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ORIGINAL ARTICLE

Environmental characterisation of coal mine waste rock in the field: an example from New Zealand

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Abstract Characterisation of mine waste rock with respect to acid generation potential is a necessary part of routine mine operations, so that environmentally benign waste rock stacks can be constructed for permanent storage. Standard static characterisation techniques, such as acid neutralisation capacity (ANC), maximum potential acidity, and associated acid–base accounting, require laboratory tests that can be difficult to obtain rapidly at remote mine sites. We show that a combination of paste pH and a simple portable carbonate dissolution test, both techniques that can be done in the field in a 15 min time-frame, is useful for distinguishing rocks that are potentially acid-forming from those that are acid-neutralising. Use of these techniques could allow characterisation of mine wastes at the metre scale during mine excavation operations. Our application of these techniques to pyrite-bearing (total S = 1–4 wt%) but variably calcareous coal mine overburden shows that there is a strong correlation between the portable carbonate dissolution technique and laboratory-determined ANC measurements (range of 0–10 wt% calcite equivalent). Paste pH measurements on the same rocks are bimodal, with high-sulphur, low-calcite rocks yielding pH near 3 after 10 min, whereas high-ANC rocks yield paste pH of 7–8. In our coal mine example, the field tests were most effective when used in conjunction with stratigraphy. However, the same field tests have potential for routine use in any mine in which distinction of acid-generating rocks from acid-neutralising rocks is required. Calibration of field-based acid–base accounting characteristics of the rocks with laboratory-based static and/or kinetic tests is still necessary.

Keywords Acid rock drainage · Acid–base accounting · Mine · Overburden · Pyrite · Calcite

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Introduction

Acid rock drainage is a common phenomenon associated with weathering of pyrite-bearing rocks (Sengupta 1993; Lottermoser 2003). Acid drainage can be accentuated by disturbance of pyrite-bearing rocks during mining, and this can result in significant environmental problems around mine sites (Sengupta 1993; Lottermoser 2003; Jambor 2003). In the past, little attention was paid to acid generation, and long-term environmental problems have arisen, associated with water discharges from old mine excavations and from piles of pyrite-bearing waste rocks. Nowadays, handling and long-term storage of pyrite-bearing waste rocks are important issues in any mine site, and details of waste rock stack construction are an integral part of mine design even before mines are opened (Sengupta 1993; Tran et al. 2003; Williams et al. 2003).

One of the most common approaches to waste rock stack construction is to encapsulate the most pyrite-rich rocks in the core of a waste rock stack (Cravotta et al. 1994; Lottermoser 2003; Williams et al. 2003; Bussiere et al. 2004). This potentially acid-forming rock is surrounded and/or capped by waste rocks with lower potential for generating acid, or even some acid neutralisation capacity (ANC; Cravotta et al. 1994; Lottermoser 2003). A key part of this process is detailed knowledge of the relative acid generation potentials of waste rocks during the mining process, so that each truckload of waste is deposited in the correct position in growing waste rock stacks. Hence, it is desirable to be able to characterise waste rocks at the outcrop or truckload scale in a rapid and cost-effective manner.

In this study, we address the above issues for a coal mine that has pyrite-bearing overburden. The overburden will be deposited in waste rock stacks for permanent storage in a very wet climate, so proper characterisation of the waste rocks is essential for long-term environmental stability. The strata overlying the coal seams to be mined have some stratigraphic predictability, so some generalisations on potential acid generation can be made at the mine planning stage. However, facies changes are common in the overburden at the metre to ten metre scales. Hence, it is desirable that the acid generation potential and the ANC of the waste rocks be known at the metre scale, with some degree of confidence, during the mining process. The mine site is remote from areas of population, so specialist laboratory services are difficult to access, and it can take much time to obtain results (days at least). To address this issue of rapid characterisation of waste rocks during mining, which is a world-wide problem, we have devised and calibrated a set of simple criteria for quick (ca. 15 min) determination of acid generation and neutralisation potential of waste rocks in the field. This paper describes these techniques, their reliability, and their limitations for our coal mine example, but we suggest that the approach has more general application for other mines as well.

General setting

The coal mine for which this study was initiated is a new pit associated with the Stockton coal mining area, on the west coast of the South Island of New Zealand (Fig. 1a). The Stockton area currently produces > 1 Mt/year of high-quality low-ash bituminous coal, principally for coking. The mine pits, including the new pit for this study, are at an altitude of 700 m above sea level, in a harsh subalpine environment with rugged topography. The area receives up to 7,000 mm annual precipitation distributed evenly through the year, with a mean annual temperature of ca. 8°C.

