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Alkali roasting method for purifying graphite concentrate

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Abstract

The graphite purification tests were conducted by GTK Mintec on the flotation concentrate produced by the University of Oulu from a graphite sample extracted in Kaukkala, Pälkäne. The test work included sample preparation and purification by alkaline roasting and acid leaching in different process conditions. The carbon grade in the bulk feed material was 79%. The carbon grades were higher in the coarser size fractions, e.g., 83.8% in +75 µm fraction, 81.4% in 20–75 µm fraction and only 61.3% in -20 µm fraction.

Totally seven purification tests were performed for three different size fractions. The carbon grades in the end products were 96.1–99.9%. Based on the results, lower solids content in alkaline roasting together with longer leaching time improved the quality of the concentrates. Also, the carbon grades could be upgraded by repeating the purification process. Further testing would be required to optimize the process conditions. In addition, it would be beneficial to do the tests for the unsieved bulk sample, too.

Keywords
Graphite, purification

Geographical area
Finland

Other information
The work was part of the BATCircle2.0 Project, funded by Business Finland

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1 INTRODUCTION

The graphite purification tests were conducted by GTK Mintec on the flotation concentrate produced by the University of Oulu from a graphite sample extracted in Kaukkala, Pälkäne. The test work included sample preparation and purification by alkaline roasting and acid leaching in different process conditions. The objective of this test work was to ascertain the attainable purity levels of graphite concentrates, without optimizing process conditions. This work is part of Task 3.5.1 "Hydrometallurgical approach for graphite purification" in the BATCircle2.0 Project, funded by Business Finland.

2 SAMPLES

About 10 kg of graphite flotation concentrates were received from the University of Oulu. The samples were homogenized at first by mixing and then the material was divided for smaller batches for the test work. Also, small sub samples were divided for the chemical XRF and Leco C and S analyses (Table 1) and for the particle size distribution analyses which was determined by the wet and dry sieving (Rotap). The carbon grade in the graphite concentrate was 79.4% and the main impurities were SiO_2 8.1%, Fe 3.9%, Al_2O_3 2.4% and sulphur 2.1%. The detailed feed analyses are shown in Appendix 1. Based on the sieve analyses, the fineness of graphite concentrate was D80~100 µm.

Table 1. Graphite flotation concentrate, chemical feed analyses.

Sample	C (Leco) %	S (Leco) %	SiO2 (XRF) %	Al2O3 (XRF) %	MgO (XRF) %	Fe (XRF) %
Graphite concentrate	79.40	2.10	8.10	2.37	0.84	3.88

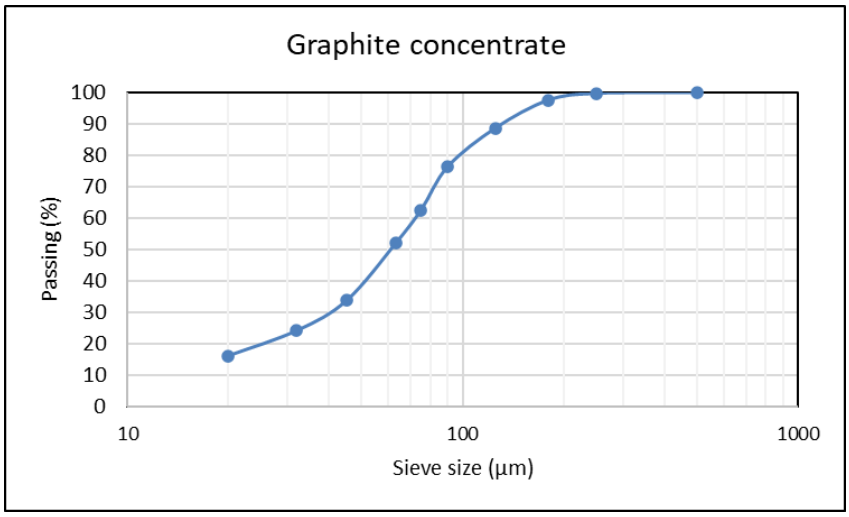

Figure 1. Graphite flotation concentrate, particle size distribution.

Later, about 1 kg sample was sieved for three size fractions for the purification tests. The carbon grades were higher, 81.4–83.8%, in the coarse +75 µm and 20–75 µm fractions in comparison to -20 µm fraction where it was only 61.3%. The fine -20 µm fraction contained much more impurities, e.g., SiO_2 content was 12.6% and Fe 8.8%. In the coarser fractions, SiO_2 and Fe contents were lower, about 6% and 2–3%, respectively.

Table 2. Graphite flotation concentrate, chemical analyses by size fractions.

