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ABSTRACT
Evaluating the Homogeneity
of Solidification/Stabilization Treated
Waste Soils by Chemical Analysis Method

by
Mei Liu

Solidification/Stabilization technology has been widely used for waste treatment, especially for heavy metals, non-volatile wastes, radioactive wastes and soils. Homogeneity is a very important criterion for this kind of technology.

This study focuses on the development of a method to evaluate the homogeneity of S/S treated soil. A chemical analysis method, atomic absorbance, is employed to evaluate the distribution of S/S reagent in a soil-cement mixture and thus the uniformity of the soil-cement mixture. Also, a fluorescent tracer method is discussed in this study, which can be used to estimate the homogeneity of the soil-cement surface. The fluorescent tracer method might be used as a field testing method. The homogeneity of the surface is expected to be representative of the uniformity of the whole bulk. In our study, the chemical analysis method is the basis for comparison for the fluorescent tracer method.

**EVALUATING THE HOMOGENEITY OF
SOLIDIFICATION/STABILIZATION TREATED
WASTE SOILS BY CHEMICAL ANALYSIS METHOD**

**by
Mei Liu**

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Waste Soils by Chemical Analysis Method

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CHAPTER 1 INTRODUCTION

1.1 Introduction to CFS Techniques

Chemical fixation and solidification/stabilization, CFS for short, technology has existed commercially for more than 20 years, but only recently has it gained much attention, principally due to the growing number of statutes and regulations that require its use. The regulations promulgated under the Resource Conservation and Recovery Act (RCRA) have forced industry to look at alternative approaches for the disposal of hazardous wastes. Essentially, RCRA has stipulated standards for the siting, design and operation of the disposal facilities. CFS is one of the techniques that can fixate or encapsulate wastes into a solid, cementitious matrix[1].

Solidification/stabilization techniques chemically and physically bind the waste with a solidification agent [1]. Together, solidification/stabilization treatment systems: (1) improve the handling and physical characteristics of the waste; (2) decrease the surface area across which transfer or loss of contained pollutants can occur; and (3) limit the solubility of or detoxify any constituents contained in the waste [2].

Solidification techniques have been used in Japan and Europe for many years. In the United States, solidification techniques have been practiced primarily with radioactive wastes, while less environmentally acceptable methods have usually been used for hazardous wastes [3]. The products of CFS treatment meet certain regulatory requirements for physical stability and leaching of hazardous constituents.

Mixing is a critical element of any CFS process. The effects of mixing on the final chemical and physical properties of CFS solids are also important, as it seems obvious that the thorough dispersion of the CFS reagents in the waste is very important. This will be further discussed in the later section on the CFS mechanism. In fact, if the CFS reagent is not mixed well in the treated mixture, part of the

hazardous waste will not be fixed by this reagent, and the efficiency of the solidification or fixation will be poor. Any waste which is not mixed with the CFS reagent could result in a major leaching problem.

1.2 Objective of Study

The objective of this study is to develop a test method to evaluate how well mixed the CFS treated waste soil is. Two different methods are employed. The first method measures the dispersion of the CFS reagent by chemical analysis. The second method measures the distribution of a tracer in the CFS reagent. A fluorescent additive is used as a tracer and is well-mixed with the CFS reagent first; in this study, Type I Portland Cement is used since it is the most common case. The distribution of the CFS reagent or tracer can be used to assess how well mixed the sample is.

For this study, the chemical analysis method was developed from both the EPA AA Test Method for Ca component in soil and the ASTM standard method for Cement content of soil-cement mixture. The major component of Portland Cement is CaO, while the soil used in this study, consisting of Kaolin clay and Ottawa sand, does not contain CaO, or is non-detectable. Therefore, the dispersion of cement in the CFS product is measured directly by determination of the dispersion of calcium oxide (CaO).

The tracer is a fluorescent powder which is thoroughly mixed with the CFS reagent (Portland cement in this study) prior to the application of the CFS reagent to the waste. The distribution of the tracer can be observed by viewing under ultraviolet (UV) light in a dark room. The statistical evaluation of the homogeneity of the surface is accomplished by computer video image analysis.

The tracer method can be easily used to evaluate the mixing index of CFS products either on site or in the mixing process. It is a simple and economic method.

On the other hand, the chemical analysis method is both complex and expensive, and is affected by the different kind of CFS reagents used and waste contaminants.

There is an assumption that the degree of uniformity of the surface reflects the homogeneity of the whole bulk. Obviously if bad mixing occurs in the surface, the bulk of the product is probably not mixed well. This study tests this assumption as well.

CHAPTER 2 BACKGROUND

2.1 Knowledge of Soil Cement

Soil cement is defined as a mixture of soil and measured amounts of Portland cement and water compacted to a high density. It can further be defined as a material produced by blending, compacting, and curing a mixture of soil/aggregate, Portland cement, possibly admixtures including pozzolans, and water to form a hardened material with specific engineering properties. Soil cement is sometimes referred to by other terms, including soil stabilization, cement-treated aggregate base, and rammed earth [4].

Almost any inorganic type of soil is suitable for soil cement. Soil cement material has many applications. For about 60 years, soil cement has proved its effectiveness as a base material for pavements. Today, it is being used for many other applications and, considering its versatility and economy, soil cement use should continue to grow. It is summarized as following:

- **Pavements:** Soil cement's primary application is as a base material underlying bituminous and concrete pavements and parking lots [4].
- **Slope Protection:** Based on laboratory studies that indicated soil cement made with sandy soils could produce a durable erosion-resistant facing. According to the U.S. Bureau of Reclamation research report, soil cement has been used successfully as slope protection for channels and streambanks exposed to lateral flows.
- **Liners:** Soil cement is not only an economic liner material but also becomes less permeable during exposure to various wastes [5].
- **Foundation stabilization:** Soil cement has been used as massive fill to provide foundation strength and uniform support under large structures [4].

2.2 Mixing Procedures for Soil Cement

The performance of such projects has depended on the proper design and quality of the soil-cement material produced during construction. There are two methods specified for cement stabilized soil:

- Mix-in-place procedure, using various traveling machines that both pulverize and mix in-situ soil material with cement and water
- Central-plant procedure, using a stationary pugmill or other continuous-type mixer that combines the Portland cement and water with stockpiled, borrowed soil/aggregate material

Actually, there are many companies that claim their mixer can do an excellent job of mixing a variety of soil materials with Portland cement[32]. However, the overall quality of the mixed materials can be affected by several other factors:

- accuracy in spreading Portland cement on the in-place soil material
- the number of passes required to thoroughly mix the Portland cement, soil, and water into a uniform material with optimum moisture content
- mixer grade control - mixing depth will affect the final product
- stabilizing a wide range of silty and clayey soils that are difficult.

2.3 Solidification/Stabilization Techniques for Contaminated Soil

2.3.1 History of solidification/stabilization Techniques

Solidification/stabilization binding processes have developed from man's attempt to better navigation or transportation. In ancient times (3,000 B.C.) the Chinese Dschou dynasty had customs for road construction [6]. In the United States, road construction methods were refined to include stabilization with lime. Waste treatment by S/S processes can be tracked back to the disposal of low-level radioactive waste in the

1950s. These processes were optimized. Guidance for S/S treatment processes involving low-level radioactive treatment appeared in the later 1970s and 1983 [7].

2.3.2 S/S Techniques for Contaminated Soils

Contaminated soil can be treated by solidification stabilization reagents to form a soil cement material; therefore, contaminated soil can be reused. The treatment of contaminated soil is a special case of CFS technology. Waste-soil treatment is becoming increasingly more important in the CFS field as remedial programs become more active. The wastes are generated in ways very different from the typical waste water treatment sludge, and so their compositions, especially with regard to metal speciation and distribution, are different. Natural soils contain clay, rock, silt, sand and many natural organic substances. When they become contaminated, it is usually by infiltration of metal species (and other hazardous components) in solution. Such contaminants may originate in accidental spills, from deliberate dumping, or leaching of older landfills. Because of the structure and characteristics of soil, soluble salts are often distributed throughout large, hard species of clay and porous rock. The uniform distribution of CFS reagent is more important for contaminated soil cases.

It is widely assumed that very thorough and intimate mixing is needed to assure that a reaction between contaminated soil and CFS reagent will take place and solidification will be complete. However, it is known that this does not happen with most in situ technology. The uniformity of mixing of solids is always evaluated by experience. Some research was done on solid mixing homogeneity many years ago [8]. It used a radioactive tracer to measure the uniformity of concrete.

Compared to a radioactive tracer, a fluorescent tracer is a better choice for solidification systems because it is safer and less hazardous. In this study, an

inorganic fluorescent powder is used [9]. It is very easy to detect under UV light, so that the amount being used is very small.

CHAPTER 3 LITERATURE REVIEW

3.1 Contaminated Soil

Soil contamination is a common consequence of a range of activities essential to modern industrial societies. Metal present in excessive concentrations in soil may give rise to two kinds of adverse effects. Plant growth may be restricted or prevented, and/or the health of humans or grazing animals may be affected. Examples of metals which affect plant growth are copper, nickel, and zinc; effects on health may result from the presence of metals such as arsenic, cadmium, lead and mercury. Many other metals may also be present on contaminated sites, such as chromium, iron, manganese and so on.

There are many other kinds of contaminants in soil, such as organic compounds, inorganic compounds, and radioactive materials. There are also various general types of technologies to clean up different contaminants, such as physical treatment, chemical treatment, and biological treatment. These technologies summarized in Table.1. The major treatment alternatives are discussed below.

For metal contaminants, solidification/stabilization is the best choice, but this technology requires uniformity of wastes in the soil and homogeneity of the mixing with the solidifying or stabilizing reagents.

3.2 Treatment Alternatives

Soil clean-up operations have been undertaken for several years. There are various techniques employed in the treatment of contaminated soil either on-site or off-site, such as thermal treatment, extraction, biological, and chemical stabilization/solidification techniques.

3.2.1 Thermal Treatment Techniques

3.2.1 Thermal Treatment Techniques

A thermal treatment process removes organic or volatile contaminants by indirectly or directly heating the soils and solids to a temperature sufficient to vaporize the hazardous component. The vapors in the desorber off-gas are treated either by oxidation in a high temperature combustion chamber or by condensation and conventional treatment of the small amount of the resultant condensate. A thermal separation system (indirectly heating) for treatment of contaminated soils at temperature of 340-455°C has been described [10]. This technology was demonstrated in the EPA-SITE (Superfund Innovative Technology Evaluation) program [11]. Demonstration of the technology on soil contaminated with volatile organic compounds [12] was funded by the U.S. Army Toxic and Hazardous Materials Agency.

Two new processes using direct heating of soil to volatilize contaminants at low temperatures have been described [13]. One was demonstrated in the EPA-SITE program, and the other is scheduled to treat Wankegan Harbor sediment.

Thermal treatment for decontaminating soils and solids can be selected based on projected technical performance and cost. Obviously, it can not be used to treat heavy metal or other non-volatile contaminants.

3.2.2 Extraction Techniques [14-20]

Supercritical fluid (SCF) extraction is another technique for separating relatively non-volatile materials.

Typically in SCF extraction a solvent gas such as carbon dioxide, at high pressure and moderate temperature, is contacted with a solid or liquid phase. Slight changes in the system temperature or pressure can cause large changes in the solvent density and consequently in its ability to make relatively nonvolatile components soluble. Thus, manipulation of system temperature and pressure can be useful in extraction and

separation of substances. The many advantageous properties of SCF extraction have opened up new technologies in environmental control; the subject has been reviewed by Groves et al., and Modell.

3.2.3 Bioremediation Techniques

There is a growing interest in biological technology for reclamation of contaminated land and water. In situ bioremediation is based on the stimulation of the natural degradation processes in the soil. There are some disadvantages of this method such as:

- can only be applied to biodegradable components
- may not work for subsoil with low permeabilities
- can be inhibited by toxic components
- soil can be clogged by excessive bacterial growth
- residual concentrations will remain in the subsoil
- treatment necessary for a relatively long period
- long-term effects insufficiently understood

In-situ bioremediation is a technique under development, and experience on a demonstration scale has been gained mainly in the United States. The two main conditions for a successful in situ biodegradation are high soil permeability and biodegradable contaminants. There are a number of obstacles to the further development and optimization of this technology, especially in the areas of availability, oxygen supply and behavior, and inoculation of microorganisms.

3.2.4. Solidification and Stabilization Techniques

a. General Concept

In the past few years, there has been a growing interest in the chemical stabilization/solidification technology. It has been tested under the EPA's new Superfund Innovative Technology Evaluation (SITE) program.

Stabilization is one of the most economical methods for neutralizing hazardous contaminants and improving a waste's physical characteristics. Because of its performance, stabilization has been designated best demonstrated available technology (BDAT) for treating many listed wastes, especially those containing heavy metals. Recent research also indicates some stabilization reagents are effective in reducing the leachability of some organics [21,27].

Stabilization can reduce transportation and landfill costs. Stabilization is a process where formulated reagents combine with waste to maintain contaminants in their most immobile form. The technology's goal is reducing a waste's solubility or chemical reactivity through the addition of specific reagents and mixing. A wide range of reagents is available for stabilizing both organic and inorganic contaminated wastes. Examples include oxidation-reduction agents, complexing agents, and such chemical absorbents as ion-exchange resins, activated carbon and organophilic clays.

Solidification is a physical process that converts waste into an easily handled solid, significantly reducing its leaching potential. The resulting product is a solid, impermeable matrix with high structural integrity.

Various techniques are available for solidifying waste products. The technology's limitations are few and primarily result from large amounts of organic contaminants in the untreated material. In some cases, organics can reduce the effectiveness of the process. This typically results in lower compressive strengths in the final material [21].

In general, chemical treatment of hazardous waste consists of two stages, stabilization and solidification. Stabilization converts the toxic components to stable forms that will resist leaching. Solidification, on the other hand, can be accomplished

by two different processes: encapsulation, where a chemical reaction agent physically surrounds the waste particles, or chemical fixation, where a chemical reaction occurs between the waste and solid matrix. The Portland cement method is an example of the former. Once a hazardous waste is successfully solidified, it can be disposed of in the landfill.

Solidification/stabilization techniques have been widely used in low level radioactive waste disposal. Their application to hazardous wastes is becoming more common, however, and many vendors are studying and developing processes that are directly applicable to hazardous waste-contaminated soils and sludges.

Waste solidification/stabilization systems that are potentially useful in remedial action activities are as follows:

- Pozzolan-Portland cement systems
- Lime-fly ash pozzolan systems
- Thermoplastic microencapsulation
- Sorption
- Organic binding

An extensive discussion of these and other solidification/stabilization techniques is presented in the document entitled "Handbook for Stabilization/Solidification of Hazardous Wastes" [22]. Also, Malone et al. [23] present detailed information concerning the solidification/stabilization of hazardous wastes. Physical and chemical testing and technology evaluation procedures are discussed in another report [24]. The most significant challenge in applying solidification/stabilization treatment in situ for contaminated soil is achieving complete and uniform mixing of the solidifying/stabilizing agent with the soils.

In situ mixing of solidifying/stabilizing agents with contaminated sludges or soil is difficult. However, uniformity of the waste-reagent matrix is also important. Proper

mixing or degree of contact between the waste, soil, and solidification/stabilization reagent depend on the following parameters [25].

- Viscosity of the agent
- Permeability of the waste materials and soils surrounding them.
- Porosity of the waste materials and soils.
- Spatial distribution of the wastes in the surrounding material (i.e., soil, rocks).
- Rate of the reactions.

We summarize some of the advantages and disadvantages for solidification/stabilization techniques as follows:

The advantages of this treatment technology are as follows:

- Additives and reagents are widely available and relatively inexpensive.
- The resulting solidified material may require little or no further treatment if proper conditions are maintained.
- Leaching of contaminants is greatly reduced.