The coal occurs in the Eocene Brunner coal measures, a fluvial to marginal marine sequence that rests on Paleozoic basement in the Stockton area (Fig. 1b). The coal measures are overlain conformably by marine sediments deposited during middle Tertiary marine transgression (Fig. 1b; Nathan et al. 1986; Lever 2001). Sediments of the Eocene to early Oligocene Rapahoe Group (Fig. 1b) immediately overlie the coal measures. In the Stockton area, the Kaiata Formation of the Rapahoe Group forms the principal overburden discussed in this study. The Kaiata Formation is dominated by marine mudstones, but has some sandy facies near its base where it overlies the coal-bearing sequence.

The Kaiata Formation rocks are made up of detrital quartz, albite, muscovite, and kaolinite, with minor detrital biotite. Diagenetic alteration of biotite has yielded some smectite and/or vermiculite clay. Calcite occurs as foraminifera tests (Fig. 2) and cement within the sedimentary matrix, and ankeritic carbonate was detected in one sample (X-ray diffraction confirmation). Organic material is scattered through the sediments, locally imparting a brownish colour to the fresh rock. This organic material is most abundant near the base of the formation. Calcite is common in the upper part of the examined section (>40 m above the base) where the abundant cement and fossils, combined with relative paucity of organic matter, gives the rock a pale grey colour. Interlayering of grey and brown rocks on the 1–10 m scale attest to local changes in relative proportions of these constituents. Pyrite is a common component of rocks near the base of the Kaiata Formation (Weber et al. 2006a, b). Authigenic pyrite framboids are scattered through the sediments, and both framboids and coarser-grained pyrite crystals are commonly associated with organic material. Coarse pyrite crystals partially fill chambers in foraminifera tests, localised by decayed organic matter (Fig. 2).

Methods

This study was conducted before the opening of the new mine, so outcrop was limited. Instead, we focussed on drill core extracted during the late mine planning stage. This core, from 11 holes, provides continuous sections through the Kaiata Formation overburden and into the Brunner coal measures. The cored overburden

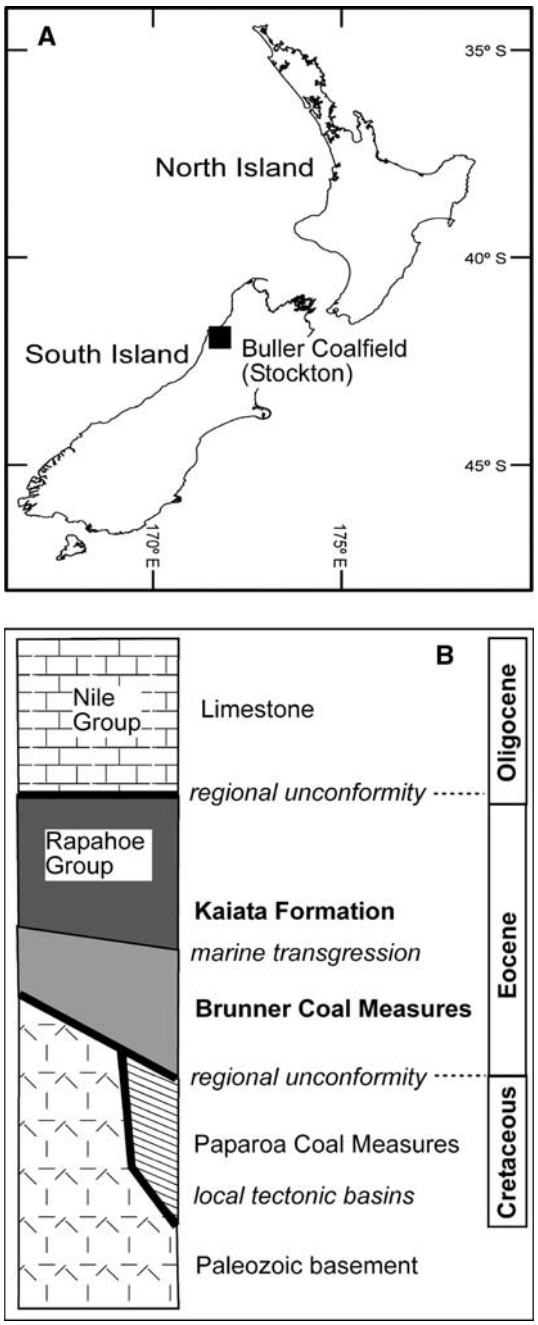

Fig. 1 Geographic and stratigraphic location for the Stockton coal mine, New Zealand. **a** Location of the Buller coalfield, which includes the Stockton mine, on the West Coast of the South Island of New Zealand. **b** Stratigraphic setting (after Lever 2001) of the Brunner coal measures in which the Stockton mine is developed, and the Kaiata Formation which forms the overburden evaluated in this study

is unweathered, although some oxidation during storage (2 years) had almost certainly occurred (Weber et al. 2004, 2006a). Representative samples of Kaiata Formation were taken from cores at intervals of 2–10 m above the boundary with the Brunner coal