Size	Mass		C (Leco)		S (Leco)		SiO2 (XRF)		Al2O3 (XRF)		Fe (XRF)	
fraction	g	%	%	rec. %	%	rec. %	%	rec. %	%	rec. %	%	rec. %
+75 µm	374.1	38.7	83.8	41.0	0.90	19.5	5.64	30.6	1.93	34.2	1.96	21.9
20-75 µm	434.1	44.9	81.4	46.3	1.50	37.7	6.44	40.5	2.01	41.3	2.83	36.6
-20 µm	158.2	16.4	61.3	12.7	4.68	42.8	12.60	28.9	3.28	24.6	8.81	41.5
Calculated feed	966.4	100.0	79.0	100.0	1.79	100.0	7.14	100.0	2.19	100.0	3.47	100.0
Unsieved feed			79.4		2.10		8.10		2.37		3.88	

3 GRAPHITE PURIFICATION TESTS

The purification tests were based on Lu & Forssberg (2002). The graphite purification method included three steps: an alkaline roasting, a water wash and a sulphuric acid leaching. In total, seven purification tests were done for the sieved graphite concentrate (three size fractions). Table 3 details each test's parameters. The main variables were the liquid solid ratio in alkaline roasting and leaching time and mixing speed in acid leaching. Otherwise, the test conditions were somewhat similar.

Table 3. Feed samples and the test parameters applied in alkaline roasting and acid leaching.

Test	Feed	Alkaline roasting				Acid leaching			
		Base	L/S	T (C°)	Time (h)	Acid	L/S	rpm	Time (h)
1	+ 75 µm	NaOH 25 %	2:1	250	2	H2SO4 10 %	5:1	300	2
2	20-75 µm	NaOH 25 %	2:1	250	2	H2SO4 10 %	5:1	300	2
1.1	Test 1 residue	NaOH 25 %	2:1	250	2	H2SO4 10 %	5:1	300	2
2.1	Test 2 residue	NaOH 25 %	2:1	250	2	H2SO4 10 %	5:1	300	2
3	+ 75 µm	NaOH 25 %	5:1	250	2	H2SO4 10 %	5:1	320	3
4	20-75 µm	NaOH 25 %	5:1	250	2	H2SO4 10 %	5:1	400	3
5	-20 µm	NaOH 25 %	5:1	250	2	H2SO4 10 %	5:1	600	3

Alkaline roasting was carried out with 25% sodium hydroxide at 250°C, roasting time 2 h and solid liquid ratio 2:1 or 5:1. After roasting, the graphite was washed with deionized water to dilute soluble impurities and extra alkali.

Figure 2. Graphite concentrate placed in the crucible and mixed with sodium hydroxide.

Figure 3. Graphite concentrate after alkaline roasting at 250°C.

Figure 4. Water wash and filtration of alkaline roasted graphite concentrate.

The last stage involved acidic leaching with 10% sulfuric acid at room temperature to remove acid-soluble impurities. The used leaching time was 2 or 3 hours and the liquid solid ratio was 5:1. Finally, the graphite was washed and neutralized with ionized water and then dried and weighted. The mixing speed used varied between 300 and 600 rpm, depending on the test.

Figure 5. On the left, acid leaching with 10% H_2SO_4, material mixed with the magnetic stirrer. In the middle, filtration of graphite concentrate and on the right leaching residual.

4 TEST RESULTS

The main results from graphite purification tests are shown in Tables 4 and 5. For the coarse +75 µm fraction, the highest carbon grade 98.6% was achieved when Test 1 residual was treated again under the same process conditions. For the middle fraction 20–75 µm, the purest end product was produced in Test 4, where the carbon grade was 99.9%. The lower solids content in alkaline roasting together with one hour longer leaching time in Tests 3 and 4 improved the quality of the concentrates about 0.8–3.7%-units higher in comparison to Tests 1 and 2 (Figure 6).

Two coarser fractions had the higher C feed grades and also presented the higher C grade in the residue. Although, the C upgrade was the highest, 34.8%-units for the finest fraction, which was the most impure one with the feed grade of 61.3 %.

Table 4. Feed samples, test parameters and the carbon grades in the end products.

Test	Feed	Alkaline roasting				Acid leaching			Feed	Residue	Upgrade
		Base	L/S	T (C°)	Time (h)	Acid	L/S	Time (h)	C %	C %	%
1	+ 75 µm	NaOH 25 %	2:1	250	2	H2SO4 10 %	5:1	2	83.8	96.8	13.0
2	20-75 µm	NaOH 25 %	2:1	250	2	H2SO4 10 %	5:1	2	81.4	96.2	14.8
1.1	Test 1 residue	NaOH 25 %	2:1	250	2	H2SO4 10 %	5:1	2	96.8	98.6	1.8
2.1	Test 2 residue	NaOH 25 %	2:1	250	2	H2SO4 10 %	5:1	2	96.2	99.8	3.6
3	+ 75 µm	NaOH 25 %	5:1	250	2	H2SO4 10 %	5:1	3	83.8	97.6	13.8
4	20-75 µm	NaOH 25 %	5:1	250	2	H2SO4 10 %	5:1	3	81.4	99.9	18.5
5	-20 µm	NaOH 25 %	5:1	250	2	H2SO4 10 %	5:1	3	61.3	96.1	34.8

The removal of impurities is detailed in Table 5. Most of the remaining impurities in the purified graphite concentrates were SiO_2 (about 0.01–1.9%) and Al_2O_3 (0–0.5%). The Fe content was about 0.03–0.4%, and S content was 0.02–0.5%.

