

# Mineralogical and geochemical investigations of silicate-rich mine waste from a kyanite mine in central Virginia: implications for mine waste recycling

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**Abstract** A kyanite mine in central Virginia produces a silicate-rich waste stream which accumulates at a rate of 450,000–600,000 tons per year. An estimated 27 million tons of this waste stream has accumulated over the past 60 years. Grain size distribution varies between 1.000 and 0.053 mm, and is commonly bimodal with modes typically being 0.425 and 0.250 mm and uniformity coefficients vary from 2.000 to 2.333. Hydraulic conductivity values vary from 0.017 to 0.047 cm/s. Mineralogy of the waste stream consists of quartz, muscovite, kyanite and hematite. Muscovite grains have distinct chemical compositions with significant Na<sub>2</sub>O content (1.12–2.66 wt%), TiO<sub>2</sub> content (0.63–1.68 wt% TiO<sub>2</sub>) and Fe content, expressed as Fe<sub>2</sub>O<sub>3</sub> (up to 1.37 wt%). Major element compositions of samples were dominated by SiO<sub>2</sub> (87.894–90.997 wt%), Al<sub>2</sub>O<sub>3</sub> (6.759–7.741 wt%), Fe<sub>2</sub>O<sub>3</sub> (1.136–1.283 wt%), and K<sub>2</sub>O (0.369–0.606 wt%) with other components being <1.000 wt%. Elements of environmental concern (V, Cr, Ni, Cu, Zn, As, Ag, Sn, Sb, Ba, Hg, Tl, and Pb) were detected; however, the concentrations of all elements except Ni were below that of the kyanite quartzites in the region from which the waste is derived. Both major and trace element compositions indicate minimal variation in composition. The waste stream has potential for recycling. Muscovite is suitable for recycling as a paint pigment or other industrial applications. Muscovite and hematite are commonly

intergrown and are interpreted to be material where much of the elements of environmental concern are concentrated. Reprocessing of the waste stream to separate muscovite from other components may enable the waste stream to be used as constructed wetland media for Virginia and nearby states. Recycling of this mine waste may have a positive impact on the local economy of Buckingham County and aid in mitigation of wetland loss.

**Keywords** Mine waste · Recycling · Mineralogy · Kyanite · Virginia

## Introduction

Mine waste is a major issue globally that impacts the environment in numerous ways including landscape disturbance (e.g., Bian et al. 2009; Hancock and Turley 2006; Brown 2005; Hamilton 2000; Hancock et al. 2000) and water resources (e.g., Meck et al. 2006; Dinelli et al. 2001; Pain et al. 1998; Ribet et al. 1995). Mineralogical studies of mine waste are important for understanding pollution pathways (Marescotti et al. 2008; Krekeler et al. 2008; Dold and Fontbote 2001, 2002; Hudson-Edwards et al. 1999; Foster et al. 1998; Davis et al. 1993). The integration of mine waste studies involving mineralogy and bulk properties is key towards developing strategies for mine waste management, including recycling of waste materials.

Recycling of waste is critical for reducing the environmental impact of mining operations globally and may have economic benefit for disadvantaged regions where mining occurs or has occurred historically. A critical factor in implementing the recycling of mine waste is understanding environmental mineralogical, geochemical and bulk physical properties. These properties must be determined in

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order to assess the potential environmental impact that these materials may have if recycled into large scale environmental applications or consumer products. If mine waste material is assessed to be free of contaminants, it may be suitable for use in such applications as a constructed wetland media, sand filtration for wastewater treatment, soil amendments, or secondary industrial mineral applications.

Kyanite Mining Corporation of Dillwyn, Virginia is the sole producer of the aluminosilicate mineral kyanite in the United States and the largest producer of the mineral in the world. The company is one of the largest employers in Buckingham County, which is an economically disadvantaged area. Kyanite is used in a variety of foundry applications and some ceramic applications. According to the United States Geological Survey (2010) minerals yearbook, the mineral has a 2009 estimated annual market value of approximately \$256 US per metric ton.

The mine extracts kyanite quartzite from Willis Mountain of Dillwyn in the Piedmont province of central Virginia. The Willis Mountain kyanite ore deposits were once thought to be produced from simple prograde metamorphism of aluminum-rich sandstones; however, further studies have interpreted the protolith as being igneous rocks altered by high-sulfur hydrothermal activity (Owens and Pasek 2007). Further geologic details of the region can be found in Krekeler et al. (2009) and references therein. Of the kyanite quartzite, 20–30% is industrial mineral grade kyanite. Information from Kyanite Mining Corporation states that continuous operations can produce more than 150,000 metric tons of processed commercial grade kyanite annually.

During processing of the ore, waste minerals are separated into three distinct and well-sorted streams. A pyrite-rich waste stream is separated into sealed silos or more commonly is sold. A second waste stream of magnetic separate dominated by magnetite and pyrrhotite accumulates in a stock pile and an estimated 3.57 million metric tons of this material has accumulated since the late 1940s (Krekeler et al. 2009). The largest waste stream is silicate-rich material that is dominantly quartz and muscovite. For every metric ton of commercial grade kyanite obtained, three to four metric tons of silicate-rich waste is produced. By multiplying the annual tonnage of kyanite produced (150,000 metric tons) by a factor of three to four, an estimated annual accumulation of this waste stream is 450,000–600,000 metric tons. A crude estimate accounting for variation in production over the past 60 years indicates that approximately 27 million tons of this waste stream has accumulated. The purpose of this paper is to investigate the environmental mineralogical, geochemical and bulk properties of the silicate-rich waste stream to evaluate its recycling potential.

## Materials and methods

Fifteen samples of the waste were acquired in April of 2008. For grain size analysis, approximately 60–100 g of each sample was passed through a set of 8 in. brass sieves whose opening size varied from 5.600 to 0.038 mm. The material retained by each sieve was weighed electronically and its mass was recorded and converted to percentages.

Hydraulic conductivity ( $K$ ) was measured using a falling head permeameter. Approximately 190 cm<sup>3</sup> of unaltered sample was poured into the permeameter. Sample material was then slowly wetted to prevent the trapping of air pockets which would lead to inappropriately low values of  $K$ . The permeameter was filled with water to 40 cm above the bottom of the sample. Increments of 2 cm were marked on the outside of the permeameter from the 40 cm  $h_0$  height down to 30 cm. Times for the water level to fall between the 2 cm increments were measured upon removal of the bottom plug.

