

## THE REMOVAL OF ARSENIC(V) FROM ACIDIC SOLUTIONS

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### Summary

The residual concentrations of dissolved arsenic(V) in 1.5% sulphuric acid solutions treated with iron salts and alkaline earth metal compounds have been measured. The results are plotted on triangular composition diagrams covering the composition ranges As 0.2% to 1.2%, Fe 1.0% to 4.0%, alkaline earth metal 3.0% to 6.5%. The results indicate that ferrous iron and lime are the most effective in removing 99.9% of the arsenic over a wide composition range to give residual dissolved arsenic concentrations of ca. 0.5 ppm. Ferric iron is more effective when used in conjunction with mixed calcium and magnesium hydroxides than with lime alone. Ferric iron and dolomite offered the least effective treatment.

The effect of lime particle size on the residual dissolved arsenic concentration following ferrous iron-lime treatment was studied. The optimum lime particle size range for arsenic removal was 50–100  $\mu\text{m}$ .

Leaching tests were carried out on samples of arsenic bearing sludge from the ferrous iron and lime treatment process mixed with sand chalk and clay soils. The presence of 5,000 ppm of acetic acid in the aqueous leachant did not appear to significantly affect the leaching test results and, after passage through contaminated and uncontaminated soils arsenic concentrations in the leachate were generally below 0.05 ppm. Ferrous iron and lime appeared to be suitable for treating the arsenic solutions and the resulting sludge seemed to be suitable for land disposal.

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### Introduction

A recent review [1] indicates that ferric salts may be used to effect better than 90% removal of arsenic from effluents containing up to ca. 300 ppm of arsenic. The purpose of the work reported here is to establish the efficiency of iron hydroxide treatment for effluent from an electrolytic copper reclamation process which contains 3,000 ppm (0.3%) arsenic (V) in 1.5% sulphuric acid solution. In addition, the combination of iron salts and alkaline earth metal hydroxides or carbonates which is most effective in removing arsenic was to be established, and the leaching properties of the resulting arsenic bearing sludge were to be investigated.

Solutions of  $\text{As}_2\text{O}_5$  in 1.5% sulphuric acid were used as models for the process effluent and these were mixed with solutions of iron(II) or iron(III) to give various iron to arsenic ratios. Varying quantities of alkaline earth metal

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hydroxides or carbonates were then added and the solid phase subsequently separated by centrifugation. The supernatant was analysed for arsenic and the results expressed on a triangular diagram for the concentration ranges: arsenic 0.2 to 1.2%, iron 1.0 to 4.5%, alkaline earth metal 3.0 to 6.5%. The combinations of iron and alkaline earth metals selected for study were as follows.

- (i) ferric sulphate plus calcium hydroxide (lime);
- (ii) ferric sulphate plus 1:1 calcium hydroxide (lime) and magnesium hydroxide (laboratory reagent);
- (iii) ferric sulphate plus magnesium calcium carbonate (dolomite);
- (iv) ferrous sulphate plus calcium hydroxide (lime).

Attempts to assess the effects of the particle size of the alkaline earth metal salts on arsenic removal efficiency were hampered by the discovery that all the available samples of dolomite or lime were in the form of fine powders. Apparently larger particles of material proved to be agglomerates of finer particles which could be separated by prolonged sieving. However, some experiments were carried out using the different particle size ranges collected in a sieve tower despite the fact that some of the particles were agglomerates.

In addition to the arsenic removal experiments the leaching of the arsenic containing sludge produced by the most effective arsenic removal process was investigated. Samples of sludge were mixed with sand, clay or chalk soils and contacted with water or 5,000 ppm acetic acid solution, the arsenic content of the leachate were then contacted with samples of uncontaminated soil to assess the capacity of the soils for removing arsenic from the leachates.