Table 5. SiO_2, Al_2O_3, S and Fe removal after purification tests.

Test	Feed SiO2 %	Residue SiO2 %	Feed Al2O3 %	Residue Al2O3 %	Feed S %	Residue S %	Feed Fe %	Residue Fe %
1	5.64	1.74	1.93	0.51	0.90	< 0.02	1.96	0.17
2	6.44	1.92	2.01	0.43	1.50	< 0.02	2.83	0.13
1.1	1.74	0.82	0.51	0.23	< 0.02	0.08	0.17	0.09
2.1	1.92	0.53	0.43	0.12	< 0.02	< 0.02	0.13	0.03
3	5.64	1.14	1.93	0.41	0.902	0.06	1.96	0.13
4	6.44	0.005	2.01	0.001	1.5	0.12	2.83	-
5	12.6	1.596	3.28	0.414	4.68	0.51	8.81	0.399

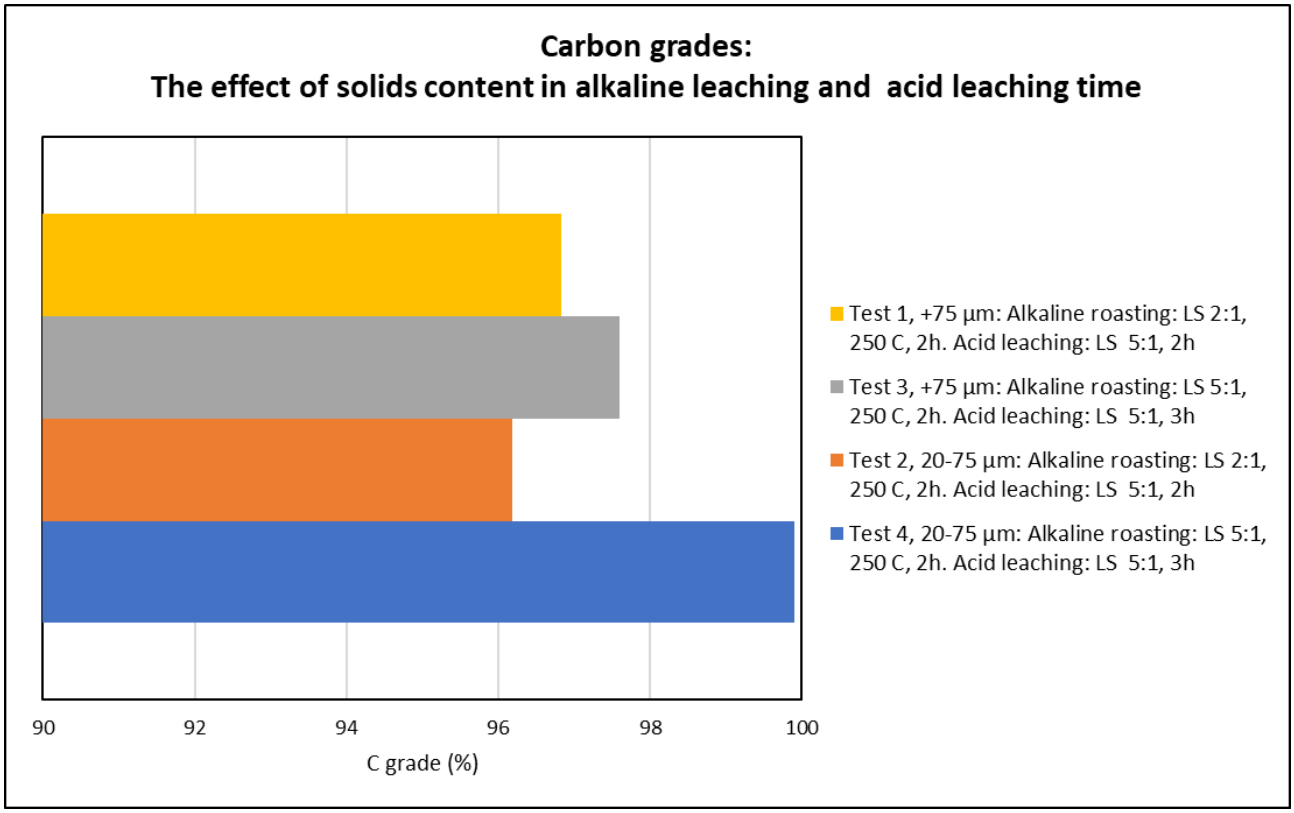

Figure 6. Carbon grades: the effect of solids content in alkaline leaching and acid leaching time.*
**It should be noted that slightly different mixing speed might have some effect too.*

5 CONCLUSIONS

The graphite purification tests were performed for the flotation concentrate produced by the University of Oulu. The carbon grade in the bulk feed material was 79 %. The carbon grades were higher in the coarser size fractions, e.g., 83.8% in +75 µm fraction, 81.4% in 20–75 µm fraction and only 61.3% in -20 µm fraction.