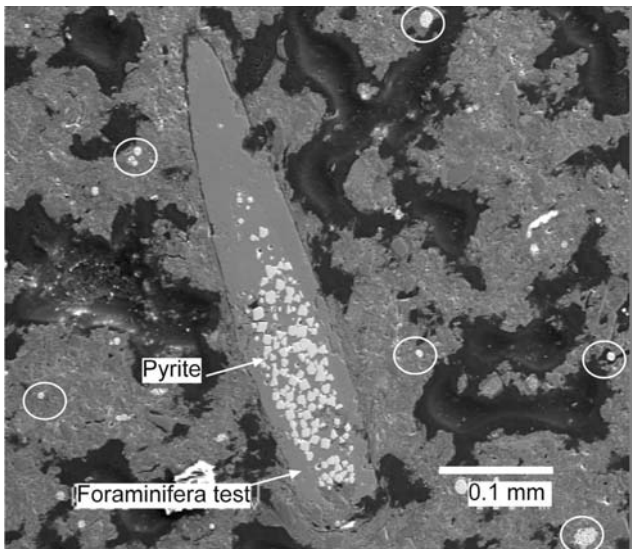

Fig. 2 Backscatter electron image (from electron microprobe) of Kaiata Formation mudstone, showing a large calcite foraminifera test (*grey centre*) with coarse crystalline pyrite (*white*) in body cavities. Irregular framboids of microcrystalline pyrite (*white*, *some circled*) are scattered through the *grey mudstone*. *Black zones* are glue-filled desiccation cracks formed during sample preparation

measures, which is characterised by a distinctive change from fluvial sandstone to finer-grained marine sediments. A total of 104 samples were taken for this study, although not all samples were analysed in detail. Care was taken during sampling to avoid material that may have developed secondary sulphate minerals during core storage.

Total sulphur and total carbon contents of a set of 88 samples were determined by a Carlo Erba Elemental Analyser EA1108. Analyses were done on 2 mg subsamples, which were oxidised and volatilised by flash combustion. Average results were determined from two or three subsamples for each sample. Six subsamples were analysed from one sample, yielding results with a standard deviation of 0.12 wt% for sulphur and 0.05 wt% for carbon. Consequently, we consider the method has instrumental uncertainty of better than ±0.3 wt%. Total sulphur can be used to calculate maximum potential acidity (MPA) by multiplying by 3.125, to yield a result in wt% equivalent calcium carbonate required to neutralise the potentially generated acid (Sengupta 1994; Lottermoser 2003). This calculation assumes that all the sulphur is contained in pyrite, which is a reasonable approximation (Weber et al. 2004, 2006a, b).

Acid neutralisation capacity of a representative set of samples was determined by the method of Sobek et al. (1978). This method involves back-titration to pH 7 after addition of acid to pulverised sample and

heating the reacting solution. Calcium carbonate (calcite) is the dominant neutralising mineral in most rocks, but some neutralisation by other minerals can occur as well (Sobek et al. 1978; Sherlock et al. 1995; Weber et al. 2005). Results were calculated as equivalent wt% calcium carbonate that would be required to neutralise the added acid; i.e. the amount of reacted calcium carbonate assuming calcite was the only reacting mineral. This allows direct comparison with the MPA (above) for the same rock. The difference between ANC and MPA determined in this manner, with the same units, gives an estimate of the net acid producing potential (NAPP; Sobek et al. 1978; Lottermoser 2003; Jambor 2003; Weber et al. 2005).

Estimates of the MPA of representative samples were also obtained using paste pH tests (after Sobek et al. 1978). Small fragments (20 g) of rock were disaggregated in 10 ml distilled water in a 50-ml container, and left for 10 min before measuring pH with a digital meter. This method is suitable for field use (Sobek et al. 1978), although all measurements were done in a laboratory in this study. Full equilibration is not attained in 10 min, but samples left overnight (ca. 10 h) typically change by <0.3 pH units only.

Estimates of calcite content of samples were determined using a ''carbonate bomb'' apparatus first described by Muller and Gastner (1971). The device measures the pressure of CO_2 gas generated by addition of acid to a sample. One gram of pulverised sample was placed in a small pile on the base of a 200-ml glass jar. Three millilitres of 3 mol/l HCl was added to the other side of the base of the glass jar, ensuring the acid does not come in contact with the sample. An Edwards Barocel Pressure Sensor was inserted into the jar, through a rubber stopper that sealed the jar, and the ambient pressure in the jar was recorded. Then, the jar was tilted so that the acid came into contact with the sample, and the calcite reacted. When the reaction ceased (as indicated by stabilised pressure), the pressure difference arising from the CO_2 gas generation was recorded. Visible reaction times in this study were less than 1 min, but dolomite may take up to 15 min to react (Muller and Gastner 1971). The proportions of calcite in the samples were estimated from pressure differences using a calibration line produced from synthetic samples made with known proportions of calcite (Fig. 3a).

