The impoved densification of sludge from neutralized acid mine drainage.

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SYNOPSIS

The paper describes an investigation aimed at finding a solution to the accumulation of acid mine drainage in the old workings at Coronation Collieries, Kromdraai. The problem involved the neutralization of the acid in the water, without the large volumes of sludge and the large amounts of dissolved solids that usually result from neutralization. Pilot-plant work on the site using a lime-neutralization process developed at Bethlehem Steel Corporation (U.S.A.) showed this to be a feasible method. A sludge of 22 per cent density was obtained under optimum operating parameters. The amount of suspended solids in the overflow water was very high, but this does not give rise to concern because the full-scale plant at Kromdraai will practise secondary sedimentation by running the thickener overflow through large dams with a theoretical retention time of 25 days.

An analysis of the theory underlying the process is also made.

SAMEVATTING

Die verhandeling beskryf 'n ondersoek met die oog op 'n oplossing van die probleem van die ophoping van suur mynafloop in die ou delfplekke by Coronation Collieries, Kromdraai. Die probleem behels die neutralisering van die suur in die water sonder die groot volumes slyk en die groot hoeveelhede opgeloste vaste stowwe wat gewoonlik met die neutralisering gepaard gaan. Proefaanlegwerk op die terrein met gebruik van 'n kalkneutraliseringsproses wat by die Bethlehem Steel Corporation (V.S.A.) ontwikkel is, het getoon dat dit 'n uitvoerbare metode is. 'n Slyk met 'n digtheid van 22 persent is met optimale bedryfsparameters verkry. Die hoeveelheid gesuspendeerde vaste stowwe in die oorloopwater was baie hoog, maar dit wek geen kommer nie omdat die volskaalse aanleg by Kromdraai sekondêre sedimentasie sal toepas deur die oorloop van die verdikmiddel deur groot damme met 'n teoretiese retensietyd van 25 dae te laat loop.

Die teorie wat ten grondslag van die proses lê, word ook ontleed.

When mining operations expose to oxygen and water the pyritic material usually associated with coal, a chemical reaction takes place. This reaction, which is invariably catalysed by bacterial action[1], results in the formation of water-soluble iron sulphates. Unless the water coming into contact with the oxidizing sulphides originally had a high alkalinity, the end result is the draining of acid water from the workings. Several authors[2, 3] have formulated the reactions, which are given here in simplified form:

$$2FeS_2+7\ O_2+2H_2O \rightarrow 2FeSO_4 +2H_2\ SO_4,$$

or, when oxidized further,

$$2FeS_2+7\tfrac{1}{2}\ O_2+H_2O \rightarrow Fe_2(SO_4)_3 +H_2\ SO_4.$$

When mining operations at Coronation Collieries, Kromdraai, ceased some years ago, the adits to the mine were closed off in an attempt to contain the acid mine drainage (AMD) in the old workings and prevent environmental pollution. However, water eventually accumulated in the mine to the extent that some of the 'mud' outcrops and adit plugs could no longer retain the water, and seepage occurred at several places.

Although sealing and flooding of the mine must deprive the oxidation reaction of fresh air, a certain amount of acidity is still formed because the seepage water has a pH value of about 2,7. The more detailed analysis given in Table I shows that the water requires treatment before it can be discharged into a public stream.

It is unpractical to treat the AMD at each of the relatively small and uncontrollable seepage points, and if these seepages are to be stopped, the level of water in the mine has to be lowered. Reduction of the pressure behind the adit plugs would also reduce the danger of these plugs bursting and causing a flood of acid water, which would eventually enter Loskop Dam.

The task of assessing the extent of the problem and of preventing the pollution of public streams was given to the Civil Engineering Department of the Anglo American Corporation. Valuable data were collected on the rate of accumulation and on the volumes of water present in various sections of the mine. Discussion of these aspects lies beyond the scope of this paper, but the outcome of the investigation was that the mine can, and must be, emptied at one point, and that the water, after adequate treatment, should be discharged into a local stream.