The graphite purification method included three steps: an alkaline roasting, a water wash and a sulphuric acid leaching. Totally seven purification tests were done for the sieved graphite flotation concentrate (three size fractions). The main variables were the liquid solid ratio in alkaline roasting and leaching time and mixing speed in acid leaching.

The coarser fractions had the higher C feed grades and also presented the higher C grade in the residue. Although, the C upgrade was the highest, 34.8%-unit for the finest fraction, which was the most impure one. The carbon grades in the end products varied between 96.1 and 99.9%. Based on the results, lower solids content in alkaline roasting together with longer leaching time improved the quality of the concentrates. Also, the carbon grades could be upgraded by repeating the purification process. Anyhow, further testing would be needed to optimize the process conditions. It would also be beneficial to do the tests for the unsieved bulk sample.

REFERENCE

Lu, X. & Forssberg, E. 2002. Preparation of high-purity and low-sulphur graphite from Woxna fine graphite concentrate by alkali roasting. Minerals Engineering - MINER ENG. 15. 755−757. Available at: https://doi.org/10.1016/S0892-6875(02)00172-3

Appendix 1

Chemical feed analyses

CRS Laboratories

REPORT OF XRF-ANALYSES

Customer	: Tero Korhonen / GTK Mintec
Order	: ID92793
Method	: 180X-O
Date	: 22.2.2022
Comment	: BF Batcircle2.0 M 3.1.3-3.1.4 Gra. Pilot / Grafiittirikaste

Contents (%)	
	Graphite concentrate
	92793-1
SiO2	8.10
TiO2	0.132
Al2O3	2.37
Cr2O3	0.018
V2O3	0.033
FeO	4.99
MnO	0.032
MgO	0.84
CaO	0.486
Rb2O	0.0000
SrO	0.0040
BaO	0.007
Na2O	0.45
K2O	0.54
ZrO2	0.008
P2O5	0.021
CO2	291.2
OxSumm	99.30
Cu	0.097
Ni	0.020
Co	0.000
Zn	0.129
Pb	0.017
Ag	0.001
S	2.89
As	0.000
Sb	0.003
Bi	0.003
Te	0.000
Y	0.0009
Nb	0.0017
Mo	0.034
Sn	0.001
W	0.001
Cl	0.070
Th	0.0000
U	0.0000
Cs	0.002
La	0.002
Ce	0.002
Ta	0.001
LOI	0.0000
Ga	0.0009
Si	3.78
Ti	0.079
Cr	0.013
V	0.023
Fe	3.88
Mn	0.025
Mg	0.51
Ca	0.347
Ba	0.007
Leco C	**79.4**
Leco S	**2.10**

CRS Laboratories

REPORT OF XRF-ANALYSES

Customer : GTK Tero Korhonen
Order : ID 103772
Method : 180X-0
Date : 05.08.2022
Comment : BF BatCircle 2.0 M 3.1.3-3.1.4 Gra pilot

Contents (%)

	+75µm	20-75µm	-20µm
	103772-1	103772-2	103772-3
SiO2	5.64	6.44	12.6
TiO2	0.116	0.097	0.172
Al2O3	1.93	2.01	3.28
Cr2O3	0.013	0.013	0.037
V2O3	0.036	0.026	0.026
FeO	2.52	3.64	11.3
MnO	0.020	0.015	0.017
MgO	0.88	0.65	0.81
CaO	0.192	0.263	0.52
Rb2O	0.0000	0.0000	0.0000
SrO	0.0024	0.0032	0.0038
BaO	0.006	0.007	0.014
Na2O	0.08	0.12	0.25
K2O	0.52	0.417	0.53
ZrO2	0.007	0.006	0.009
P2O5	0.016	0.016	0.046
CO2	307.3	298.5	224.8
OxSumm	96.50	96.20	94.40
Cu	0.029	0.047	0.336
Ni	0.006	0.008	0.028
Co	0.001	0.002	0.002
Zn	0.025	0.048	0.263
Pb	0.007	0.008	0.009
Ag	0.001	0.001	0.003
S	1.12	1.68	4.95
As	0.000	0.000	0.000
Sb	0.004	0.005	0.010
Bi	0.002	0.002	0.002
Te	0.000	0.000	0.001
Y	0.0009	0.0012	0.0023
Nb	0.0018	0.0020	0.0013
Mo	0.016	0.033	0.068
Sn	0.002	0.003	0.003
W	0.001	0.001	0.000
Cl	0.005	0.004	0.008
Th	0.0015	0.0000	0.0010
U	0.0000	0.0000	0.0000
Cs	0.001	0.001	0.001
La	0.002	0.002	0.004
Ce	0.001	0.002	0.004
Ta	0.001	0.000	0.000
LOI	0.0000	0.0000	0.0000
Ga	0.0005	0.0003	0.0007
Si	2.64	3.01	5.89
Ti	0.070	0.058	0.103
Cr	0.0087	0.0088	0.025
V	0.024	0.018	0.018
Fe	1.96	2.83	8.81
Mn	0.016	0.012	0.014
Mg	0.53	0.39	0.49
Ca	0.137	0.188	0.370
Ba	0.006	0.006	0.012
Leco C	83.8	81.4	61.3
Leco S	0.902	1.50	4.68