For powder X-ray diffraction, approximately 5 g of material was milled in a SPEX aluminum oxide mini-mill for 10 min. Samples were analyzed using a Scintag X-1 powder diffractometer equipped with a Peltier detector, using Cu  $K\alpha$  radiation (0.1548 nm) operated at 40 kV and 35 mA. Scan parameters were 2° to 65°  $2\theta$  at 0.02° steps for 0.5 s count times. Minerals were identified using the computer program Jade and PDF cards (quartz, muscovite, hematite, kyanite).

SEM investigation was conducted using a Zeiss Supra 35 VP FEG equipped with an energy dispersive spectrometer (EDS). All images were captured in variable pressure mode using N<sub>2</sub> gas to prevent charging. Sample grains were mounted on carbon sticky tabs on aluminum stubs and were uncoated.

Chemical analysis of major elements was conducted using a Beckman Spectraspan V direct-current argon plasma (DCP) spectrometer. Trace element concentrations were completed using a Varian inductively coupled plasma mass-spectrometer (ICPMS). Ten powdered rocks of known compositions ranging from shale to andesite were used as reference standards. Sample powders used for X-ray diffraction investigation were also used for bulk chemical analysis of major and trace elements. For DCP analysis, samples and standards were weighed to 100.0 ± 0.5 mg, thoroughly mixed with 500.0 ± 0.5 mg of LiBO<sub>2</sub> spectrafux, transferred into graphite crucibles, and fused in an oven at 950°C for 20 min. A blank was created by putting ~500 mg of flux directly into ~50 g of nitric acid stock. The 1:5 sample-to-flux ratio resulted in proper dissolution of all samples and standards after fusion. The fused samples and standards were placed in ~50 g of nitric acid stock and allowed to dissolve overnight on a

shaking table. This solution was used to analyze trace elements. Major element solutions were created by diluting ~2 g of the trace solution in ~25 g of nitric acid stock. A new blank was created by adding ~500 mg of flux to ~50 g of nitric acid. After major and trace element solutions were created, they were run through the DCP spectrometer. Major solutions were analyzed three times while trace solutions were analyzed twice. All solutions were analyzed for 15 counts. Results for each solution were then averaged. Results for phosphorus weight percents were not accurate when analyzed as a major element. Therefore, phosphorus was analyzed again using the trace solution instead of the diluted major element solution.

For ICP-MS analysis, samples were prepared somewhat differently than for DCP analysis by combining 35 mg sample or reference standard with 85 mg sodium tetraborate and potassium carbonate flux, transferring it into graphite crucibles and fusing at 950°C for 30 min. To keep the dissolved solid content of solutions less than 1,000 ppm for instrument longevity, the fused samples or standards were dropped into 120 ml 1% trace-metal grade nitric acid and allowed to dissolve overnight on a shaking table. A blank was created by dropping 85 mg flux directly into 120 ml 1% nitric acid. Trace element solutions were analyzed with the ICP-MS to create three replicates per solution with 30 readings each.

**Results**

**Bulk properties**

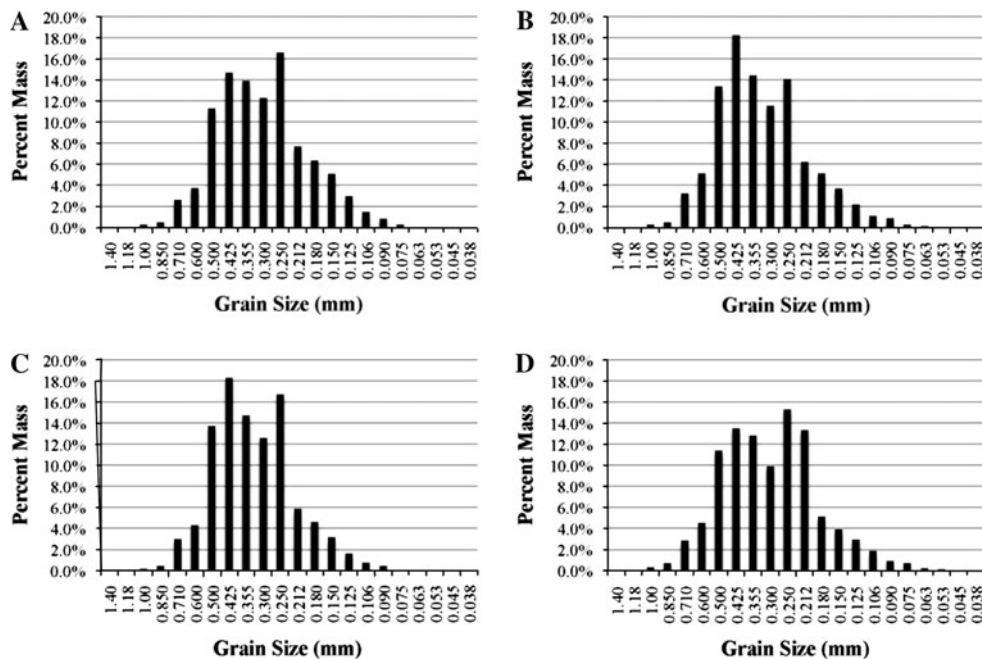
Grain size data are presented in Table 1 and representative histograms are presented in Fig. 1. Grain size varied from 1.000 to 0.053 mm for all samples. Fourteen of the 15 (93%) samples produced a bimodal histogram with modes at 0.425 and 0.250 mm. These modes comprised 13.5–19.7%, and 13.7 and 17.1% of the mass of the samples, respectively. Coefficients of uniformity (Cu) varied minimally with values between 2.000 and 2.333, with a mean of 2.148 and a standard deviation of 0.177.

Hydraulic conductivity values (Table 1) show some variation with values between 0.019 and 0.047 cm/s having a mean value of 0.035 cm/s with a standard deviation of 0.008 cm/s. Because linear regression analysis of  $\ln(h/h_0)$  against time for multiple trials of each sample resulted in  $R^2$  values from 0.9984 to 1.000, Darcy’s Law applies. Permeameter measured hydraulic conductivity values are higher than those estimated based on grain characteristics. The Hazen method (Hazen 1911), based on grain size, returned values ranging from 0.018 to 0.025 cm/s. The Shepherd method (Sheperd 1989), based on grain size and shape, underestimated  $K$  by an order of magnitude with a range of 0.005–0.007 cm/s.