The disadvantages of this treatment technology are as follows:

- Volume of the treated material may increase with the addition of reagents.
- Delivering reagents to the subsurface and achieving uniform mixing and treatment in situ may be difficult.
- Volatilization and emission of volatile organic compounds may occur during mixing procedures.

b. Cement-Based Stabilization/Solidification

Much of the stabilization/solidification that now occurs in the USA is based on the chemistry of lime or cement. CFS technology can be grouped as either inorganic stabilization/solidification - cement-based and pozzolanic; or organic stabilization/

solidification - thermoplastic and organic polymerization. Inorganic stabilization/solidification with cements and pozzolans has been used for hazardous waste treatment more often than the other technologies.

Cement-based stabilization/solidification is a process in which waste materials are mixed with Portland cement. Water is added to the mixture, if it is not already present in the waste material, to ensure the proper hydration reactions necessary for bonding the cement. The wastes are incorporated into the cement matrix and, in some cases, undergo physical - chemical changes that further reduce their mobility in the waste - cement matrix. Typically, hydroxides of metals are formed, which are much less soluble than other ionic species of the metals.

Cement-based stabilization/solidification has been applied to plating wastes containing various metals such as cadmium, chromium, copper, lead, nickel, and zinc. Cement has also been used with complex wastes containing PCBs, oils, and oil sludge; wastes containing vinyl chloride and ethylene dichloride; resins; stabilized/solidified plastics; asbestos; sulfides; and other materials [26]. Studies performed under the BDAT programs on contaminated soil showed cement-based stabilization/solidification effective for arsenic, lead, zinc, copper, cadmium, and nickel [26].

Because laboratory leaching tests usually involve standardized aqueous solutions (neutral, buffered, or dilute acidic solutions) rather than site-specific solutions, the results of the laboratory tests may not directly duplicate leaching in the field. As previously mentioned, laboratory leaching tests run with standard solutions can be used to compare the relative leachability of waste constituents under similar test conditions and similar solutions.

Depending on the physical and chemical properties of the waste and leaching solution, the kinetics of contaminant transport (or leaching) in a porous medium are controlled by advective or dispersive/diffusive mechanisms. Advection refers to the

hydraulic flow and subsequent solute transport of highly soluble contaminants in response to a hydraulic gradient. Dispersion refers to the transport of contaminants via mechanical mixing in the pore solution and molecular diffusion (the transport for adsorbed or chemically bonded constituents is generally considered to be controlled by molecular diffusion at the particular surfaces within the waste form, rather than by advection or dispersion).

Major selected remediation techniques, including some other treatments, possibly suitable for clean-up of contaminated soils are summarized in Table.1. Obviously, it is required to have completed and uniform mixing of the contaminated soils or other wastes with the reagent in CFS techniques.

Table 1. Selected remediation techniques possibly suitable for clean up of contaminated soils [33,34,35,36,37]

Type of treatment technology	Treatment category	Function	Possible application	Possible limitation
Thermal Stripping	In Tank; In situ	Separation	Compounds of low water solubility and high volatility	Limited to organics with Henry's Law constant greater than 3.0×10^{-3} atm-m ³ /mole and boiling point less than 800 °F; most effective for soils with low contents of organic matter and moisture
Soil Vacuum Extraction (SVE)	In situ; Prepared Bed	Separation	Volatile organics and toxic metals; May be enhanced by the use of steam	Soil heterogeneity (e.g., permeability, texture); Not applicable to saturated materials or miscible compounds
Fluidized Bed	In-Tank	Volume Reduction; Detoxification	Halogenated and nonhalogenated organics; Inorganic cyanides	High maintenance requirements; Waste size and homogeneity requirements; Applicable to wastes with low sodium and metal contents
Biological Treatment	In-Tank; Prepared Bed; In situ	Detoxification	Biodegradable organic wastes	Maintenance of optimum environmental conditions for biological activity; Requires large amounts of compost materials mixed with only about 10% wastes

Cement Solidification	In-Tank; In situ	Storage; Immobilization	Metal cations; Latex and solid plastic wastes	Incompatible with large amounts of dissolved sulfate salt or metallic anions such as arsenates or borate; Setting time increased by per-cente of organic matter, lig-nite,silt, or clay; Requires complete and uniform mixing of soils and reagents; May reduce soil permeability and increase run-off
Lime Solidification	In-Tank; In situ	Storage; Immobilization	Metals; waste oils; and solvents	Long-term stability unknown; Incompatible with broates, sulfates, carbohydrates; Re-quires complete and uniform mixing of soils and reagents; May reduce soil permeability and increase run-off

3.3 Mixing Problem

Mixing is that unit operation in which energy is applied to some material for the purpose of altering the initial particle arrangement so as to effect a more desirable particle arrangement. The objective of this treatment is usually to blend two or more materials into a more homogenous mixture.

Based on the theory of probability and resulting from a study of mixing kinetics [28], application of analytical methods brought a new relationship between mixing time and mixing completion. Two kinds of models they used are shown in Figures 1 and 2. Figure 1 shows the initial state, in which two clearly defined layers of materials A and B are loaded.

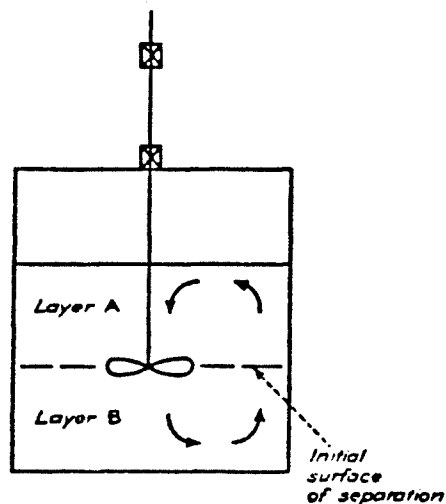


Figure 1. Simple mixer showing circulation pattern of mixture

The function of the agitation, once started, is to extend the initial plane or surface of separation between the layers. It is obvious that the extension of the interface between component A and B will vary quantitatively with time. A quantitative relationship between the instantaneous magnitude of the surface of separation and the mixing time was derived by Brothman [28].

Another approach from a practical point of view is given by Brothman, C. N. Wollan, and S. M. Feldman[28]. One of the basic statements is that if the volume of the batch were divided into a large number of units of equal volume (see Figure 2), a determination of the mixing condition existing could be made in terms of the uniformity of dispersion of the generated surface of separation among these units.

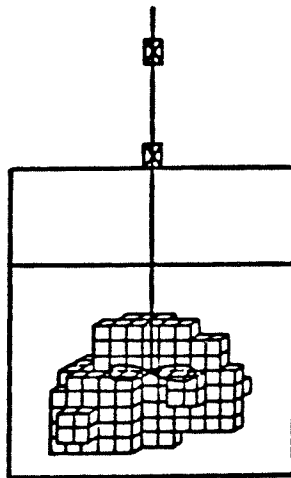


Figure 2 The mix broken down into a large number of equal sized cubes.

A practical and usable quantitative relationship between the dispersion of the elements and the time of mixing was brought out. According to their research, they conclude that (1) After any given elapsed mixing time, while there may exist a practical level of dispersion or mixture, nevertheless, in the absolute sense there will at all times be varying composition ratios among the component subdivisions of the batch volume. (2) If the properties of the "mix" components were reasonably close, the same device could be expected to give roughly identical performance with respect to the

time required to achieve mixing, providing the same degree of permeation of the batch by any given component is required.

These two models show that when you want to evaluate the homogeneity of a mixture, you should choose samples randomly in the whole system and evaluate each sample's properties. If each sample has the same properties, the mixture is uniform. Which kind of property should be chosen? Should we choose all physical properties or chemical properties to evaluate the homogeneity? And how to evaluate the properties which are chosen?

We want to develop a practical method to evaluate well-mixed CFS products. There are two methods employed in our study. One of them is a bulk or batch homogeneity test, another is a 'surface' homogeneity test. In our study, all the mixing parameters are fixed except mixing time. The test samples are chosen randomly for evaluation of homogeneity.

It is not necessary to measure all kinds of properties for determine the uniformity of the mixture. This will become clear when the concept of homogeneity is defined.

3.4 Homogeneity of Mixture & Mixing (Uniformity) Index [29]

3.4.1 Homogeneity

The objective of waste solidification is to convert it into a stable monolithic form which minimizes the probability of hazardous release to the environment during interim storage, transportation, and final disposal. The solidified waste should be of such chemical, mechanical, thermal, and radiolytic stability that its integrity can be assured over the time required for the decay of contained radio nuclides and chemical contaminants to acceptable levels.

One of the most fundamental physical properties required for any kind of immobilized waste form is homogeneity. This property is important in the

solidification process and during long-term storage. Furthermore, chemical and physical properties of immobilized waste such as density, porosity, leaching rate, matrix degradation, permeability, compression strength, radiation damage, thermal conductivity, etc. cannot be easily understood if the matrix is not homogeneous. Often during waste immobilization in laboratory scale the homogeneity condition can be assumed. However, in the scale up of the immobilization process this condition is mandatory and cannot necessarily be assumed.

In laboratory scale procedures, emphasis is given to the necessity of well homogenizing the mixture in order to obtain a uniform and reproducible product. In certain cases, compression tests are conducted on samples taken from a real site product. However, reliable, statistical tests are seldom applied to them.

Not all physical and chemical properties have to be uniform throughout the waste form. If the waste form is homogenous with respect to a certain property, it does not mean necessarily that is homogeneous with respect to some other properties. On the other hand, it is known that some properties do correlate in the sense that if one observes homogeneity for one property, the other will also be homogeneous. So a careful selection of a set of properties has to be made in order to assure that once acceptable levels of homogeneity hold for this set, then all relevant properties can be considered homogeneous.

No matter what set of properties is considered, these properties have to be measured according to statistical criteria.

3.4.2 Measuring Uniformity

Except for cases where a coating of one ingredient with another takes place, the theoretical end result of mixing will not be an arrangement in which one type of particle is directly next to a different type. With easily distinguishable particles which

can be counted, the variation between spot samples of a known size (i.e., number of particles) can be theoretically predicted for a random mixture and used as a guide to determine how closely random blending of the ingredients is approached [30]. Where individual particles cannot be easily distinguished and particle counts are not practical, various types of analyses can be made on spot samples to determine batch uniformity. Recent advances in instrumental analysis have made it much easier to give rapid and numerous analyses which are of great benefit for statistical analyses. Some of these analysis methods are X-ray fluorescence, flame spectrometry, polarography, and emission spectroscopy. Also, radioactive tracer methods have been used [30].

Obviously, the sampling procedure is important for evaluating the homogeneity. Method of sampling, location, size and number of samples, method of sample's analysis, and fraction of the batch removed for sampling all contribute to how well the sampling study reflects the actual conditions.

Regardless of the analytical methods chosen, whether gravimetric, volumetric, electrometric, particle counts, optical or other, it is very important that the data be objectively analyzed via statistical methods should there be any question as to the adequacy of the mixture. The analytical error should be small compared with the variation in the composition (or other property) between spot samples. Weidenbaum described many different ways of measuring uniformity [30].

We choose the flame spectrometric method (AA) and the fluorescent tracer method in our study, and use statistical methods while analyzing our data from both methods.

3.4.3 Mixing (Uniformity) Index^[31]

A mixture could be defined as homogeneous if any sample of the mixture has the same composition and properties as any other. It is necessary to specify the scale of homogeneity in terms of size of sample chosen for analysis. The scale of homogeneity

which should be employed in evaluating a particular mixing process will depend largely on the use to which the final mixture will be put.

The deviation of the mixture from true homogeneity can be used as a measure of the uniformity, which is defined as the mean deviation of the volume concentration of one component in a number of randomly selected samples (of fixed volume) from the mean concentration of component A in the mixture [31].

$$D_v = \sqrt{\frac{\sum (C_A - C_A^m)^2}{n C_A^{m,2}}}$$

where, C_A is the concentration of A in a random sample of volume v , and C_A^m is the mean concentration of A in the mixture, then D_v is the fractional root-mean-deviation, which D_v can be used directly as a measure of the mixture uniformity. A difficulty arises in its use, however, in that its value for the unmixed state is dependent upon the volume fraction of A added.

If D_v^o represents the value of D_v at no mixing, it was shown that,

$$D_v^o = \sqrt{\frac{1 - C_A^m}{C_A^m}}$$

Further, the Uniformity or Mixing Index was calculated by the value of I_v , which is the ratio of D_v to D_v^o . It can be used as measurement of mixture homogeneity, which has a value of unity for the completely unmixed state, and a value of zero for the completely mixed condition.

$$I_v = \frac{D_v}{D_v^o}$$

Also, I_v is independent of n , the number of sample taken, provided the samples are truly random and representative of the mixture. An analogous expression has been derived by Danckwerts [32].

According to these relations, the Mixing Index at any stage in a mixing process can be determined from (1) analysis of a number of random samples for one component being mixed, and (2) the knowledge of the mean concentration of that component.

In our study, the mixing index is employed, but the samples are taken randomly with the same mass not the same volume, because the weight percentage is used as the concentration expression.

CHAPTER 4 EXPERIMENTS

4.1 Experiment I. Titration Method for Cement Content of Soil-Cement Mixtures

In order to determine the cement content of soil-cement mixtures, the ASTM method D-806-89 is employed in this study. Because this method is used to determine the cement content in a hardened mixture of cement with soil, it is based on the determination by chemical analysis of the calcium oxide (CaO) content of the sample.

a. Apparatus

- (1) Analytical Balance: An Analytical balance with Class S weights.
- (2) Filter Paper: Filter paper including Whatman No. 1, 11 and 15 cm in diameter; Whatman No. 41, 15 cm in diameter; and Whatman No. 2 11 or 15 cm in diameter.
- (3) Fifty-milliliter-pipet.
- (4) Miscellaneous Apparatus: Supplementary equipment, such as electric ovens, hot plates, a small riffle, a No. 40-(425 μ m-)sieve with bottom pan and cover, a cast iron mortar and pestle.

b. Reagents

- (1) Purity of reagents: reagent grade chemicals be used in all tests.
- (2) Potassium Permanganate, Standard Solution (0.1N): Prepare and standardize a 0.1N KMnO_4 solution.
- (3) Ammonium Nitrate Solution: Dissolve 20g of NH_4NO_3 in 1 liter distilled water.
- (4) Hydrochloric Acid (1+3): Add 200ml of HCl (sp. gr. 1.19) to 600 ml of distilled water.
- (5) Hydrochloric Acid (1+1): Add 25ml of HCl (sp. gr. 1.19) to 25 ml of distilled water.
- (6) Ammonium Oxalate solution (5%): 50g of ammonium Oxalate

(7) Ammonium Hydroxide, NH_4OH (sp. gr. 0.90)

(8) Sulfuric Acid(1+10): Add 50ml H_2SO_4 (sp. gr. 1.84) to 500 ml of distilled water

c. Samples

(1) Samples of the following are selected for the test

- Soils - Consist of Kaolin clay and Ottawa sand (clay : sand = 1 : 2)
- Cement - Portland Type I Cement
- Soil-cement Mixture - Ratio is listed in Table 2

(2) The gross laboratory sample of each component shall be approximately 200g.

d. Procedure

- (1) Dry 25g of each of the samples in an oven to constant weight at 119°C to remove free moisture. Reduce the samples to pass a No. 40 sieve.
- (2) Weight out on the analytical balance, the following amounts of the samples: raw soil 5g; soil-cement mixture 5g; cement 1g. Place each of the weighted samples in a 250ml beaker. Add 50 ml of HCl (1+1) to each sample, cover and boil gently for 5 minutes on the hot plate.
- (3) Add 25ml of hot water to the beakers, stir, allow to settle momentarily, and then decant the contents through a Whatman No.1 filter paper. The filtrate should be received in a 250 ml volumetric flask. Wash it using hot water.
- (4) Dilute in a volumetric flask to 250ml with cold water. Agitate the flask to mix the contents thoroughly, then remove a 50ml aliquot and transfer to the original 250ml beaker using a 50ml pipet. Dilute to 100ml. Make the solution slightly ammoniacal, boil 1 to 2 min. and allow the hydroxide to settle.
- (5) Filter the hydroxides through an 11 cm Whatman No.1 (or No 41) filter paper, receiving the filtrate in the 600ml beaker. Wash the original 250ml beaker into the filter once with a stream of hot NH_4NO_3 solution (20g/l), and follow by washing

the hydroxides precipitate once or twice with hot NH_4NO_3 solution. Set the filtrate aside, and place the paper with a rod, and wash the hydroxides down into the original beaker, using a stream of hot NH_4NO_3 solution to remove most of the precipitate from the filter paper. Treat the paper with 20ml of hot HCl (1+3), directly dumping the acid over the paper with a glass rod. Wash the paper three times with hot water. Dilute the solution to 75ml.