Appendix 2

Graphite purification test reports

GRAPHITE PURIFICATION TEST REPORT							
Sample:	Graphite concentrate +75 µm						
Project:	Batcircle 2.0						
Date:	8.8.2022 5.10.2022						
Author:	MKP						
Test no.:	Test 1						
Alkaline Roasting							
NaOH		25	w-%	pasutettu kahdessa erässä			
Temperature		250	°C				
Time		2	h				
L/S ratio		2:1				yht.	
Sample		74.97	g	80.22	g	155.19	g
NaOH		150.05	g	160.44	g	310.49	g
Water wash							
Deionized hot water		2000	mL				
Time		30	min				
1 step	liete 60C	12.24	pH	Koko näytemäärä pestään kerralla			
2 step	liete 60C	11.68	pH				
3 step	liete 60 C	11.25	pH				
4 step		10.78	pH	lopuksi huuhtelu runsaalla kylmällä vedellä			
Acid Leaching							
H2SO4		10	w-%	103 g H2SO4+897g H2O			
Temperature		25	°C				
Time		2	h	300 rpm			
L/S ratio		5:1					
H2SO4		795	g	755 ml			
	loppusakka	133.12	g	suodos keltainen			

GRAPHITE PURIFICATION TEST REPORT								
Sample:	Graphite concentrate 20-75 µm							
Project:	Batcircle 2.0							
Date:	8.8.2022 5.10.2022							
Author:	MKP							
Test no.:	Test 2							
Alkaline Roasting								
NaOH		25	w-%	pasutettu kolmessa erässä				
Temperature		250	°C					
Time		2	h					
L/S ratio		2:1						yht.
Sample		54.95	g	52.62	g	51.44	g	159.01
NaOH		110	g	105.3	g	102.9	g	318.2
Water wash								
Deionized hot water		2000	mL					
Time		30	min					
1 step	liete 60C	12.02	pH	Koko näytemäärä pestään kerralla				
2 step	liete 60C	11.7	pH					
3 step	liete 60 C	10.98	pH					
4 step		10.59	pH	lopuksi huuhtelu runsaalla kylmällä vedellä				
Acid Leaching								
H2SO4		10	w-%	103 g H2SO4+897g H2O				
Temperature		25	°C					
Time		2	h	300 rpm				
L/S ratio		5:1						
H2SO4		795	g	755 ml				
	loppusakka	130.09	g	suodos keltainen				

GRAPHITE PURIFICATION TEST REPORT							
Sample:	Test 1 residual, repeat of Test 1						
Project:	Batcircle 2.0						
Date:	21.11.2022-25.11.2022						
Author:	MKP						
Test no.:	Test 1.1						
Alkaline Roasting							
NaOH		25	w-%				
Temperature		250	°C				
Time		2	h				
L/S ratio		2:1				yht.	
Sample		55.18	g			55.18	g
NaOH		110.5	g			110.5	g
Water wash							
Deionized hot water		2000	mL				
Time		30	min				
1 step	liete 60C	12.06	pH	Koko näytemäärä pestään kerralla			
2 step	liete 60C	11.68	pH				
3 step	liete 60 C	10.57	pH				
4 step		9.8	pH	lopuksi huuhtelu runsaalla kylmällä vedellä			
Acid Leaching							
H2SO4		10	w-%	103 g H2SO4+897g H2O			
Temperature		25	°C				
Time		2	h	300 rpm			
L/S ratio		5:1					
H2SO4		275.9	g	262 ml			
	loppusakka	52.57	g	suodos keltainen			

GRAPHITE PURIFICATION TEST REPORT							
Sample:	Test 2 residual, repeat of Test 2						
Project:	Batcircle 2.0						
Date:	23.11.2022-25.11.2022						
Author:	MKP						
Test no.:	Test 2.1						
Alkaline Roasting							
NaOH		25	w-%				
Temperature		250	°C				
Time		2	h				
L/S ratio		2:1				yht.	
Sample		55.24	g		g	55.24	g
NaOH		110.5	g		g	110.5	g
Water wash							
Deionized hot water		2000	mL				
Time		30	min				
1 step	liete 60C	12.02	pH	Koko näytemäärä pestään kerralla			
2 step	liete 60C	11.41	pH				
3 step	liete 60 C	10.49	pH				
4 step		10.14	pH	lopuksi huuhtelu runsaalla kylmällä vedellä			
Acid Leaching							
H2SO4		10	w-%	103 g H2SO4+897g H2O			
Temperature		25	°C				
Time		2	h	300 rpm			
L/S ratio		5:1					
H2SO4		276.2	g	263 ml			
	loppusakka	52.36	g	suodos keltainen			

