Fly Ash–Cement Mixtures for Solidification and Detoxification of Oil and Gas Well Sludges

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Oil and gas well sludges usually consist of drill mud and formation cuttings, as well as various hydrocarbons. They are in a semiliquid state and vary widely in composition. The present environmental regulations in Canada and the United States require these sludges to be disposed of in an environmentally sound manner. Laboratory studies were conducted to determine the feasibility of solidifying and stabilizing oil and gas well sludges using portland cement, fly ash, lime, and sodium silicate mixtures. Oil well sludge samples from a site in Alberta, Canada, were mixed with various proportions and different combinations of the solidifying agents. Compressive strength tests were conducted to determine an optimum formulation of cement and fly ash that may be used to solidify the sludge. Hydraulic conductivity and microtoxicity tests were conducted on the solidified samples. The test results indicate that oil and gas well sludges in a semiliquid state with a solid content greater than 30 percent and having moderate toxicity can be effectively solidified using cement and fly ash mixtures with 15 percent of each by weight of solids in the sludge. Addition of 1 percent sodium silicate by weight of cement accelerates the strength development. Solidification also reduces the toxicity of sludges.

Portland cement, a binding reagent, has been effectively used alone or with some chemical or mineral admixtures, or both, for solidification and stabilization of a wide variety of wastes (*1–4*). Relatively fewer studies have been conducted with cement fly ash as the primary binding reagent in waste management, particularly related to the treatment of oil and gas well sludges.

The sludges generated during the drilling of wells for exploration and production of oil and gas mostly consist of drilling mud and well cuttings. These sludges are usually in a semiliquid state and have a complex composition. They contain a wide range of inorganic, organic, and polymeric compounds. Usually sludges have a large proportion of bentonite and water. However, chlorides of sodium and potassium, sulphates, lignosulphonates, oil emulsions, and certain heavy metals may also be present, making the sludge toxic. If left untreated, various components of the sludge may leach into the surrounding soil and groundwater, causing severe pollution problems.

It is estimated that there are currently thousands of old or orphan oil and gas well drilling sumps in Alberta that contain drilling mud sludge. Furthermore, a large number of wells are drilled each year, and about 60 percent of these wells have sumps 3 to 15 m deep to contain the waste drilling mud and well cuttings. About 50 percent of the sumps have two to four compartments for storing different types of fluids and cuttings. Usually the depth of a sump is about 5 m, and it contains sludge in semiliquid form. The solid cuttings are generally stacked on the banks. These sludges are classified as water bound, salt water bound, and oil bound and may also have varying amounts of hydrocarbons. Heavy oil sludges are generally in the form of wet sands or asphalt sands and are effectively used as road material. The semiliquid sludges from oil and gas wells generally require treatment before disposing in landfills. Most of the hydrocarbons are separated from the sludges by floatation, and the sludges are kept exposed to ambient conditions for clarification and stabilization by natural processes of evaporation, oxidation, and sedimentation.

Most of the water-bound sludges containing significant amounts of solids can be effectively solidified and stabilized by using portland cement. Because of its cohesive and adhesive characteristics, portland cement converts the sludge to a weak mortar or soil-cement–like material. The sorptive properties of portland cement can also reduce the toxicity of sludges (*2,5–7*). On solidification with cement-based materials, the water content of the sludge decreases; in addition, the physical and chemical characteristics alter decreasing leachate migration through the pores of the solidified sludges. Thus, the possibility of contamination from the solidified sludges should be considerably reduced or at least mitigated. It is thus expected that by treating sludges with mixtures of portland cement, fly ash, and other additives, oil and gas well sludges can be solidified and stabilized for disposal by acceptable methods. This paper describes a study conducted to evaluate formulation mixtures using portland cement, fly ash, lime, and other materials for solidifying and detoxifying the oil and gas well sludges encountered in Alberta, Canada.

MATERIALS AND METHODS

Sludge Type

The sludge samples were visually examined to explore the feasibility of their solidification by using cement and fly ash mixtures. Highly aqueous, oil bound, and highly toxic sludges with a hydrocarbon content greater than about 2 percent are not amenable for treatment with portland cement (*2,8*) and were not considered in this study.

