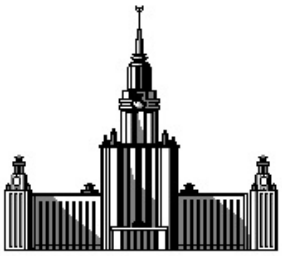
Московский государственный университет имени М.В. Ломоносова
Химический факультет

Кафедра физической химии

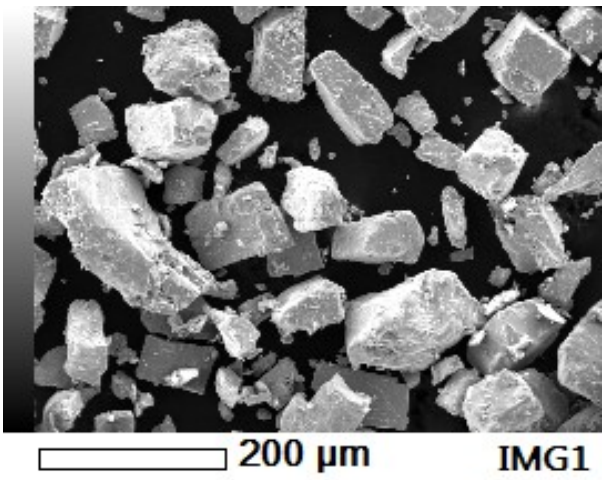


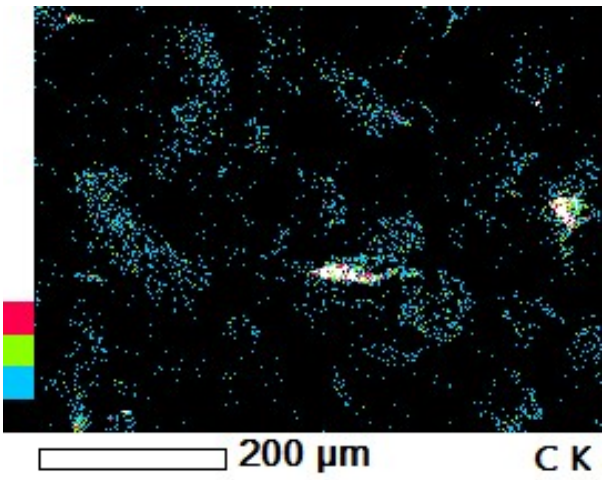
Сканирующая электронная микроскопия в режиме низкого вакуума

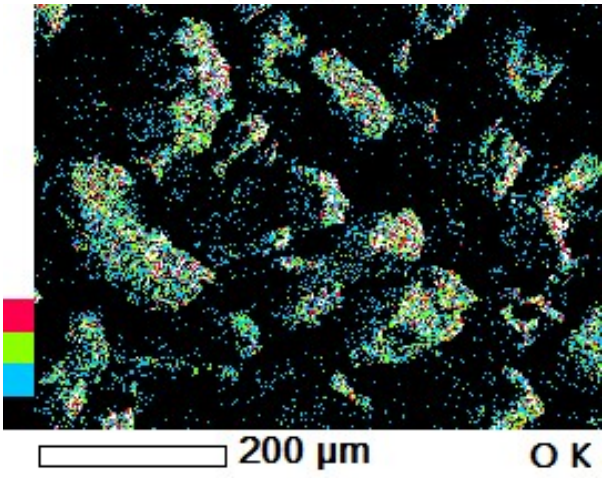
Работа выполнена студентом 515 группы
Финенко А.А.