The Anglo American Research Laboratories, having been involved in the problem through determining some water qualities and flowrates, were then asked to find a practical

TABLE I
AVERAGE ANALYSES OF KROMDRAAI ACID DRAINAGE

pH	2,7
Acidity as $CaCO_3$, mg/l	1000
Conductivity, μS/m	18,0 × 10^4
Total dissolved solids, mg/l	1750
Ferrous iron, mg/l	0-140
Ferric iron, mg/l	30-200
Total iron, mg/l	175
Aluminium, mg/l	90
Calcium, mg/l	30
Magnesium, mg/l	10
Sodium, mg/l	5
Potassium, mg/l	3
Sulphate, mg/l	1150
Chloride, mg/l	5

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TABLE II

ANALYSES OF AMD EFFLUENT AFTER NEUTRALIZATION WITH LIME AND CLARIFICATION

pH	7,5
Total dissolved solids, mg/l	1800
Suspended solids, mg/l	60
Ferrous iron, mg/l	1
Ferric iron, mg/l	30
Aluminium, mg/l	< 1
Calcium, mg/l	450
Magnesium, mg/l	20

and economic process for the treatment of AMD.

The commonest and oldest method for the treatment of acid water is the neutralization of the water with an alkali such as lime[4]. On neutralization, a chemical precipitate is formed that settles slowly to form a sludge containing about 99 per cent water, and disposal of this voluminous sludge often presents problems. Another disadvantage of this neutralization process is that it does not reduce the undesirably high content of dissolved solids in the water; in fact, the total dissolved solids (TDS) are often higher after neutralization and clarification than before (Table II).

As effluents containing more than about 1200 mg/l of TDS were considered unsuitable for discharge into a public stream, attention had to be given to processes by which the TDS of the water could be reduced. The following processes were investigated: bacterial sulphate reduction[5], various desalination processes, and precipitation of the sulphates with a barium salt[6]. None of these methods was found to offer a solution to the Kromdraai problem. Bacterial sulphate reduction appeared to be feasible but required the transportation of excessive amounts of organic matter (e.g., sewage sludge). The desalination, ion-exchange, and barium-precipitation processes were ruled out as being too sophisticated and expensive for the removal of salts from water that has to be discharged to waste. (This statement does not apply to the costs involved in rendering saline water suitable for, say, domestic purposes.) A further disadvantage of the desalination processes is that undesirable salts remain in solution—concentrated about five-fold—which still have to be disposed of. Unless the iron present in the AMD is removed beforehand (e.g., by neutralization and clarification), this metal interferes with the operation of the resins used in most ion-exchange processes and the membranes used in the reverse-osmosis process.

Consideration was also given to the possibility of neutralizing the AMD with lime and disposing of the clarified effluent by land irrigation. About 1000 ha of land were available for this purpose. Advice[7, 8] received on this aspect was fairly favourable; in fact, Fölscher[8] even claimed that, if the soil is properly treated with an alkali, unneutralized AMD can be applied direct to the land. However, the idea of disposal by irrigation was not carried further because of the statement at this juncture by the Department of Water Affairs that a permit would not be required for the discharge to a public stream of water with TDS of about 1800 mg/l, provided the pH value and suspended-solids content of the water complied with the specifications of the Water Act.

Although a disposal point had been found for the neutralized and clarified water, the problem was not yet entirely solved since, as previously mentioned, the sludge separated on neutralization is very voluminous and requires considerable space for disposal. When allowed to settle by gravity, the sludge normally occupies 10 per cent of the original volume of water, but, if the sedimentation is continued in the same container, layer upon layer, further densification takes place to about 4 per cent of the original volume[9]. However, this 2,5-fold densification would still not be sufficient because the mine contained 10^4 Ml of AMD with 10^4 tonnes of precipitable solids. At 2,5 per cent solids, the sludge from this volume of AMD would occupy 400 Ml. The construction of a surface dam for the disposal of this volume of sludge would have been too costly, and the use of the old workings for sludge disposal would have been shortsighted in that the disposed sludge would prove a nuisance if another profitable coal seam and the pillars of the already-mined seam should be open-cast in future. Therefore, the feasibility of the neutralization of AMD depended on the possibility of concentrating the waste solids to an extent where the space required for disposal would be considerably less.