- (6) Make the solution slightly ammoniacal and boil 1 to 2 min. Allow the precipitate to settle, then decant through a Whatman No.1 paper as before, receiving the filtrate in the 600ml beaker previously set aside. Wash and police the beaker in which precipitation took place, finally washing the precipitate on the filter three or four times with NH_4NO_3 solution. Discard the hydroxides precipitate. Add 2ml of NH_4OH (sp. gr. 0.9) to filtrate- which will now have a volume of 250-350ml. Heat the solution to boiling and add 10ml of hot oxalate saturated solution. Keep the mixture near boiling until precipitate becomes granular, then set aside on a warm hot plate for 30 min. or more. Before filtering off the calcium oxalate, verify completeness of precipitation, and make sure that a slight excess of NH_4OH is present.
- (7) Filter the mixture through an 11 cm or 15 cm Whatman No. 2 (or No. 42) filter paper, making sure that all the precipitate is being retained. Thoroughly clean with a rubber policeman the beaker in which the precipitation took place, and transfer the contents to the filter eight to ten times with hot water (not over 75ml) using a stream from the wash bottle.
- (8) Carefully open the filter paper and wash the precipitate into the beaker in which the precipitation was done. Dilute to 200ml and add 10ml of H_2SO_4 (1+1). Heat the solution just short of boiling, and titrate it with the standard KMnO_4 solution

to a persistent pink color. Add the filter paper and macerate it. Continue the titration slowly until the pink color persists for 10 sec.

- (9) Blank: Make a blank determination, following the same procedure and using the same amounts of all reagents.

The results are shown in the Table 2, 3 and Figure 3. Although there is a linear relationship between the CaO weight percent and the cement content in the soil mixture, this chemical analysis method is too complex and requires too much time, especially in the extraction step. Based on the linear relationship between cement content and CaO weight percent, another chemical analysis method was developed for this study, the Flame Atomic Absorption Spectroscopy Method for Calcium, which represents the cement component in the mixture. Also, an extraction procedure is substituted for the EPA digestion method [EPA 3150, 7140], since it requires less time and is efficient at extracting calcium.

4.2 Experiment II. Homogeneity Evaluation

4.2.1 Sample Preparation

a. Apparatus

- (1) Balance or scale, sensitivity 0.1g.
- (2) Mixer: electrical driven mechanical mixer, Hobart Mixer is employed in this study
- (3) Paddle: readily removable, made of stainless steel, see Figure III.3.
- (4) Mixing Bowl: removable mixing bowl with capacity of 5 liters, 10 liters.
- (5) Sampler: glass tube 20x1(long x diameter, cm) marked with same height.
- (6) Oven:
- (7) Mortar and pestle

b. Materials

- (1) Portland I Cement

- (2) Kaolin Clay
- (3) Ottawa sand
- (4) Water (Deionized)
- (5) Powder fluorescent tracer

c. Procedure for Mixing Pastes

- (1) Place the dry paddle and dry bowl in the mixing position in the mixer. Then introduce the materials into the bowl and mix in the following manner.
- (2) Mix the specific amount of fluorescent tracer with Portland cement; this should be checked under UV light in a dark room to ensure complete mixing.
- (3) Add water both to soil and cement according the ratio: clay : water = 1 : 0.3, and cement : water = 1: 0.5 individually.
- (4) Start the mixer at middle speed, and mix each of them completely in different mixer, soils mixed in 10-liter-volume Hobart Mixer, and Portland cement mixed in a 5-liter-volume Hobart mixer.
- (5) Mix the two layers of pastes and place soil paste at the bottom and cement paste at the top. Mixing speed is controlled at medium level.
- (6) Stop mixing at different mixing times, such as 5 seconds, 10 seconds, and so on.
See Table 4.

d. Procedure for Taking Samples

- (1) Each sample is taken by inserting a glass tube into the mixed pastes at a random location withdrawing approximately 5 grams.
- (2) Put all tubes in a oven at temperature 110°C for 4 hours.
- (3) Smash each sample in a mortar with a pestle. Mix each sample completely and take 1 gram from each sample for chemical analysis.

Each sample is tested both by Atomic Absorption Spectroscopy and by Fluorescent Imaging System [9] individually. Repeat for the same composition mixed with different mixing times. The data are shown in Table 2, 3, and 4.

4.2.2 Procedure for Atomic Absorbance Test

Generally, based on the AA test, the cement content distribution can be measured by the Ca distribution. Therefore, we designed our measurement by: (1) Take samples from each batch randomly, extract Ca from sample [EPA 3050, 7140], test Ca concentration in the mixture with AA method. (2) Calculate mixing index for each sample.

The Atomic Absorbance Method is a modified chemical analytical method ASTM C 114). A representative 1- to 2-g (dry weight) sample is digested in nitric acid and hydrogen peroxide. The digestate is then refluxed with either nitric acid. Dilute hydrochloric acid is used as the final reflux acid for the flame AA analysis of calcium.

a. Apparatus and Materials

- (1) Conical Phillips beaker: 250ml
- (2) Watch glass
- (3) Drying oven
- (4) Thermometer: range of 0 to 200°C
- (5) Whatman No. 41 filter paper (or equivalent)
- (6) Perkin-Elmer 305 B AA Instrument

b. Reagents

- (1) ASTM Type II water (ASTM D1193)
- (2) Concentrated nitric acid, reagent grade (HNO_3)
- (3) Concentrated hydrochloric acid, reagent grade (HCl)
- (4) Hydrogen peroxide (30%) (H_2O_2)

- (5) Lanthanum (0.1%) solution
- (6) Stock standard Calcium solution 1000 ppm

c. Sample Collection, Preservation, and Handling

All sample containers were prewashed with detergents, acid, and Type II water.

All samples were collected randomly from the whole mixer at each mixing condition. The manner was described above.

d. Procedure

- (1) Mix the sample thoroughly to achieve homogeneity. For each digestion procedure, weigh to the nearest 0.0001g and transfer to a conical beaker a 1.000-to 2.000-g portion of sample.
- (2) Add 10 ml of 1:1 HNO₃, mix the soil-cement, and cover with a watch glass. Heat to 95°C and reflux for 10 to 15 minutes without boiling. Allow the sample to cool, add 5 ml of concentrated HNO₃, replace the watch glass, and reflux for 30 minutes. Repeat this last step to ensure complete oxidation. Using a ribbed watch glass, allow the solution to evaporate.
- (3) After the last step has been completed and the sample has cooled, add 2 ml of type II water and 3 ml of H₂O₂. Cover the beaker with a watch glass and return the covered beaker to the hot plate for warming and start the peroxide reaction. Heat until effervescence subsides and cool the beaker.
- (4) Continue to add 30% H₂O₂ in 1-ml aliquots with warming until the effervescence is minimal or until the general sample appearance is unchanged. (DO NOT ADD MORE THAN 10 ml 30% H₂O₂.)
- (5) Filtration: Filter through Whatman No. 41 filter paper (or equivalent) and dilute to 100 ml with type II water. Particulate in the digestate removed by filtration.
- (6) The dilute digestate solution was ready for Ca analysis.

e. AA Operation Conditions

Light source: Hollow Cathode

Lamp Current: 7.0 ma

Wavelength: 422.7 nm

Bandpass: 1.0 nm

Flame Description: Air-Acetylene

The sensitivity is about 0.05 ppm., and the linear range for Ca is up to 3.0 ppm at the instrumental parameters described above. Because silicon, aluminum, phosphate and sulfate depress calcium's sensitivity, lanthanum at a concentration of 0.1% is added to all samples and standards to control these interferences brought by cement and soil samples.

The results are shown in Tables 5 to 10 and Figures 5 to 10. The concentration of calcium has a linear relationship with cement content in the soil-cement mixture by the AA test method (see Figure 4). Therefore, the cement distribution can be determined by evaluation of calcium distribution in the soil-cement mixtures.

4.2.3 Fluorescent Tracer Method

The experimental procedure for this method is described in the work by Gao [9].

CHAPTER 5
RESULTS AND DISCUSSION

5.1 Results

5.1.1 Results from Titration Method

In this experiment, soil-cement mixtures with different compositions of cement are prepared first. The composition of each sample is listed in the Table 2.

Table 2. Composition of Soil-Cement

Sample No.	Cement Wt. (g)	Kaolin Wt. (g)	Ottawa Wt. (g)	Water Wt. (g)
1	0.0	66.7	133.3	40
2	20.0	46.7	93.3	40
3	40.0	40.0	80.0	40
4	60.0	33.3	66.7	40
5	80.0	26.7	53.3	40
6	100.0	20.0	40.0	40
7	120.0	13.3	26.7	40
8	140.0	6.7	13.3	40
9	160.0	0.0	0.0	40

Under this experiment, CaO content in well-mixed soil-cement mixtures is determined by titration method, ASTM 114-c, Figure 3 shows that the composition of CaO in a hardened soil-cement mixture is proportional to the cement content in the soil-cement mixture. All of the data are listed in Table 3.

**Table 3. Titration Method Data
Cement content of the Soil-cement Mixture**

	A	B	C	D	CaO% (wt)
Cement(Portland I)	8.6	0.1	0.1	0.2	11.9
Soil(sand:clay=2:1)	0.16	0.1	0.1	1.0	0.0168
Soil-Cement 40% Cement	13.78	0.1	0.1	1.0	3.83
Soil-Cement 60% Cement	25.1	0.1	0.1	1.0	7.01
Blank	0.10				0.0

where,

$$\text{CaO\%} = ((A - B) \times 0.028 / D) \times 100$$

A = vol of KMnO_4 Titrated

B = vol. of Blank

C = 0.1 N (concentration of HNO_3)

D= for soil or soil-cement

= 0.2 for cement

5.1.2 Results from AA Test Method

• Standardizing Sample's Data

Based on the above results, there is a linear relationship between the percentage of CaO and the cement composition; thus we can use the CaO component to represent the cement content in soil-cement. Also, the distribution of CaO can represent the distribution of cement.

In order to shorten the chemical analysis time, we chose the atomic absorption spectroscopy method for the measurement of the distribution of calcium instead of the

titration method, and we developed the extraction procedure. Figure 4 shows the concentration of calcium in soil-cement standards is proportional to the cement composition in soil-cement mixture. All the samples are prepared the same as for the titration method. (see Table 3)

• **Ca Distribution Tests**

In this series of experiments, calcium component is measured by the Flame Atomic Absorbance. Figure 5 to 10 show the calcium distribution in each sample for different mixing times. The mixing conditions are listed in Table 4.

Table 4 Mixing Conditions of Each Sample

Test No.	Cement wt %	Soil wt % (clay:sand =2:1)	Water wt %	Mixing Time (seconds)
1	15	65	20	5
2	15	65	20	10
3	15	65	20	20
4	15	65	20	30
5	15	65	20	45
6	15	65	20	60

All the parameters are kept at the same value same except mixing time. Also, the type of agitator and motor speed are kept the same. As mixing time increases, the concentrations of calcium at different locations match more closely. That means the

mixing degree increases as mixing time increases, or we can say that the sample's uniformity increases as mixing time increases at these mixing conditions.

5.1.3 Results for Mixing Index

Figure 11 shows the mixing index of each sample at different mixing times as determined by the AA test method. All data are shown in Tables 5 to 10.

The lower the mixing index, the more homogenous the mixing. Figure 11 shows that at short mixing time, the mixing is poor. When mixing time increases at the same conditions, the uniformity becomes better. However, there may exist an over mixing problem, that is although mixing time increased, the mixing index increased, or the uniformity decreased. Figure 11 shows that when mixing time was larger than 20 seconds the mixing index increased slightly. Therefore, after a certain mixing time (about 20 seconds in this series of experiments), the mixing index keeps a constant value.

5.1.4 Results from Fluorescent-Tracer Method

The results are shown in Figures 13 to 16. The standard deviation method [Ref. 9] is employed in this method to evaluate all the data.

The fluorescent tracer method gave the same results as AA test method shown in Figure 11 and Figure 12. Although the tracer method used in this study only can measure the surface uniformity of each sample, the results are equivalent to chemical analysis for the whole mixture. We thus conclude that at our mixing conditions, the homogeneity of the surface can be used to represent the bulk homogeneity. Therefore, it is possible to develop a fluorescent tracer method for field use rather than requiring chemical analysis. It will also not be necessary to take samples by driving holes in a hardened field.

Figure 13 to 16 show that when standard deviation is used as the homogeneity measurement, it is not dependent upon the number of samples when the illuminative intensity criterion is chosen suitably. [See Ref. 9]

Figures 17 to 21 show that when the number of samples taken is large enough, the standard deviation (or mixing index) is constant.

5.2 Discussion

5.2.1 Discussion of AA Method

Since calcium in soil-cement mixture is proportional to cement composition (see Figure 3 and 4), it is possible to evaluate the distribution of cement in a soil-cement mixture by measuring calcium's distribution in each sample. The results are similar to the titration method, but take less time to analyze and need fewer chemicals.

It is important that the extraction should be complete each time. We employ the EPA digestion method to modify the calcium concentration determination. The results are the same as expected and similar to the other evaluation method [29,33]. Therefore, the modified AA test is acceptable.

5.2.2 Comparison of AA to Fluorescence Method

For evaluating the homogeneity of the hardened soil-cement product, the chemical analysis method (AA) and the fluorescent tracer method give almost the same results. However, both of them have some limitations. Such as:

- For AA Method: If calcium exists both in the CFS reagent and in the wastes, this method will fail. In fact, this method shows that if there is a specific compound or element contained in only one dispersive phase, a good way to evaluate the uniformity is to analyze for this special compound or element. Obviously, it is difficult to

practice this method in the waste-treatment field because there are many unknown wastes in contaminated materials which might interfere with the chemical analysis.

•For Fluorescent tracer method: It only can be used as surface test. Therefore, the results will be affected both by the surface condition and the validity of the assumption that the homogeneity of the surface is proportional to the bulk or batch homogeneity. Also, the sensitivity of the imaging system is very important and will determine the results of whole testing including the amount of tracer.

5.3 Suggestions for Future Work

X-ray fluorescence can be used to analyze for many kinds of metals directly. It is an especially convenient method for solid samples. It would avoid the extraction efficiency problem, and it can analyze for many metals at the same time.

The purpose of the chemical analysis method employed in this study is to verify the fluorescent tracer method and the assumption that bulk uniformity can be evaluated with surface homogeneity. But if the fluorescent tracer method is possible to be used as an evaluating measurement, it needs to be verified not only for other kinds of solidification/stabilization reagents but also for various kinds of waste. In addition, numerous factors should be considered in the study, such as solvent, extraction efficiency, interactions between solidification/stabilization reagents and contaminants, pH value of the product, and detection limit of each method.