## Experimental section

### *Arsenic removal experiments*

A stock solution of ca. 2% arsenic in 1.5% sulphuric acid was prepared from  $\text{As}_2\text{O}_5$  (Technical Grade Reagent) and its arsenic content checked by analysis. Stock solutions of 10% iron(III) as ferric sulphate and of 5% iron(II) as ferrous sulphate were also prepared using reagent grade laboratory chemicals and 1.5% sulphuric acid as solvent. Aliquots of these stock solutions were mixed and made up to 10 ml with 1.5% sulphuric acid to give combinations of iron and arsenic concentrations in the ranges 0.2 to 1.2% arsenic and 1.0 to 4.5% iron. Varying amounts of alkaline earth metal compounds were added as solids to these solutions and the mixtures stirred for 5 minutes (20 minutes in the case of iron(II) solutions to allow oxidation of the iron to occur). The mixtures were then allowed to settle for 1 hour (45 minutes for iron(II) giving the same overall contact time of 1 h 5 min) and the supernatant was removed and centrifuged for 15 minutes at 3,500 r.p.m. in an MSA minor model centrifuge. A 5 ml aliquot of the centrifuged liquor was diluted to 10 ml with 10% hydrochloric acid and submitted for analysis.

The alkaline earth metal compounds used in the arsenic removal experiments were calcium hydroxide obtained as class A lime (to BS 890) from Staveley Lime Products, Buxton, dolomite obtained as 'Dolodust' (80–100 mesh) from

Steeley Limited, Mineral Division, Worksop (chemical assay CaO, 30.5%; MgO, 21.0%; SiO<sub>2</sub>, 6.0%; Fe<sub>2</sub>O<sub>3</sub>, 0.6%; Al<sub>2</sub>O<sub>3</sub>, 0.3%) and an equimolar mixture of calcium and magnesium hydroxides comprising lime and laboratory reagent grade magnesium hydroxide. The lime was sieved to the particle size range 250 to 315 microns but the bulk of the particles were probably agglomerates of finer particles and no sources of lime of large particle size could be identified.

### *Leaching experiments*

The three soils used for the leaching experiments were sand from Pusey Pits near Stanford in the Vale, chalk from Frilsham Agricultural Lime near Abingdon and Gault Clay obtained from a road construction site at Steventon near Abingdon. An arsenic bearing sludge was prepared by mixing solutions with lime to give 0.4% As, 2.0% Fe<sup>II</sup> and 3.99% Ca. After centrifugation the sludge was mixed with the three different soils to the extent of 20% w/w wet weight. A sample of wet sludge was also mixed with dried clay to the extent of 20% w/w. The moisture content of the three soils was: sand 0%, chalk 13% and clay 27% w/w as measured by drying at 80°C for 24 h. The moisture content of the sludge determined in a similar manner was 61%.

In the cases of sand and chalk soils two 100 gram samples of the contaminated soils were placed in two glass columns of 1.55 cm internal diameter and compacted to give a bed depth of ca. 10 cm. Four similar columns were prepared using 100 gram samples of uncontaminated soils, two each for sand and chalk. The contaminated soil columns were leached with demineralised water or 5,000 ppm acetic acid solution at the rate of 2 bed volumes or 150 ml per week and 5 ml samples of leachate taken at intervals during the experiment. The rest of the leachate was delivered onto the tops of columns containing the uncontaminated soil of the same type and the leachates from these columns were also collected for analysis.

The impermeability of the wet clay rendered it unsuitable for column leaching tests and a different approach was used in this case. A 50 g sample of contaminated clay (both wet and dry clay was used) was shaken for 24 hours with 100 ml of water or 5,000 ppm acetic acid. After this time the mixture was centrifuged, the supernatant decanted and, after removal of a 5 ml sample for analysis, this liquid was placed in a flask containing 50 g of uncontaminated clay. The decanted supernatant was replaced with an equal volume of fresh water or acetic acid and both flasks shaken for 35 h, centrifuged and the supernatant removed. This process was repeated until four 100 ml aliquots of leachant had been successively contacted with contaminated and uncontaminated clay. Samples of leachates from each stage of the experiment were then analysed for arsenic.