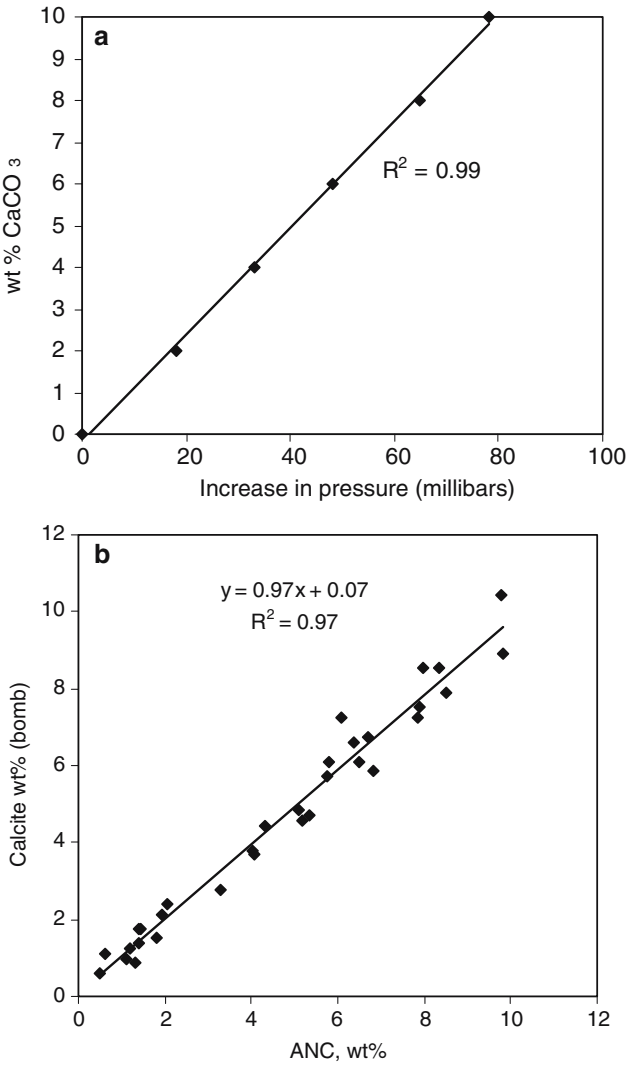

Fig. 3 Calibration of the carbonate bomb of Muller and Gastner (1971). **a** Calibration of the increase in vapour pressure from reaction of acid with synthetic samples containing different proportions of calcite. **b** Comparison between acid neutralisation capacity derived by the method of Sobek et al. (1978) and calcite % estimated using the carbonate bomb test

Stratigraphic variations of Kaiata Formation composition

Pyrite is visible in hand specimen in many Kaiata Formation samples, especially those from near the base of the unit. The total S content of Kaiata Formation rocks (Fig. 4a) reflects these hand specimen observations. Most of the Kaiata rocks contain at least 1.5 wt% total S, with some rocks near the base having up to 4 wt% total S (Fig. 4a). There are similar trends in the Kaiata Formation for total C content as for the total S (Fig. 4b). Total C is up to 4 wt% near the base of the unit, and decreases up-section to ca. 2 wt% (Fig. 4b). Above ca 40 m above the base, total C is slightly elevated again at ca. 2.5–3 wt% (Fig. 4b).

The ANC generally increases up-section through the Kaiata Formation, from low levels near the base (<1 wt% $CaCO_3$ equivalent) to as high as 10 wt%