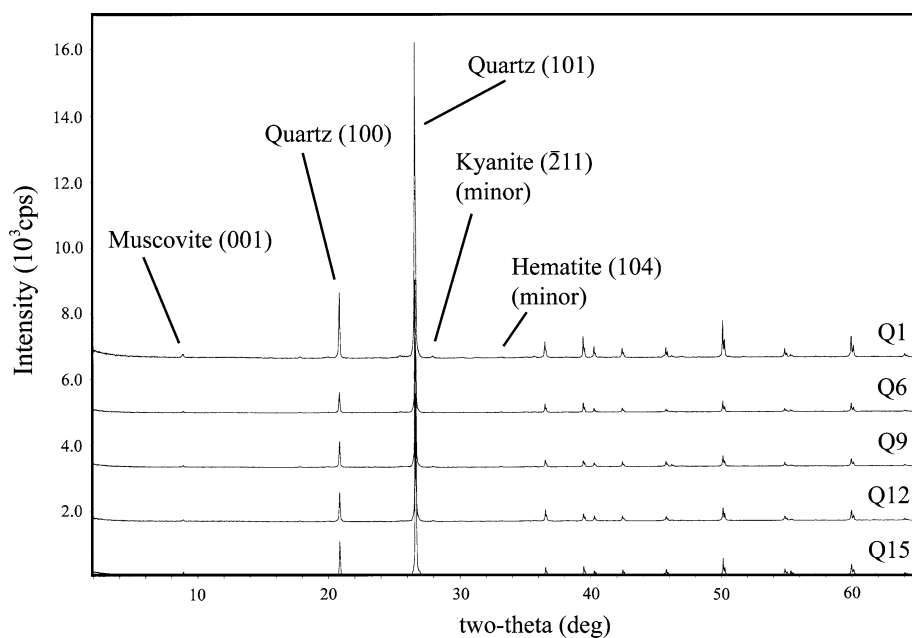
**Table 1** Summary chart of grain size data and hydraulic conductivity values for samples

Sample	Grain size range (mm)	Grain size modes (mm)	Coeff. uniformity	$d_{50}$ (mm)	$d_{10}$ (mm)	$d_{10}$ (cm)	Measured conductivity (cm/s)	Shepherd estimate (cm/s)	Hazen estimate (cm/s)
Q1	1.00–0.075	0.425, 0.250	2.17	0.285	0.150	0.015	0.0258	0.0054	0.0180
Q2	1.00–0.063	0.425, 0.250	2.00	0.310	0.175	0.018	0.0334	0.0061	0.0245
Q3	1.00–0.075	0.425, 0.250	2.14	0.330	0.175	0.018	0.0410	0.0067	0.0245
Q4	1.00–0.075	0.425, 0.250	2.33	0.305	0.150	0.015	0.0344	0.0059	0.0180
Q5	1.00–0.063	0.425, 0.250	2.14	0.320	0.175	0.018	0.0368	0.0064	0.0245
Q6	1.18–0.075	1.18–1.00, 0.355, 0.250	2.17	0.290	0.150	0.015	0.0373	0.0055	0.0180
Q7	1.00–0.063	0.425, 0.250	2.33	0.300	0.150	0.015	0.0420	0.0058	0.0180
Q8	1.00–0.063	0.425, 0.250	2.33	0.320	0.150	0.015	0.0409	0.0064	0.0180
Q9	1.00–0.090	0.425, 0.250	2.00	0.330	0.175	0.018	0.0474	0.0067	0.0245
Q10	1.00–0.090	0.425, 0.250	2.14	0.325	0.175	0.018	0.0461	0.0065	0.0245
Q11	1.00–0.075	0.425, 0.250	2.14	0.340	0.175	0.018	0.0279	0.0070	0.0245
Q12	1.00–0.075	0.425, 0.250	2.00	0.315	0.175	0.018	0.0291	0.0062	0.0245
Q13	1.00–0.075	0.425, 0.250	2.00	0.330	0.175	0.018	0.0337	0.0067	0.0245
Q14	1.00–0.075	0.425, 0.250	2.14	0.330	0.175	0.018	0.0193	0.0067	0.0245
Q15	1.00–0.053	0.425, 0.250	2.17	0.275	0.150	0.015	0.0260	0.0051	0.0180
Min.	n.a.	n.a.	2.00	0.275	0.150	0.015	0.0193	0.0051	0.0180
Max.	n.a.	n.a.	2.33	0.340	0.175	0.018	0.0474	0.0070	0.0245
Avg.	n.a.	n.a.	2.15	0.314	0.165	0.017	0.0347	0.0062	0.0219
SD	n.a.	n.a.	0.12	0.019	0.013	0.001	0.0081	0.0006	0.0033

**Fig. 1** Representative grain size histograms (a), (b), (c), and (d) correspond with samples Q1, Q5, Q9, and Q15, respectively. Histograms show the multimodal nature of samples with modes at 0.425 and 0.250 mm



**Fig. 2** Representative powder X-ray diffraction patterns of selected samples with major peaks of respective minerals labeled. Patterns show nominal variation