Most of the tests were conducted on the two semiliquid sludge samples from Amerada Hess. Toxicity tests on this sludge indicated that it was nontoxic. To study the effect of portland cement and other additives on the toxicity of the sludge, a mixture of the Amerada Hess sludge and another sludge from an oil well of Shell Canada were considered. The composition and the nature of the contaminants in the sludges could not be obtained from industry. A

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few physical tests were conducted to characterize the sludge samples for their treatment with cement fly ash mixtures. The combined water and organic content of the sludge from Amerada Hess ranged from 120 to 220 percent. The solids content of the sludge was used as a base to express the amount of stabilizing agent used for the tests. The Amerada Hess sludge was designated as S_1. For toxicity tests, two composite samples marked S_2 and S_3 were made using 90 and 80 percent Amerada Hess sludge and 10 and 20 percent high liquid (aqueous) Shell sludge, respectively. The Shell sludge was found to be highly toxic in its virgin state. The composite samples S_2 and S_3 thus had moderate toxicity and were in a semiliquid state and thereby amenable to treatment with cement–fly ash mixtures.

Materials Used

Normal portland cement (ASTM Type 1 or CSA Type 10), Alberta fly ash obtained from the Forestburg power plant, and hydrated lime in various proportions were used as solidifying agents. Sodium silicate (water glass) was used as a dispersant for the cement. Demineralized water and the necessary reagents and bacterial culture were used for microtoxicity testing. The physical and chemical characteristics of the fly ash used are presented in Table 1.

The following combinations of materials were used to prepare trial mixes for solidification of the sludge:

1. Only portland cement at 20, 30, 40, and 50 percent by weight of solids in the sludge;
2. Portland cement as in A plus 1 percent lime by weight of solids in the sludge;
3. Portland cement as in A plus 1 percent sodium silicate by weight of the cement used (a mix with 10 percent cement was also tried.);
4. Portland cement plus fly ash mixed in equal proportions by weight and the mixture at 20, 30, 40, and 50 percent by weight of solid material in the sludge; and
5. Mixtures of lime and fly ash in proportions of 1:5 by weight at 10 to 50 percent by weight of solids in the sludge.

Table 2 describes the designations and details of the stabilizing mixtures, reported in this study.

TABLE 1 Chemical Analysis and Physical Properties of Fly Ash (Unclassified) from Forestburg Power Plant

Chemical Analysis	
Silicon dioxide, SiO_2	61.41
Aluminum oxide, Al_2O_3	20.69
Iron oxide, Fe_2O_3	4.44
Total ($SiO_2+Al_2O_3+Fe_2O_3$)	86.54
Sulphur trioxide, SO_3	0.24
Calcium oxide, CaO, total	6.91
Available alkalies	0.54
Loss on ignition	0.50
Moisture content	--
pH	10.8
Physical Properties	
Fineness-Retained on 45μm (no. 325) sieve	22.2
Strength Activity Index with cement:	
% of control at 7 days	82.3
% of control at 28 days	85.5
Strength Activity Index with lime, MPa	8.0
Specific Gravity	1.92
Increase of drying shrinkage at 28 days	0.01%
Uniformity Requirements:	
- Specific gravity variation from average	-2.0%
- Fineness, 45μm sieve, variation from average	+2.4%
- Soundness: Autoclave expansion (%)	0.05
- Quantity of Air-entraining Agent (%)	0.178
Uniformity (%)	+20.3
- Reactivity with cement alkalies:	
Reduction at 14 days (%)	73.1

Test Methods

The sludge samples for the tests were kept in properly sealed plastic pails. The combined water content plus organic content of the sludges was determined, and the solids content of the uniformly mixed sludge was computed. For solidification, predetermined quantities of the various stabilizing agents were thoroughly mixed with the sludge. No additional water was used for mixing. The details of the test procedures used in this study are described elsewhere (*9,10*).

The sludge mixed with various stabilizing agents was tested to determine its compressive strength. Cylindrical specimens that were 25 mm in diameter and about 51 mm long were cast in plastic molds. The samples were air sealed with polyethylene sheet and cured in a fog room for 1, 3, 7, and 28 days. Before testing, the specimens were weighed and their length and diameter were measured. Compression tests were conducted with an INSTRON machine. After the test, the diameter of the failed specimen and the water content of the tested specimen were determined. For each test parameter, three samples were tested. As the test results were consistent, the average value was considered.