Future work should also try to verify the conclusion of this study that the bulk or batch uniformity can be measured by surface homogeneity. For instance, the surface condition, such as smoothness, might affect the results.

Table 5 5 Sec. Mixing Time Data

weight (g)	Ca % (wt)	$C_A - C_A^m$	$(C_A - C_A^m)^2$
1.0019	11.5	3	9
1.002	5.4	-3.1	9.61
1.002	4.4	-4.1	16.81
1.002	8.3	-0.2	0.04
1.0017	6.3	-2.2	4.84
1.002	4.9	-3.6	12.96
1.002	8.2	-0.3	0.09
1.0016	6.6	-1.9	3.61
1.0019	14	5.5	30.25
	$\bar{c} = 7.73$		$\Sigma = 87.21$

Table 6 10 Sec. Mixing-Time Data

weight (g)	Ca % (wt)	$C_A - C_A^m$	$(C_A - C_A^m)^2$
1.001	5.1	-3.4	11.56
1.0019	11.5	3	9
1.007	7.1	-1.4	1.96
1.0015	9.8	1.3	1.69
1.0016	6.5	-2	4
1.0015	10.3	1.8	3.24
1.007	10.2	1.7	2.89
1.0012	10.5	2	4
1.009	9.5	1	1
	$\bar{c} = 8.94$		$\Sigma = 39.34$

Table 7 20 Sec. Mixing-Time Data

weight (g)	Ca % (wt)	$C_A - C_A^m$	$(C_A - C_A^m)^2$
1.002	8.7	0.2	0.04
1.0018	9.6	1.1	1.21
1.0017	8.8	0.3	0.09
1.002	8.7	0.2	0.04
1.002	8.3	-0.2	0.04
1.002	8.2	-0.3	0.09
1.0019	9	0.5	0.25
1.0016	6.9	-1.6	2.56
1.002	8.9	0.4	0.16
	$\bar{c} = 8.57$		$\Sigma = 4.48$

Table 8 30 Sec. Mixing-Time Data

Weight (g)	Ca % (wt)	$C_A - C_A^m$	$(C_A - C_A^m)^2$
1.0019	9.2	0.7	0.49
1.002	10.7	2.2	4.84
1.0017	8.6	0.1	0.01
1.002	8.6	0.1	0.01
1.0021	8.6	0.1	0.01
1.002	8.7	0.2	0.04
1.0018	8.4	-0.1	0.01
1.002	10.9	2.4	5.76
1.0019	8	-0.5	0.25
	$\bar{c} = 9.08$		$\Sigma = 4.48$

Table 9 45 Sec. Mixing-Time Data

Weight (g)	Ca % (wt)	$C_A - C_A^m$	$(C_A - C_A^m)^2$
1.002	9.98	1.48	2.1904
1.0019	6.39	-2.11	4.4521
1.002	7.88	-0.62	0.3844
1.002	9.38	0.88	0.7744
1.0021	8.89	0.39	0.5382
1.0018	9.88	1.38	1.9044
0.0017	9.68	1.18	1.3924
1.0016	8.59	0.09	0.0081
1.0021	9.98	1.48	2.1904
	$\bar{c} = 8.96$		$\Sigma = 13.83$

Table 10 60 Sec. Mixing-Time Data

Weight (g)	Ca % (wt)	$C_A - C_A^m$	$(C_A - C_A^m)^2$
1.002	6.99	-1.51	2.2801
1.002	8.28	-0.22	0.0484
1.0016	7.16	-1.34	1.7956
1.002	6.59	-1.91	3.6481
1.0018	7.68	-0.82	0.6724
1.002	8.29	-0.21	0.0441
1.002	7.39	-1.11	1.2321
1.002	8.78	0.28	0.0784
1.0021	8.51	0.01	1E-04
	$\bar{c} = 7.74$		$\Sigma = 9.80$

Table 11 Mixing Index

Mixing Time (sec.)	D_v	D_v^o	$I_v = D_v/D_v^o$
5	0.366	3.28	0.11
10	0.246	3.28	0.075
20	0.083	3.28	0.025
30	0.132	3.28	0.040
45	0.146	3.28	0.044
60	0.123	3.28	0.037

