Stabilization of phenolics in foundry waste using cementitious materials

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Abstract

A series of experiments were conducted to stabilize the phenolics in foundry sands from Kansas using four different types of binders – Portland cement, fly ash, kaolinite, and bentonite. Strength and leachability of stabilized mixes of foundry sand were analyzed to assess their feasibility in construction and geotechnical applications. The results suggest that compressive strength was acquired relatively faster in fly ash than in cement and, in general, it varied inversely with the proportion of foundry sand in the stabilized mix. Lesser amounts of phenolic compounds leached from fly ash-stabilized mixes than from cement-stabilized mixes. The leachate analyses for both total phenolics and 2,4,6-trichlorophenol indicate that increasing percent replacement of foundry sands enhances stabilization. These results are supported by scanning electron micrographs which showed increased porosity in the case of cement-stabilized mixes. The swelling potential and instability of bentonite-stabilized mixes rendered the leachate quality unpredictable. The general conclusion that fly ash stabilizes phenolics better than Portland cement may lead to development of a cost-effective solution for stabilizing phenolics in foundry sands and may have important implications in the construction industry.

Keywords: Waste stabilization; Cementation; Fly ash; Leachability; Foundry waste

1. Introduction

Foundry sands are commonly used in casting processes by iron foundries to form molds in which molten iron is poured. After cooling, the sand molds are broken and the finished iron products are removed. Either clay or chemically based binder is added to the sands to maintain the shape of the mold during pouring and cooling.

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While clay binders typically include bentonite at the 5–10% level, chemical binders involve resins which are usually synthetic organic polymers. The exact composition of the binder system, including resin, catalyst, and solvent, governs the nature of contamination of the foundry sands. In general, contamination of foundry sands originates from unreacted materials, catalysts, solvents, and soluble polymers. For instance, the clay-bonded (CBS) and resin-bonded (RBS) foundry sands studied in this paper contained 2,4,6-trichlorophenol. In view of the EPA regulation which limits the phenolic compounds in drinking water to 1 µg/l, any utilization of foundry sands must insure stabilization of 2,4,6-trichlorophenol. In many cases, the foundry sands are discarded in landfills at an average disposal cost of $35 per ton. The disposal problem is currently not significant in states like Kansas, which only produces about 12,000 tons per year, but it is significant in states like Illinois, which produces over 800,000 tons per year [1]. It is believed that disposal in landfills will no longer be feasible in the future because of the increasingly stringent regulations controlling waste disposal and also because of the rapid closure of available landfills [2, 3]. Foundries are responding to the problem by developing markets for utilizing the waste sand in the construction industry and also by installing sand reclamation systems that allow reuse of the sands.

The American Foundry Society [4] recently studied a number of applications for foundry sands including geotechnical fill applications such as drainage aggregate, filtration media, slurry and utility trench backfill, and final landfill cover. In general, the active clay content and the amount of material smaller than 200 mesh size (0.075 mm) are believed to be the important properties determining the appropriate uses for the material. Lessiter [2] studied the properties of high-performance concrete mixes designed with up to 35% of regular sand replaced with foundry sands. Their results indicated that construction products with foundry sands passed all the ASTM requirements for strength tests.

Although the mechanical behavior of stabilized foundry sands was well documented in literature, their leachability behavior was not studied adequately with respect to the binder used in the stabilization process. A growing number of research activities in solidification and stabilization (S/S) processes now enable us to quantify the leachability behavior of stabilized foundry sands. Holmes [5] presented a method of quantifying leachability with respect to the binders used in a comparative study of three separate S/S treatment technologies. Cocke and Mollah [6] reviewed the binding chemistry and leaching mechanisms of hazardous substances, particularly heavy metal pollutants, in cementitious S/S systems in terms of the surface and solution chemistries of the pollutants. Butler et al. [7] and Ivey et al. [8], among several others, reported results from experimental techniques, such as electron microscopy and NMR spectroscopy, to characterize cement-solidified hazardous wastes. Several such studies in recent literature documented the S/S issues pertinent to individual contaminant-binder systems. A comprehensive review of these studies may be found in Conner [9], Colombo et al. [10], and Means et al. [11].

Only a few studies addressed the effect of phenol on microstructure and leaching properties of stabilized products. Of direct relevance to this paper is the work by Vipulanandan and Krishnan [12] who studied the potential of a thermosetting poly-

ester polymer to solidify/stabilize phenol. The effects of phenol concentrations on microstructure of the polyester polymer and cement were investigated in their study. Vipulanandan and Krishnan concluded that phenol inhibits the setting time of the polyester polymer and cement, and that the phenol leached from cement matrices is very much dependent on curing time and initial phenol content. The polyester polymer was found to be very effective in rapidly stabilizing phenol. Binders other than cement and polyester polymer were not explored in their study. In a later study, Vipulanandan and Krishnan [13] concluded that phenol retards the initial and final setting times of cement by interfering with normal cement hydration reactions and by preventing the formation of calcium hydroxide during the initial period of setting and hardening.

The purpose of this study was to evaluate the effectiveness of four binders – Portland cement, fly ash, kaolinite, and bentonite – in the stabilization of phenolic compounds in foundry sands as reflected in the mechanical properties and leachates of the stabilized products. The study compared the effectiveness of the various binders in stabilizing phenolics. Portland cement and fly ash were chosen because of their importance in the construction industry, and bentonite and kaolinite were chosen to evaluate the feasibility of utilizing the foundry sands as backfill materials after mixing with clays.