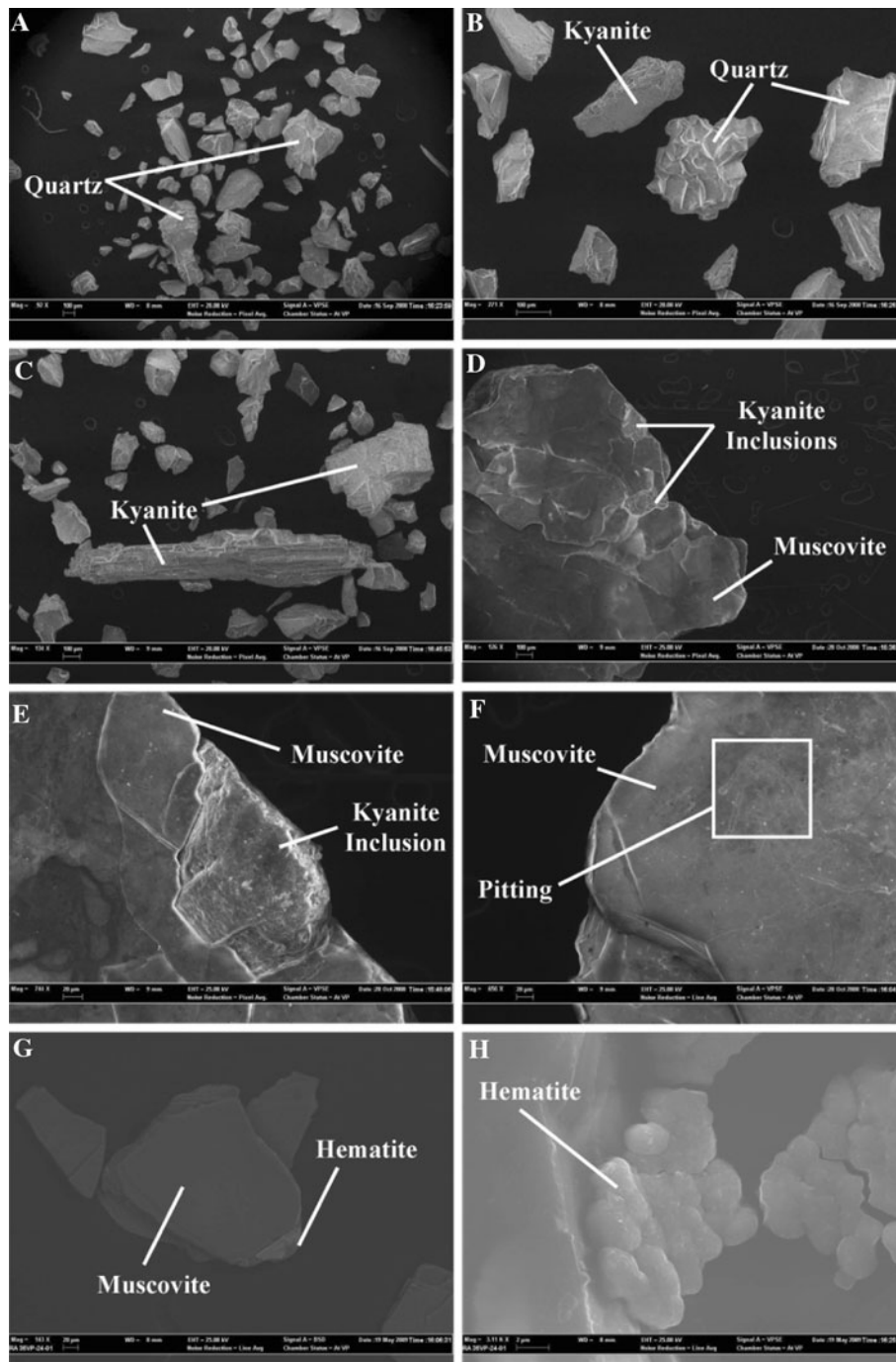


## Mineralogy

XRD patterns of the samples are very similar and show little variation in relative peak intensity of minerals (Fig. 2). Quartz is the dominant mineral present with strong (100), (101), (110), (102), (200) and (112) peaks that agree with PDF# 046-1045. Muscovite consistently shows trace (001) and (002) peaks which vary slightly in d-spacing from approximately 0.990–1.050 nm. Minor or trace peaks of the (104) hematite with a d-spacing of

(0.318 nm) consistent with PDF# 033-0664 occurs in many samples. Minor amounts of kyanite are also observed in many of the XRD patterns with a peak consistent with (11) of PDF# 011-0046.

SEM investigation indicates that the mine waste has a wide variety of grain morphologies (Fig. 2). Quartz and kyanite grains are most common and are dominantly angular and subhedral and compose approximately 95–99% of the grain population. Quartz grains are largely equant, and approximately 90% of kyanite grains are blade-



**Fig. 3** Representative SEM images of the mine waste were produced under analytical conditions shown respectively. **a** Quartz grains and lesser amounts of muscovite. Quartz grains are anhedral to subhedral, angular, and commonly with conchoidal fractures. Most grains are equant or bladed. Aspect ratios vary from approximately 1–5. Grain diameters vary from approximately 50 to 750  $\mu\text{m}$ , although most vary from approximately 200 to 500  $\mu\text{m}$ . **b** Quartz grains and a kyanite grain. The quartz grains are very angular, and have conchoidal fractures. Grain textures vary from anhedral to subhedral. The quartz grains are largely equant with diameters that vary from approximately 100 to 400  $\mu\text{m}$ . **c** Kyanite grains that are dominantly angular and subhedral. Kyanite grains are blade-shaped or tabular, and aspect

ratios vary from approximately 3–9. Grain diameters vary from approximately 200 to 400  $\mu\text{m}$ . **d** Large muscovite grain with kyanite inclusions. The grain has near euhedral to anhedral (hkl) faces. **e** A kyanite inclusion in a muscovite grain that is approximately 260  $\mu\text{m}$  in diameter, blade-shaped, and angular. There is a distinct contact boundary between the muscovite grain and the kyanite inclusion. **f** A muscovite grain that is platy, subhedral, and approximately 580  $\mu\text{m}$  in diameter. Small holes represent pitting as the result of processing. **g** A low magnification back scatter image of a muscovite grain with hematite intergrowth at the lower right portion of the grain. **h** A broken botryoidal texture of hematite commonly observed

shaped or tabular with other examples being equant. Grain diameters of quartz and kyanite commonly vary from approximately 200 to 400  $\mu\text{m}$  (Fig. 3).

Muscovite grains compose approximately 1–5% of the total grain population and are of the typical platy habit of the mineral, often with subhedral or well rounded edges, presumably from crushing and grinding during ore processing. Muscovite grains tend to be larger than other minerals and vary in maximum dimensions from approximately 500 to 2000  $\mu\text{m}$ . Approximately 10% of the muscovite grains contain blade-shaped kyanite inclusions with diameters being approximately 250  $\mu\text{m}$ . Contact boundaries between the muscovite and kyanite inclusions are distinct. Hematite is intimately associated with muscovite and occurs as platy intergrowths in approximately 10% of muscovite grains. Hematite occurs with a flat, smooth, platy texture as well as a less common

botryoidal texture. EDS analysis of hematite detected no impurities above the detection limit of most elements of approximately 0.1 wt%.

EDS analysis of muscovite grains shows a distinct chemical composition (Table 2).  $\text{K}_2\text{O}$  content varies from 7.16 to 10.69 wt%,  $\text{Na}_2\text{O}$  content varies from 1.12 to 2.66 wt%. The muscovite has distinctive  $\text{TiO}_2$  content (0.63–1.68 wt%  $\text{TiO}_2$ ) and has appreciable Fe content expressed as  $\text{Fe}_2\text{O}_3$  (up to 1.37 wt%) (Table 3).