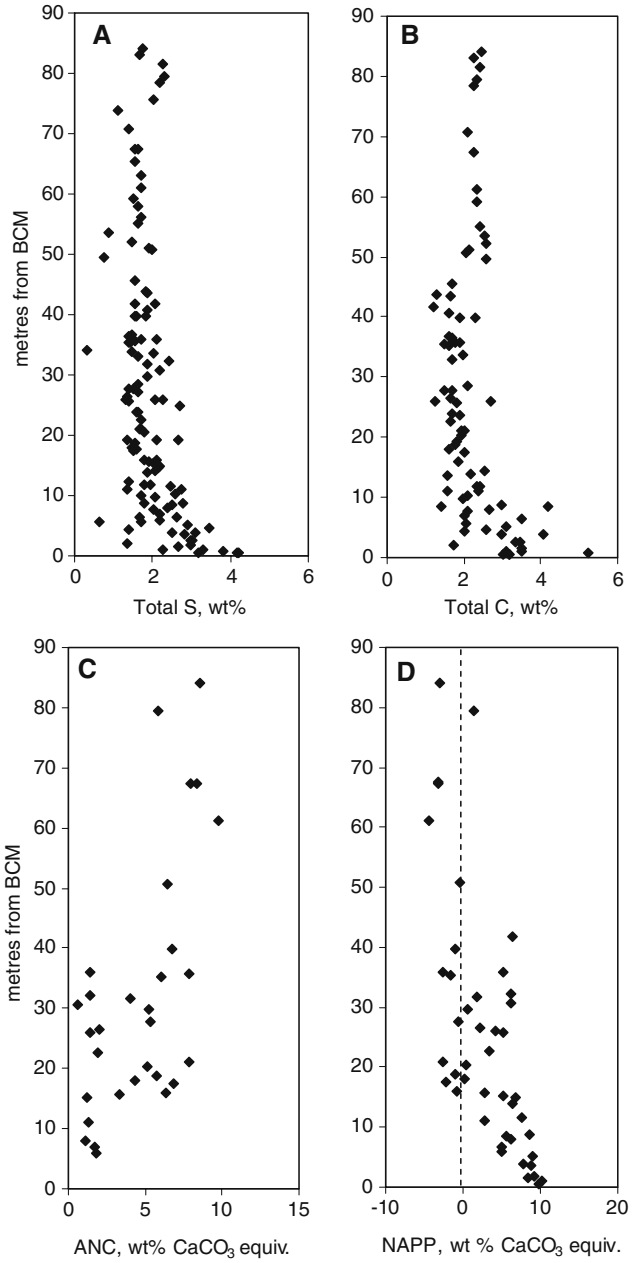

Fig. 4 Variations in key environmental parameters with distance up the Kaiata Formation, measured as metres above the top of the Brunner coal measures (*BCM*).**a** Total sulphur. **b** Total carbon. **c** Acid neutralisation capacity (*ANC*), as determined by the Sobek et al. (1978) method. **d** Net acid producing potential (*NAPP*), determined as difference between maximum potential acidity (calculated from total S) and ANC. Positive NAPP, to *right* of *dashed line*, is acid generating

$CaCO_3$ equivalent (Fig. 4c). The carbon content in the upper part of the section (Fig. 4b) mainly reflects the elevated calcite content that is evident petrographically and in the ANC data (Fig. 4c). The ANC is highly variable in the interval 10–40 m above the base (Fig. 4c). The NAPP of the Kaiata Formation, derived from the difference of MPA (calculated from total S) and the ANC, decreases up-section through the formation (Fig. 4d). NAPP is strongly positive in many samples in the lower 40 m of the formation, implying that up to 10 wt% $CaCO_3$ (equivalent) is required to neutralise acid potentially formed by these rocks (Fig. 3d). Beyond 40 m above the base, most rocks have negative NAPP, implying minor excess of calcite over pyrite (Fig. 4d).

In addition to the similar patterns of variation with stratigraphic height, both total S and total C are highly variable in the lower 30 m of the Kaiata Formation (Fig. 4a, b). However, there is only a crude positive correlation ($R^2 = 0.48$) between total S and total C through the Kaiata Formation (Fig. 5). The elevated carbon content near the base of the formation (Fig. 4b) largely reflects abundance of detrital organic matter in these rocks. The elevated total S near the base of the formation reflects the abundance of pyrite that has been localised by some of this organic matter (Fig. 2). Estimates of the amount of organic matter in some of these rocks could theoretically be made by subtracting the calcite carbon component. However, this calculation yields several negative organic carbon contents (as high as 1 wt% C), presumably as a result of compounding analytical errors, so this calculation is of little value.

Carbonate bomb and paste pH results

The carbonate bomb device was used on a representative set of core samples for comparison to ANC

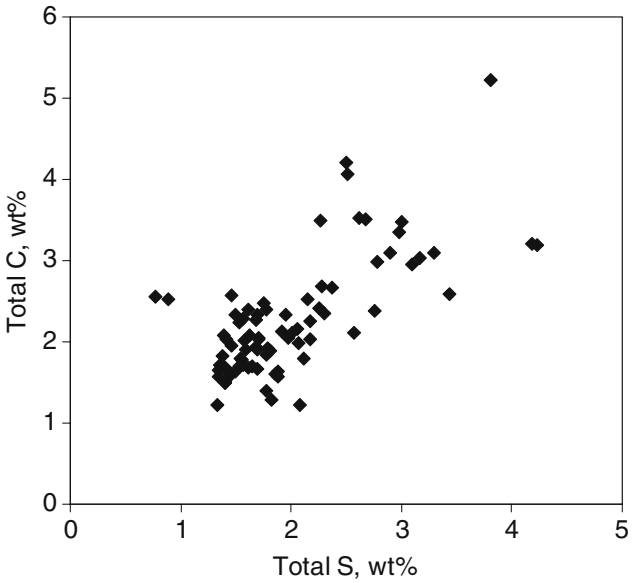

Fig. 5 Relationship between total sulphur and total carbon contents of Kaiata Formation rocks