2. Experimental program

The experimental program was designed to address the strength as well as the leachability of the foundry waste stabilized with the four types of binders. The purpose of the experiments was to observe the differences among the binders under a given set of conditions, and not to optimize the mix designs to obtain desired strength and leachability. Tests were conducted on both resin-bonded and clay-bonded foundry sands obtained from two representative foundries in Kansas. The grain size distributions of the sands are shown in Fig. 1. Also shown in the figure is the grain size distribution of a clean silica sand which was used in control experiments.

In the experiments with cement and fly ash, mixtures were prepared using 0%, 25%, 50%, 75%, and 100% levels of replacement of silica sand by foundry sands. Initial experiments with Class 'F' fly ash were unsuccessful because it lacked cementitious properties to form a stable mix; therefore, subsequent experiments (reported in this paper) were restricted to Class 'C' fly ash only. The ratio of water to the cementitious binder (analogous to water–cement ratio) was chosen to be 1.0 in the case of Portland cement and 0.35 in the case of fly ash. These ratios were found to yield consistent and desirable range of strengths in trial experiments. The samples were formed in PVC pipes, 2.85 cm in diameter and 5.72 cm long. The mixtures of the sands and the binders were poured into these pipes and then vibrated on a vibrating table to minimize air pockets. After setting for one day, the sides of the PVC pipes were split lengthwise to remove the sample. The samples were then cured for a specified period of time under moist conditions. For each of the replacement levels, compression strengths were obtained after 3, 7, 14, 28, and 56 d in order to evaluate the differences due to curing time.
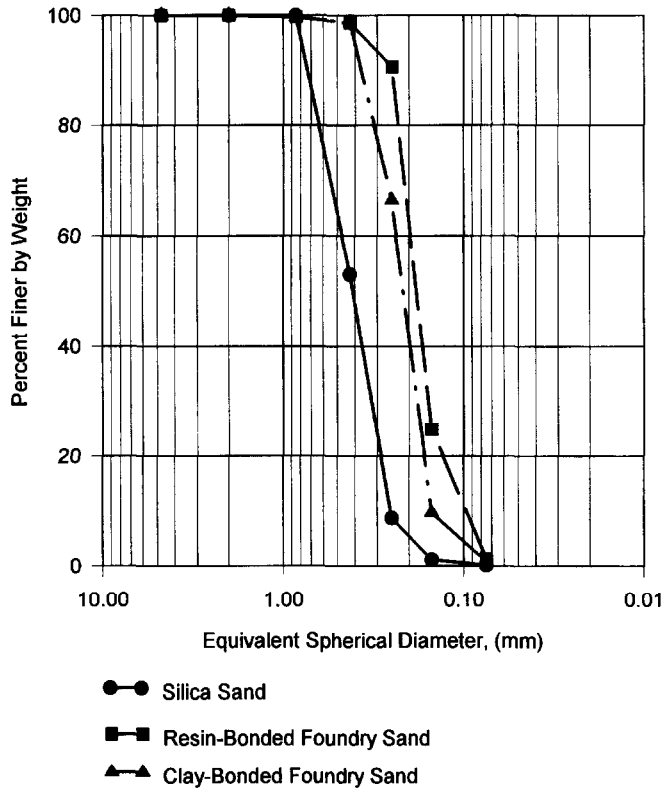
Fig. 1. Particle size distribution for silica sand, CBS, and RBS.

In the experiments with kaolinite and bentonite, compaction curves were obtained first for samples with 50% of the clay mineral and 50% of the clean silica sand. The foundry sands then replaced 50% and 75% of the clean sand in the mixtures to observe their effect on compaction behavior. Harvard miniature compaction, which allows for the same size of samples as in cement and fly ash stabilization, was used to obtain the compaction curves (moisture–density relationships) for all these mixtures. The Harvard compaction procedure involves kneading a soil sample in a metal mold with a hand-held spring-loaded tamper. By repeating the test on separate samples with various moisture contents, the moisture–density relationship was obtained. A detailed test procedure is given in ASTM STP 479 [14].

Samples were soaked individually in distilled, deionized water for 96 h and the solutions were then tested to observe the leachability of stabilized mixes. The samples for obtaining leachabilities were those which were cured for long periods of time and the strengths of which were stabilized. This approach was adopted instead of toxicity characteristic leaching procedure (TCLP) because of two reasons. First, for a given size of the stabilized sample, the microstructure, and the permeability and diffusion characteristics are different for each of the four binders. The TCLP proce-

dure involves breaking up the waste to pass a 9.5 mm sieve, and therefore, it does not allow observation of leachate differences due to the structural differences of the four binders. Second, the purpose of this experimental program is to compare the feasibility of using each of the four binders and not to assess the absolute leachabilities to check compliance with environmental regulations.

Two types of analytical tests were conducted on each of the liquid samples by different laboratories. The first test was intended to determine concentrations of total phenolic compounds. It resorts to the 4-aminoantipyrine colorimetric method to determine phenol and, ortho- and meta-substituted phenols. It does not determine those para-substituted phenols where the substitution is an alkyl, aryl, nitro, benzoyl, or aldehyde group. The method essentially involves reaction of steam-distillable phenols with 4-aminoantipyrine in the presence of potassium ferricyanide to form a colored antipyrine dye. This dye is then extracted from aqueous solution with $CHCl_3$ and the absorbance is measured at 460 nm. With this method, phenols can be measured in the concentration from 1.0 μg/l to over 250 μg/l, with a sensitivity of 1 μg/l. In the second test, 2,4,6-trichlorophenol was identified as the significant phenolic compound in the EPA list of chlorinated phenols, in both resin-bonded and clay-bonded foundry sands. 2,4,6-trichlorophenol was extracted from leachate with methylene chloride. The solution was then analyzed on a HP 5890A gas chromatograph. The detection limit in this test was about 1.5 μg/l.