Date:	9.-13.2.2023								
Author:	MKP								
Test no.:	Test 3								
Alkaline Roasting									
NaOH		25	w-%						
Temperature		250	°C						
Time		2	h						
L/S ratio		5:1				yht.			
Sample		42.8	g			42.8	g		
NaOH		214	g			214	g		
Water wash									
Deionized hot water		1500	mL						
Time		30	min						
1 step	liete 60C	12.17	pH						
2 step	liete 60C	11.93	pH						
3 step	liete 60 C	11.22	pH						
4 step		10	pH	lopuksi huuhtelu runsaalla kylmällä vedellä					
Acid Leaching									
H2SO4		10	w-%	103 g H2SO4+897g H2O					
Temperature		25	°C	käytössä harmaa Heidolph magneettisekoitin					
Time		3	h	320 rpm	toivottua 600-800 rpm ei pysty toteuttamaan,				
L/S ratio		5:1		koska magneetti ei pysynyt paikoillaan					
H2SO4		214	g	201,6 ml					
	loppusakka	37.00	g						
	loppuliuos	190.1	g	177 ml					

GRAPHITE PURIFICATION TEST REPORT							
Sample:	Graphite concentrate 20-75 µm						
Project:	Batcircle 2.0, 50404-4021029						
Date:	9.-14.2.2023						
Author:	MKP						
Test no.:	Test 4						
Alkaline Roasting							
NaOH		25	w-%				
Temperature		250	°C				
Time		2	h				
L/S ratio		5:1				yht.	
Sample		40.34	g			40.34	g
NaOH		201.7	g			201.7	g
Water wash							
Deionized hot water		1500	mL				
Time		30	min				
1 step	liete 60C	12.65	pH				
2 step	liete 60C	11.81	pH				
3 step	liete 60 C	10.74	pH				
4 step	20 C	10.87	pH	lopuksi huuhtelu runsaalla kylmällä vedellä			
Acid Leaching							
H2SO4		10	w-%	103 g H2SO4+897g H2O			
Temperature		25	°C				
Time		3	h	400 rpm	toivottua 600-800 rpm ei pysty toteuttamaan, koska magneetti ei pysynyt paikoillaan		
L/S ratio		5:1		käytössä IKA sininen magneettisekoitin			
H2SO4		201.7	g	190 ml			
	loppusakka	32.59	g				
	loppuliuos	184.19	g	170 ml			

GRAPHITE PURIFICATION TEST REPORT								
Sample:	Graphite concentrate -20 µm							
Project:	Batcircle 2.0, 50404-4021029							
Date:	11.4.2023							
Author:	MKP							
Test no.:	Test 5							
Alkaline Roasting								
NaOH		25	w-%					
Temperature		250	°C					
Time		2	h					
L/S ratio		5:1				yht.		
Sample		41.38	g			41.38	g	
NaOH		206.9	g			206.9	g	
Water wash								
Deionized hot water		1500	mL					
Time		30	min					
1 step	liete 60C	12.27	pH					
2 step	liete 60C	11.48	pH					
3 step	liete 60 C	10.71	pH					
4 step	20 C	10	pH	lopuksi huuhtelu runsaalla kylmällä vedellä				
Acid Leaching								
H2SO4		10	w-%	103 g H2SO4+897g H2O				
Temperature		25	°C					
Time		3	h	600 rpm	kapea 400 ml dekka			
L/S ratio		5:1						
H2SO4		206.9	g	192 ml				
	loppusakka	24.95	g					
	loppuliuos	178.7	g	162 ml				

Appendix 3

Graphite purification tests
chemical analyses

CRS Laboratories		
XRF-ANALYSES		
Customer	: GTK Tero Korhonen	
Order	: ID 111771	
Method	: 181X-0	
Date	: 11.10.2022	
Comment	: BatCircle 2.0 50404-4021027	
Contents (%)		
	Residual	Residual
	Test 1	Test 2
	111771-1	111771-2
SiO2	1.74	1.92
TiO2	0.063	0.051
Al2O3	0.51	0.43
Cr2O3	0.0042	0.0025
V2O3	0.016	0.0088
FeO	0.22	0.17
MnO	0.008	0.004
MgO	0.40	0.23
CaO	0.052	0.080
Rb2O	0.0008	0.0004
SrO	0.0000	0.0000
BaO	0.006	0.006
Na2O	0.02	0.03
K2O	0.212	0.116
ZrO2	0.001	0.001
P2O5	0.002	0.002
CO2	355.1	352.7
OxSumm	100.10	99.30
Cu	0.001	0.004
Ni	0.001	0.001
Co	0.000	0.003
Zn	0.002	0.004
Pb	0.006	0.006
Ag	0.000	0.000
S	0.023	0.039
As	0.000	0.000
Sb	0.000	0.000
Bi	0.001	0.001
Te	0.002	0.002
Y	0.0006	0.0005
Nb	0.0010	0.0010
Mo	0.0017	0.0021
Sn	0.000	0.000
W	0.001	0.001
Cl	0.004	0.003
Th	0.0009	0.0009
U	0.0000	0.0000
Cs	0.001	0.001
La	0.001	0.001
Ce	0.001	0.001
Pr	0.000	0.000
Nd	0.00	0.00
Sm	0.00	0.00
Eu	0.00	0.00
Gd	0.00	0.00
Tb	0.00	0.00
Dy	0.00	0.00
Ho	0.00	0.00
Er	0.00	0.00
Tm	0.00	0.00
Yb	0.00	0.00
Lu	0.00	0.00
Hf	0.001	0.001
Ta	0.000	0.000
LOI	0.0000	0.0000
Br	0.0000	0.0000
Leco C	96.83	96.187
Leco S	< 0.02	< 0.02