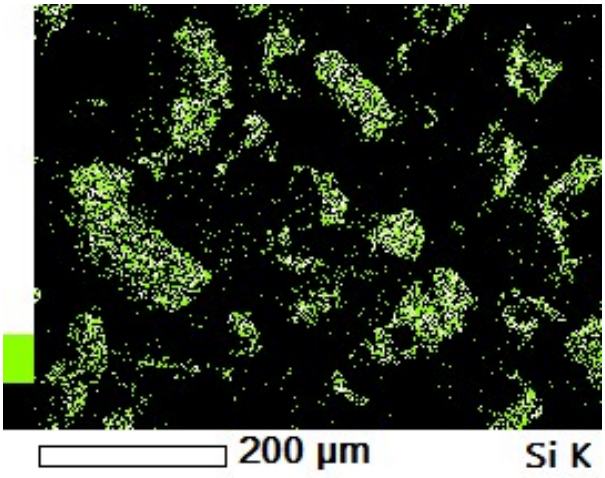
Москва
2017

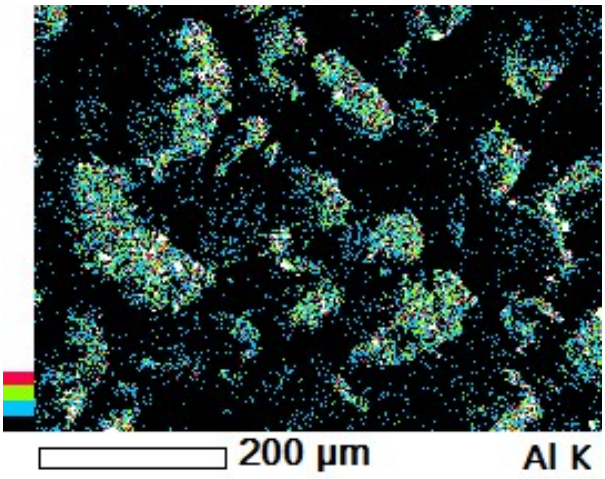
Результаты эксперимента

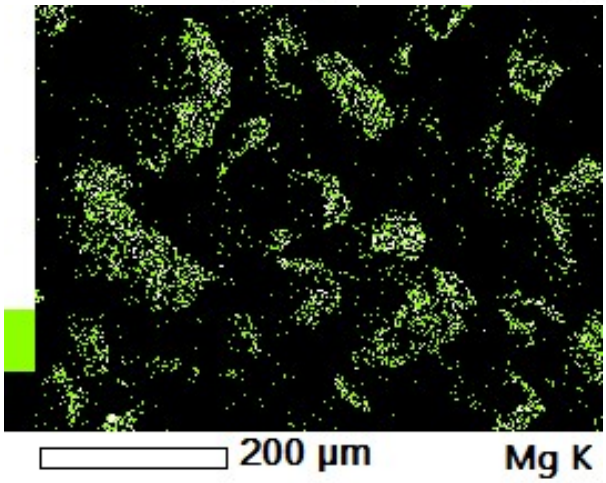


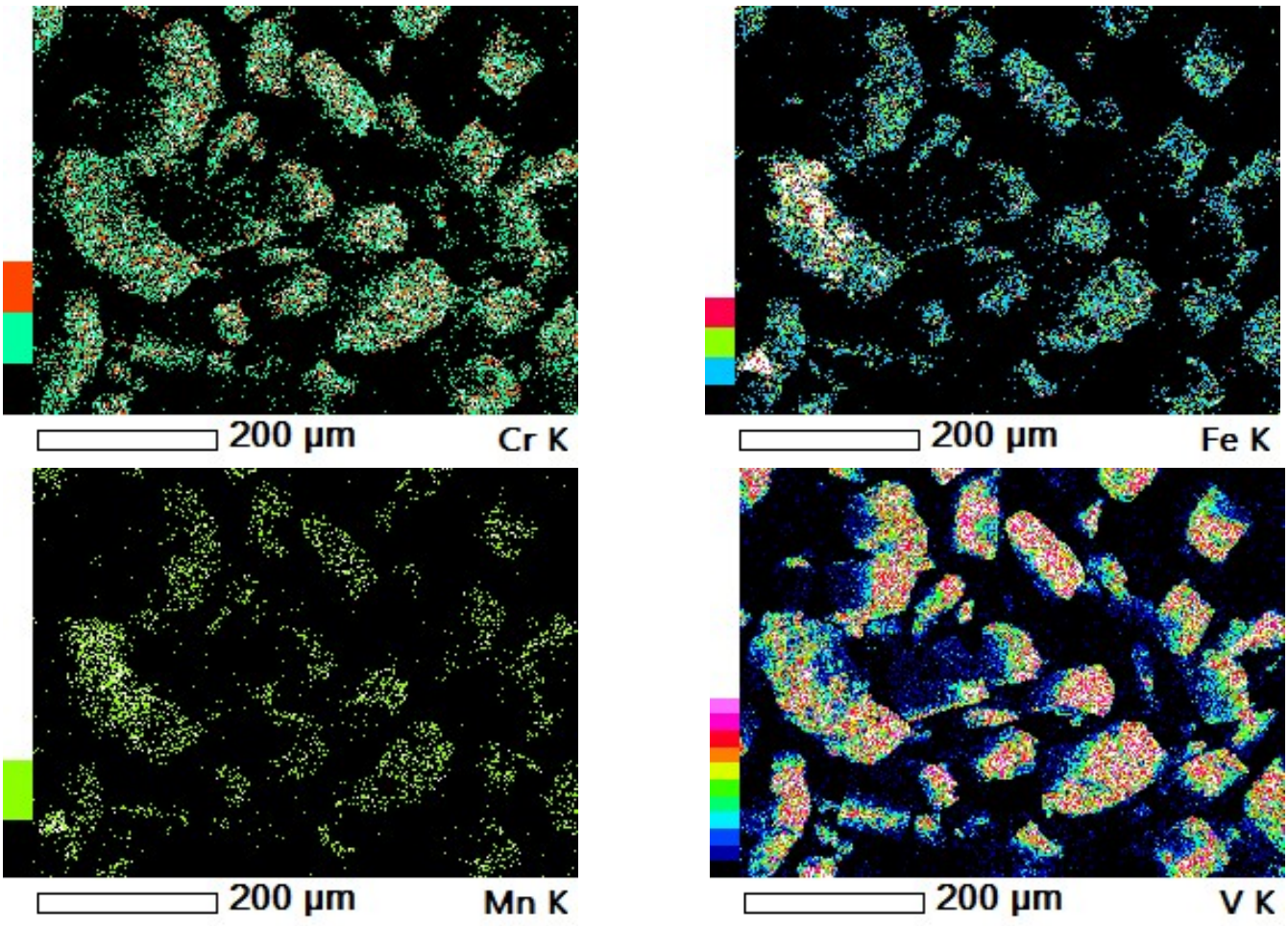