3. Test results and discussion

3.1. Mechanical properties

The compressive strengths of stabilized mixes for various replacement levels of foundry sands and at various times are shown in Figs. 2–5. The stabilization due to Portland cement is shown in Figs. 2 and 3 for clay-bonded and resin-bonded foundry sands, respectively. Only the trends in the reduction of strength should be implied from these figures since the absolute values of strength depend on important factors such as water–cement ratio. Although the clean silica sand was replaced by foundry sands in various proportions, the water–cement ratio was kept constant in these experiments. In general, the clay-bonded foundry sand reduced the strength of the stabilized mixes more than the resin-bonded foundry sands. This is attributed to the fact that the bentonite present in the clay-bonded sands absorbed the water and limited the availability of water to cement, thereby reducing the effective water–cement ratio. A similar observation is made in the context of fly ash stabilization (Figs. 4 and 5). The drastic reduction in strength with an increase in clay-bonded foundry sand replacement is apparent in the cases of both fly ash and cement.

A notable difference between cement- and fly ash-stabilized mixes is in the setting time. In general, the cement-stabilized mixes acquired their strength considerably slower than fly ash-stabilized mixes. This is seen in Fig. 6 which presents the strengths of stabilized mixes with respect to time as proportions of maximum strength achieved after 56 d. After seven days of curing, the cement-stabilized RBS reached

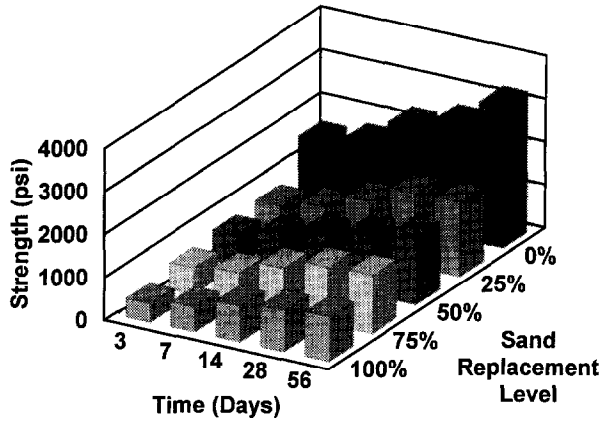## Portland Cement and Clay-Bonded Sand

Fig. 2. Compressive strength vs. time for all replacement levels of CBS in Portland cement.
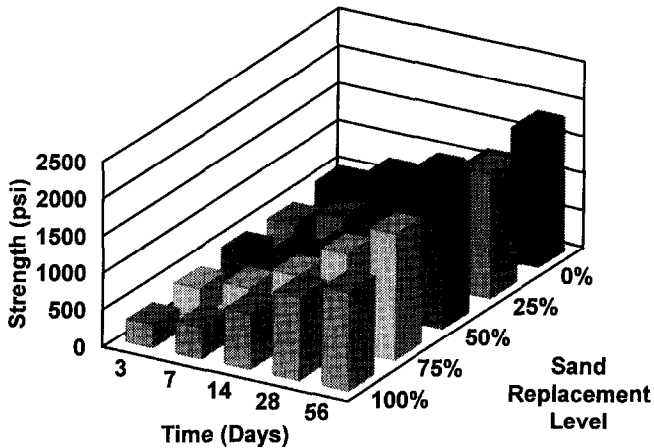
Portland Cement and Resin-Bonded Sands

Fig. 3. Compressive strength vs. time for all replacement levels of RBS in Portland cement.

only 30% of the peak strength whereas its fly ash counterpart achieved 80% of its peak strength. Earlier studies by Vipulanandan and Krishnan [12] indicated that phenolics inhibit setting of cement. They also implied that curing time is an important factor governing the leachability of phenols. Based on these results, the difference in setting time of fly ash and cement observed in the present study is believed to be responsible for the leachability behavior of these mixes, discussed in a later section.

A property equivalent to compressive strength of cement and fly ash stabilized mixes in the case of bentonite and kaolinite stabilization is the compaction behavior. The maximum dry density to which a soil mix can be compacted at a specified

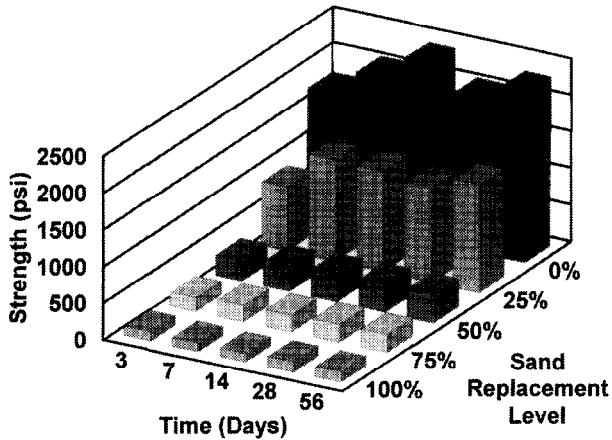## Class C Fly Ash and Clay-Bonded Sand

Fig. 4. Compressive strength vs. time for all replacement levels of CBS in fly ash.