Two tests were conducted to determine the hydraulic conductivity, k, of the solidified sludge after 28 days of curing in a double-ring fixed-wall permeameter (*9*) before commencement of water permeation. The height of the sample was 110 mm, and a hydraulic gradient of 20 was used for the permeability tests.

Microtoxicity tests were conducted in accordance with the Canadian standard procedures on untreated as well as solidified sludge samples with a Microtox Instrument. The following procedure was used to prepare samples for microtoxicity testing (*11,12*):

1. After the strength test on solidified sludge, a 1:1 solution wt/wt using demineralized water of weight equal to that of the solid sample was prepared.
2. The 1:1 solution was shaken for 12 hr.
3. The sample was then centrifuged to clarify.
4. The pH of the sample was adjusted to lie in the range of 6.0 to 8.8, using 0.1 N H_2SO_4 or 0.1 N NaOH.
5. The sample was then stored at 4°C. The microtox test was conducted within 72 hr of the sample preparation.
6. By using salt water bacteria (lumin acid) and the microtox instrument, the value of EC 50, an index to measure the toxicity, was determined.

First, the microtox test was conducted on the raw sludge sample. If found toxic, the sludge was solidified using the stabilizing com-

TABLE 2 Details of Stabilizing Mixtures and Solidifying Agents

Test series	Solidifying agent designation	Amount of solidfying agent by weight of solids in the sludge
A only normal portland cement	A20	20% cement
	A30	30% cement
	A40	40% cement
	A50	50% cement
B Cement+Lime at 1% by weight of solids in the sludge	B20	20% cement+1% lime
	B30	30% cement+1% lime
	B40	40% cement+1% lime
	B50	50% cement+1% lime
C Cement+sodium silicate at 1% by weight of cement	C10	10%cement+0.01%sodium silicate
	C20	20%cement+0.02%sodium silicate
	C30	30%cement+0.03%sodium silicate
	C40	40%cement+0.04%sodium silicate
	C50	50%cement+0.05%sodium silicate
D Cement+fly ash in equal proportion by weight	D20	10%cement+10%flyash
	D30	15%cement+15%flyash
	D40	20%cement+20%flyash
	D50	25%cement+25%flyash

pounds. The microtox test was then conducted on the solidified material to assess the effectiveness of solidification on reducing the toxicity of the sludge.

RESULTS AND DISCUSSION

Compressive Strength

The results of compressive strength tests on treated and solidified sludges are shown in Figures 1 through 4 and Tables 3 and 4.

The solidified sludge for effective disposal in a landfill should have some minimum strength to ensure that it can support the weight of the overburden placed on the top. Thus, a minimum compressive strength of about 140 kPa (20 psi) was adopted to meet the requirements (*2*).

It is observed from Figures 1 through 4 and Tables 3 and 4 that compressive strength increases with age and with an increase in the amount of the solidifying agent. The addition of lime to cement to improve the solidification is insignificant. However, the addition of sodium silicate at 1 percent by weight of cement accelerates the strength development at all the ages up to 28 days. The increase in strength with sodium silicate addition is even greater with richer mixes starting from the early age of 1 day. Sodium silicate gels rapidly by reacting with the calcium hydroxide released from hydration of portland cement. The rapid gelation of cement mixtures with sodium silicate stabilizes the sludge and prevents the deleterious materials in the sludge from affecting the setting and hardening of cement (*2,13*). Better dispersion of cement in the mix, with sodium silicate, leads to improved hydration, which results in higher strength. Furthermore, the rapid gel formation from sodium silicate reduces the mobility of the substances present in the sludge that may interfere with cement hydration.

With the addition of fly ash, the early-age strength is considerably reduced. However, at 28 days, the effect of pozzolanic action of fly ash becomes prominent as is evident by marked increases in 28-day strength of cement and fly ash mixtures compared with those of the corresponding mixtures with cement alone. The relative increase in the 28-day-strength sample, which contained 10,