CRS Laboratories			
XRF-ANALYSES			
Customer	: GTK Tero Korhonen		
Order	: ID 115285		
Method	: 181X-0		
Date	: 16.1.2023		
Comment	: BatCircle 2.0 Test 1 and 2 follow up		
Contents (%)			
	Residual	Residual	
	Test 1.1	Test 2.1	
	115285-1	115285-2	
SiO2	0.82	0.53	
TiO2	0.034	0.026	
Al2O3	0.23	0.12	
Cr2O3	0.0031	0.0013	
V2O3	0.0088	0.0038	
FeO	0.11	0.04	
MnO	0.004	0.001	
MgO	0.21	0.07	
CaO	0.013	0.021	
Rb2O	0.0002	0.0002	
SrO	0.0000	0.0000	
BaO	0.005	0.005	
Na2O	0.01	0.01	
K2O	0.113	0.042	
ZrO2	0.001	0.001	
P2O5	0.001	0.001	
CO2	361.6	365.8	
OxSumm	100.20	100.70	
Cu	0.000	0.000	
Ni	0.001	0.001	
Co	0.001	0.003	
Zn	0.001	0.001	
Pb	0.005	0.005	
Ag	0.000	0.000	
S	0.025	0.017	
As	0.000	0.000	
Sb	0.000	0.000	
Bi	0.001	0.001	
Te	0.002	0.002	
Y	0.0004	0.0002	
Nb	0.0008	0.0009	
Mo	0.0009	0.0011	
Sn	0.000	0.000	
W	0.000	0.001	
Cl	0.003	0.002	
Th	0.0006	0.0008	
U	0.0000	0.0000	
Cs	0.001	0.001	
La	0.001	0.001	
Ce	0.000	0.000	
Pr	0.000	0.000	
Nd	0.00	0.00	
Sm	0.00	0.00	
Eu	0.00	0.00	
Gd	0.00	0.00	
Tb	0.00	0.00	
Dy	0.00	0.00	
Ho	0.00	0.00	
Er	0.00	0.00	
Tm	0.00	0.00	
Yb	0.00	0.00	
Lu	0.00	0.00	
Hf	0.001	0.001	
Ta	0.001	0.001	
LOI	0.0000	0.0000	
Br	0.0000	0.0000	
Leco C	98.61	99.76	
Leco S	0.0802	< 0.02	

CRS Laboratories
Thermo performX XRF results

Batch Batcircle 2.0 Test 3 and 4 residual
Method X_UQ_3600W Oxides
Template
Report date 17.2.2023
Comment GTK Tero Korhonen

Sample Name	Al2O3	BaO	CaO	Cr2O3	Fe2O3	K2O	MgO	MnO	Na2O	P2O5	Rb2O	SiO2	SrO	TiO2	V2O5	ZrO2
	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)
	PP-XRF12	PP-XRF12	PP-XRF12	PP-XRF12	PP-XRF12	PP-XRF12	PP-XRF12	PP-XRF12	PP-XRF12	PP-XRF12	PP-XRF12	PP-XRF12	PP-XRF12	PP-XRF12	PP-XRF12	PP-XRF12
Test 3 121263-1	0.409	0.006	0.034	0.003	0.19	0.191	0.261	0.007	0.029	0.004	0.001	1.138	< 0.001	0.036	0.016	< 0.001
Test 4 121263-2	0.001	< 0.001	< 0.001	< 0.001	< 0.001	< 0.001	< 0.001	< 0.001	< 0.001	< 0.001	< 0.001	0.005	< 0.001	< 0.001	< 0.001	< 0.001

Sample Name	Ag	As	Bi	Ce	Cl	Co	Cs	Cu	Dy	Er	Eu	Ga	Gd	Hf	Ho	La
	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)
	PP-XRF12	PP-XRF12	PP-XRF12	PP-XRF12	PP-XRF12	PP-XRF12	PP-XRF12	PP-XRF12	PP-XRF12	PP-XRF12	PP-XRF12	PP-XRF12	PP-XRF12	PP-XRF12	PP-XRF12	PP-XRF12
Test 3 121263-1	< 0.001	< 0.001	< 0.001	< 0.001	< 0.001	0.003	0.002	0.004	< 0.001	< 0.001	< 0.001	< 0.001	< 0.001	< 0.001	< 0.001	< 0.001
Test 4 121263-2	< 0.001	< 0.001	< 0.001	< 0.001	< 0.001	< 0.001	< 0.001	< 0.001	< 0.001	< 0.001	< 0.001	< 0.001	< 0.001	< 0.001	< 0.001	< 0.001