The arsenic determinations were carried out by Analytical Services Section, Chemical Analysis Group, Environmental and Medical Sciences Division, Harwell using standard atomic absorption techniques.

Removal of arsenic(V) from acid solutions

*Lime and ferric sulphate*

The concentrations of arsenic in the supernatant liquors of solutions treated with ferric sulphate and lime are presented in the form of a triangular diagram in Fig. 1. The percentage removal of arsenic which corresponds with these figures is shown in Fig. 2. The observed arsenic concentrations range from 0.4 to 3,820 ppm and the percentage removal figures from 4.5 to 99.99%. Thus the performance of ferric sulphate/lime treatment in removing arsenic from acid solution is somewhat variable. In broad terms Figs. 1 and 2 may be divided into two regions. One is defined by Ca concentrations of 4.0 to 6.5% in which arsenic removal is better than 95% and arsenic levels are, for the most part,

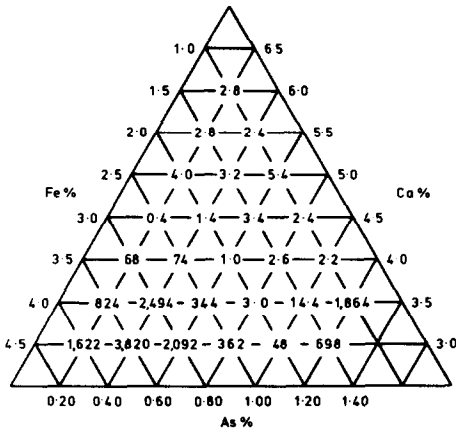


Fig. 1. Concentrations of arsenic (w/v ppm) in supernatant following ferric sulphate/lime treatment.

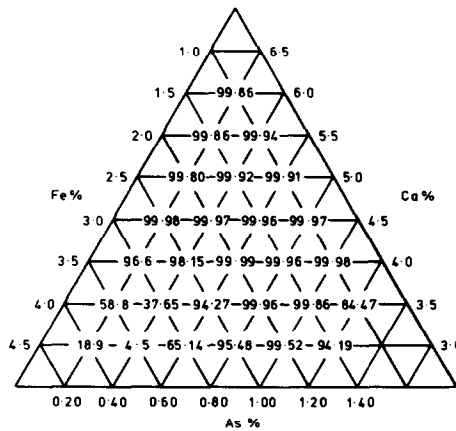


Fig. 2. Percentage arsenic removal following ferric sulphate/lime treatment.

below 5 ppm. The second region defined by Ca concentrations of 3.0 to 3.5% contains variable percentage removals of arsenic and concentrations ranging up to several thousand ppm.

More detailed examination of Figs. 1 and 2 reveals a region defined by the concentration ranges As 0.2 to 0.8%, Fe<sup>III</sup> 1.0 to 2.0%, Ca 5.0 to 6.5% in which better than 99.8% arsenic removal was obtained with corresponding residual arsenic concentrations in solution which were generally below 5 ppm. A second region defined by the concentration ranges As 0.2 to 0.6%, Fe<sup>III</sup> 3.0 to 4.5%, Ca 3.0 to 3.5% corresponds with less than 70% arsenic removal and residual arsenic concentrations up to 3,820 ppm. Thus it would appear that

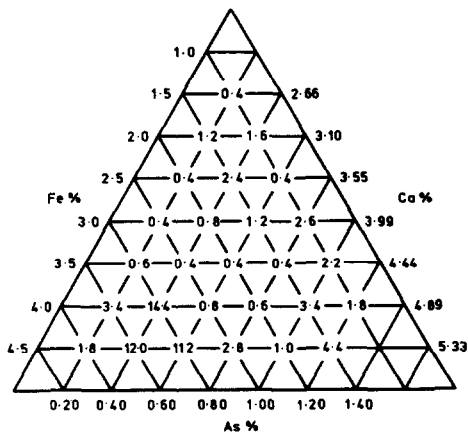


Fig. 3. Concentrations of arsenic (w/v ppm) in supernatant following ferrous sulphate/lime treatment.