When limestone is used, instead of lime, for the neutralization of AMD, a somewhat higher sludge density can be obtained[10]. The curves in Fig. 1 show that, although sedimentation occurs slowly, the sludge densifies to 2,7 per cent solids when limestone is used as the alkali. However, laboratory tests had shown that the reaction rate is much slower for limestone than for lime[9], and a larger neutralization vessel would therefore be required. It can be seen from Fig. 2 that, despite the longer reaction time and larger volumes of oxidation air applied during these tests, the pH value seldom rose above 6,5. Therefore, any unoxidized iron would remain in solution and would contaminate the final effluent. Limestone's price advantage (being a cheaper reagent than lime) did not apply to Kromdraai because the only limestone available had to be transported over a considerable distance. The problems and costs involved in the handling of two raw materials instead of one precluded the use of a mixture of lime and limestone, and the benefits shown in Figs. 1 and 2 could therefore not be obtained.

At this stage in the search for a suitable treatment process, it was learnt that the Bethlehem Steel Corporation[11] had succeeded in densifying sludge after lime neutralization. Rather than waste time on the less promising methods of vacuum and pressure filtration, it was decided to conduct tests on the feasibility of this process for Kromdraai AMD.

In addition to the conventional neutralization of AMD by means of a lime slurry and subsequent liquid-solid separation, Bethlehem Steel's process incorporated the recirculation of settled sludge to a conditioner, where it was mixed with the lime slurry before being added to the AMD. The precipitate that formed when the AMD was neutral-

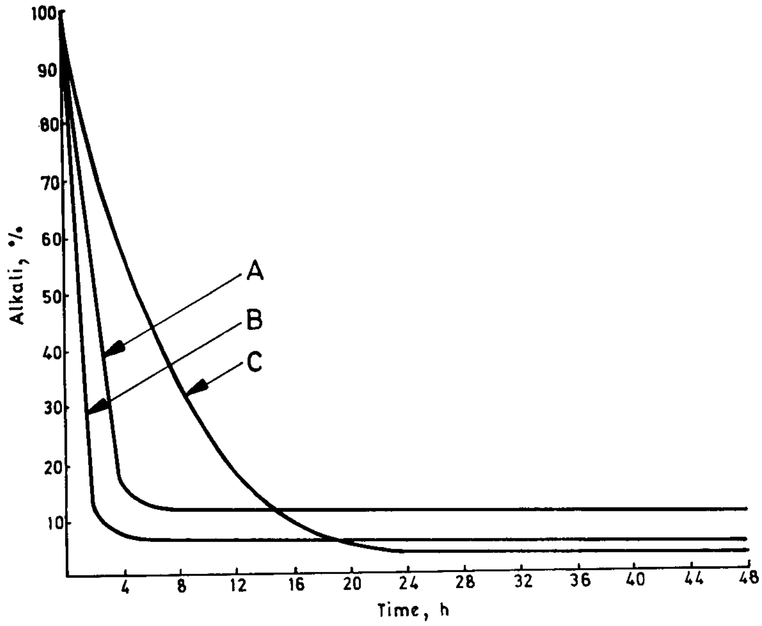

Fig. 1—Settling rate of precipitate from Kromdraai neutralized water
A—Lime 0,875 g/l Solids in sludge 0,8%
B—Limestone 0,83 g/l + lime 0,47 g/l Solids in sludge 1,7%
C—Limestone 1,10 g/l Solids in sludge 2,7%

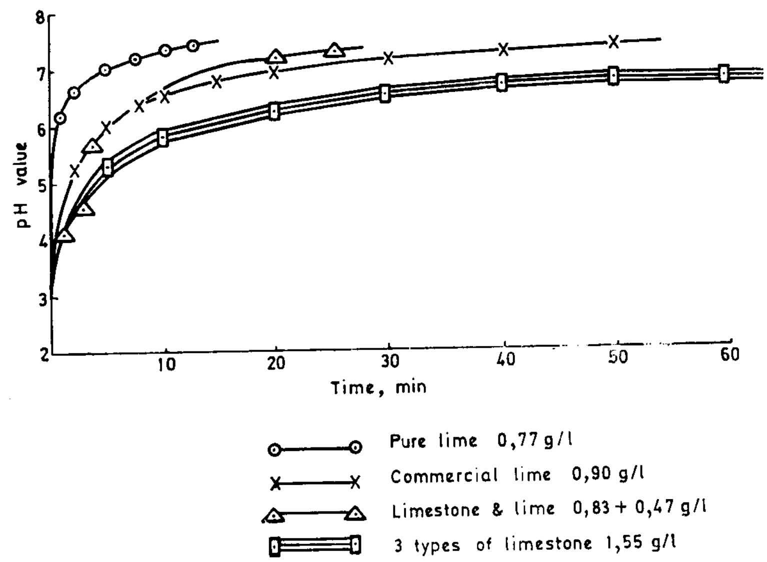

Fig. 2—Neutralization of Kromdraai acid drainage with different types of lime and limestone