determined by the more laborious Sobek et al. (1978) laboratory method. The same general trends of variations in calcite wt% (equivalent) are seen through the Kaiata Formation in results of the carbonate bomb tests (Fig. 6a) as are seen for the Sobek et al. (1978) ANC tests (Fig. 4c). Overall, there is a strong positive correlation ($R^2 = 0.97$) between calcite contents determined by this carbonate bomb and ANC (Fig. 3b). These data show that the carbonate bomb test is a useful proxy for the Sobek et al. (1978) method.

Paste pH results through the Kaiata Formation (Fig. 6b) are less obviously similar to the total S data that are used to generate MPA estimates (Fig. 4a). The paste pH data are generally bimodal: those that became distinctly acidic, and those that remained with neutral pH (Fig. 6b). Those samples that acidified in the paste pH test are mostly from the lower 30 m of the Kaiata Formation (Fig. 6b), confirming the high pyrite content that the high total S in many of these lower samples implies (Fig. 4a). This relationship between acid paste pH and high total S is even more pronounced in Fig. 7a, which shows that acid paste pH typically results from rocks with total S > 2 wt%. These acidifying rocks are from the lower part of the Kaiata Formation (mainly < 30 m from base; Fig. 7a).

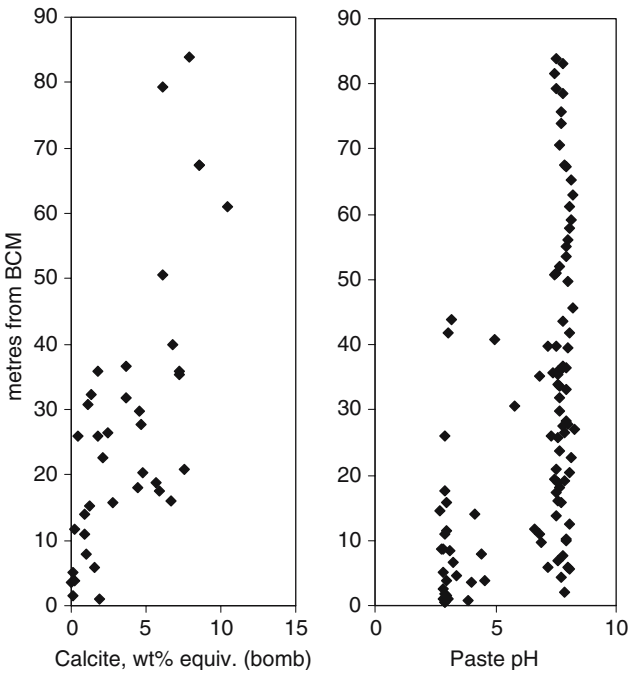

Fig. 6 Variations in environmental parameters determinable in the field, with distance up the Kaiata Formation, measured as metres above the top of the Brunner coal measures (*BCM*). **a** Calcite % from the carbonate bomb test of Muller and Gastner (1971). **b** Paste pH