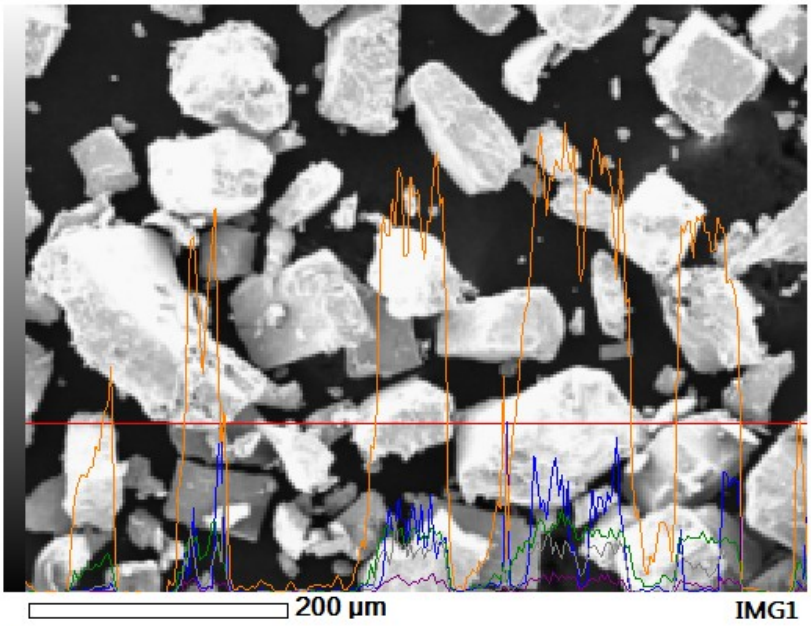


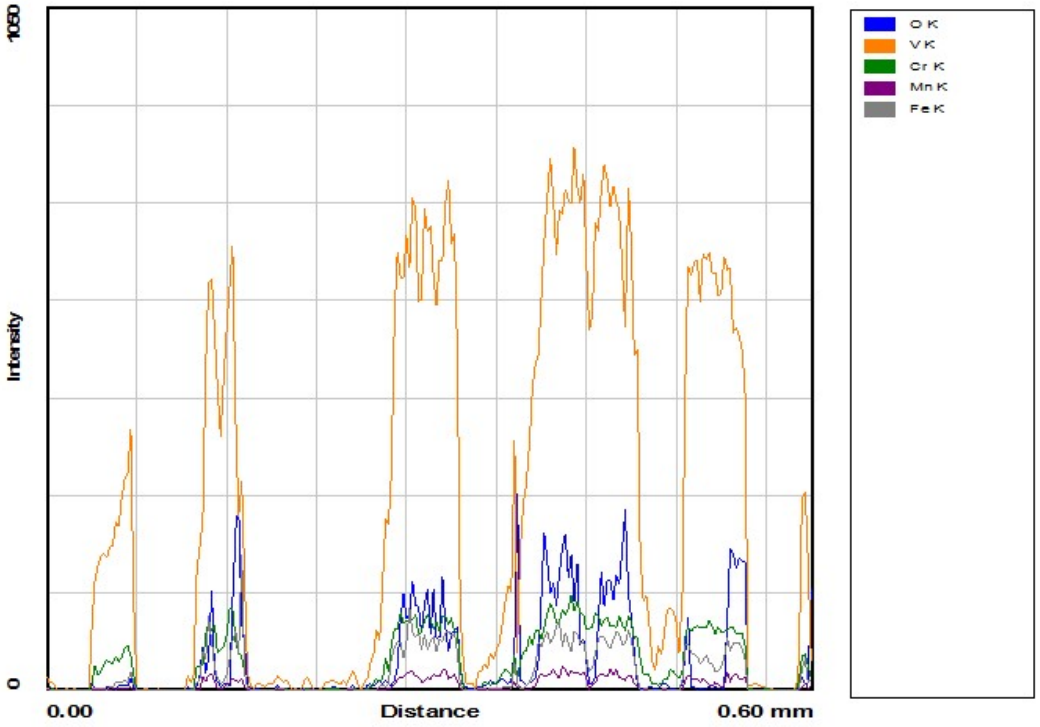




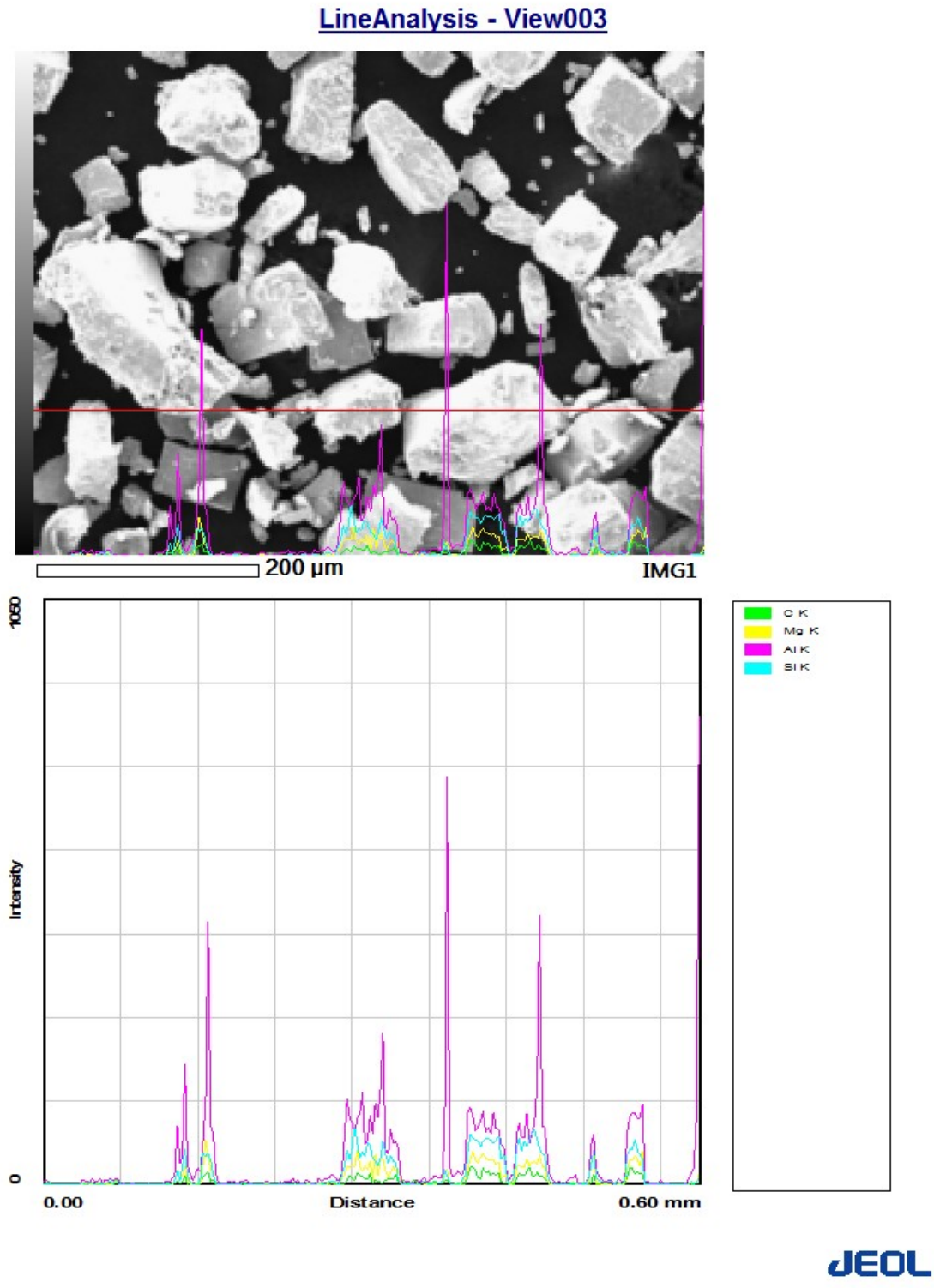
Судя по карте распределений элементов, самым распространенным элементов является ванадий. Ванадий равномерно распределен по частицам. Помимо ванадия в частицах присутствуют магний, крмений, алюминий, хром, железо, магний и кислород. В качестве примесного элемента присутствует углерод.