ized with this sludge-lime mixture was of much higher density than when lime was added direct to the AMD. It was claimed that densities of 15 to 40 per cent solids could be achieved, depending on the ratio of ferrous to ferric iron in the AMD. Densities of such a magnitude appeared to offer an ideal solution to the Kromdraai sludge-disposal problem because, at 20 per cent solids, the sludge from the volume of AMD in the mine would occupy only 50 Ml, and even less after it had been densified further in surface disposal dams.

Tests to ascertain the densities obtainable with sludge from the Kromdraai AMD were highly desirable, and, as electric power and accommodation for plant operators were still available at the abandoned mine, it was decided to construct and operate a pilot plant on site. The main advantage of tests on site was that an abundant supply of AMD was available and that its ratio of ferric to ferrous iron remained unchanged (the ferrous iron might have become partially oxidized during transportation). The availability of enough AMD also made it possible to increase the size of the plant to a scale from which scaling-up could be undertaken with reasonable accuracy.

DESCRIPTION OF THE PLANT

As shown in Fig. 3, the pilot plant comprised the following.

(a) A 180-litre mechanically stirred drum in which hydrated, air-separated lime was mixed with some of the final supernatant effluent to form a 20 per cent lime slurry.

(b) The lime slurry was pumped by chemical feeder to the 60-litre conditioner tank into which it was proportioned at a rate depending on the pH value required for the neutralized water. The recirculated underflow sludge from the thickener was also pumped into this conditioner tank, which was slowly stirred mechanically.

(c) The lime-sludge mixture at a pH value of about 10 was then used for neutralization of the AMD. The pilot plant was sited close to an old borehole into the mine, where the acid water was conveniently obtainable and could be pumped at a metered rate to the plant.

(d) This metered AMD was neutralized in two 50-litre tanks in series. Air, as oxidant and also as a means of agitation, was blown into these vessels.

(e) The neutralized water, with its precipitated solids and the recirculating sludge, was then passed into a 1,5 m-diameter thickener tank, which had a rake operating at 0,83 rev/min. Liquid-solids separation was accomplished in the thickener so that a relatively clear water overflowed to waste (except for a small portion used for the make-up of lime slurry).

All the underflow sludge was pumped back to the conditioner tank, but, when the desired concentration of solids in the system had been reached, calculated amounts of excess sludge (equal to the amount gained by precipitation from the incoming AMD) were run to waste.