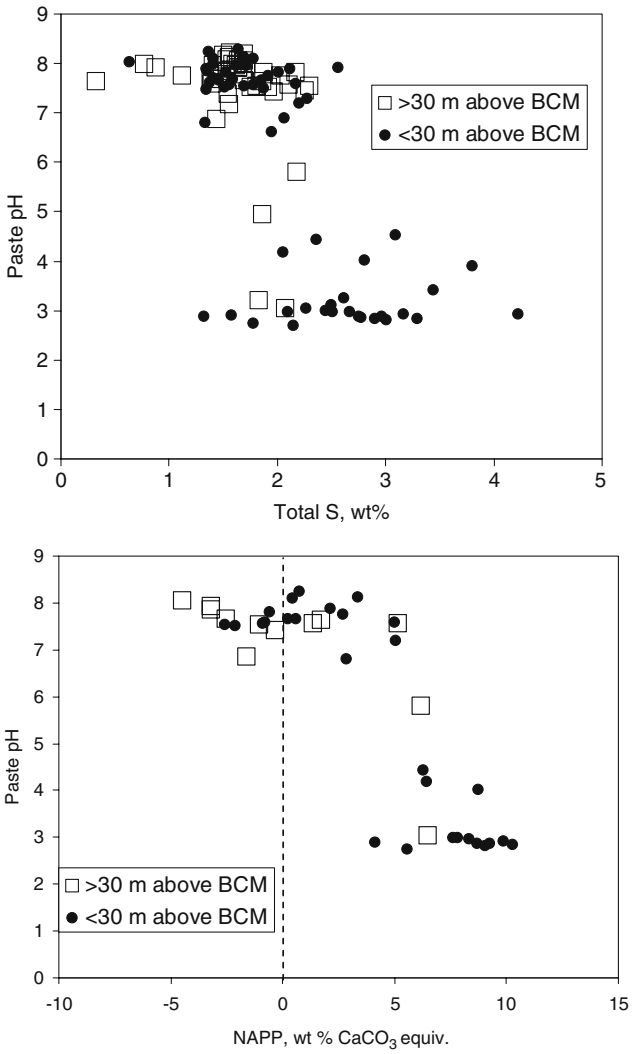

Fig. 7 Relationships between paste pH and **a** total sulphur, and **b** net acid producing potential in the Kaiata Formation. Samples above 30 m from the base (*BCM*) are shown as *squares*; other samples are *black dots*

Some samples with total S < 2 wt% also yield low paste pH (Fig. 7a), and these samples are from the lower part of the Kaiata Formation where calcite is less abundant (Figs. 4b, c, 6a, 7a). Consequently, these samples have relatively high positive NAPP (Fig. 7b).

Discussion

Proxies for ANC and MPA

Results described in the previous section show that the carbonate bomb test is an effective proxy for the Sobek et al. (1978) ANC test, with the added advantage that the carbonate bomb procedure is much simpler. The ANC test reputedly involves neutralisation of acid by

minerals other than calcite (Sobek et al. 1978; Sherlock et al. 1995). In contrast, the carbonate bomb test measures evolved CO_2, and therefore reflects carbonate mineral dissolution only. The strong correlation between ANC and carbonate bomb results implies a linear relationship that passes close to the origin (Fig. 3b). This suggests that any non-carbonate neutralisation reactions that occurred during ANC determinations are negligible. Otherwise the linear relationship would have an intercept on the ANC axis in Fig. 3b that was representative of additional neutralisation reactions in the ANC test that did not occur in the carbonate bomb test. Additional neutralisation potential may be present in some of the studied rocks as ankeritic carbonate, which dissolves more slowly (Muller and Gastner 1971; Skousen et al. 1997). However, the fast neutralisation reactions quantified in this study provide a useful, and conservative, approximation to environmental neutralisation capacity.

The paste pH results provide a general proxy for MPA based on total S content (Figs. 4a, 6b). However, the distinction between high- and low-MPA samples is more subtle because of the bimodal nature of the results (Fig. 7a). Also, there is considerable overlap in total S contents between samples yielding neutral paste pH and samples yielding acid paste pH (Fig. 7a). Paste pH is useful as a general indicator of NAPP, in that strongly negative NAPP samples have neutral paste pH, and strongly positive NAPP samples have acid paste pH (Fig. 7b). There is an acidification ''lag'' for weakly positive NAPP samples in the paste pH test (Fig. 7b). This lag has been quantified, with more sophisticated laboratory-based kinetic tests, to require up to 356 min for onset of acidification (Weber et al. 2006a). Paste pH acidification does not occur until samples have NAPP greater than ca 3 wt% $CaCO_3$ equivalent (Fig. 7b). Some of the discrepancy between paste pH results and MPA and NAPP based on total S contents may be because total S can overestimate MPA if some of the S is contained in organic matter, rather than pyrite, although this has been shown to be minor (Weber et al. 2006a, b). Formation of soluble acidic sulphate salts in the core during storage may have made the material more reactive to the paste pH test, and this extra reactivity would probably overshadow any overestimation of pyritic S. Because of these uncertainties, over-interpretation of details of paste pH results should be avoided.

A combination of carbonate bomb and paste pH results has potential to discriminate between acid-forming and neutralising waste rocks (Fig. 8). This combination of the data defines a trajectory from acid-forming rocks at the base of the Kaiata Formation, to acid-neutralising rocks at the top of the overburden section (Fig. 8). A conservative cut-off between acid-forming and acid-neutralising rocks can be defined with 3 wt% $CaCO_3$ equivalent (carbonate bomb) and neutral paste pH (Fig. 8).