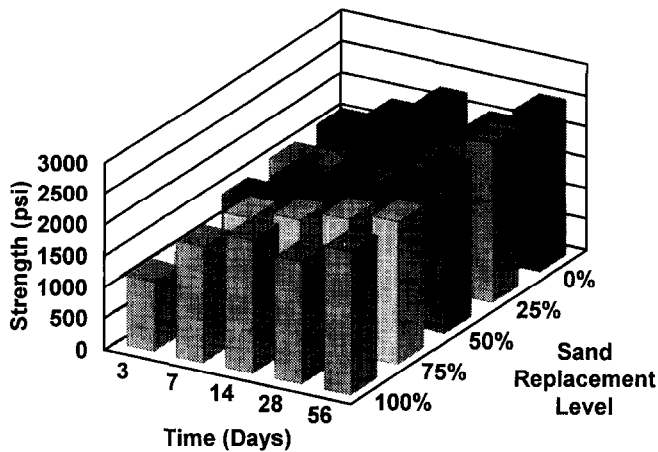## Class C Fly Ash and Resin-Bonded Sand

Fig. 5. Compressive strength vs. time for all replacement levels of RBS in fly ash.

molding water content governs its worth as a backfill material. Although the shear strength of the stabilized mixes is another important factor in geotechnical applications, we limit our discussion here to the compaction behavior only, in view of the anticipated application of the foundry sands as backfill materials. Figs. 7 and 8 show the compaction curves for the individual mixtures of the two clay binders and the foundry sands. To understand the effects of foundry sands on compaction behavior, it is important to bear in mind the fundamental mechanisms involved in soil compaction, first conceived by Hogentogler [15]. These are generally grouped under
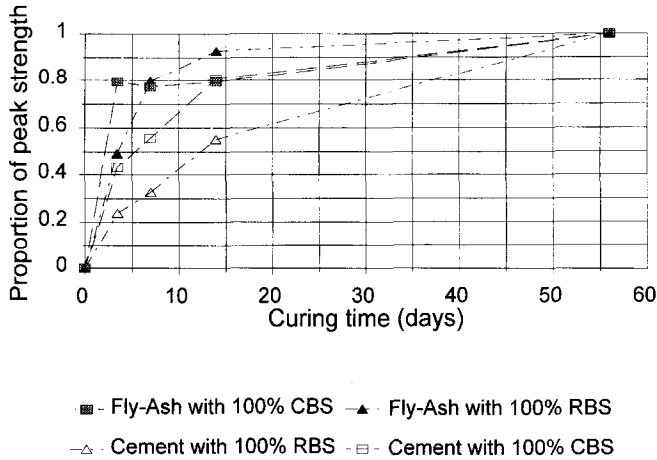
Fig. 6. Proportions of peak strength achieved vs. time for fly ash and cement at 100% replacement level of CBS and RBS.

four stages of wetting, namely, hydration, lubrication, swelling, and saturation. During the initial hydration stage, water is adsorbed by the soil particles as cohesive films. Increased addition of water will lead to the lubrication stage wherein the water will act as a lubricant yielding a closer rearrangement of solid particles in the vicinity of maximum dry density. Subsequent addition of water will result in swelling of the soil with some quantities of air remaining in the swelling stage, and in saturation of the soil with the air completely displaced by water in the saturation stage. It is seen in Figs. 7 and 8 that lower densities were achieved when foundry sand was substituted for clean silica sand in the case of both kaolinite and bentonite. A shift in the maximum dry density is also seen in the case of kaolinite. These are attributed mostly to the differences in grain size distribution of the clean silica sand and the foundry sands (Fig. 1), since it is well known that grain size distributions of sands govern their compaction densities to a large extent. A more significant result, however, is that clay-bonded and resin-bonded sands yielded almost the same compaction curves, indicating that the amount of bentonite in clay-bonded sand did not significantly alter the moisture availability during the processes of hydration, lubrication, swelling, and saturation. Similarly, greater concentrations of phenolics in RBS, when compared with CBS, do not seem to affect any of these four processes during compaction.

3.2. Leachability

The data on total phenolics obtained from leachability experiments were normalized to account for the differences in the volumes of leachates collected for each stabilized mix. This normalization was important to account for unequal amounts of foundry sands used with each of the four binders. To provide a basis for comparison among the four binders, treatment efficiencies were calculated representing
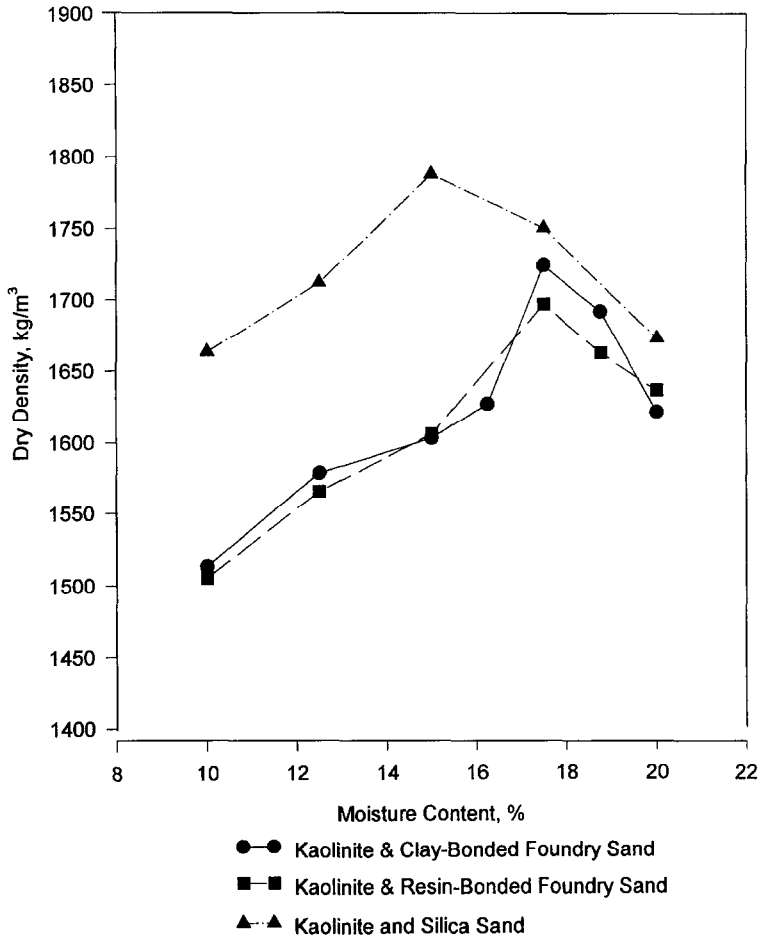
Fig. 7. Harvard compaction curves for kaolinite-stabilized foundry sands.