The loading of solids on the

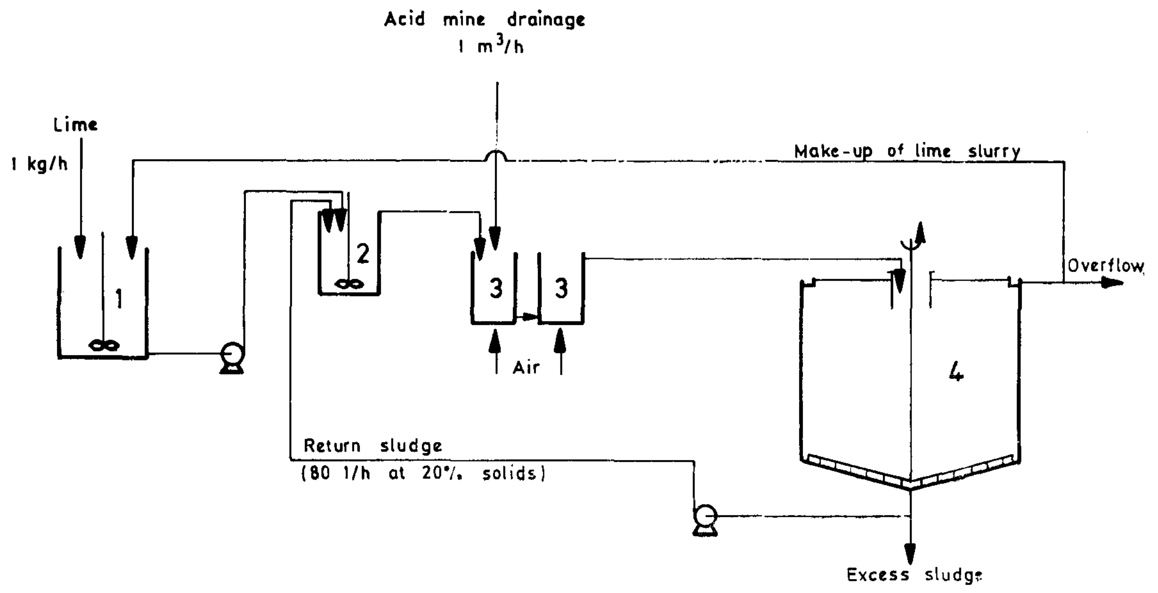

Fig. 3—Flow diagram for the pilot plant
1—180-litre tank for lime slurry
2—60-litre conditioner
3—50-litre neutralization tanks
4—2600-litre thickener

thickener, sludge recirculation ratio, conditioner retention time, and conditioner stirrer speed were the variables studied during the operation of the plant. However, these factors were not varied to extremes because the work by Kostenbader and Haines[11] had already indicated the direction in which optimum conditions should be attained. Furthermore, conditions at the abandoned mine were not very convenient for the continuous operation of a pilot plant, and only sufficient data were collected for the design of a full-scale plant.

RESULTS OF THE EXPERIMENT

The results from the test runs, which were conducted over a period of three months, showed that sludge with a density of 22 per cent can be obtained under optimum operating parameters. This concentration is in agreement with the results obtained by the Bethlehem Steel Corporation at a mine where the AMD had a ferric to ferrous iron ratio similar to that of Kromdraai. Kostenbader and Haines[11] found that the maximum attainable sludge density is very dependent on this ratio. Table III, which was taken from their paper, shows how the higher densities result from acid water having higher concentrations of ferrous iron.

When the operating parameters for the highest sludge density were being established, the retention time in the sludge conditioner was not shortened to the 1 minute that Kostenbader and Haines found to be adequate. Results obtained at Kromdraai showed that this retention time should be at least 20 minutes, and preferably 30 minutes. Comparison of the densities obtained during runs 1, 4, and 5, and also during runs 2 and 3 (Table IV) shows that lower densities are obtained at the shorter retention times in the conditioner. Addition of lime to the return sludge in the conditioner did not seem to affect the alkali consumption of the AMD, the quantity used during the pilot-plant tests being 0,9 g per litre of AMD, as in the laboratory neutralization tests, where lime was added direct to the AMD.

However, a condition in the mixing tank that did affect the sludge density attained was the degree of mixing. Rapid stirring of the lime-sludge mixture had a detrimental effect on sludge density. Good results were obtained when the peripheral velocity of the mechanical stirrer was 1,3 m/s. Speeds of 1,5 m/s could be tolerated, but it was found that the density of the sludge was reduced if the tips of the impeller blades rotated at a speed of 4 m/s. This conclusion was not clearly indicated by the results because the densities obtained during runs 3 and 4 were affected not only by a change in stirrer speed but also by different retention times in the conditioner. However, the effect of retention time on sludge density

TABLE III

EFFECT OF FERROUS TO FERRIC IRON RATIO OF AMD ON CONCENTRATION OF SETTLED SLUDGE (PRODUCED BY KOSTENBADER AND HAINES[11])

Water source	Ferrous iron as av. % of total iron	Maximum concentration of settled solids, %
Mines 32-33 AMD (source discharge)	90	40
Synthetic AMD	> 95	50
Synthetic steel-plant waste	> 95	45
Steel-plant waste	> 95	45
Mines 32-33 AMD (shipped samples)	70 (range 45-90)	22
Mine 32, supply-shaft AMD	30	15
Mine 31 AMD (shipped samples)	2	18

TABLE IV

PILOT-PLANT RESULTS FOR AMD SLUDGE DENSIFICATION

Run	1	2	3	4	5	6	7	8
Alkali used	lime	lime	lime	lime	lime	lime	lime	lime-stone
Thickener loading, kg solids/m² day	240	285	250	240	235	230	240	215
Flocculant dosage, mg/l	—	—	—	—	—	—	0,5	—
Conditioner retention time, min	85	10	15	22	48	41	41	24
Stirrer peripheral velocity, m/s	1,5	4,0	4,0	1,3	1,3	1,3	1,3	1,3
Sludge density, % solids	23,0	11,5	15,0	21,0	22,5	18,5	19,5	12,0

did not appear to be substantial, especially at times longer than 20 minutes, so that stirrer speed must have affected the sludge density as well.