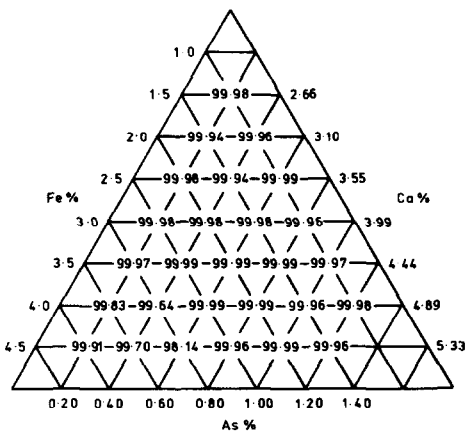


Fig. 4. Percentage arsenic removal following ferrous sulphate/lime treatment.

ferric sulphate/lime treatment will be effective for treating solutions of 0.2 to 1.0% arsenic(V) in 1.5% sulphuric acid provided the concentration range Fe 1.0 to 2.0% and Ca 4.0 to 6.0% is used. Use of the concentration ranges Fe 3.0 to 4.0% and Ca 3.0 to 3.5% is likely to prove especially ineffective in removing arsenic from acid solution.

#### *Lime and ferrous sulphate*

The concentrations of arsenic in the supernatant liquors of solutions treated with ferrous sulphate and lime are presented in Fig. 3 and the corresponding percentage arsenic removals are shown in Fig. 4. In these experiments the lime concentration was increased in reverse order to that used in the ferric/lime experiments. Arsenic concentrations in the supernatant ranged from 0.4 to 112 ppm and the percentage arsenic removal was generally better than 99%. The best results for solutions containing 0.2 to 0.8% As were obtained using 2.0 to 2.5% Fe<sup>II</sup> with 4.89 to 3.55% Ca and the worst using 3.0 to 4.0% Fe<sup>II</sup> and 4.89 to 5.33% Ca.

Comparing the results obtained using iron(II) and lime with those obtained using iron(III) and lime it appears that iron(II)/lime treatment is effective in removing arsenic over a greater range of concentrations than is iron(III)/lime treatment. In addition, lower concentrations of arsenic remain in solution after iron(II) treatment than after iron(III) treatment as a general rule.

#### *Dolomite and ferric sulphate*

The concentrations of arsenic in the supernatant liquors of solutions treated with ferric sulphate and dolomite are presented in Fig. 5 and the corresponding percentage arsenic removals are shown in Fig. 6. The observed arsenic concentrations ranged from 4.4 to 5,140 ppm and the percentage removal from 1.95 to 99.99%. Compared with lime and ferric or ferrous sulphate treatment, dolomite and ferric sulphate treatment appears to be generally ineffective. Arsenic concentrations in excess of 1,000 ppm appear throughout Fig. 5 and the instances where better than 99% arsenic removal was obtained correspond with comparatively high arsenic concentrations in solution.

The best result in terms of both percentage arsenic removal and residual arsenic concentration was found at the point As 0.432%, Fe 1.0%, Ca 5.5% (Ca + Mg 8.74%). Generally the best percentage removal was obtained in the region As 0.432 to 1.08%, Fe 1.0 to 1.5%, Ca 4.0 to 5.5%. Poor percentage removals were obtained for As, 0.216% and in the region As 0.216 to 0.648%, Fe 2.5 to 4.0%, Ca 3.0 to 4.5%. The use of lime in conjunction with iron salts appears to be a far better arsenic removal method than the use of dolomite and iron(III).