the percentage of total phenolics immobilized due to the stabilization process. The treatment efficiencies (TE) were used in the existing literature [5], and involve normalizing the contaminants leached from stabilized products with respect to the contaminants leached from untreated waste. *TE* may therefore be expressed as:

$$TE = \frac{C_u - C_t}{C_u} \times 100,$$ (1)

where C_u is the leachate contaminant mass per unit weight of the untreated waste, and C_t is the leachate contaminant mass per unit weight of the waste stabilized by a binder. C_u may be expressed as
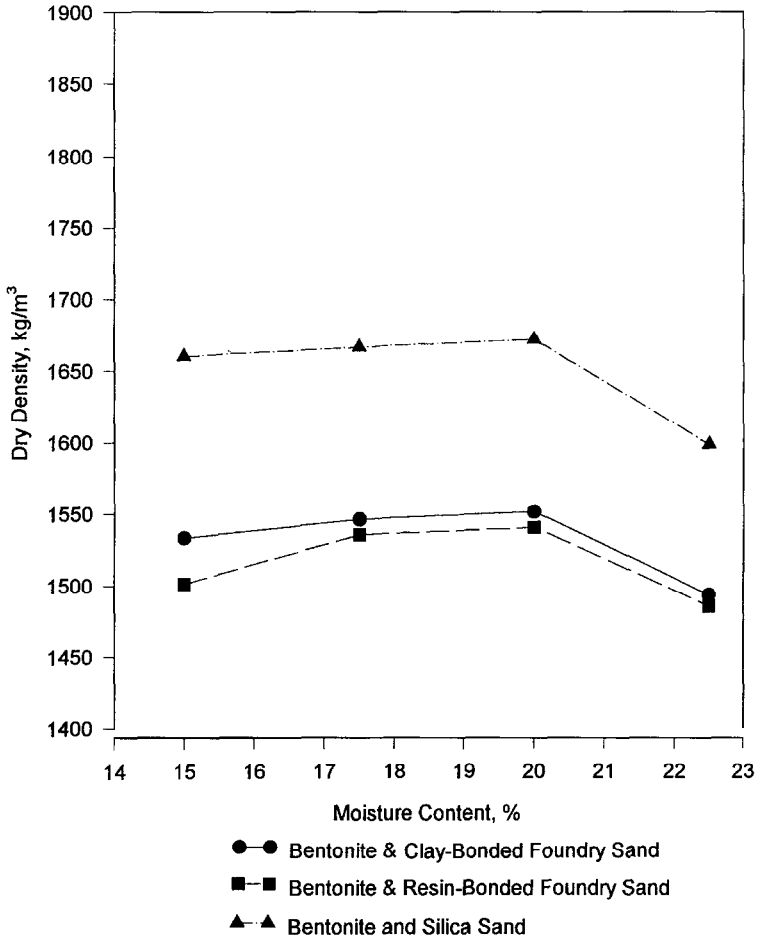
$$C_u = \frac{C_{ut}}{W_{ut}}$$ (2)

Fig. 8. Harvard compaction curves for bentonite-stabilized foundry sands.

where C_{ut} is the total leachate mass and W_{ut} is the weight of the untreated waste extracted. C_t may similarly be expressed as

$$C_t = \frac{C_{tt}}{W_{tt} \times S_t \times F_t} \tag{3}$$

where C_{tt} is the total leachate mass, W_{tt} is the weight of the stabilized (and undried) waste, S_t is the solids proportion of the stabilized waste, and F_t is the weight fraction of the waste in relation to the total weight of the stabilized product, calculated as

$$F_t = \frac{\text{weight of waste}}{\text{weight of waste} + \text{weight of binder}}. \tag{4}$$
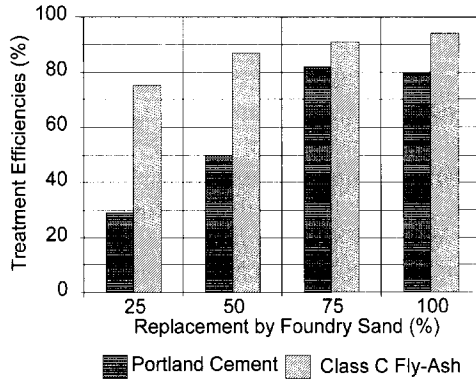
Fig. 9. Treatment efficiencies for Portland cement and fly ash at four replacement levels of RBS.