Kostenbader and Haines[11] found the optimum recycle ratio for sludge to be between 25 and 30 to 1. This ratio is based on the mass of solids returned against the mass of solids precipitated from solution. At Kromdraai, the ratio was mostly kept at 21 to 1, but when during run 6 it was reduced to 16 to 1, a reduction in sludge density was experienced.

At Bethlehem, it was found that a retention time of 10 minutes in the neutralization-oxidation tank ensured oxidation and precipitation of the ferrous iron at any pH value above 7,2. At Kromdraai, it was found that even a retention time of 5 minutes in the two-stage neutralization step was sufficient for the removal of iron from solution. The analysis given in Table V shows that a large proportion of the iron was precipitated as ferrous iron. However, as can be seen from the analysis in Table II, very little iron remained in solution at a pH value of 7,5; the ferric iron shown to be present was not in solution but was present as suspended matter.

Despite the relatively low rising velocity of 0,57 m/h in the thickener, the suspended matter in the overflow water was as high as 60 mg/l. The addition of an economic amount of flocculant to the thickener feed did not reduce the quantity of fine particles in the overflow, probably because the flocculant dosage of about 0,5 mg/l was still insufficient for the high concentration of solids (approximately 15 g/l) in the thickener feed. The only effect noticed from the addition of flocculant was a reduction in the sludge density, as shown by the result of run 7 (compared with run 5). However, the high content of suspended solids in the overflow does not cause concern; in the full-scale plant at Kromdraai, which has a daily capacity of 14 000 m³, secondary sedimentation will be achieved by the running of the thickener overflow through large dams with a theoretical retention time of 25 days.

The data obtained from the pilot-plant tests were insufficient to provide a straightforward answer to the effect of the solids loading on the thickener. As good results were obtained at loading rates of 240 kg/m² day, the full-scale plant was designed accordingly. The big plant will probably prove to be over-designed; at Bethlehem, loadings of about 500 kg/m² were found to be possible. The application of the Coe and Clevenger formula[12], as converted by Turner and Glasser[13], to the sedimentation rate of the sludge produced in the pilot plant also showed that a solids loading of 500 kg/m² day need not prove excessive. As the other major components of the full-scale plant (namely, the conditioner and neutralization vessel) may also prove to be over-designed, it should be possible to increase the throughput to more than the proposed flow of 14 000 m³/h.

Whether calcium carbonate, when used as an alkali in the sludge-recirculation process, will produce a sludge of higher density than that formed by calcium hydroxide was tested in a run with limestone, instead of with lime. The result of this test (run 8) shows that the density was, in fact, much lower with limestone, indicating that the mode of formation of the precipitate in the recirculation process is different from the precipitation of solids by the direct addition of alkali to AMD.

THEORETICAL CONSIDERATION OF SLUDGE DENSIFICATION

The analysis given in Table V shows that iron is the main constituent of the chemical precipitate formed during the neutralization of AMD. In the precipitate, the iron is present in the form of a hydrous iron oxide, which, according to Laitinen[14], can undergo extensive hydrolysis. The iron hydroxide particles can be visualized as being surrounded by an envelope of firmly bound water.