#### *Lime magnesium hydroxide and ferric sulphate*

The concentrations of arsenic in the supernatant liquors of solutions treated with ferric sulphate and an equimolar mixture of lime and magnesium hydroxide are presented in Fig. 7 and the corresponding percentage removals

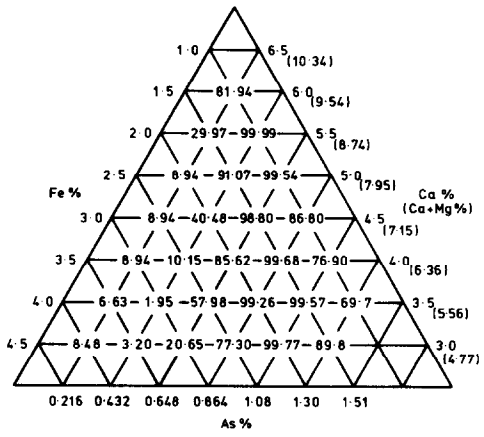
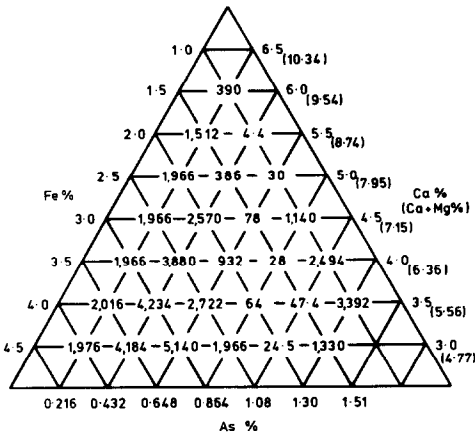


Fig. 5. Concentrations of arsenic (w/v ppm) in supernatant following ferric sulphate/dolomite treatment.

Fig. 6. Percentage arsenic removal following ferric sulphate/dolomite treatment.

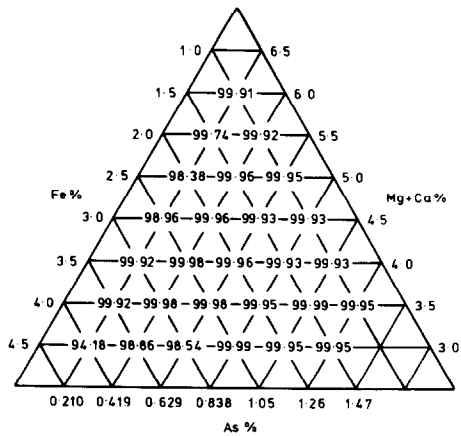
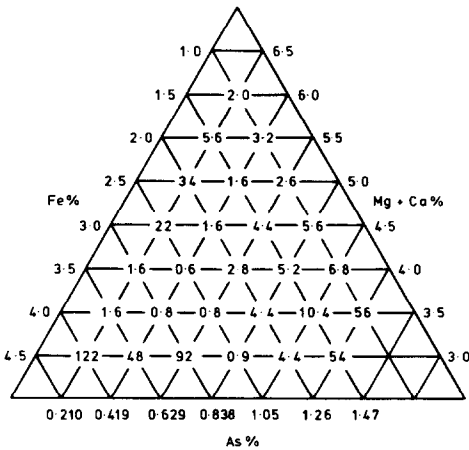


Fig. 7. Concentrations of arsenic (w/v ppm) in supernatant following ferric sulphate/1:1 lime magnesium hydroxide treatment.

Fig. 8. Percentage arsenic removal following ferric sulphate/1:1 lime:magnesium hydroxide treatment.

are shown in Fig. 8. The concentrations of arsenic in the supernatant ranged from 0.6 to 122 ppm and the percentage arsenic removals were generally in excess of 98%. As with the ferric sulphate/lime treatment the poorest results were obtained at alkaline earth metal concentrations of 3.0 to 3.5%. The best results were obtained in the region As 0.419 to 0.629%, Fe 2.5 to 3.0% and Ca + Mg 3.5 to 4.0%.

Taken as a whole the results obtained with ferric sulphate, lime and magnesium hydroxide are not quite as good as those obtained with ferrous sulphate and lime. However they are generally better than those obtained with ferric sulphate and lime especially in the region As 0.2 to 1.0%, Fe 1.5 to 4.0%, alkaline earth metal 3.0 to 3.5%. Thus treatment with ferric sulphate, lime and magnesium hydroxide offers advantages over treatment with ferric sulphate and lime alone but not over treatment with ferrous sulphate and lime.