#### Bulk chemistry

The bulk chemical composition of the waste is essentially uniform with no functional trends of co-variation or distinct separate populations existing. Major element analysis of the silicate waste indicates compositions are dominated by four elements. The samples consist primarily of  $\text{SiO}_2$

**Table 2** EDS analyses of muscovite

	1–1	1–2	1–3	1–4	1–5	1–6	1–7	1–8	1–9	1–10	1–11	1–12	1–13	1–14
$\text{SiO}_2$	47.88	48.08	47.49	48.04	48.04	47.95	48.67	48.10	47.81	48.65	48.25	48.53	48.13	48.05
$\text{TiO}_2$	1.37	1.57	1.59	1.29	1.27	1.31	1.30	1.10	1.22	1.11	1.25	1.31	1.30	1.41
$\text{Al}_2\text{O}_3$	39.10	38.34	38.72	37.75	37.83	37.81	37.62	38.71	38.51	38.23	38.00	37.47	38.22	37.10
$\text{Fe}_2\text{O}_3$	0.45	0.47	0.46	0.46	0.64	0.30	0.40	0.38	0.59	0.00	0.41	0.45	0.36	0.41
$\text{MgO}$	0.39	0.58	0.57	1.01	0.74	0.85	0.78	0.67	0.70	0.86	0.91	0.85	0.73	1.17
$\text{Na}_2\text{O}$	1.58	1.64	1.42	1.45	1.39	1.62	1.78	1.82	2.13	1.63	1.83	1.49	1.72	1.17
$\text{K}_2\text{O}$	9.22	9.32	9.75	10.01	10.09	10.15	9.45	9.22	9.05	9.52	9.35	9.89	9.54	10.69
Total	100.00	100.00	100.00	100.00	100.00	100.00	100.00	100.00	100.00	100.00	100.00	100.00	100.00	100.00
	1–15	3–1	3–2	3–3	3–4	3–5	3–6	3–7	3–8	3–9	3–10	6–1	6–2	6–3
$\text{SiO}_2$	47.65	55.01	56.22	51.42	50.94	51.75	51.03	52.70	51.43	51.62	52.00	48.50	48.90	48.30
$\text{TiO}_2$	1.02	1.66	1.06	0.87	0.93	0.86	1.20	0.67	1.68	0.63	0.90	1.29	1.14	1.02
$\text{Al}_2\text{O}_3$	39.23	30.95	32.81	36.04	36.00	36.06	35.90	35.52	35.26	36.70	35.28	39.70	39.91	39.71
$\text{Fe}_2\text{O}_3$	0.45	1.06	0.72	0.33	0.50	0.34	0.61	0.71	1.37	0.55	1.02	0.46	0.28	0.32
$\text{MgO}$	0.79	1.10	0.61	1.49	1.26	1.21	1.55	1.37	0.72	1.11	1.15	0.30	0.64	0.35
$\text{Na}_2\text{O}$	1.90	1.19	1.24	1.49	2.01	1.41	1.12	1.88	1.39	1.36	1.40	2.19	1.66	2.23
$\text{K}_2\text{O}$	8.96	9.02	7.35	8.35	8.36	8.38	8.60	7.16	8.15	8.03	8.25	7.58	7.47	8.07
Total	100.00	100.00	100.00	100.00	100.00	100.00	100.00	100.00	100.00	100.00	100.00	100.00	100.00	100.00
	6–4	6–5	6–6	6–7	6–8	6–9	6–10	6–11	Max	Min	Average	SD		
$\text{SiO}_2$	48.43	49.56	48.90	47.66	48.72	48.27	50.22	49.75	56.22	47.49	49.52	2.11		
$\text{TiO}_2$	1.05	1.65	1.18	1.20	1.21	1.40	1.42	1.49	1.68	0.63	1.22	0.26		
$\text{Al}_2\text{O}_3$	39.30	36.82	39.35	39.55	38.78	39.40	36.37	36.35	39.91	30.95	37.46	1.96		
$\text{Fe}_2\text{O}_3$	0.57	0.50	0.57	0.55	0.60	0.71	0.46	0.57	1.37	0.00	0.53	0.24		
$\text{MgO}$	0.62	1.69	0.32	0.58	0.38	0.38	1.76	1.84	1.84	0.30	0.89	0.42		
$\text{Na}_2\text{O}$	2.35	1.54	1.42	2.66	2.12	1.52	1.41	1.82	2.66	1.12	1.67	0.36		
$\text{K}_2\text{O}$	7.67	8.24	8.26	7.81	8.19	8.31	8.36	8.18	10.69	7.16	8.72	0.89		
Total	100.00	100.00	100.00	100.00	100.00	100.00	100.00	100.00						

The first digit in analysis designation indicates the sample and the second is the analysis number. Descriptive statistics are provided for all analyses

**Table 3** Chemical analyses of mine waste samples as determined by ICP-MS and DCP. Major element concentrations are expressed in weight percent. Minor and trace elements are expressed as parts per million (ppm)