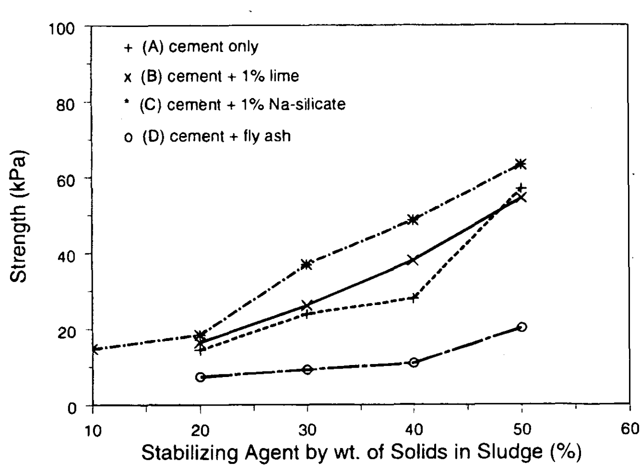

FIGURE 1 1-day compressive strength versus amount of stabilizing agent mixed with sludge.

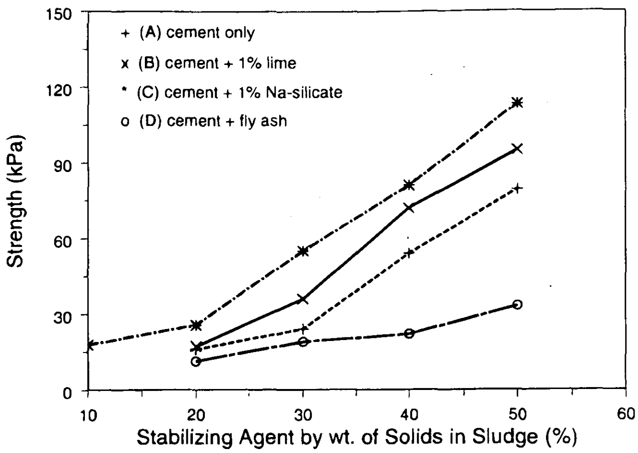

FIGURE 2 3-Day compressive strength versus amount of stabilizing agent mixed with sludge.

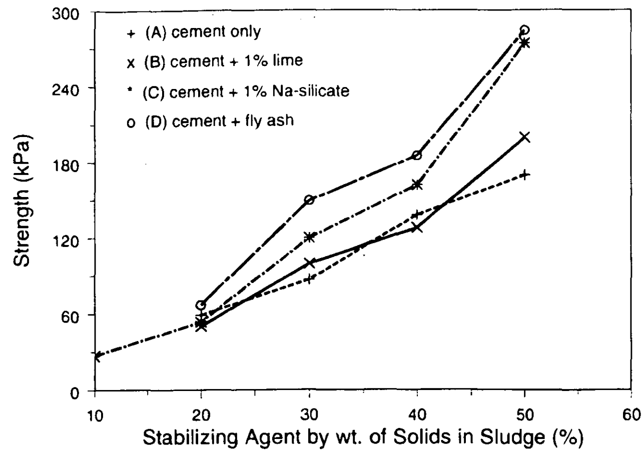

FIGURE 4 28-day compressive strength versus amount of stabilizing agent mixed with sludge.

15, 20, and 25 percent fly ash as cement replacement, is 12, 72, 34, and 67 percent, respectively. Alberta ashes are classified as Class F per ASTM C 618. They possess pozzolanic properties and are also somewhat self-cementitious. During cement hydration, calcium hydroxide is released, which reacts with fly ash to produce cementitious compounds similar to those produced by cement hydration. Pozzolanic reactions are relatively slow and depend on cement hydration. The ultimate strength or long-term strength of the sludge samples solidified with cement and fly ash would be higher than that of those solidified with cement alone (*1,14*).

A few trial tests, Series E, were conducted using lime and fly ash mixtures. These mixtures containing 1:5 lime and fly ash did not solidify the sludges effectively, even after 7 days, and were not considered for further investigations. Similarly, preliminary tests on mixtures containing 5 percent cement and 5 percent fly ash as well as 10 percent cement alone by weight of solid material in the sludge were not found to be effective in solidifying the sludge sample. However, the addition of sodium silicate to the 10 percent cement mixture hardened the sludge within 24 hr.