First inspection of the ANC and MPA data through the Kaiata Formation (Fig. 4a, c, d) suggests a strong stratigraphic control on potential acidification. This implies that a simple stratigraphic boundary could be drawn between acid-forming and acid-neutralising rocks at, say, 30 m above the base of the formation (Fig. 4a, c, d). However, there are many samples near the base of the formation that are potentially acid-neutralising, and some in the upper part of the section that are potentially acid-forming (Fig. 4a, c, d). Hence, a stratigraphic cut-off for waste rock stack construction is an oversimplification. Interlayering of potentially acid-forming and potentially acid-neutralising rocks between 10 and 50 m above the base of the Kaiata Formation is indicated in Fig. 8. Paste pH and carbonate bomb data provide an effective and simple means of distinguishing these rocks, especially when used in the context of the stratigraphy.

Field techniques

All the data for this study were gathered in a laboratory, but the paste pH and carbonate bomb techniques,

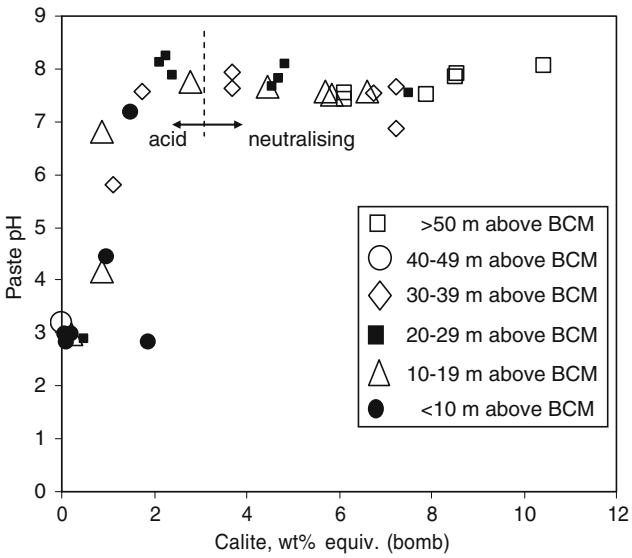

Fig. 8 Combined results of carbonate bomb test (proxy for acid neutralisation capacity) and paste pH (proxy for maximum potential acidity) for Kaiata Formation samples. *Symbols* are for different distances above the base of the formation (*BCM*), as indicated in the *legend*. *Dashed line* indicates probable distinction criteria for acid-neutralising rocks (to *right*) and acid-generating rocks (to *left*)

in combination, are ideally suited for rapid field characterisation of waste rocks. Paste pH is the simplest test to do in the field, as it needs only basic measurements of sample and solution amounts, followed by pH measurement with a field pH meter. Sample preparation, equilibration, and measurement take less than 15 min. Several samples can be prepared at the same time, and left to stand while more samples are prepared, then pH of all samples measured in quick succession. Battery-powered field scales could be used for weighing samples. Alternatively, use of sample volume, in a calibrated sampling cup, rather than sample weight, would eliminate this need for weighing materials in the field.

The carbonate bomb is more complex than paste pH, but is still readily adaptable for field measurement. The pressure sensor is battery-powered and readily portable, and the whole apparatus weighs less than 3 kg. Sample preparation could be streamlined, as for paste pH, by use of a calibrated sampling cup, rather than weighing samples. However, unlike paste pH, it would be more difficult to test several samples simultaneously, unless a number of pressure sensors were purchased and carried in the field.

Initial and on-going calibration of the field techniques against laboratory-based static and/or kinetic tests is likely to be necessary throughout the life of a mine, as geological variations outside initial calibrations are highly likely. However, these calibrations can be conducted as part of background quality assurance procedures, at a slower rate than is demanded by day-to-day mine operations.

Conclusions

A portable calcite-dissolution quantification test based on pressure of evolved carbon dioxide gas is a useful proxy for ANC determined in a laboratory by the Sobek et al. (1978) method. There is a high degree of correlation of results ($R^2 = 0.97$) between these two techniques in samples taken from calcareous overburden on a coal mine in this study. Acid neutralisation reactions involve minerals other than carbonates in these rocks, for both techniques. Paste pH determined on the coal mine overburden yields bimodal results, with pyrite-rich, calcite-poor rocks having paste pH near 3, and calcite-rich rocks having paste pH near 7. Hence, paste pH is a crude proxy for MPA that has been determined from analyses of total sulphur content. A combination of paste pH and the portable calcite-dissolution quantification test has potential for distinguishing acid-generating from acid-neutralising rocks in mine overburden. The advantage of these two techniques is that they can be used in the field, and analyses can be obtained in about 15 min. Hence, real-time environmental characterisation of mine waste rocks at the metre scale is possible as a routine part of mine excavation operations. This can enable efficient construction of waste rock stacks that encapsulate all acid-forming rocks within acid-neutralising rocks. On-going calibration of the field tests with laboratory-based techniques is still required, but this can be done on a longer time scale than routine mine operations.

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