#### *Effect of particle size range*

The results of the experiments using different particle sizes of lime to precipitate arsenic are summarised in Table 1. Despite the uncertainty over whether the particle size ranges used consisted of discrete particles or agglomerates of finer particles, there did seem to be an improvement in arsenic removal when the finer sieve fractions were used. The data for the sieve fractions 63–90, 91–125 and 250–315  $\mu\text{m}$  showed decreasing concentrations of residual dissolved arsenic with decreasing lime particle size for both ferric and ferrous iron treatment. The data for the sieve fraction  $<63$  ppm were somewhat inconsistent but taken as a whole there was a clear correlation between lime particle size and arsenic removal.

A plot of the residual arsenic concentration against the reciprocal of the mean particle diameter for the sieve fractions used is shown in Fig. 9. Assuming spherical particles the total surface area of a given mass of particulate material is inversely proportional to the particle diameter, this plot shows that the residual dissolved arsenic concentration decreases linearly as the surface area of lime exposed to the solution increases, over the limited range studied. The graph also shows that, when finer particulate lime is used, the effectiveness of ferric iron in removing arsenic approaches that of ferrous iron. In fact the line for ferric iron converges to meet that for ferrous iron at an arsenic concentration of 0 ppb and a  $1/d$  value corresponding with an average particle diameter of 57  $\mu\text{m}$ . The results obtained with the  $<63$   $\mu\text{m}$  particle size fraction suggest that there is in fact no further advantage in using particle

TABLE 1

Effect of lime particle size on arsenic removal

Particle size range ( $\mu\text{m}$ )	Arsenic concentration in $\text{Fe}^{\text{III}}$ /lime*	Supernatant (ppm) in $\text{Fe}^{\text{II}}$ /Lime**
$< 63$	0.41	0.60
63–90	0.42	0.27
90–125	0.87	0.44
250–315	1.40	0.80***

\*Composition: As, 0.4%;  $\text{Fe}^{\text{III}}$ , 2.0%; Ca, 4.5%.

\*\*Composition: As, 0.4%;  $\text{Fe}^{\text{II}}$ , 2.5%; Ca, 4.0%.

\*\*\*Composition: As, 0.4%;  $\text{Fe}^{\text{II}}$ , 2.0%; Ca, 3.99%.



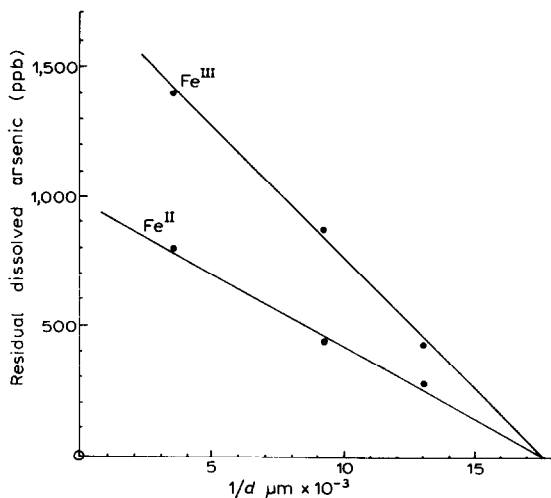


Fig. 9. Graphs of residual dissolved arsenic as a function of  $1/d$ , where  $d$  is the average particle diameter ( $\mu\text{m}$ ) for the sieve fraction of lime used to remove arsenic.

sizes below ca.  $60 \mu\text{m}$  and that a limiting residual arsenic concentration of the order of ca. 500 ppb is to be expected. A further objection to the use of very fine particles, and which may account in part for the erratic results obtained for the  $<63 \mu\text{m}$  fraction, is that more complete conversion of these particles to carbonate may occur by reaction with atmospheric  $\text{CO}_2$ . The results obtained for dolomite treatment suggest that alkaline earth carbonates are considerably less effective than the hydroxides in removing arsenic.