	Q1	Q2	Q3	Q4	Q5	Q6	Q7	Q8	Q9	Q10	Q11	Q12	Q13	Q14	Q15	Min	Max	Average	SD
SiO <sub>2</sub>	87.894	88.424	88.060	89.071	88.841	89.235	89.596	88.573	89.195	90.997	88.724	87.930	90.613	88.165	88.591	87.894	90.997	88.927	0.914
TiO <sub>2</sub>	0.187	0.207	0.211	0.187	0.196	0.199	0.187	0.192	0.200	0.189	0.201	0.190	0.191	0.188	0.220	0.187	0.220	0.196	0.010
Al <sub>2</sub> O <sub>3</sub>	6.874	6.979	6.759	6.889	6.769	7.165	7.014	6.978	7.078	7.246	6.794	6.913	6.944	7.741	7.627	6.759	7.741	7.051	0.292
Fe <sub>2</sub> O <sub>3</sub>	1.197	1.175	1.154	1.282	1.186	1.258	1.246	1.202	1.283	1.241	1.149	1.277	1.260	1.136	1.147	1.136	1.283	1.213	0.054
MnO	b.d.	b.d.	b.d.	b.d.	b.d.	b.d.	b.d.	b.d.	b.d.	b.d.	b.d.	b.d.	b.d.	b.d.	b.d.	0.000	0.000	0.000	n.r.
MgO	b.d.	0.035	0.025	b.d.	b.d.	b.d.	b.d.	b.d.	b.d.	b.d.	b.d.	b.d.	b.d.	b.d.	0.021	0.021	0.035	0.027	0.007
CaO	b.d.	b.d.	b.d.	b.d.	b.d.	b.d.	b.d.	b.d.	0.227	b.d.	b.d.	b.d.	b.d.	b.d.	b.d.	0.227	0.227	0.227	n.r.
Na <sub>2</sub> O	b.d.	b.d.	b.d.	b.d.	b.d.	b.d.	b.d.	b.d.	0.110	b.d.	b.d.	b.d.	b.d.	b.d.	b.d.	0.110	0.110	0.110	n.r.
K <sub>2</sub> O	0.606	0.572	0.544	0.466	0.524	0.410	0.403	0.380	0.398	0.369	0.540	0.408	0.420	0.403	0.540	0.369	0.606	0.466	0.080
P <sub>2</sub> O <sub>5</sub>	0.021	0.022	0.031	0.033	0.023	0.012	0.042	0.060	0.052	b.d.	0.035	0.064	0.059	0.054	0.057	0.012	0.064	0.040	0.017
LOI	0.651	0.644	0.633	0.636	0.626	0.623	0.640	0.629	0.644	0.618	0.642	0.621	0.621	0.572	0.602	0.572	0.651	0.627	0.020
Total	97.430	98.058	97.417	98.564	98.165	98.902	99.128	98.014	99.187	100.660	98.085	97.403	100.108	98.259	98.805	97.403	100.660	98.546	0.950
Be	0.29	0.29	0.28	0.28	0.28	0.29	0.28	0.27	0.27	0.27	0.27	0.28	0.29	0.28	0.29	0.27	0.29	0.28	0.01
V	53.16	56.25	52.54	53.49	53.44	53.67	55.30	54.63	54.17	53.63	56.02	56.86	56.96	54.35	57.36	52.54	57.36	54.79	1.56
Cr	7.26	7.72	7.95	7.00	6.84	7.44	7.44	7.94	7.73	7.38	7.41	7.75	8.41	7.77	7.27	6.84	8.41	7.55	0.40
Ni	9.72	5.98	5.69	8.40	10.51	7.61	13.45	9.75	14.36	10.59	8.95	4.95	7.44	12.31	12.22	4.95	14.36	9.46	2.85
Cu	13.68	15.61	14.38	37.62	13.86	15.36	15.75	16.46	14.41	12.39	16.87	14.56	13.90	14.34	15.37	12.39	37.62	16.30	6.01
Zn	2.91	1.70	2.09	1.76	0.57	2.16	5.54	4.29	1.27	1.13	2.42	2.01	1.17	1.98	5.05	0.57	5.54	2.40	1.46
As	0.75	0.12	0.20	0.10	0.08	0.14	1.75	0.29	0.12	0.05	0.33	0.12	0.07	0.13	0.26	0.05	1.75	0.30	0.44
Se	b.d.	b.d.	b.d.	b.d.	b.d.	b.d.	b.d.	b.d.	b.d.	b.d.	b.d.	b.d.	b.d.	b.d.	b.d.	b.d.	b.d.	b.d.	b.d.
Rb	8.46	8.46	7.34	8.24	7.16	8.12	7.76	6.97	7.97	6.06	7.30	7.78	6.38	7.85	7.69	6.06	8.46	7.57	0.71
Sr	45.62	43.82	44.05	42.96	40.85	43.48	45.24	44.78	43.15	43.32	46.25	42.02	43.03	39.20	43.64	39.20	46.25	43.43	1.80
Zr	86.82	77.04	82.44	79.75	79.91	66.67	80.25	69.69	65.85	56.70	65.38	65.06	62.27	73.29	79.40	56.70	86.82	72.70	8.81
Ag	0.08	0.04	0.29	0.09	0.11	0.08	0.04	0.04	0.01	0.36	0.09	0.06	0.06	0.20	0.06	0.01	0.36	0.11	0.10
Cd	b.d.	b.d.	b.d.	b.d.	b.d.	b.d.	b.d.	b.d.	b.d.	b.d.	b.d.	b.d.	b.d.	b.d.	b.d.	b.d.	b.d.	b.d.	b.d.
Sn	4.30	5.18	4.84	5.10	4.93	4.78	5.32	4.15	4.57	3.82	4.26	4.62	3.93	6.56	5.38	3.82	6.56	4.78	0.69
Sb	2.68	0.12	0.14	0.09	0.10	0.09	0.33	0.10	0.08	0.08	0.08	0.08	0.07	0.09	0.13	0.07	2.68	0.28	0.67
Ba	202.13	209.79	193.04	195.09	197.36	191.39	198.08	184.99	185.43	177.28	218.98	209.48	215.28	217.81	215.57	177.28	218.98	200.78	13.22
Hg	0.17	0.14	0.06	0.06	0.04	0.05	0.06	0.07	0.05	0.07	0.08	0.08	0.16	0.00	0.00	0.00	0.17	0.07	0.05
Tl	0.73	0.35	0.42	0.26	0.21	0.31	0.28	0.06	0.10	0.04	0.01	0.11	0.03	0.17	0.11	0.01	0.73	0.21	0.19
Pb	26.79	18.57	17.15	16.26	14.70	23.60	19.39	16.58	14.88	16.68	16.66	27.11	26.51	16.76	15.23	14.70	27.11	19.12	4.52

Major element concentrations are determined by DCP analysis and minor and trace element concentrations are determined by ICPMS

(87.894–90.997 wt%), Al<sub>2</sub>O<sub>3</sub> (6.759–7.741 wt%), Fe<sub>2</sub>O<sub>3</sub> (1.136–1.283 wt%), and K<sub>2</sub>O (0.369–0.606 wt%). Other major elements present but in lesser concentrations include TiO<sub>2</sub> (0.187–0.220 wt%) and P<sub>2</sub>O<sub>5</sub> (b.d.–0.064 wt%). Several elements are present in only one or a few samples, such as CaO (b.d.–0.227 wt%), Na<sub>2</sub>O (b.d.–0.110 wt%), and MgO (b.d.–0.035 wt%). MnO was below the 0.007 wt% detection limit for all samples. Weak linear correlations exist between some major element concentrations. The strongest correlation occurs between concentrations of Fe<sub>2</sub>O<sub>3</sub> and K<sub>2</sub>O with an  $R^2$  value of 0.3257. Of the dominant major elements plotted against SiO<sub>2</sub>, the strongest correlation occurs with K<sub>2</sub>O ( $R^2 = 0.2693$ ), followed by Fe<sub>2</sub>O<sub>3</sub> ( $R^2 = 0.2241$ ), TiO<sub>2</sub> ( $R^2 = 0.0666$ ), P<sub>2</sub>O<sub>5</sub> ( $R^2 = 0.0135$ ), and Al<sub>2</sub>O<sub>3</sub> ( $R^2 = 0.0032$ ).