C_u in Eq. 2 was observed to be relatively constant at 1.54×10^{-6} for total phenolics in the case of resin-bonded sands; however, it exhibited significant variation in the case of clay-bonded sands. The overall phenolic concentrations in clay-bonded sands are, in general, known to be less than those in the case of resin-bonded sands due to the differences in foundry operations. Samples with clay-bonded foundry sands yielded consistently lower leachate masses than those with resin-bonded foundry sands, in accordance with the original phenolic concentrations in the two types of sands. To provide a sound basis for comparing stabilization potentials of each of the four binders, we restrict our discussion on leachabilities to resin-bonded sands only.

The *TE*s as given by Eq. (1) for total phenolics were calculated for each of the mixes; they are presented in Fig. 9 for resin-bonded sands stabilized with Portland cement and fly ash. In obtaining these values, detectable limits were used as the leachates in those cases where phenolics were not detected. As seen in Fig. 9, the
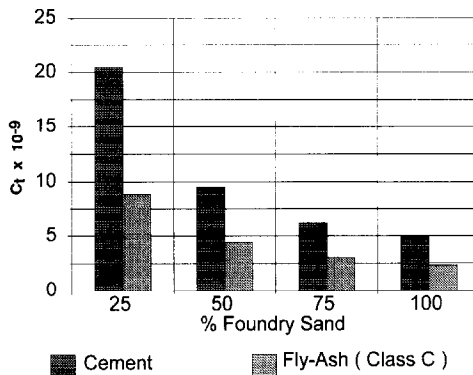

Fig. 10. Mass of 2,4,6-trichlorophenol leached (in grams) per gram of stabilized foundry sand for four replacement levels of RBS.

*TE*s ranged from 29% to 80% for Portland cement and from 75 to 94% for fly ash. In general, the results showed that fly ash is more effective than cement in stabilizing the phenolics. This observation is consistent with the earlier studies reported in Ref. [12] which suggested that phenol is not chemically bound to the cement. Furthermore, their conclusion that phenol recovery is directly dependent on curing time seems to explain the difference between fly ash and cement in this study, since faster setting times were consistently observed for all mixtures of fly ash compared with the corresponding mixtures of cement. It is also seen in Fig. 9 that the treatment efficiencies increased as the percent replacement of foundry sands increased. In other words, the total phenolics leached from each stabilized gram of foundry sand decreased as higher proportions of clean sand were replaced by foundry sand.

It is important to note that the leachates were tested after the samples were soaked for a constant period of time, i.e., 96 h. Although the samples were small in size, it is likely that the structural differences of the stabilized products might not have allowed diffusion to reach an equilibrium stage during this period in some of the mixes. The method of evaluating the leachate quality simulates the practical scenario where the stabilized product is used in a construction application and the leachate from the construction block is governed not only by the binding chemistry at a microscopic level but also by the macroscale permeability and diffusivity of the block. Therefore, the unique increase in *TE*s with increasing levels of foundry sand replacement, if confirmed by future investigations (involving larger-size samples and long-term leachates), has significant implications in the construction industry, e.g., it allows the usage of maximum proportions of foundry sand in stabilization. The ultimate acceptable levels of foundry sand replacement will, of course, be also governed by the desired strength which, as indicated earlier, was observed to decrease with increasing levels of foundry sand replacement.

Results from GC analyses on 2,4,6-trichlorophenol are presented in Fig. 10 for cement and fly ash. The concentrations of 2,4,6-trichlorophenol per each stabilized gram of foundry sand were consistently lower for fly ash than the corresponding concentrations for cement. Moreover, increasing replacement levels of foundry sand
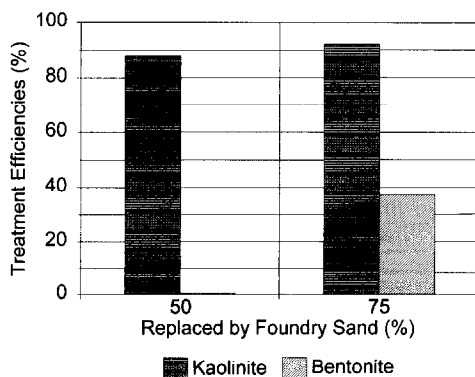

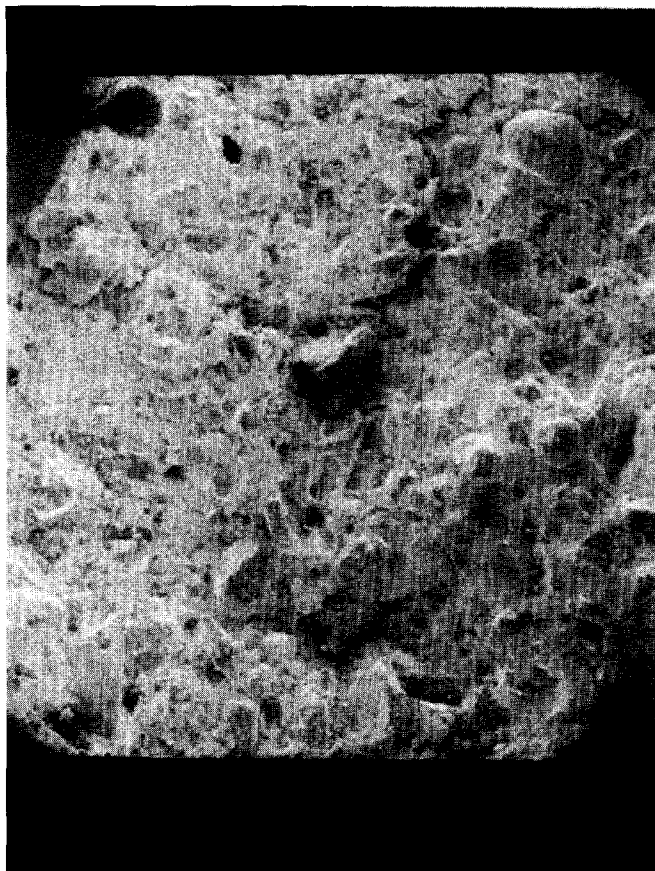Fig. 11. Treatment efficiencies for kaolinite and bentonite at two replacement levels of RBS.