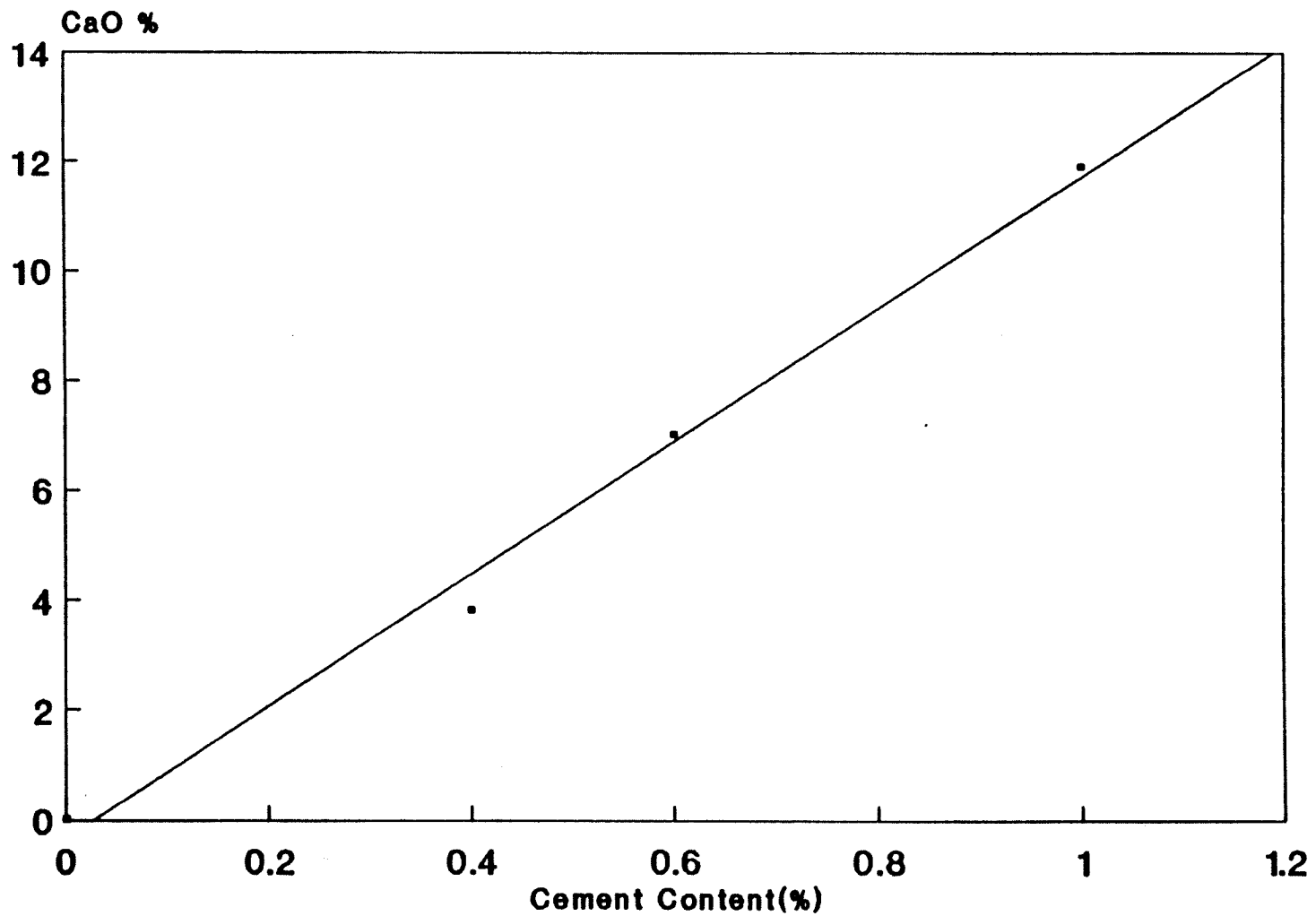


Figure 3 Titration Method Data
Cement content vs. CaO%

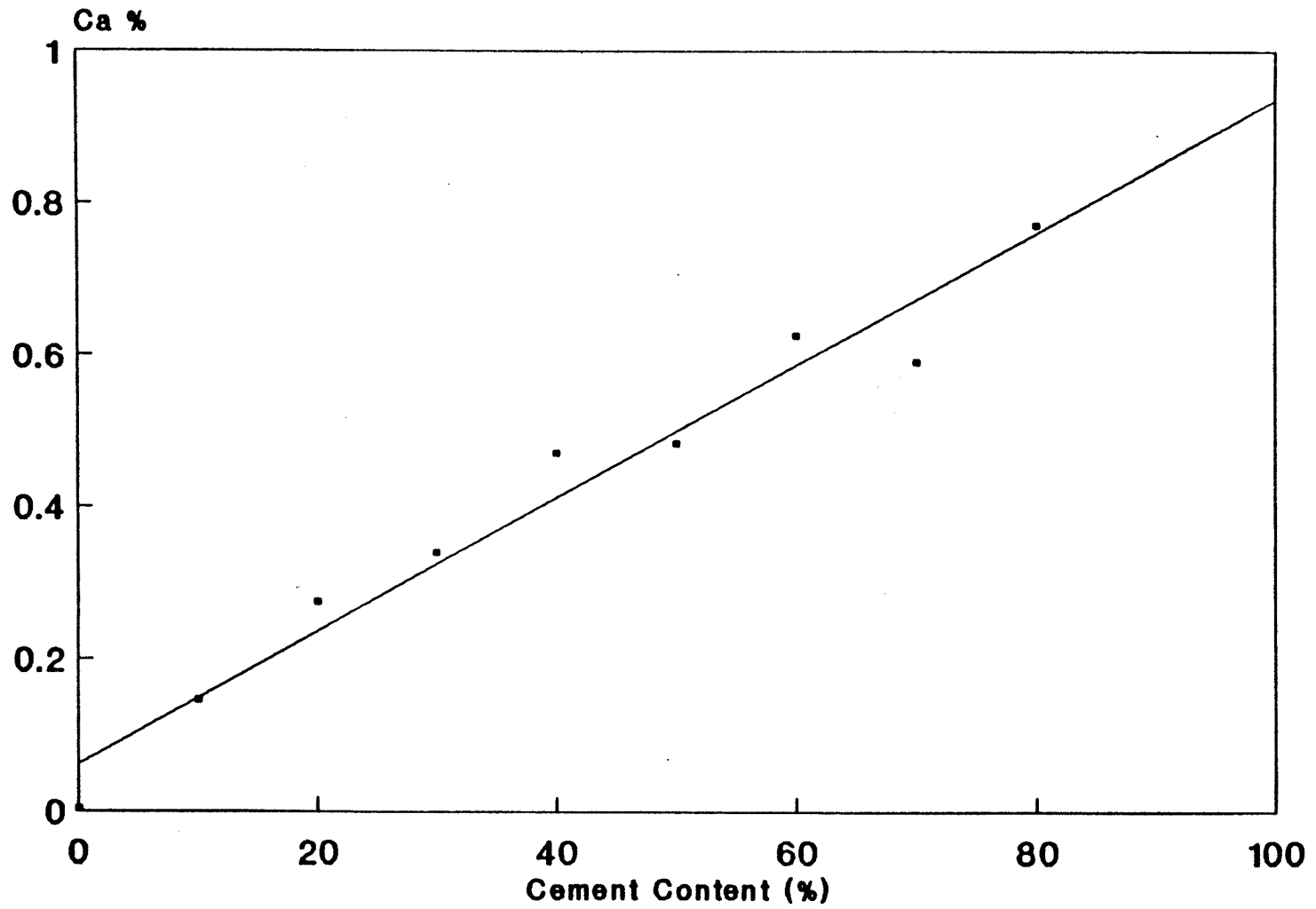


Figure 4 Cement Content vs. Ca%
AA Method for Soil-Cement Mixture

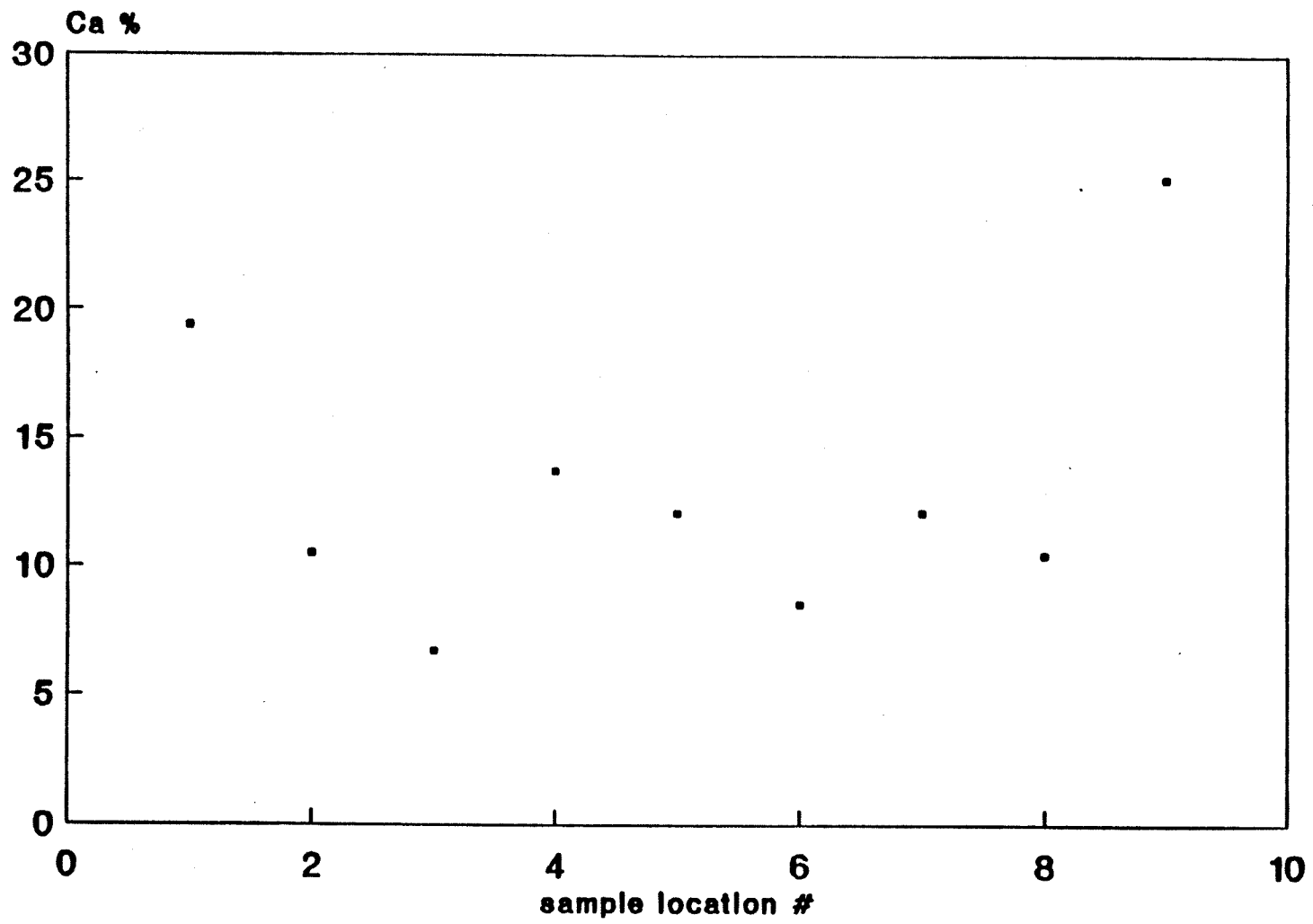


Figure 5 Ca% vs. samples' location
5 sec mixed sample

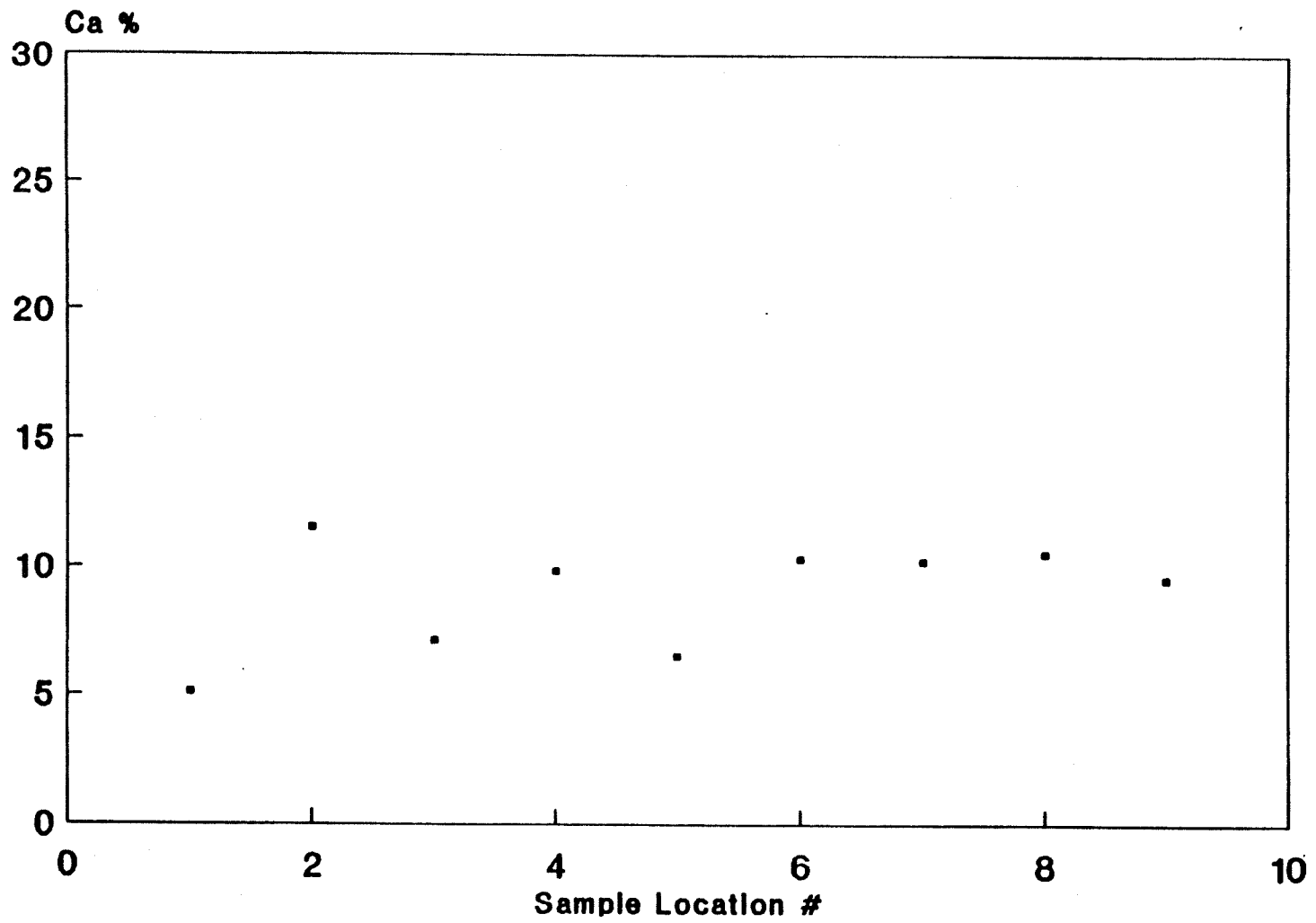


Figure 6 Ca% vs. samples' location
10 sec. mixed sample

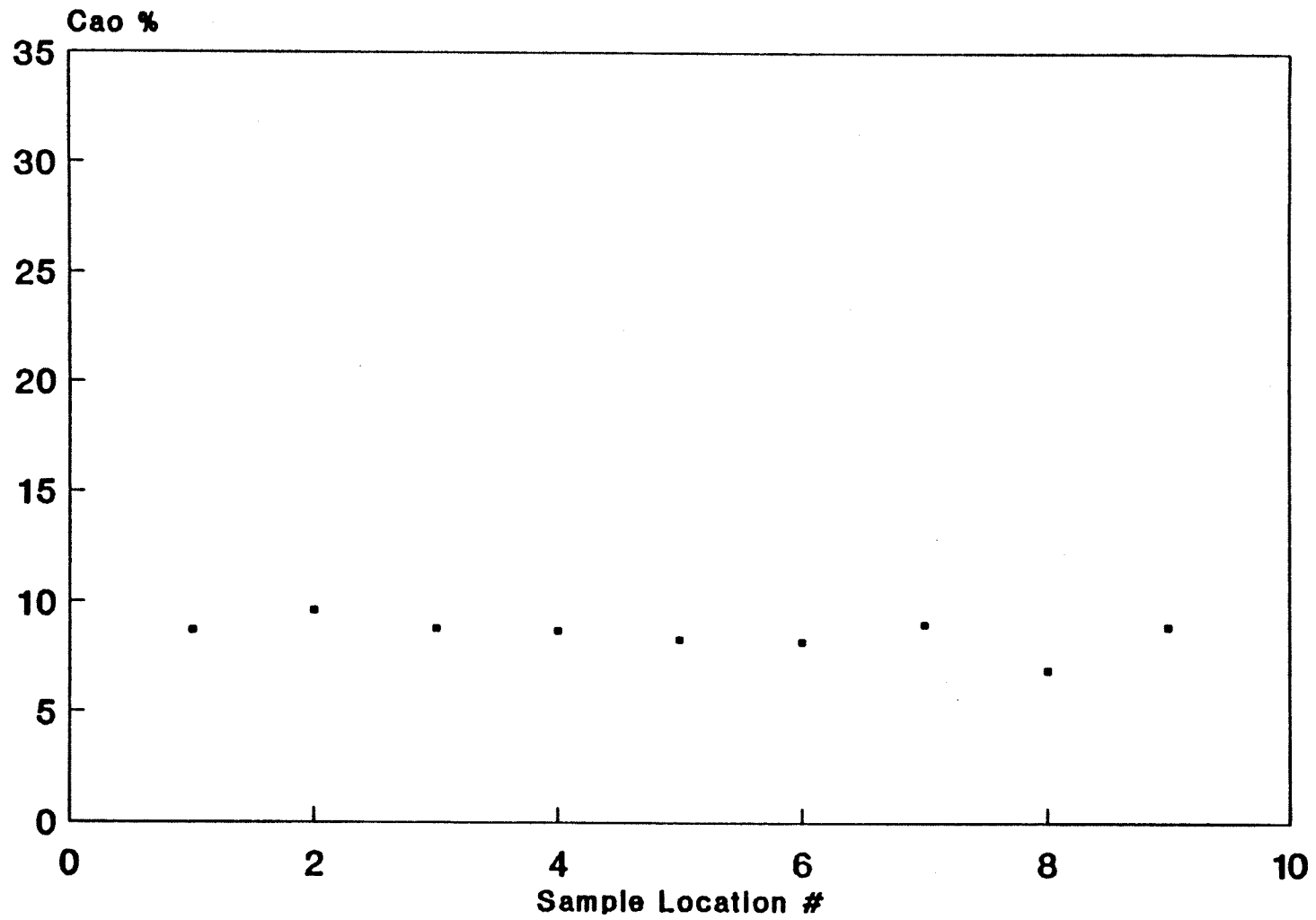


Figure 7 Ca% vs. samples' location