Trace element concentrations of the silicate mine waste analyzed by ICP-MS resulted in detectable amounts of some USEPA recognized heavy-metal toxic pollutants. These toxic trace heavy-metals include Sb, As, Be, Cr, Cu, Pb, Hg, Ni, Ag, Tl and Zn. Of these metals, only Cd and Se are below the detection limits of the mass spectrometer. Concentrations in parts per million for respective elements include Sb (0.07–2.68), As (0.05–1.75), Be (0.27–0.29), Cr (6.84–8.41), Cu (12.389–37.62), Pb (14.70–27.11), Hg (0.00–0.17), Ni (4.95–14.36), Ag (0.01–0.36), Tl (0.01–0.73), Zn (0.57–5.54), V (52.54–57.36), Rb (6.06–8.46), Sr (39.20–46.25), Zr (56.70–86.82), Sn (3.82–6.56), and Ba (177.28–218.98). No strong correlations of trace elements and major elements are observed.

## Discussion

### Bulk properties

The grain characteristics of the mine waste are dissimilar to most natural sands having a significant proportion of both platy muscovite particles and elongated kyanite particles. A variety of angular quartz textures are also common. Grain size analysis indicates that the material is uniform and moderately well sorted, a property that is often desirable for use in a range of applications. The grain size characteristics are amenable to basic sand applications. The small Cu value of the samples indicates that the material could be used in water filtration processes. Values of <4.0 are often required in constructed wetlands to prevent clogging of pores, a common problem in organic rich systems (e.g., Danish EPA 1999).

The range in hydraulic conductivity values observed may be a function of several factors. Sample heterogeneity may be one factor where variable proportions of platy particles of muscovite cause local preferential pathways to develop. Some possible sources of error related to physical

properties of the experiment include air pockets trapped within the water saturated samples, and heterogeneous zoning of grain sizes upon addition of sample to permeameter. The variable packing of platy muscovite particles and the irregular shaped kyanite and quartz grains in the waste likely contribute variation in hydraulic conductivity values. The Hazen (1911) method more closely approximates hydraulic conductivity values than the Sheperd (1989) method. This is attributed to the mixture of unusual grain shapes that occur in the material.

### Mineralogy

Powder X-ray diffraction indicates a minimal variation in the mineral composition of the waste stream and the minerals observed are those expected from the ore. The waste material is dominated by four minerals: quartz, kyanite, muscovite and hematite. Feldspar minerals are known to occur in the ore and have been observed in the magnetic waste stream yet are conspicuously absent from the silicate-rich waste stream. The silicate-rich waste stream does not have magnetite or pyrite and this attests to the efficiency of the separation process. These minerals occurring in the waste stream are sparingly soluble at earth surface conditions (e.g., Langmuir 1997; Oelkers and Schott 1999) and are environmentally benign.

Muscovite is the only mineral present in the waste with extensive solid solution. Several studies have shown that TiO<sub>2</sub> occurs in muscovite at concentrations similar to those observed in this investigation (e.g., Puziewicz and Koepke 1991; Guidotti et al. 1977). Muscovite is the primary control of TiO<sub>2</sub> content found in the samples and no TiO<sub>2</sub> polymorphs were observed. Muscovite grains also have significant sodium content consistent with solid solution with paragonite and compositions are consistent with metamorphic muscovites. Hematite is found only as intergrowths with muscovite in the waste stream and crystals are uniform in composition with no impurities above detection limit.

### Bulk chemistry

The major elemental compositions of the silicate mine waste samples are reasonably uniform and are consistent with its mineralogical content. Major element compositions are dominated by SiO<sub>2</sub>, Al<sub>2</sub>O<sub>3</sub>, Fe<sub>2</sub>O<sub>3</sub>, and K<sub>2</sub>O and all of these components are accounted for in the minerals observed. The weak correlation of Fe<sub>2</sub>O<sub>3</sub> and K<sub>2</sub>O is attributed to the intergrowths of hematite with some muscovites.

Determining if mine processing concentrates or dilutes heavy-metal content in the mine waste is problematic. One approach is to compare the chemical compositions to that



of the kyanite quartzites in the region which have been reported by Owens and Pasek (2007) and Owens and Dickerson (2001). The range of concentrations of V, Zn, Sn, Ba and Pb are all within the range of concentrations reported in the kyanite schists. Only the concentrations of Ni and Tl exceed the reported ranges by minor amounts. Concentrations of Cr are below the reported ranges. Comparisons of other elements are somewhat more problematic as only a few analyses are reported. Owens and Pasek (2007) analyzed four kyanite schists and found one with As at a concentration of 8 ppm which is much greater than the range of 0.05–1.75 ppm. Similarly, Owens and Pasek (2007) report two analyses with Sb being 3.2 and 3.4 ppm, respectively. Concentrations of Sb in the mine waste vary from 0.07 to 2.68 ppm. Other elements of environmental concern are not reported in Owens and Pasek (2007) or Owens and Dickerson (2001) and thus comparisons cannot be made.

Other elements of specific environmental concern are Cu and Hg. Cu has been recognized to occur as a poorly crystalline Cu sulfate in some regions of the mine (Krekeler et al. 2009). A specific Cu phase was not identified in the waste stream samples. The Cu sulfate phase in the mine is highly soluble and may occur diffusely throughout the waste as a result of the wet separation processing. Mercury pollution is a major environmental concern globally (e.g., Feng et al. 2009; Li et al. 2009; Burton 2009; Patra and Sharma 2000; Fitzgerald et al. 1998; Ratcliffe et al. 1996; Gilbert and Grantwebster 1995; Slemr and Langer 1992). These studies document growing concern over mercury pollution and recently an international agreement has been reached to address these concerns (Burton 2009).

Mercury pollution studies specific to the state of Virginia are not numerous. Kolker et al. (2008) investigated heavy-metal content of atmospheric fine particulate matter (PM<sub>2.5</sub>) in Shenandoah National Park and found anthropogenic enrichment of Hg. Background levels are reported in the single pg/m<sup>3</sup> range. Virginia has an extensive history of gold mining in the region (e.g., Spears and Upchurch 1997; Sweet and Trimble 1983; Sweet 1980) and Hg is a major pollutant associated with gold mining. Some of these mines such as Sprouse's Corner were in relative close proximity to the kyanite mine. Although concentrations of Hg in the mine waste are low, concentrations should be monitored to prevent unexpected release of Hg into the environment. Waste should be evaluated for mercury mobility for each specific recycling application.