Fig. 12. Scanning electron micrograph of fly ash sample prepared with clean silica sand.

yielded lower concentrations. These results support the earlier trends observed for total phenolics in Fig. 9.

The treatment efficiencies for clay-stabilized RBS are presented in Fig. 11. The *TE*s for total phenolics in bentonite-stabilized mixes were much lower than the corresponding *TE*s in kaolinite. This is, however, a misleading result. The samples stabilized by bentonite exhibited a number of cracks, and due to its swelling potential, the structure of the samples was not intact and was of an unpredictable nature. This enabled a greater opportunity for phenolics to leach out of the samples into the solution. Problems with the integrity of bentonite stabilization are well recognized in the literature [3]; bentonite stabilization presents perhaps one of the lowest potentials for foundry sand utilization. The nonpredictability of leachate quality for bentonite-stabilized samples was supported by results from GC analyses on 2,4,6-trichlorophenol which yielded comparable values of C_t for both bentonite and kaolinite (4×10^{-7} for bentonite and 3×10^{-7} for kaolinite corresponding to a replacement level of 50%).
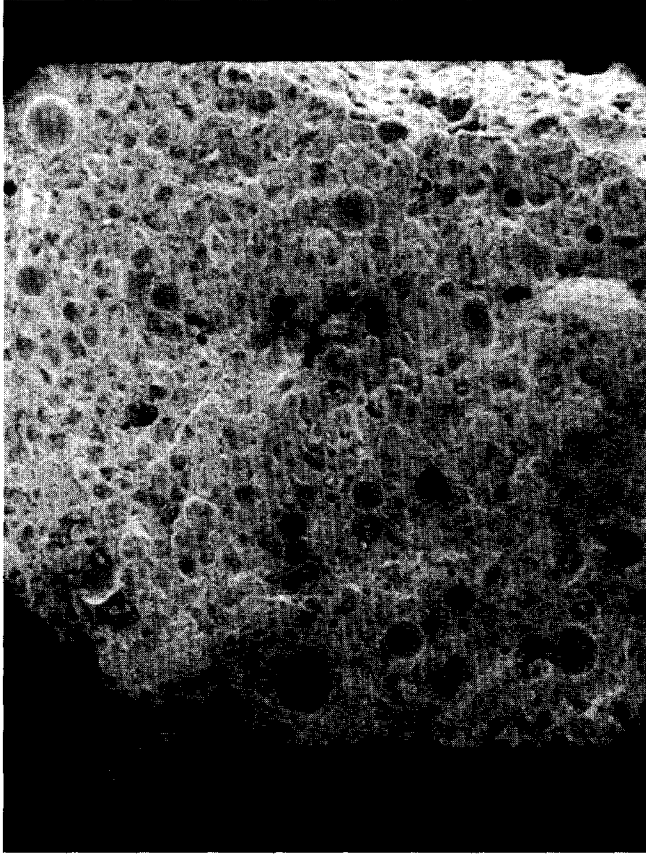
Fig. 13. Scanning electron micrograph of fly ash-stabilized sample with 100% replacement level of RBS.

3.3. Morphology

The morphologies of stabilized RBS as revealed by scanning electron microscope (SEM), are shown in Figs. 12–15. Only the mixes stabilized by cement and fly ash showed discernible features. Mixes stabilized by kaolinite and bentonite exhibited considerable heterogeneity in the porous structure and any conclusions based on a few micrographs would be misleading; therefore, they were not analyzed by SEM. A comparison of Figs. 12 and 13 indicates that fly ash remained relatively unimpacted by the presence of phenolics although blister-type voids were seen consistently in a number of other micrographs. A comparison of Figs. 14 and 15 shows a contrasting feature in cement-stabilized mixes. Large voids, similar to the one shown in Fig. 15, were seen consistently in a number of micrographs on cement-stabilized mixes. The presence of these voids explains the increased leaching observed in cement-stabilized foundry sands and confirms the earlier observation that phenolics were not binding with cement. These observations are in general agreement with the ear-
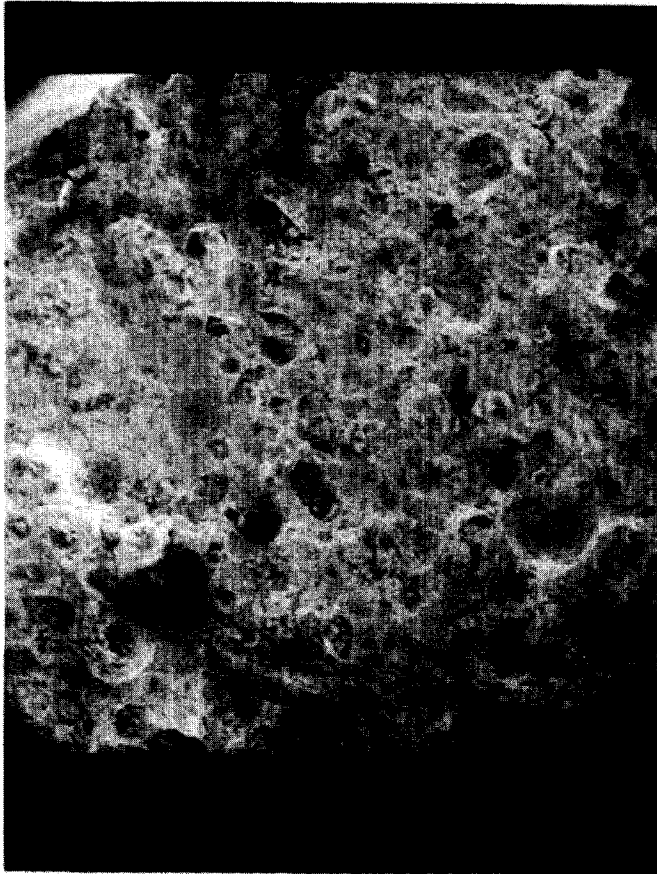
Fig. 14. Scanning electron micrograph of Portland cement sample prepared with clean silica sand.

