.c.), r (sec^2/g).

TABLE XV

BUCHNER FUNNEL TEST FOR SLUDGE A (THE TOTAL SOLID CONCENTRATION AT 34%)

	% Fly Ash Added											
	0		10		30		50		70		90	
Time (sec)	Fil. (ml)	t/v (sec/ml)	Fil. (ml)	t/v (sec/ml)	Fil. (ml)	t/v (sec/ml)	Fil. (ml)	t/v (sec/ml)	Fil. (ml)	t/v (sec/ml)	Fil. (ml)	t/v (sec/ml)
0	2	0	2	0	2	0	3	0	2	0	2.5	0
5	8.5	.59	-	-	12	.48	13	.30	13.5	.37	14.5	.42
10	19	.53	22	.46	22.5	.49	23.5	.49	27	.37	28	.39
15	27	.56	32.5	.46	31	.52	32.5	.51	37	.41	39.5	.41
20	34	.59	41.5	.48	38	.56	41	.53	46	.44	47.5	.45
25	41	.61	48	.52	45	.58	47.5	.56	54	.46	56	.47
30	47	.64	54	.56	51	.61	53	.6	61	.49	63	.50
35	52	.67	57	.61	55	.66	58	.64	63	.56	68	.53
40	53.5	.75	59.5	.67	58	.71	61	.66	65	.62	-	-
45	55	82	61	.74	-	-	62.8	.72	66	.68	73	.64
50	56	89	62	.81	62	.84	64	.82	67	.74	74	.70
55	57	95	-	-	62	.92	64.8	.89	67.2	.82	75	.76
60	57.8	1.04	63	.95	63	.98	65	.97	-	-	75.2	.83
65	-	-	63.5	1.02	63	1.01	-	-	-	-	-	-
70	58	1.21	-	-	63.5	1.14	65.8	1.12	-	-	-	-
b*	.00429		.00417		.00438		.00458		.00354		.00422	
w*	1.039		.957		.941		.917		.863		.761	
r*	2.324×10^6		2.449×10^6		2.598×10^6		2.79×10^6		2.28×10^6		$.309 \times 10^6$	

VITA

Dick Yu-Gen Chen was born in Fu-Chang Province, China, on June 2, 1945. He attended Chung Yuan Christian College of Science and Engineering, Taiwan, China, completing the requirement for a Bachelor of Science Degree in Chemical Engineering in June 1971. He also attended Seattle University, Seattle, Washington, and earned his second Bachelor of Science Degree in Clinical Chemistry in December 1972. Following graduation he worked as research assistant at Virginia Mason Research Center in Seattle, Washington. He came back for his graduate study at the University of Tennessee in September 1975, completing his Master of Science Degree in Environmental Engineering in June 1977.