The effect of a magnetic field on the precipitates produced in the arsenic removal experiments was investigated. The presence of a bar magnet close to the suspended material caused no visible movement of the solid particles or any other indication that they had magnetic properties which might be exploited in solid liquid separation processes. However, recently developed techniques such as high gradient magnetic separation which are effective for separating weakly paramagnetic substances might still be applicable. Trials in specialised equipment would be necessary to establish the applicability of these methods.

### Leaching of arsenic bearing sludges

#### *Sand dispersions*

The results of the column leaching tests with sand dispersions of sludge taken from the mixture As, 0.4%; Fe<sup>II</sup>, 2.0%; Ca, 3.99% are summarised in Table 2. The concentrations of arsenic in the column leachates were all below 0.2 ppm and were generally 0.01 ppm or less. No significant differences were apparent between the results obtained using water as the leachant and those obtained using 5,000 ppm acetic acid. The low arsenic concentrations eluted from the

TABLE 2

Results of leaching test using sand columns

Water leachant				Acetic acid leachant			
Contaminated sand		Clean sand		Contaminated sand		Clean sand	
Volume eluted (ml)	Arsenic conc. (ppm)	Volume eluted (ml)	Arsenic conc. (ppm)	Volume eluted (ml)	Arsenic conc. (ppm)	Volume eluted (ml)	Arsenic conc. (ppm)
0-5	0.01	50-55	0.01	0-5	<0.01	50-55	0.01
95-100	0.01	105-110	<0.01	95-100	0.06	105-110	0.01
145-150	0.14	160-165	<0.01	145-150	<0.01	160-165	0.01
240-245	<0.01	215-220	<0.01	240-245	0.02	215-220	0.01
330-335	<0.01			330-335	<0.01		
385-390	<0.01			385-390	<0.01		

column of contaminated sand did not allow scope for assessing the removal of arsenic from the leachate by uncontaminated sand. However, in view of these low arsenic concentrations there seems little need for a mechanism for reducing the arsenic concentration in the leachate.

#### Chalk dispersions

The results of the column leaching tests with chalk dispersions are summarised in Table 3. As with the sand columns the arsenic concentrations in leachates were below 0.2 ppm and generally 0.01 ppm or less. Again no significant differences were observed between water and 5,000 ppm acetic acid leachants although more variable results were obtained than with the sand columns. This may be due to the fissures and voids in the sample of chalk used which did not pack as homogeneously as the more fine grained sand sample.

TABLE 3

Results of leaching tests using chalk columns

Water leachant				Acetic acid leachant			
Contaminated chalk		Clean chalk		Contaminated chalk		Clean chalk	
Volume eluted (ml)	Arsenic conc. (ppm)	Volume eluted (ml)	Arsenic conc. (ppm)	Volume eluted (ml)	Arsenic conc. (ppm)	Volume eluted (ml)	Arsenic conc. (ppm)
0-5	0.01	0-5	0.01	0-5	0.01	0-5	0.14
95-100	0.01	105-110	0.01	95-100	0.01	105-110	0.01
145-150	0.02	160-165	0.01	145-150	0.01	160-165	0.01
240-245	0.02			240-245	0.01	215-220	0.02
330-335	0.01			330-335	0.01		
				385-390	0.01		

### Clay dispersions

The results of the leaching experiments on clay dispersions of arsenic bearing sludge are summarised in Table 4. Higher arsenic concentrations appeared in leachates from contaminated clay than for the other two soils studied. However, contacting these leachates with uncontaminated clay resulted in a significant drop in the arsenic levels to give residual arsenic concentrations of a few hundredths of a ppm. No large differences were apparent between the water and 5,000 ppm acetic acid leachants. Although, when dry clay was used, the acetic acid leachates produced from the contaminated clay contained up to twice the concentration of arsenic found in the water leachates, the arsenic concentrations in all the leachates treated with uncontaminated clay were of a similar value and below 0.05 ppm.