20 sec mixed sample

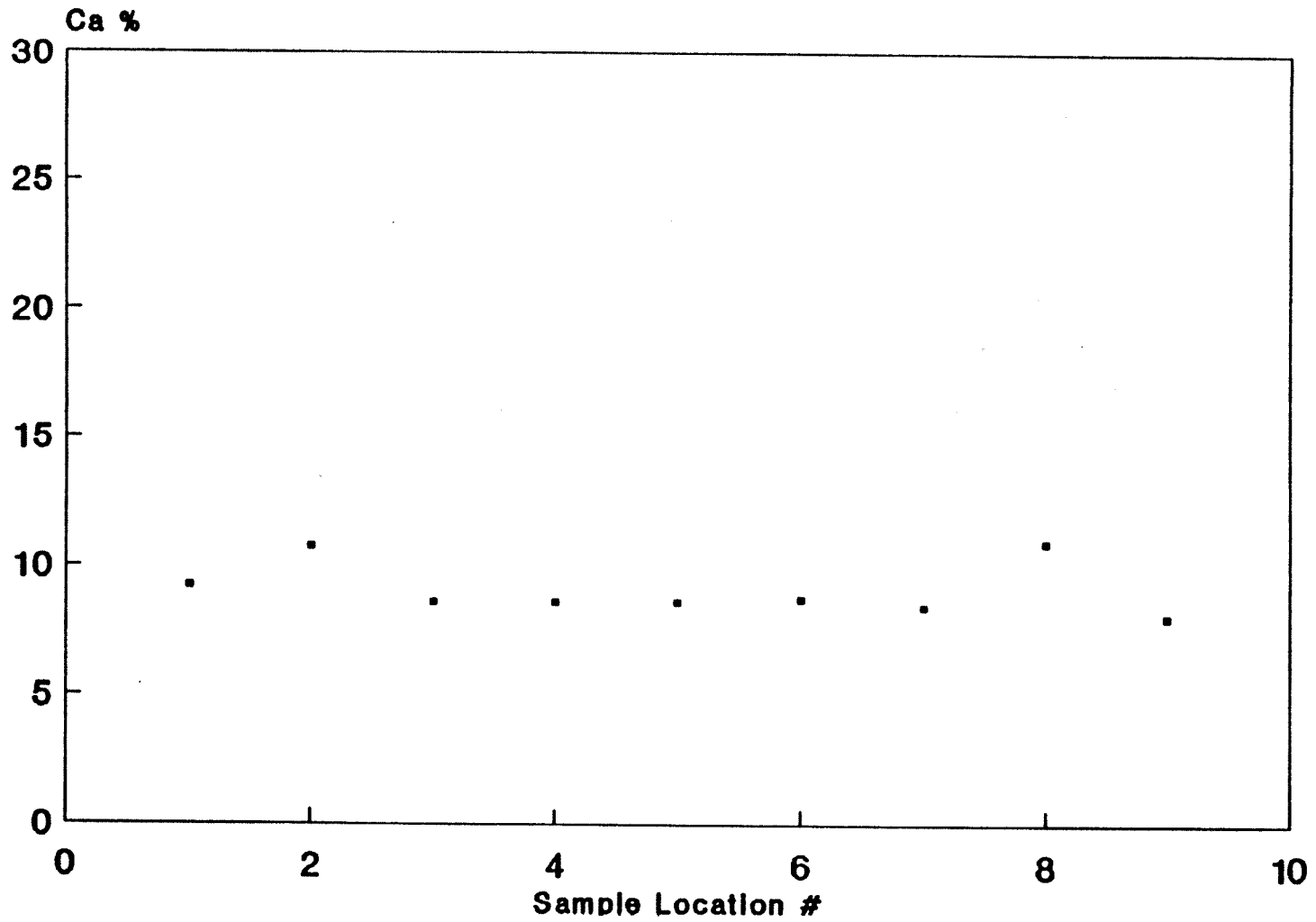


Figure 8 Ca% vs. samples' location
30 sec. mixed sample

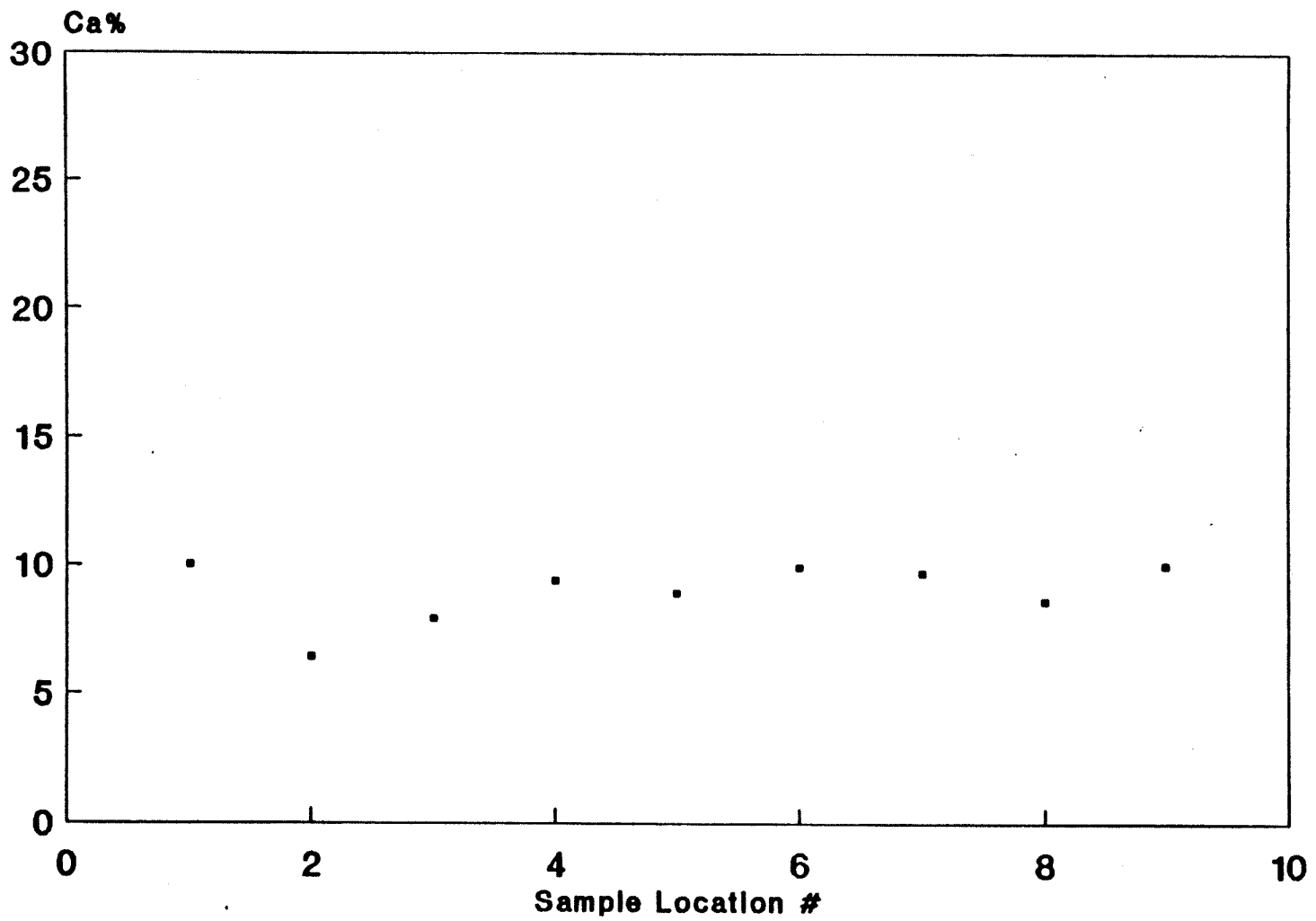


Figure 9 Ca% vs. samples' location
45 sec. mixed sample

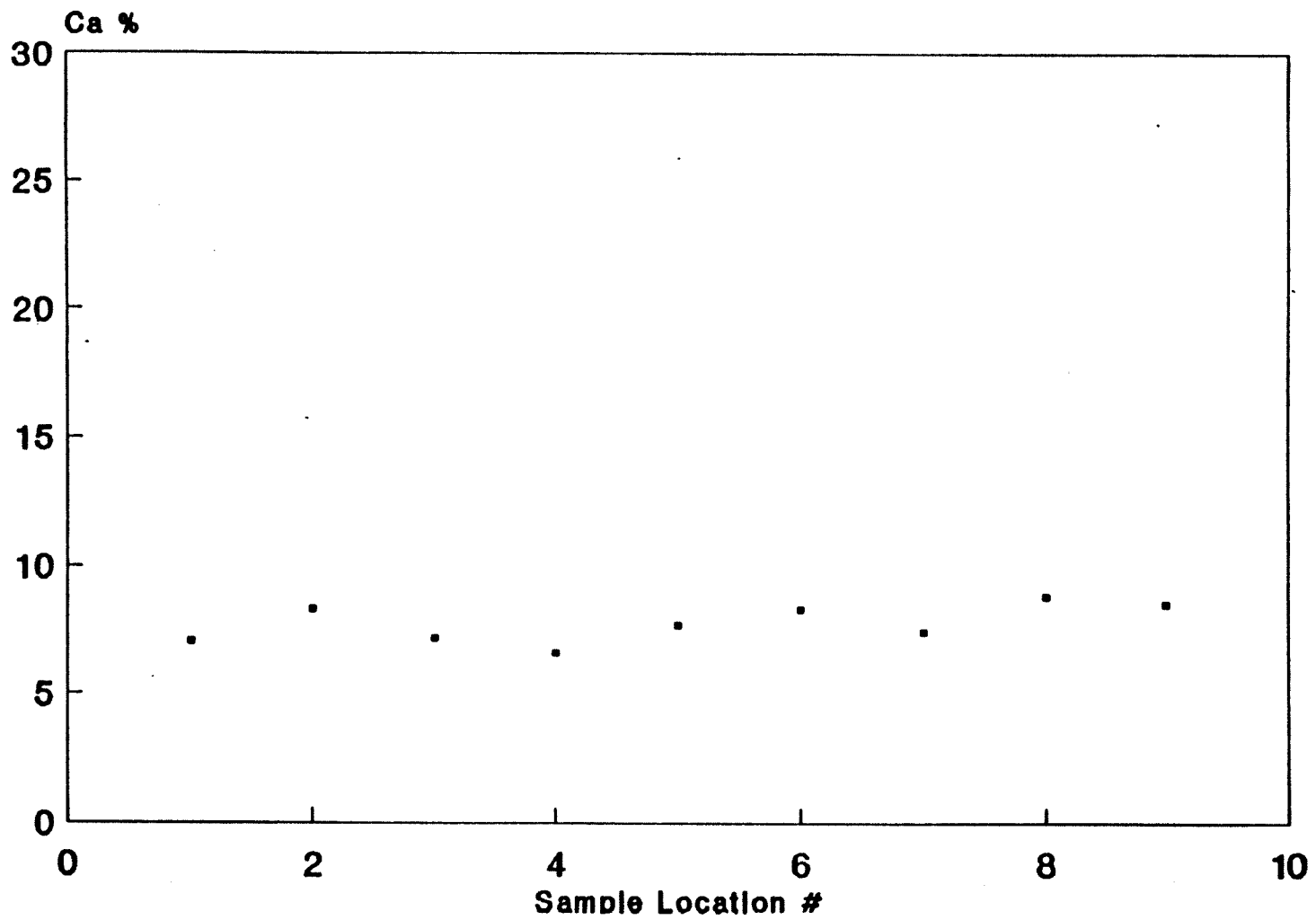


Figure 10 Ca% vs. samples' location
60 sec. mixed sample

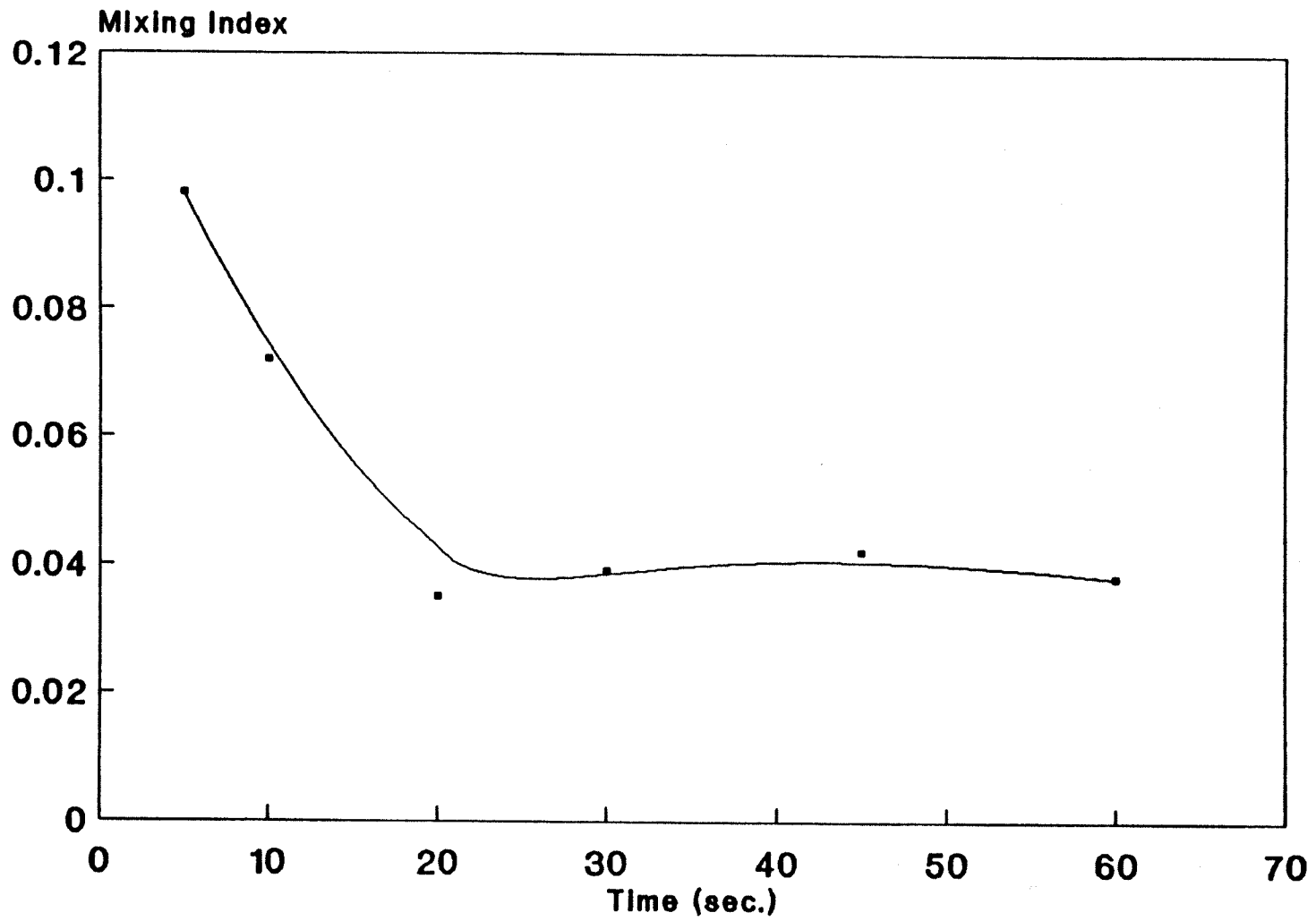


Figure 11 Mixing Index vs. Time
AA test method

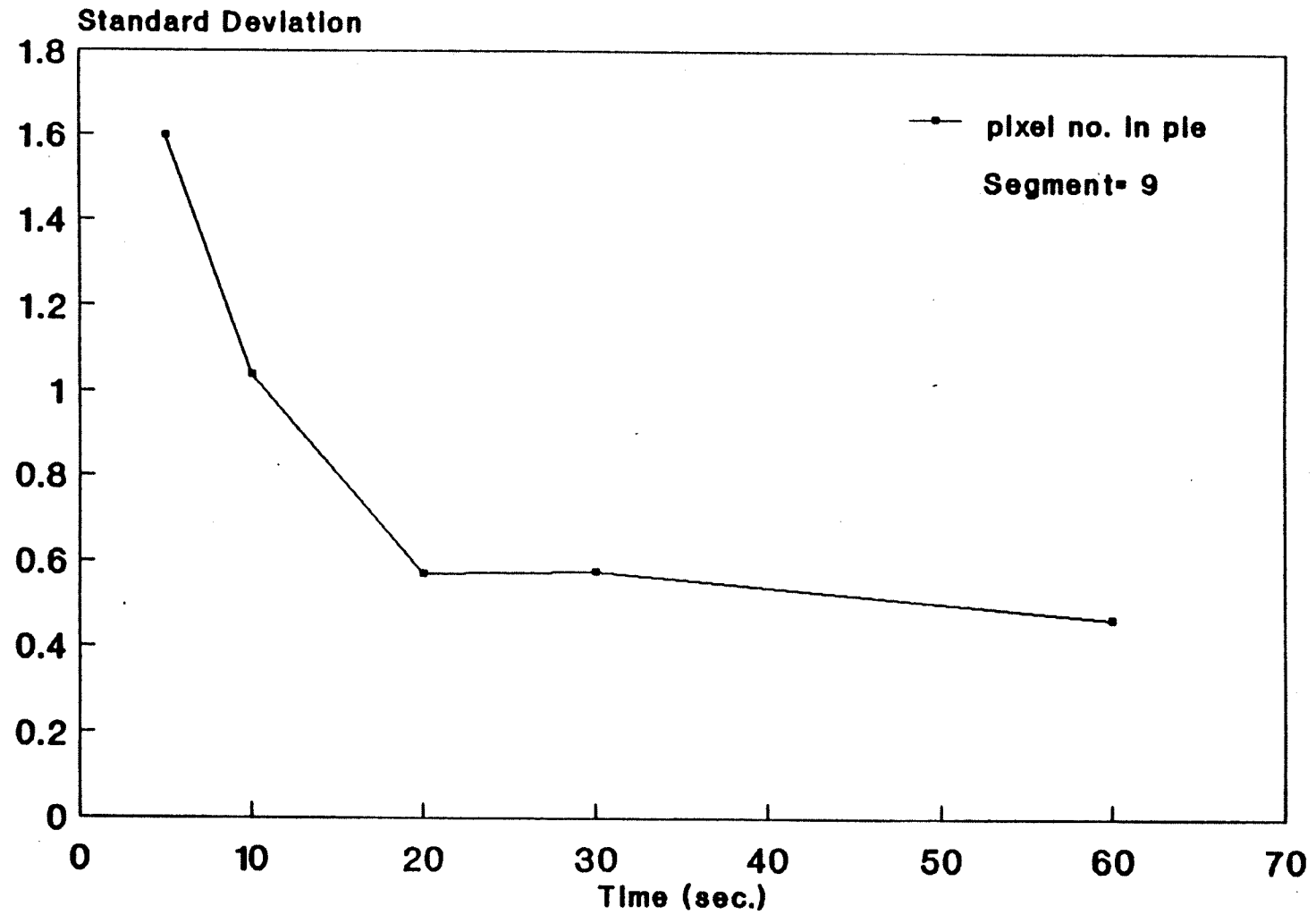


Figure 12 Std. Dev. vs. Time