The particles absorb hydroxyl ions as potential determining ions, and hydrogen or metal ions as counter ions. The hydroxyl and hydrogen or metal ions constitute an electric double-layer round the particle, probably giving it the following form:

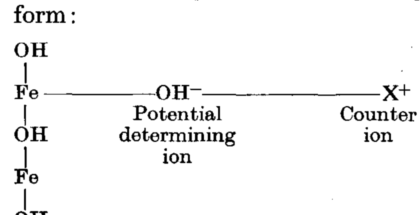

TABLE V

ANALYSES OF DENSIFIED SLUDGE (ON DRY BASIS)

Ferrous iron	5,6 per cent
Ferric iron	20,2 per cent
Aluminium	13,6 per cent
Calcium	5,5 per cent
Silica	8,9 per cent

The electrokinetic (zeta) potential of the particles, which causes the particles to repel one another, and the relatively large volume of the bound water layers, are probably the reasons for the very voluminous sludge that forms when iron hydroxide precipitates from an aqueous solution. Therefore, a denser sludge may be found if either the volume of absorbed water or the repelling forces are reduced. In the process described here, these targets must have been achieved by recirculation of the settled sludge. The following description, although by no means complete, offers a possible explanation of the mode of densification.

The rate of precipitation of solids from solution has an effect on the density of the precipitate. Laitinen[14] quotes Moser[15], who was probably the first to recognize that a slow formation rate results in a denser precipitate. In the sludge-recirculation process, the pH value of the lime-sludge mixture was only 10, as against a pH value of 12 for lime slurry. The lower concentration of hydroxyl ions in the former thus resulted in a slower precipitation rate. The mixing of the lime slurry with ten times its volume of recycled sludge diluted the lime, which also retarded the rate of increase in the pH value of the AMD. The high-density sludge obtained when limestone is used to neutralize AMD is probably also a result of a slow precipitation rate — limestone reacts much more slowly than lime does.

The recirculation of an 80 per cent liquid sludge (20 per cent solids) with its high concentration of calcium ions in solution (450 mg/l before the addition of lime) probably caused the concentration of calcium ions at the point of precipitation to be higher than when lime is added direct to AMD. This high concentration of calcium ions could, according to the Schulze-Hardy rule quoted by Van Olphen[16], result in a lower electrokinetic potential, when some of the high concentration of divalent calcium ions exchange with monovalent hydrogen counter ions. The increased charge resulting would then cause the double-layer to compress.

The absorption of ions of higher valency as counter ions also offers an explanation for the higher density obtained from acid water having a higher ratio of ferrous to ferric iron. Ferric iron precipitates at a much lower pH value than ferrous iron does. Therefore, as the pH value of the acid water is raised and ferric iron starts to precipitate, unoxidized (ferrous) iron will still be present in solution. These ferrous ions are then absorbed as counter ions instead of hydrogen, thus again giving a more compressed electric double-layer with a lower zeta potential. The presence of ferrous iron in the sludge (Table V) perhaps supports this theory of absorption, which is not necessarily contradicted by the absence of ferrous iron in the final effluent. The absorption of some of the ferrous iron occurred while the iron was still in the reduced form; the remainder was subsequently oxidized and precipitated, producing an iron-free solution.

Hydrous metallic oxides can also densify by aging (Laitinen[14]). Apparently, the precipitate is densified by a polymerization reaction that involves dehydration and formation of Fe-O-Fe linkages with chain structures containing forty to fifty atoms of iron. This phenomenon probably occurred during the operation of the pilot plant at Kromdraai, where it was found that the