Two types of clay were used in the leaching experiments, oven dried material in the form of a powder which readily dispersed in water and wet clay which was in plastic lumps. The higher leachate arsenic concentrations found for contaminated dry clay may reflect its ease of dispersion in water. The wet clay formed lumps which "encapsulated" the sludge and probably reduced its exposure to leachants. The more aggressive conditions of the shaker experiments may also be responsible for the clay leachants containing more arsenic than those from sand or chalk. However, the arsenic concentrations in all but one of the leachates contacted with clean soils were of a similar order of magnitude viz. less than 0.105 ppm (Tables 2, 3 and 4).

TABLE 4

Results of leaching tests using clay dispersions

Water leachant				Acetic acid leachant			
Contaminated clay		Clean clay		Contaminated clay		Clean clay	
Total volume added (ml)	Arsenic in supernatant (ppm)	Total volume added (ml)	Arsenic in supernatant (ppm)	Total volume added (ml)	Arsenic in supernatant (ppm)	Total volume added (ml)	Arsenic in supernatant (ppm)
<i>Dry clay</i>							
100	0.09	60	0.02	100	0.37	56	0.01
200	0.23	170	0.01	200	0.50	161	0.02
300	0.09	265	0.02	300	0.42	256	0.01
400	0.24			400	0.19		
<i>Wet clay</i>							
100	0.03	66	0.04	100	0.16	71	0.03
200	0.05	166	0.01	200	0.14	171	0.01
300	0.20	266	0.02	300		271	0.03
400	0.28			400	0.04		

## Conclusions

The experiments described here have shown that the treatment of acid solutions containing tenths of a percent of  $\text{As}^{\text{V}}$  with iron salts and alkaline earth metal hydroxides can result in residual dissolved arsenic concentrations below 1 ppm. The post treatment dissolved arsenic concentration appears to be directly related to the particle size of the alkaline earth hydroxide used. Leaching experiments indicate that concentrations of arsenic in leachates from clay, sand or chalk soils contaminated with arsenic bearing sludges are below 1 ppm and generally below 0.05 ppm. The experimental findings may be summarised as follows:

- (i) ferrous iron and lime provides the most effective treatment giving residual dissolved arsenic concentrations of below 1.0 ppm over a range of compositions along with 99.9% removal;
- (ii) ferric iron treatment is more effective in conjunction with mixed lime and magnesium hydroxide than with lime alone;
- (iii) ferric iron and dolomite are relatively ineffective for treating the arsenic solutions studied;
- (iv) generally speaking the most effective treatment was obtained with arsenic contents of 0.4% to 0.8%, iron contents of below 2.5% and calcium contents above 4.0%;
- (v) the optimum particle size for lime used in the treatment process appears to be in the range 50–100  $\mu\text{m}$ ;
- (vi) leachates from soils containing the arsenic bearing sludge from ferrous iron and lime treatment contain less than 1 ppm of arsenic and generally less than 0.05 ppm;
- (vii) the presence of acetic acid to the extent of 5,000 ppm in the aqueous leachant appears to have no substantial effect on the concentrations of arsenic in the leachate.

The results summarised above indicate that ferrous iron and lime treatment of 1.5% sulphuric acid solutions of arsenic(V) can remove 99.9% of the arsenic and leave residual dissolved arsenic concentrations of ca. 0.5 ppm. Thus the effluent from treatment would require polishing or dilution by a factor of ca. 10 to meet the World Health Organisation Maximum Permissible Concentration for arsenic in drinking water [2]. However, since it is hardly likely that the effluent would be directly used for drinking water this figure of tenfold dilution serves only to place the arsenic concentration in treated effluent into perspective. The leaching tests on the arsenic bearing sludges produced by this treatment process show that, for the most part, the arsenic concentrations in the final leachates meet the WHO MPC requirement for arsenic in drinking water. Thus it would appear that ferrous iron and lime treatment is effective for solutions of arsenic(V) in 1.5% sulphuric acid and that the resulting sludge is suitable for land disposal.

## **Acknowledgement**

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