Sample Name	Lu	Mo	Nb	Nd	Ni	Pb	Pr	S	Sb	Sm	Sn	Ta	Tb	Te	Th	Tm
	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)
	PP-XRF12	PP-XRF12	PP-XRF12	PP-XRF12	PP-XRF12	PP-XRF12	PP-XRF12	PP-XRF12	PP-XRF12	PP-XRF12	PP-XRF12	PP-XRF12	PP-XRF12	PP-XRF12	PP-XRF12	PP-XRF12
Test 3 121263-1	< 0.001	0.008	< 0.001	< 0.001	< 0.001	< 0.001	< 0.001	0.04	< 0.001	< 0.001	< 0.001	< 0.001	< 0.001	< 0.001	< 0.001	< 0.001
Test 4 121263-2	< 0.001	< 0.001	< 0.001	< 0.001	< 0.001	< 0.001	< 0.001	< 0.001	< 0.001	< 0.001	< 0.001	< 0.001	< 0.001	< 0.001	< 0.001	< 0.001

Sample Name	U	W	Y	Yb	Zn	Sum Before Norm.	Fe*	I	C	S
	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)
	PP-XRF12	PP-XRF12	PP-XRF12	PP-XRF12	PP-XRF12			PP-XRF12	CA-C	CA-S
Test 3 121263-1	< 0.001	0.02	< 0.001	< 0.001	0.003	101.158	0.133	0.002	97.6	0.064
Test 4 121263-2	< 0.001	< 0.001	< 0.001	< 0.001	< 0.001	102.538			100.1	0.12

CRS Laboratories
Thermo PerformX XRF results

Batch BatCircle 2.0 Test 5 roasting and H2SO4-leaching
Method X_UQ_3600W Oxides
Report date 15.6.2023
Client GTK / Tero Korhonen
Notes

Lab ID	Client ID	Satmagan	Fe*	C	S	Al2O3	BaO	CaO	Cr2O3	Fe	Fe2O3	K2O	MgO	Mn	MnO	Na2O	P2O5
		(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)
			calc	CA-C	CA-S	PP-XRF12	PP-XRF12	PP-XRF12	PP-XRF12	PP-XRF12	PP-XRF12	PP-XRF12	PP-XRF12	PP-XRF12	PP-XRF12	PP-XRF12	PP-XRF12
126245-1	Test 5 Residual			96.14	0.51	0.414	0.021	0.069	0.007	0.399	0.571	0.129	0.207		0.006	0.04	< 0.001

Lab ID	Client ID	Rb2O	SiO2	SrO	TiO2	V2O5	ZrO2	Ag	As	Bi	Ce	Cl	Co	Cs	Cu	Dy	Er
		(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)
		PP-XRF12	PP-XRF12	PP-XRF12	PP-XRF12	PP-XRF12	PP-XRF12	PP-XRF12	PP-XRF12	PP-XRF12	PP-XRF12	PP-XRF12	PP-XRF12	PP-XRF12	PP-XRF12	PP-XRF12	PP-XRF12
126245-1	Test 5 Residual	< 0.001	1.596	< 0.001	0.052	0.011	0.002	0.002	< 0.001	< 0.001	< 0.001	0.002	0.003	0.005	0.118	< 0.001	< 0.001

Lab ID	Client ID	Eu	Ga	Gd	Hf	Ho	La	Lu	Mo	Nb	Nd	Ni	Pb	Pr	S	Sb	Sm
		(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)
		PP-XRF12	PP-XRF12	PP-XRF12	PP-XRF12	PP-XRF12	PP-XRF12	PP-XRF12	PP-XRF12	PP-XRF12	PP-XRF12	PP-XRF12	PP-XRF12	PP-XRF12	PP-XRF12	PP-XRF12	PP-XRF12
126245-1	Test 5 Residual	< 0.001	< 0.001	< 0.001	< 0.001	< 0.001	< 0.001	< 0.001	0.027	0.002	< 0.001	0.004	0.004	< 0.001	< 0.001	< 0.001	< 0.001

Lab ID	Client ID	Sn	Ta	Tb	Te	Th	Tm	U	W	Y	Yb	Zn	Sum Before Norm.	I
		(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)
		PP-XRF12	PP-XRF12	PP-XRF12	PP-XRF12	PP-XRF12	PP-XRF12	PP-XRF12	PP-XRF12	PP-XRF12	PP-XRF12	PP-XRF12		
126245-1	Test 5 Residual	< 0.001	< 0.001	< 0.001	< 0.001	0.003	< 0.001	< 0.001	0.02	< 0.001	< 0.001	0.04	100.74	0.003