Because there is minimal co-variation amongst the elemental concentrations, identifying a particular mineral as a control for a given element of environmental concern is difficult. However, known solid solutions occur in both muscovite and hematite and these phases likely control

most elements of environmental concern. Ba is known to be a common constituent of muscovites (e.g., Graeser et al. 2003; Chabu and Boulegue 1992; Grapes 1993) and functionally has no solid solution with quartz, kyanite or hematite. Some transition metals such as V and Cr commonly substitute in hematite (e.g., Secco et al. 2008; Cabral et al. 2006) and solid solution between hematite (Fe<sub>2</sub>O<sub>3</sub>) and karelianite (V<sub>2</sub>O<sub>3</sub>) and eskolaite (Cr<sub>2</sub>O<sub>3</sub>) are well recognized (e.g., Secco et al. 2008; Koneva 2002). Hematite is interpreted as a carrier of transition metals of environmental concern.

## Recycling

Efforts to recycle mine waste have been described in the literature (e.g., Driussi and Jansz 2006). One body of work focuses on clay-sized or powder materials while other areas focus on very specific aspects of waste. Investigations of clay-sized waste materials include that of Jung et al. (2005) and Yoo et al. (2005) which have investigated coal waste in brick making. Other examples include Brunori et al.'s (2005) investigation of treated bauxite waste and the work of Souza and Mansur (2004) who investigated recycling wastes generated from slate mining and processing. Reprocessing or reining mine waste has also been proposed or investigated (e.g., Marabini et al. 1998; Smith and Williams 1996a, b; Ritcey 2005). Other approaches include investigating iron oxide materials generated as mine waste as sorption material for removal of phosphate from wastewater (Dobbie et al. 2009). For all of these examples both technical and economic requirements must be met for effective recycling to occur. Furthermore, the nature of recycling or reprocessing is case by case dependent on each individual mine. The silicate waste stream is simple and technologically easy to reprocess and use for certain recycling applications.

## Recycling muscovite

Muscovite in the waste stream could be easily separated by density difference methods. Muscovite is a common mineral used in many industrial applications. Over 1,300 US patents alone involve muscovite in some way and approximately 500 US patents involve muscovite as a pigment. Muscovite is a common pearlescent pigment in a variety of products including automobile coatings, cosmetics and plastics (Cavalcante et al. 2007). Specialized or novel pigments such as composites resistant to UV radiation can also be developed using muscovite (Kalenda et al. 2004). The chemical composition of the muscovite is reasonably uniform and this property is conducive for pigment and paint application. Common inclusions of kyanite and hematite and the distinctive TiO<sub>2</sub> content may make the

muscovite an inexpensive label marker for product authentication in certain applications.

Separation of the muscovite from other components of the waste stream may have added advantage in that the process would likely reduce the concentrations of many elements of environmental concern in the left over quartz-rich fraction. Assuming that muscovite composes 1% of the waste and using the lower bound figure of 450,000 metric tons of total waste produced, an estimated minimum of 4,500 metric tons of muscovite could be recovered. According to the United States Geological Survey 2009 minerals yearbook, production of scrap mica varied from approximately 97,000 to 110,000 metric tons. The 2008 market value was \$14 million US with production being 98,700 metric tons. This yields an average estimated price of approximately \$140 per metric ton resulting in an estimated minimum value of approximately \$630,000. Assuming a profit margin of 20% (\$126,000) annually and an initial estimated \$300,000 of infrastructure for a small wet processing facility, cash flow would be adequate to support approximately eight laborers (\$32,000 total annual compensation each), one quality control technician (\$47,000 total annual compensation) and one manager (\$60,000 total annual compensation). If a market could be developed from pigment suppliers recycling of muscovite may be not only an environmental improvement but may also have significant impact on the town of Dillwyn directly.

#### Recycling as constructed wetland media

One of the potential methods of recycling the waste is to use it as media in constructed wetlands. The mine site has rail access and large volumes of the material could be shipped to many areas in Virginia and other states. In the state of Virginia, the law prohibits any net loss of wetland acreage. Among the compensatory mitigation options available for unavoidable wetland impacts are wetland creation and restoration (Virginia. Code. §9VAC25-210-116).

The mine waste has potential for use in constructed wetlands. Arias et al. (2001) suggest that the most important property of a substrate material for use in constructed wetlands is the grain size characteristics. The Danish EPA guidelines for aggregate stipulate that  $d_{10}$  should be in the range of 0.30–2.00 mm and  $d_{60}$  should be in the range of 0.5–8.0 mm. In addition, the Cu value should be <4.0. These characteristics have been found to be effective and minimize the risk of obstruction of pores by organic matter. The grain characteristics of the mine waste generally agree with these parameters and are suitable for use with the most critical property of a low Cu (2.000–2.333) being met. Another benefit is that the mine waste is composed of

minerals of low solubility and elements of concern are largely at or below concentrations of the kyanite quartzites from which the waste is derived. No sulfide minerals or other minerals of environmental concern were observed in the waste. The bulk, mineralogical, and geochemical properties of the waste stream merit further detailed investigations as media for constructed wetlands in mesocosm systems.

The waste is sufficiently large to supply constructed wetlands for decades. Using an average density of 2.2 for the mine waste and a substrate depth of 1.5 m, approximately 33,000 tons of mine waste would supply a 1-ha constructed wetland. Approximately 18 1-ha wetlands can be made annually assuming 600,000 tons of waste are produced. The existing waste reserves would be sufficient to produce approximately 800 ha of wetlands. Crude economic estimations for this market suggest that the material may need to be sold at a nominal price (\$0.50–\$1.00 per ton) to enable it to be recycled. The nature of shipping the materials and the associated transportation costs likely will impact economic feasibility. An additional complication is the constructed wetland market in Virginia is closely tied to residential and commercial development. Although this market is currently not active compared to historical trends, future growth is expected.

Recycling the waste in either or both forms would have economic benefit to the local area. Dillwyn is the largest town in Buckingham County. As of 2000, the population of Dillwyn was 447, and Buckingham County was 15,623. As of 2007, 19.7% of individuals in Buckingham County lived below poverty level compared to the national average of 9.9% (US Census Bureau, 2000a, b). An increase of labor positions and a few technical positions would benefit the economy greatly.

#### Conclusions

The mineralogy of the silicate waste stream is generally uniform and reasonably environmentally benign having no sulfides or other minerals of environmental concern detected in the sample set. With the minor exception of Ni, elements of environmental concern occur at concentrations below that of the kyanite quartzites from which they are derived and many trace elements likely occur in grains of hematite–muscovite intergrowths which can be separated for pigment or industrial mineral recycling. The resulting modified waste stream is likely very suitable for use in constructed wetlands and testing in mesocosms should be pursued. Recycling the waste has the potential to have a positive environmental and economic impact on a community in need.

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