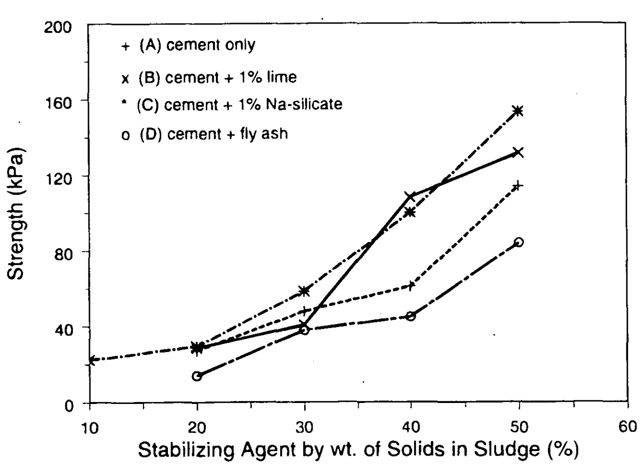

FIGURE 3 7-day compressive strength versus amount of stabilizing agent mixed with sludge.

Based on the strength results, the potential solidifying agents were identified as (a) mixtures containing 40 percent cement with or without lime and sodium silicate (A40, B40, and C40) and (b) cement and fly ash mixtures containing 10 percent cement and 10 percent fly ash (D20), 15 percent cement and 15 percent fly ash (D30), and 20 percent cement and 20 percent fly ash (D40) by weight of solid material in the sludge.

The compressive strength results of the composite samples, S_2 and S_3, 7 to 28 days, are given in Tables 3 and 4. As can be observed from the data in Tables 3 and 4, the strength values of the samples made from S_2 sludge containing 90 percent Amerada Hess sludge and 10 percent Shell sludge are much higher than those made from S_3 sludge containing 80 percent Amerada Hess sludge and 20 percent Shell sludge under similar conditions. For the sludge sample S_2, all the solidifying agents identified and tried were found suitable. However, for the sludge sample S_3, the solidifying agent that gave the specified strength of 140 kPa at 28 days was 20 percent cement and 20 percent fly ash. Even the mixture containing 15 percent cement and 15 percent fly ash, which solidified the sludge and imparted strength of 108 kPa, can be acceptable because with age the strength is anticipated to increase considerably because of the pozzolanic reactivity of fly ash.

Hydraulic Conductivity

The hydraulic conductivity test conducted on the 28-day cured and solidified samples of the composite sludge S_3 using two solidifying agents—15 percent cement and 15 percent fly ash (D30) and 20 percent cement and 20 percent fly ash (D40)—gave k values as 1.85×10^{-6} cm/sec and 2.01×10^{-6} cm/sec with D40 and D30 mixtures, respectively. A hydraulic conductivity, k, of about 10^{-6} cm/sec indicates a fairly impervious material to control the migration of leachates (*15*).

Toxicity Tests

Toxicity of virgin and solidified sludge samples was considered on an overall basis per the Canadian Standards (*11,12*). Individual toxicants or contaminants were not identified to define toxicity levels because of experimental constraints.

TABLE 3 Compressive Strength Results of Composite Sludge Sample, S_2, Containing 90 percent Amerada Hess Sludge and 10 percent Highly Liquid Shell Sludge

Solidifying Agent Designation	Amount by weight of solid content in the sludge sample	Compressive strength in kPa at	
		7 days	28 days
A40	40% cement	658.0	1221.0
B40	40% cement + 1% lime	634.0	963.0
C40	40%cement+0.04% sodium silicate	770.0	1157.0
D20	10% cement + 10% fly ash	81.0	386.0
D30	15% cement + 15% fly ash	149.0	648.0
D40	20% cement + 20% fly ash	294.0	957.0

Microtox analyses of the untreated virgin sludge samples showed Amerada Hess sludge to be nontoxic and the liquid Shell sludge to be highly toxic. The results of Microtox analyses of the composite samples are presented in Tables 5 and 6. It is observed from the data in these tables that the sludge sample S_2, which contained 90 percent Amerada Hess sludge and 10 percent highly liquid Shell sludge, showed slight toxicity in the virgin state. However, when solidified with the identified solidifying agents, no toxicity was observed, even after 7 days of curing.