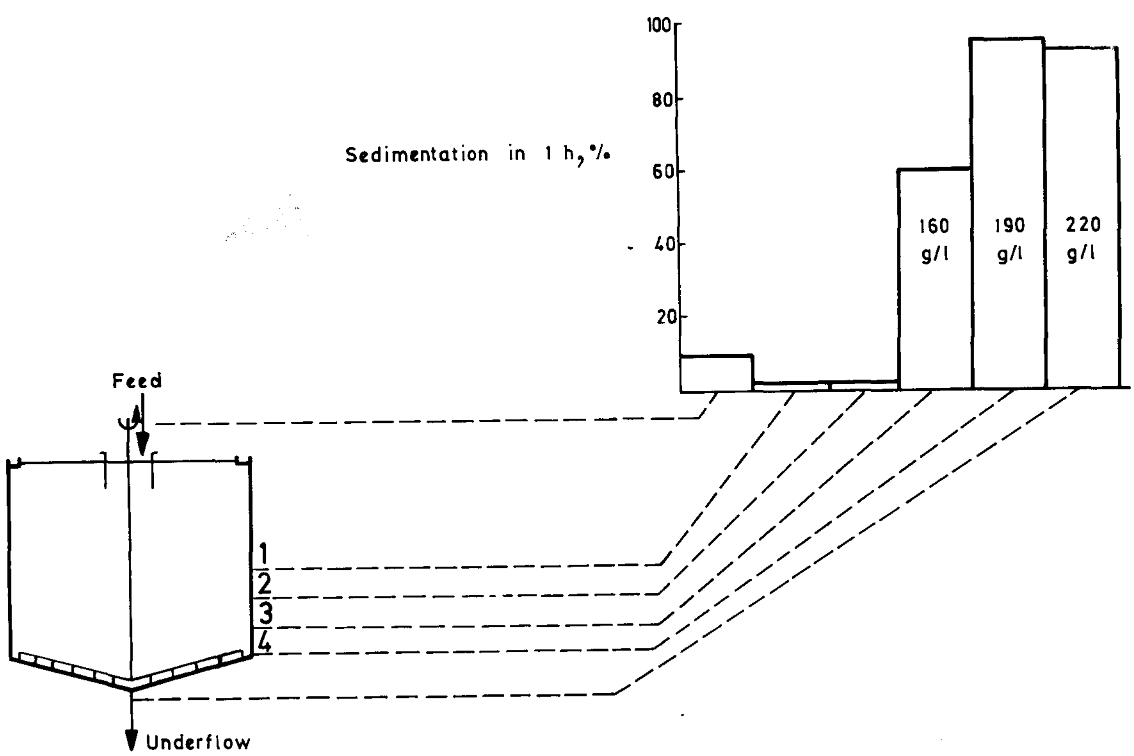

Fig. 4—Density profile of the sludge in the thickener

rake action in the thickener also densified the sludge (Fig. 4).

The addition, during one test, of an anionic polyacrylamide as a flocculant for the clarification of the thickener overflow resulted in a loss of sludge density. The reduced density was probably caused by the long-chain polymer holding the iron hydroxide particles apart, with a subsequent higher amount of interstitial water and also preventing densification on sedimentation.

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DISCUSSION

R. T. RUDD*

I have the following comments on Mr Bosman's paper.

Before giving the company permission to discharge neutralized mine drainage containing 1800 mg/l of dissolved solids into a public stream, my Department had to consider the matter from both the legal and the practical viewpoint.

The legal position is that, as the acid water does not arise from the use of water in an industrial process, a permit for its discharge is not required. However, as it can still be considered to be polluting, the following practical considerations were taken into account.

(1) The quantity of water in the mine represents roughly 3 per cent of the capacity of Loskop Dam. If some natural catastrophe were to release all this water into the dam at its pH value of 2,7, the results would be disastrous.

(2) The use of calcium to neutralize the effluent would result in the formation of calcium sulphate, which in general would not be harmful to irrigated soil applied in the quantities in which it would reach (and leave) Loskop, and could under certain circumstances even be beneficial.

(3) The water from Loskop Dam is used almost exclusively for irrigation—in fact, the Olifants River from Loskop to Phalaborwa is at present used for little else.

(4) Previous methods of disposal, in which the acid water was left out of our streams by leading it to evaporation dams and evaporating it, posed the problem that we were merely transferring the water to another site where it could possibly cause trouble in future.

(5) The diluting effect of the dam and other waters reaching it from the catchment would be sufficient to reduce the calcium sulphate level to an insignificant figure.

It was therefore considered that the method described by Mr Bosman offered the best available solution to the problem.

It was pointed out that the dissolved solids in mine water, even fairly close to Kromdraai, varied by large amounts. The old T & D.B. Colliery, for instance, discharges a water containing some 4000 mg/l of dissolved solids, while figures for the old Douglas Collieries are somewhat lower.

Similarly, the pH of the waters varies from mine to mine, and figures between 1,9 and about 4 have been reported.

In view of this, the opening remarks made by the Chairman of this symposium, Mr Van Rensburg, that each mine would have its individual problem in this respect, were most apt.

However, with the mixture of ingenuity and scientific expertise shown by the Company's work on Kromdraai, it will no doubt be able to solve these problems satisfactorily in due course.

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Mr Bosman and his team are to be congratulated on the research work carried out by them on this high-density sludge (H.D.S.) process and in establishing the parameters ap-

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