lier results reported in Ref. [12], which indicate that phenolic concentrations as low as 0.5% can cause a number of large pores in the cement matrix with a blister-type appearance.

4. Conclusions and recommendations

In this study, a comparative analysis was presented on the stabilization of phenolic compounds in foundry sands with four different types of binders. Strength and leachability of stabilized mixes were analyzed to assess the relative stabilizing potential of each of the binders. The following conclusions have been drawn from the study:

(1) Compression strength of stabilized foundry sands decreases as the replacement proportion of foundry sand increases in the mixes, and the strength is achieved relatively faster with fly ash than with cement. In general, the clay-bonded foundry
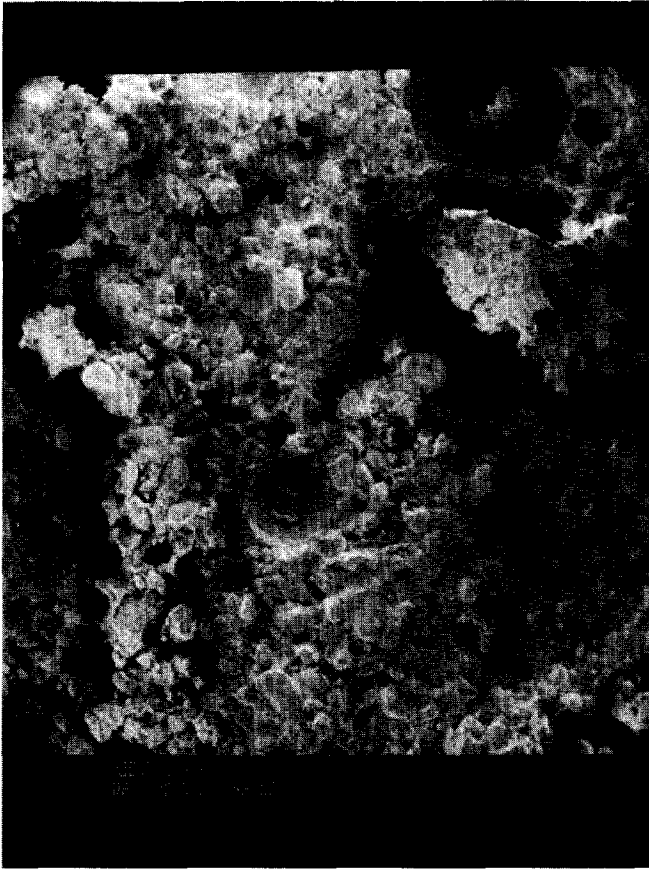
Fig. 15. Scanning electron micrograph of Portland cement-stabilized sample with 100% replacement level of RBS.

sand reduces the strength of stabilized mixes more than the resin-bonded sands, owing to the absorption of water by the clay content.

(2) Both CBS and RBS yield almost identical compaction curves when stabilized with kaolinite and bentonite.

(3) Higher treatment efficiencies are observed (in other words, lesser amounts of total phenolics leached) for fly ash-stabilized mixes than for cement-stabilized mixes; the treatment efficiencies also increase with increasing replacement levels of foundry sands.

(4) GC analyses on 2,4,6-trichlorophenol indicate the same trends as those for total phenolics, i.e., lower concentrations from fly ash than from cement, and lower concentrations for increasing replacement levels of foundry sands.

(5) The swelling potential and instability of bentonite-stabilized mixes render the leachate quality unpredictable.

(6) The morphologies as revealed by scanning electron microscopy support the analytical results on leachate quality by exhibiting relatively large voids in cement-stabilized mixes.

The third and fourth conclusions have important practical implications since they may lead to cost-effective solutions for stabilizing phenolics in foundry sands. However, it is important to validate these comparative analyses by conducting strength and leachability studies with samples similar in size to actual construction blocks and subjecting the samples to long-term environmental conditions such as freeze-thaw. The strengths and leachate qualities reported in this paper are for selected water contents and should not be interpreted as the maximum obtainable values. An optimization of the mix proportions is necessary to evaluate the maximum benefits from each of the four binders. Additives such as polyester polymer were reported in the literature as beneficial in enhancing stabilization of phenolics. The variations in binding chemistry due to such additives should be studied for each of the four binders in order to maximize its stabilization potential. Such studies might lead in the near future to a cost-effective utilization of foundry sands in the construction industry.

Acknowledgements

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