The sludge sample S_3, which contained 80 percent Amerada Hess sludge and 20 percent highly liquid Shell sludge, was quite toxic (EC 50 < 20) in the virgin state. After 7 days of curing, 40 percent cement mixtures containing (a) 40 percent cement (A40), (b) 40 percent cement and 1 percent lime (B40), and (c) 40 percent cement and 1 percent sodium silicate (C40) made the sample nontoxic. However, the sample treated with a cement–fly ash mixture consisting of 10 and 20 percent each (D20–D40) retained the toxicity until 7 days after treatment. But after 28 days of curing, the sludge samples treated with cement and fly ash mixtures D30 and D40 were nontoxic, whereas slight toxicity was observed in the sample treated with the 10 percent mixture of cement and fly ash each (D20). Pozzolanic reactions are slow and they continue over a long period of time. It is, therefore, anticipated that with a further increase in curing age, even the stabilizing agent D20 will solidify and stabilize the sludge sample S_3 to make it nontoxic.

TABLE 4 Compressive Strength Results of Composite Sludge Sample, S_3, Containing 80 Percent Amerada Hess Sludge and 20 Percent Highly Liquid Shell Sludge

Solidifying Agent Designation	Amount by weight of solid content in the sludge sample	Compressive strength in kPa at		
		7 days	14 days	28 days
A40	40% cement	91.0	105.3	134.0
B40	40% cement + 1% lime	77.0	85.5	112.0
C40	40%cement+0.04% sodium silicate	92.5	100.7	131.5
D20	10% cement + 10% fly ash	19.0	29.7	45.9
D30	15% cement + 15% fly ash	28.3	55.0	108.0
D40	20% cement + 20% fly ash	32.7	70.2	183.5

CONCLUSIONS

On the basis of the results of the experiments conducted on oil and gas well sludge samples using cement and fly ash based materials, the following conclusions can be drawn:

- Oil and gas well sludges or drilling mud in the semiliquid state with a solids content greater than 30 percent and having moderate toxicity can be effectively solidified with cement and fly ash content as low as 15 percent each by weight of solids in the sludge.
- The addition of 1 percent sodium silicate by weight of cement accelerates the strength development and also increases the strength of solidified sludge samples treated with cement as the stabilizing agent.
- At least 30 percent by weight of solids in the sludge is required to produce satisfactory stabilized products at a reasonable cost.
- The effect of lime addition at 1 percent by weight of solid content in the sludge before treatment with cement does not exhibit any significant effect on the strength of sludge treated with cement.
- The sludges solidified with cement and fly ash mixtures become reasonably impervious with a hydraulic conductivity, k, about 2.0×10^{-6} cm/sec.
- Solidification of the sludges also reduces toxicity.
- Cement and fly ash mixtures, 1:1 by proportion, at about 20 to 30 percent by weight of solids in the sludge may be used to stabilize and dispose oil and gas well sludges of moderate toxicity in a technically effective and environmentally sound manner. However, for a particular type of sludge, trial tests to obtain optimum formulation will be necessary.

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TABLE 5 Results of Microtox Test on Sludge Samples S_1, S_2, and S_3 Before Solidifying

Sludge Sample	EC* 50 in (%)	Toxicity*
S1- Amerada Hess Sludge	>90	Non toxic
S2- 90% Amerada Hess sludge + 10% highly liquid Shell sludge	60	Slightly toxic
S3- 80% Amerada Hess sludge + 20% highly liquid Shell sludge	<20	Toxic

* EC 50 value in %	Toxicity
0-50	Toxic
50-90	Slightly toxic
> 90	Non toxic

TABLE 6 Results of Microtox Test on Solidified Samples of Sludge S_2 and S_3

Solidifying Agent Designation	Amount by weight of solid content in the sludge sample	EC 50 values in % and toxicity					
		Sample S2		Sample S3			
		7 day fog curing		7 day fog curing		28 day fog curing	
		EC 50	Toxicity	EC 50	Toxicity	EC 50	Toxicity
A40	40% cement	>90	nontoxic	>90	nontoxic	>90	nontoxic
B40	40cement+ 1%lime	>90	nontoxic	>90	nontoxic	>90	nontoxic
C40	40% cement + 0.04% sodium silicate	>90	nontoxic	>90	nontoxic	>90	nontoxic
D20	10% cement + 10% fly ash	>90	nontoxic	28	toxic	60	slightly toxic
D30	15% cement + 15% fly ash	>90	nontoxic	40	toxic	>90	nontoxic
D40	20% cement + 20% fly ash	>90	nontoxic	67	slightly toxic	>90	nontoxic

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