Fluorescent Tracer Method

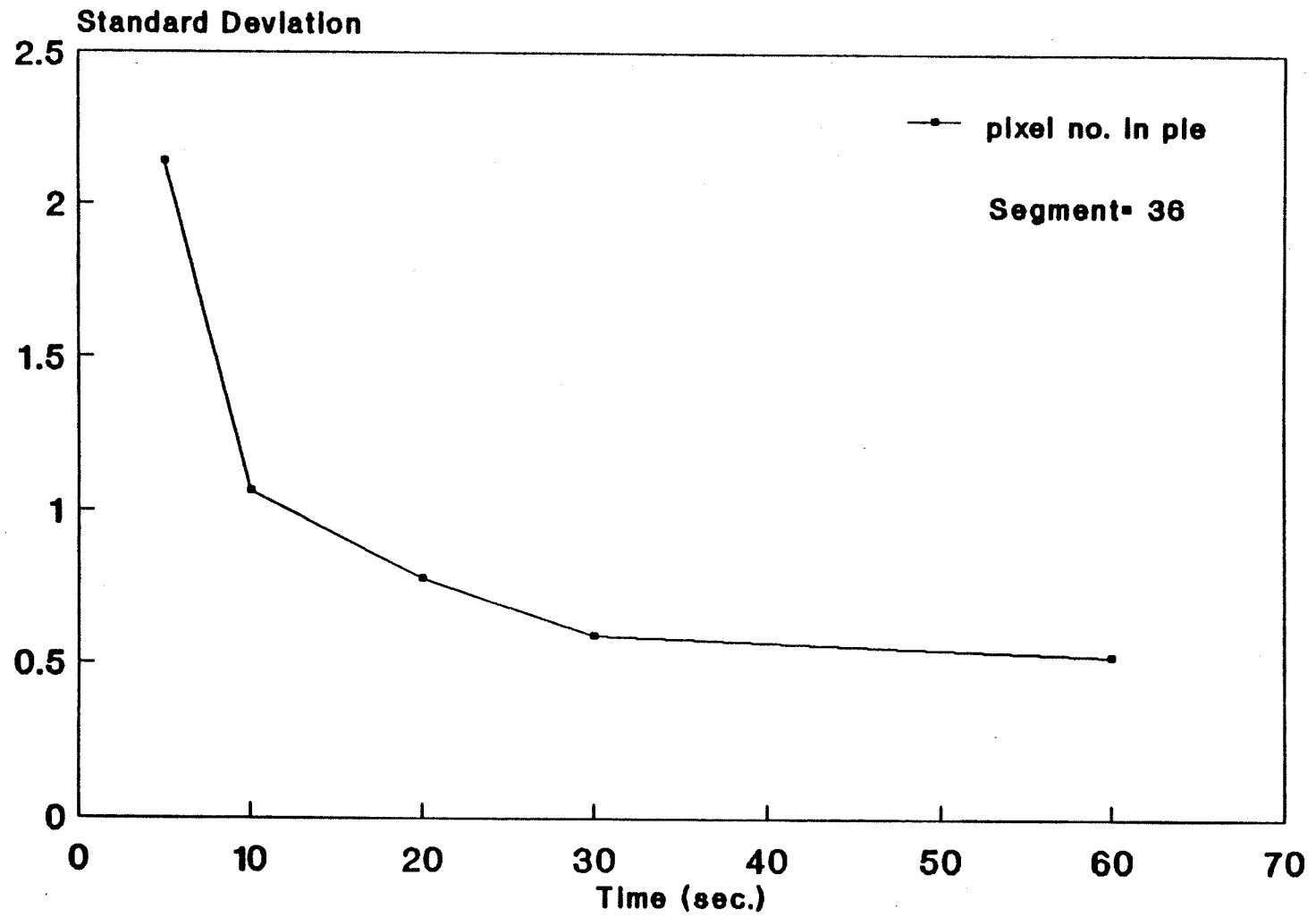


Figure 13 Std. Dev. vs. Time
Segment = 36

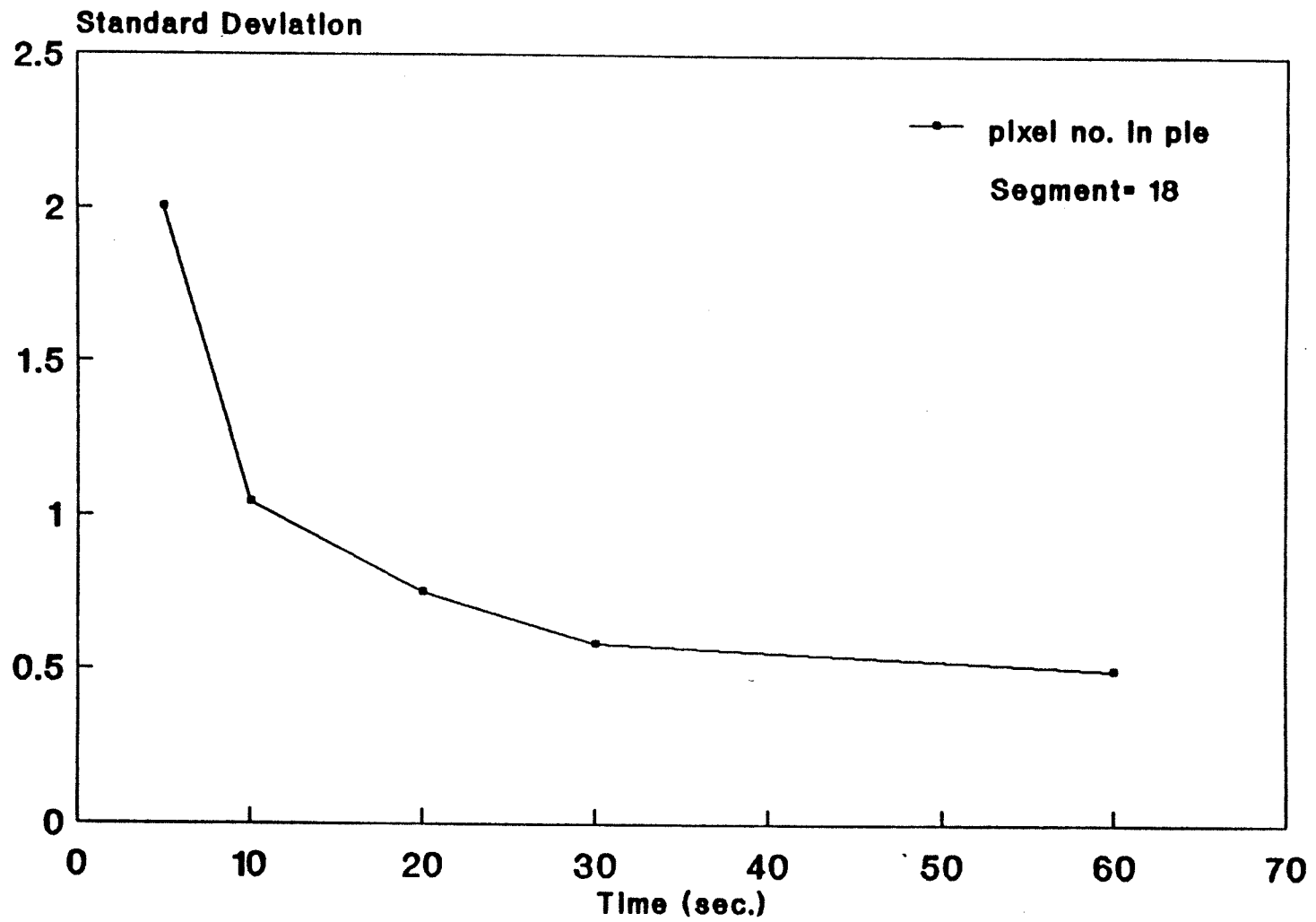


Figure 14 Std. Dev. vs. Time
Segment = 18

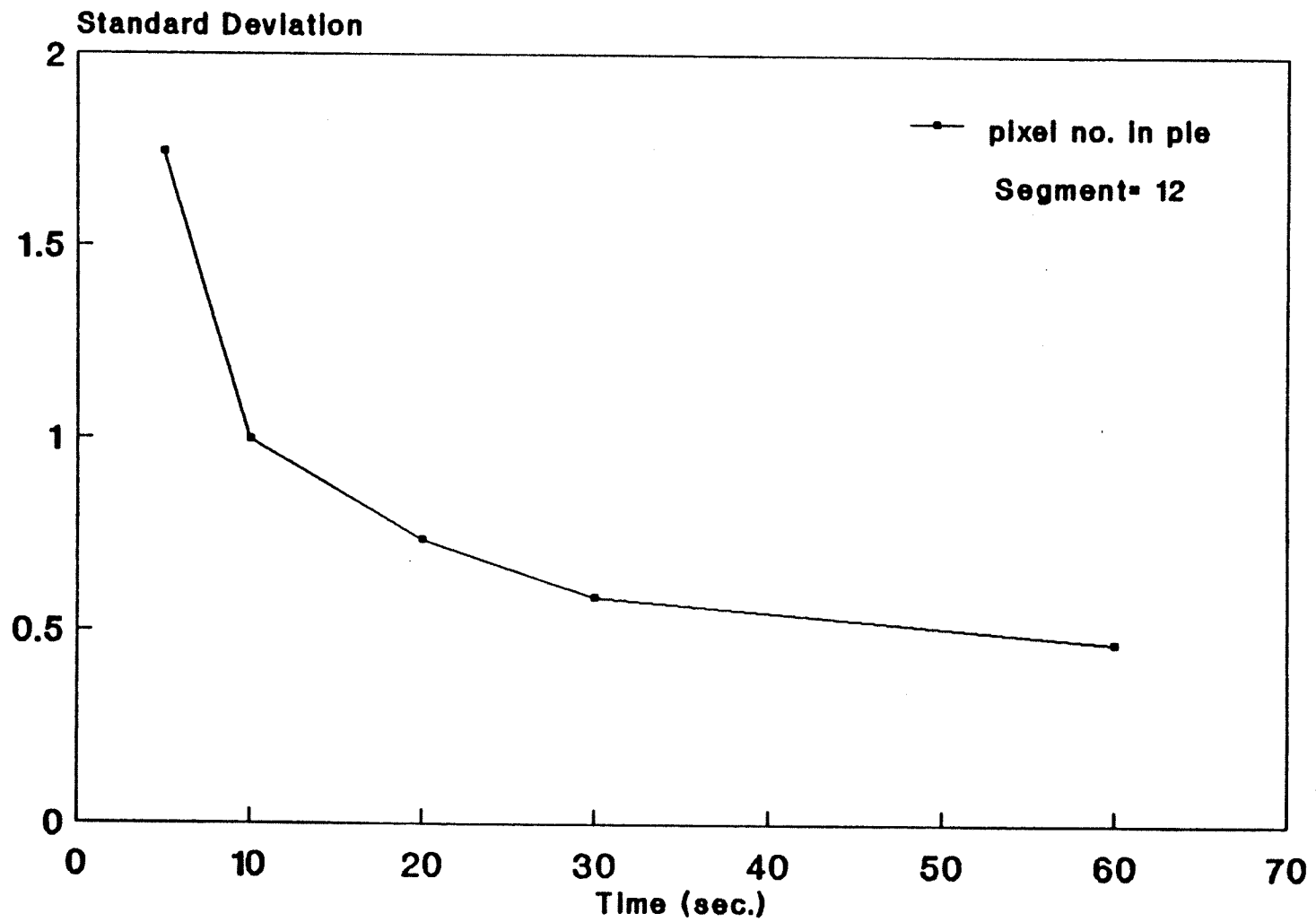


Figure 15 Std. Dev. vs. Time
Segment = 12

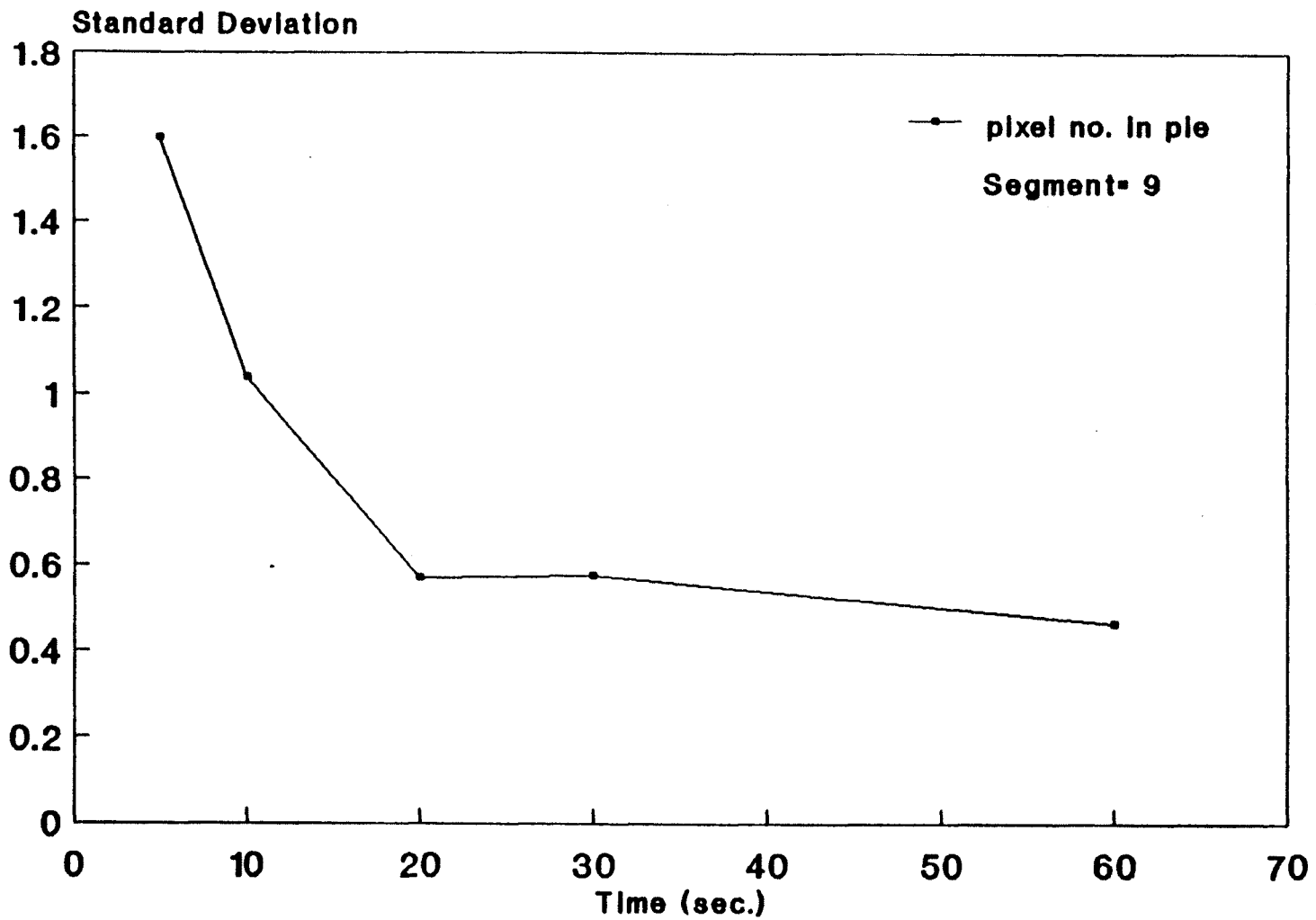


Figure 16 Std. Dev. vs. Time
Segment = 9

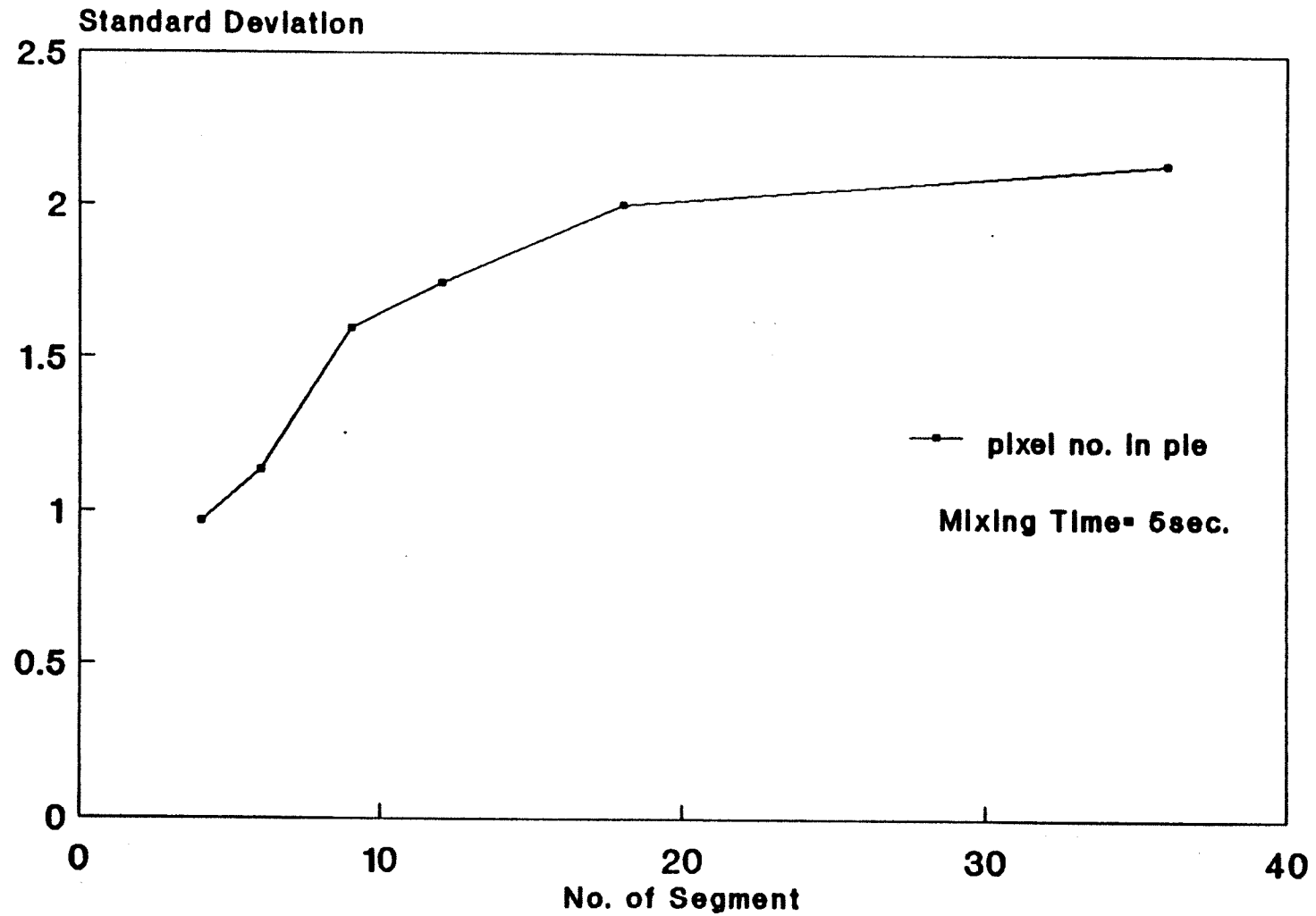


Figure 17 No. of Segment vs Std. Dev.
Imaging Test Method (5 sec.)

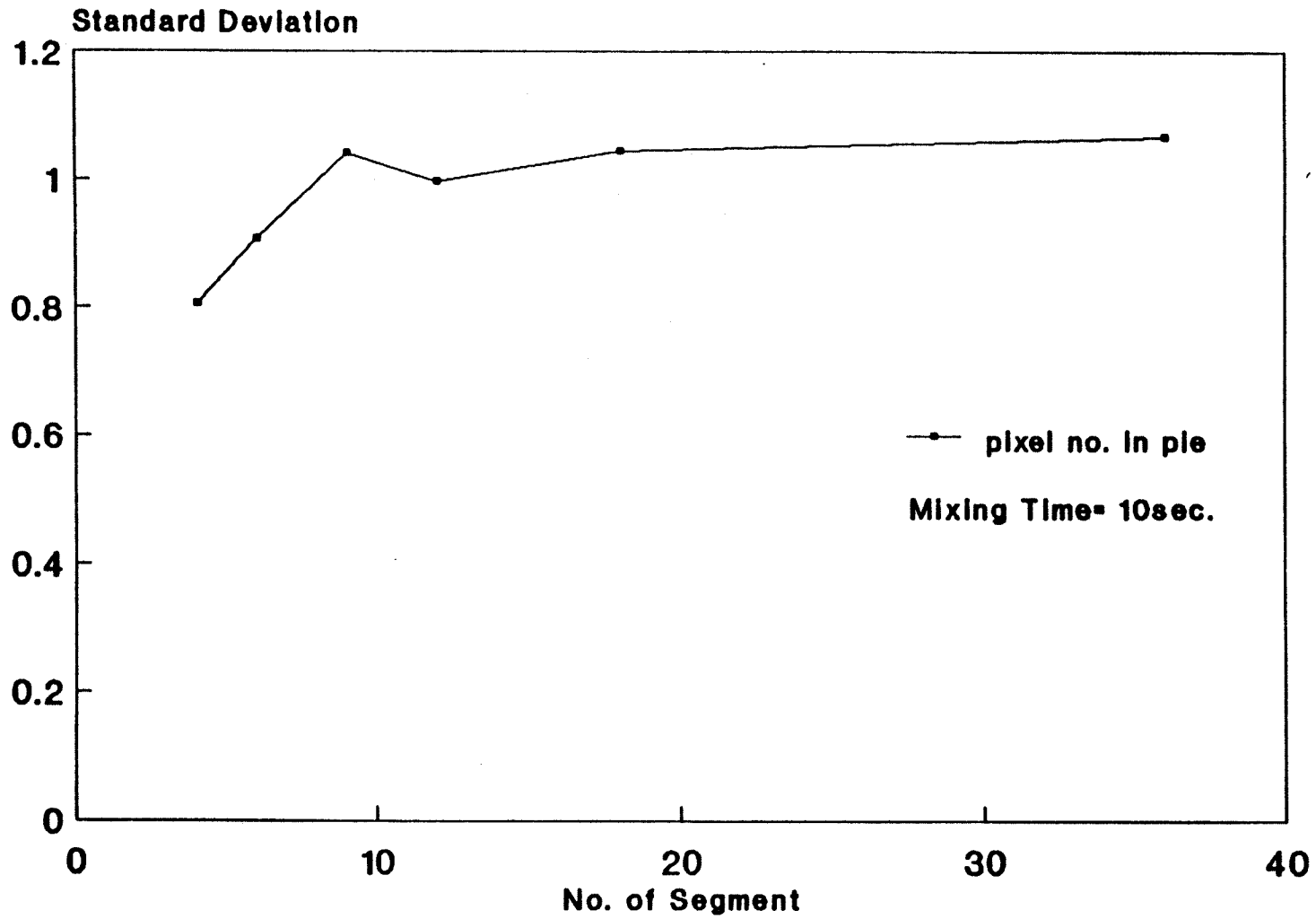


Figure 18 No. Segment vs. Std. Dev.
Imaging Test Method (10 sec.)

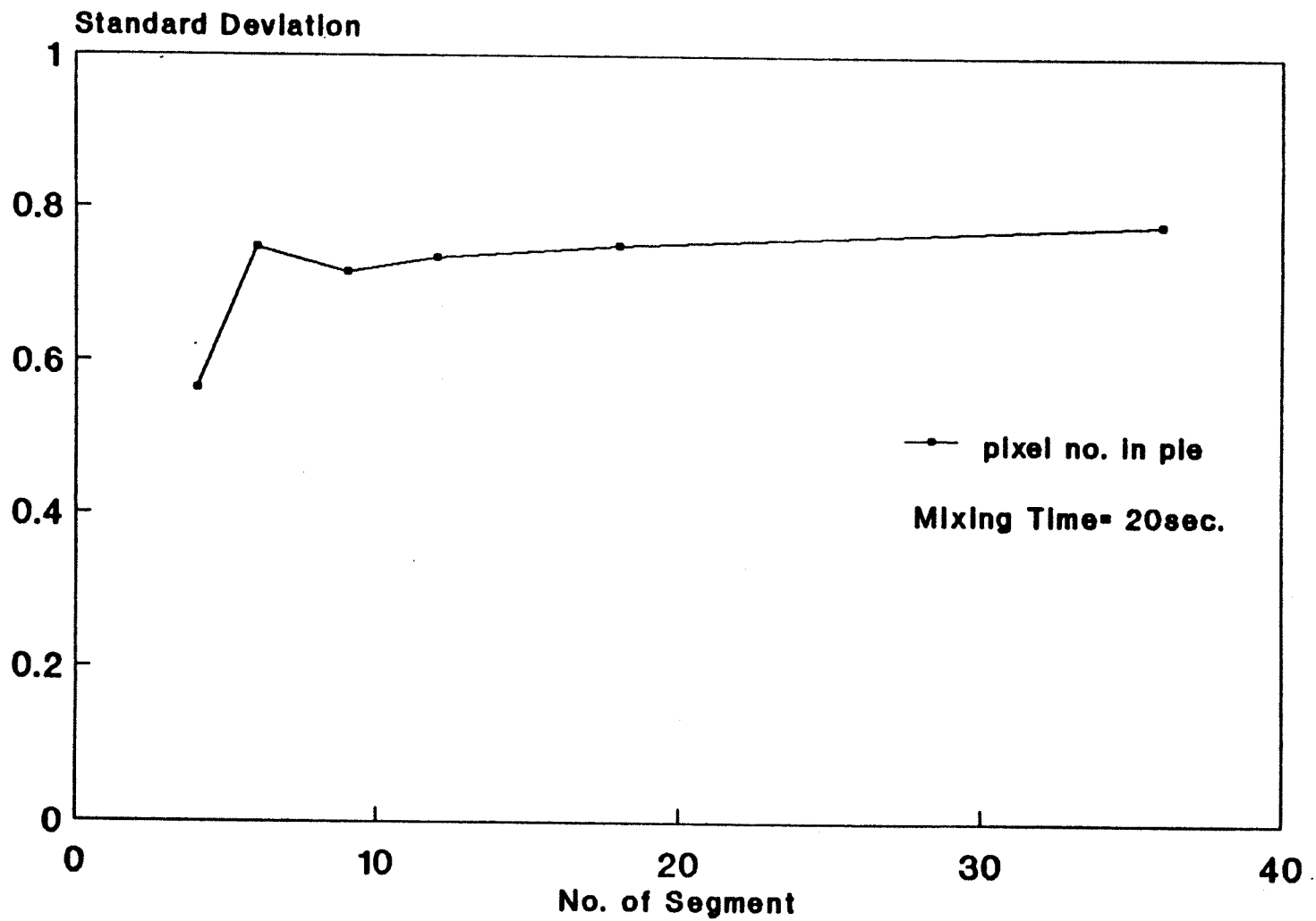


Figure 19 No. of Segment vs Std. Dev.
Imaging Test Method (20 sec.)

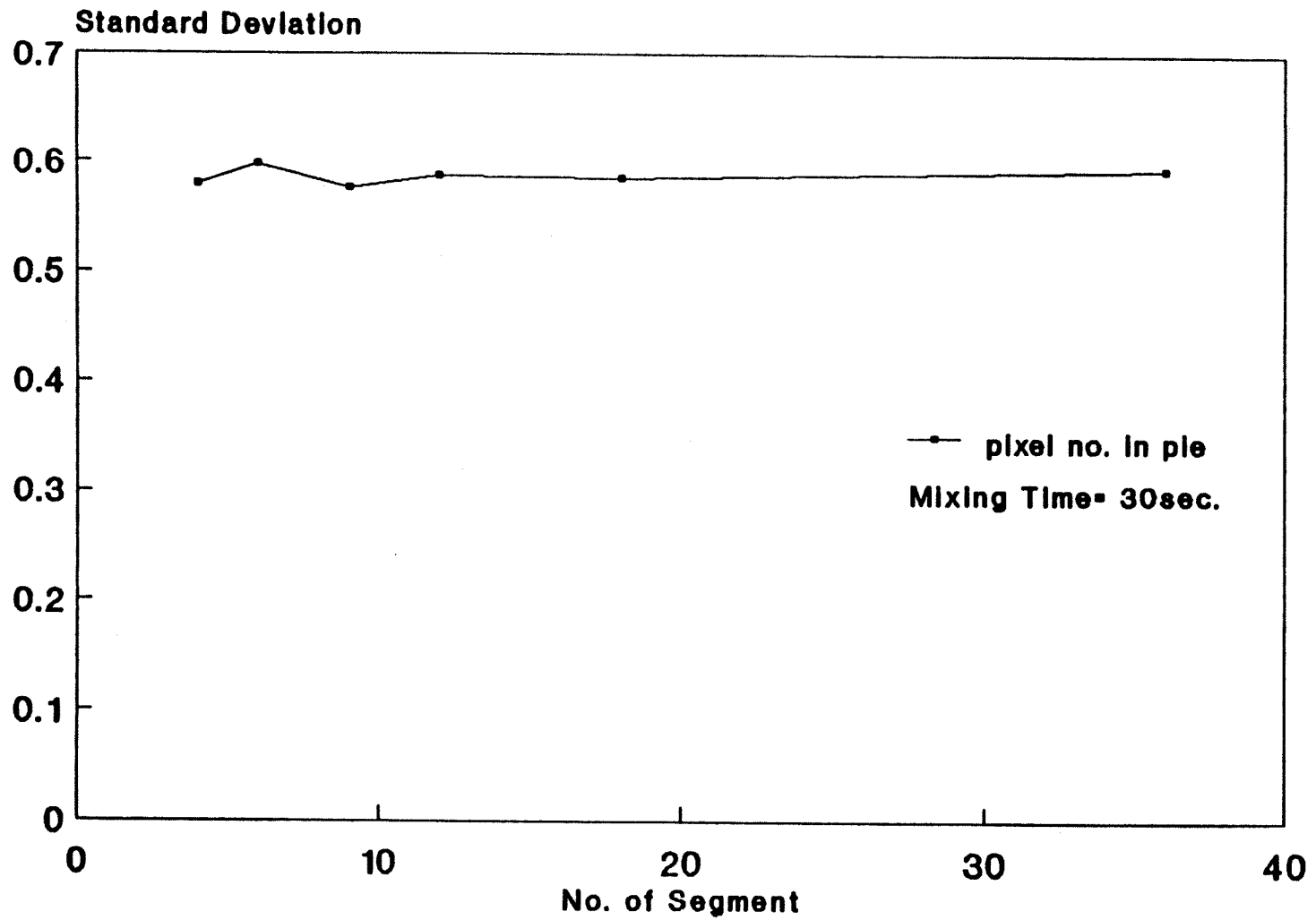
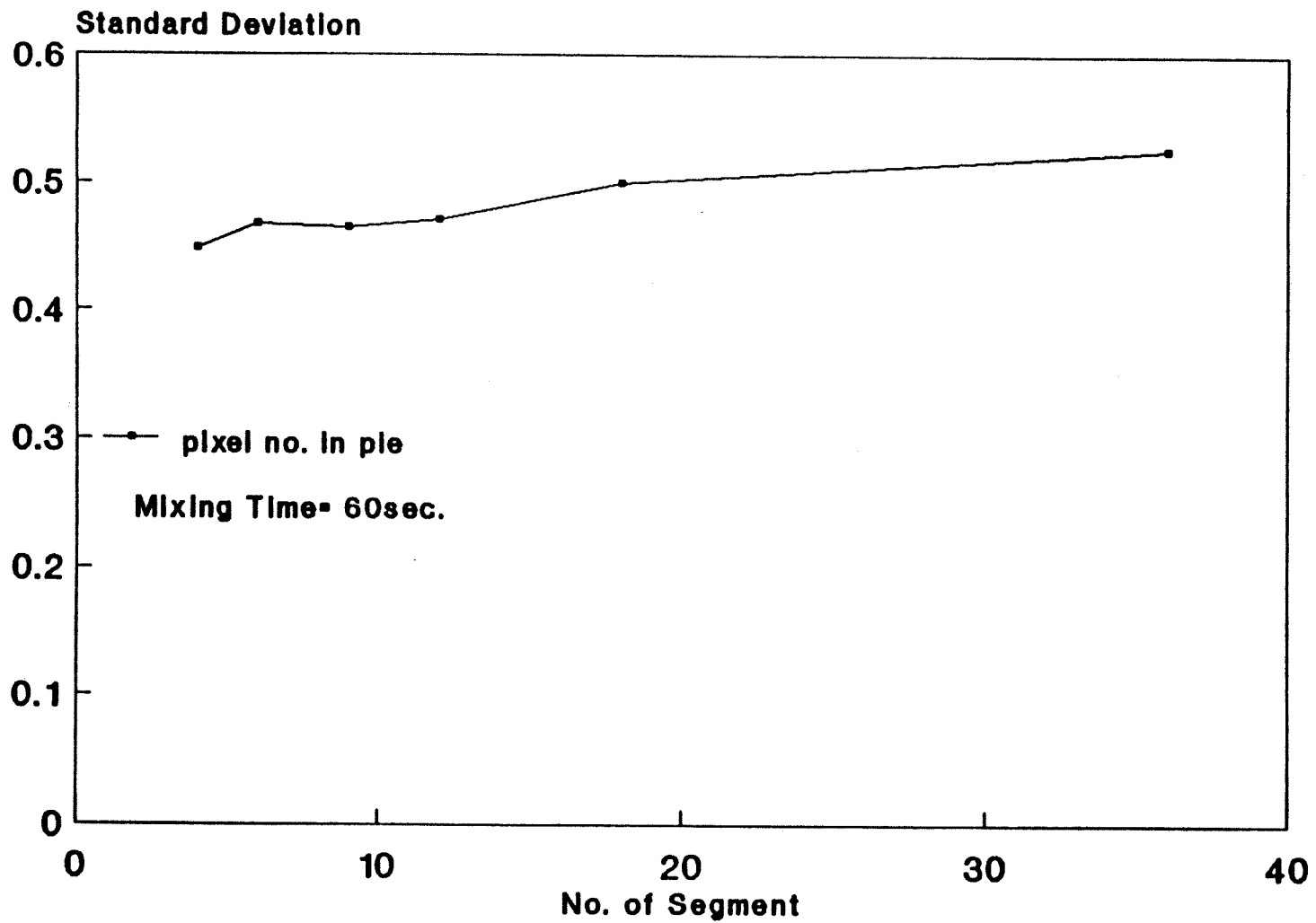


Figure 20 No. of Segment vs. Std. Dev.
Imaging Test Method (30 sec.)



**Figure 21 No. of Segment vs. Std. Dev.
Imaging Test Method (60 sec.)**

CHAPTER 6 CONCLUSIONS

The results of this study lead us to the following conclusions:

1. The formulation of a Mixing Index to characterize the degree of homogeneity in a soil-cement mixture, and the use of the AA method of analysis for determination as the cement distribution have led to a reliable and reproducible results.
2. The calcium concentration in a soil-cement mixture is proportional to the cement content; therefore, the Ca distribution in the samples can be used as the measurement for the cement distribution.
3. Mixing Index is applicable for evaluating the homogeneity of the soil-cement mixture.
4. For the process of mixing Portland I cement with soil (Kaolin : Ottawa = 1 : 2) in a Hobart Mixer with all conditions fixed except for the mixing time, it has been found that as mixing time increases, the sample's mixing index goes down, meaning that the degree of mixing increases.
5. The fluorescent tracer method is comparable to the AA method, indicating that the homogeneity of the soil cement can be evaluated from either the surface or the bulk. The surface homogeneity may be used as representative of the bulk uniformity, especially, for non-uniform cases.
6. For field testing, this is a useful conclusion since it is not necessary to take samples for analysis but instead the surface may be used to evaluate the homogeneity of solidification/stabilization treated products.

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