LineAnalysis - View003





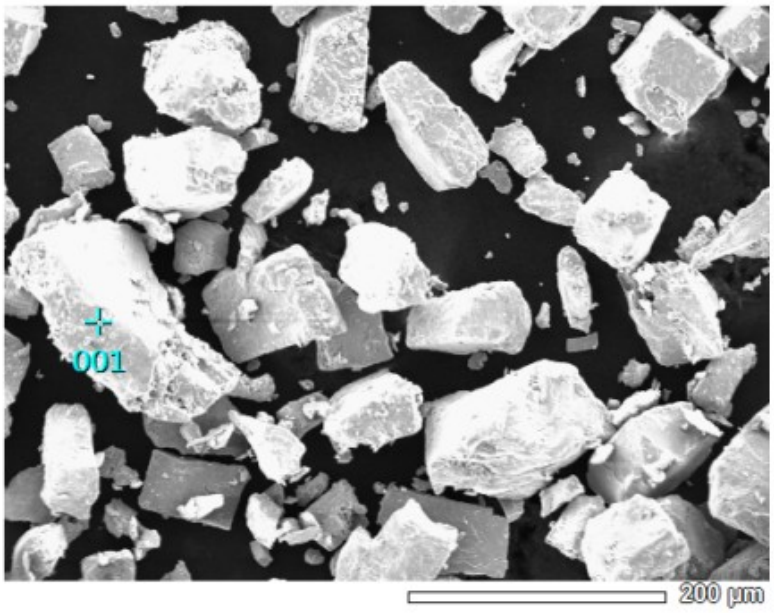
JEOL



Линейное распределение элементов показывает, что основу частиц составляют металлы ванадий, хром, железо и марганец. Сигнал алюминия идет как от частиц, так и от материала подложки.

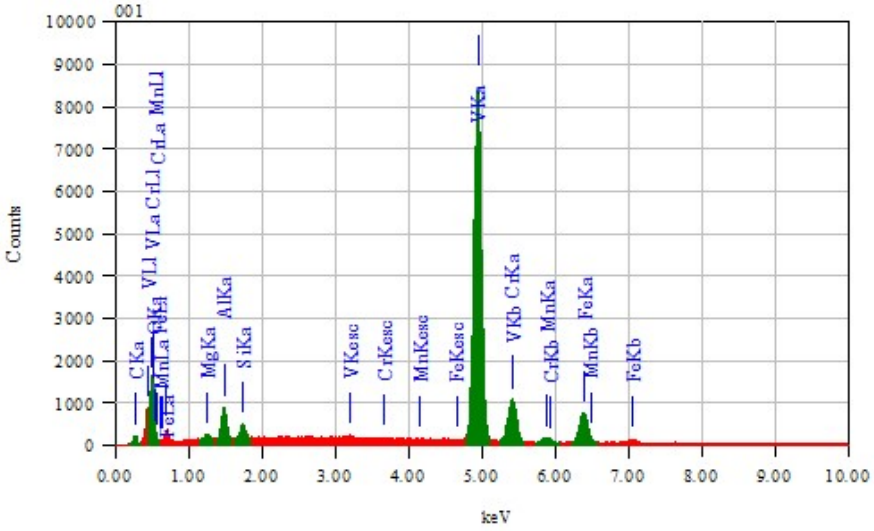
View003

JEOL 1/1



Title : IMG1

Instrument : JCM-6000
Volt : 15.00 kV
Mag. : x 200
Date : 2017/09/25



Acquisition Parameter
Instrument : JCM-6000
Acc. Voltage : 15.0 kV
Probe Current: 1.00000 nA
PHA mode : T3
Real Time : 63.19 sec
Live Time : 60.34 sec
Dead Time : 4 %
Counting Rate: 4408 cps
Energy Range : 0 - 20 ke

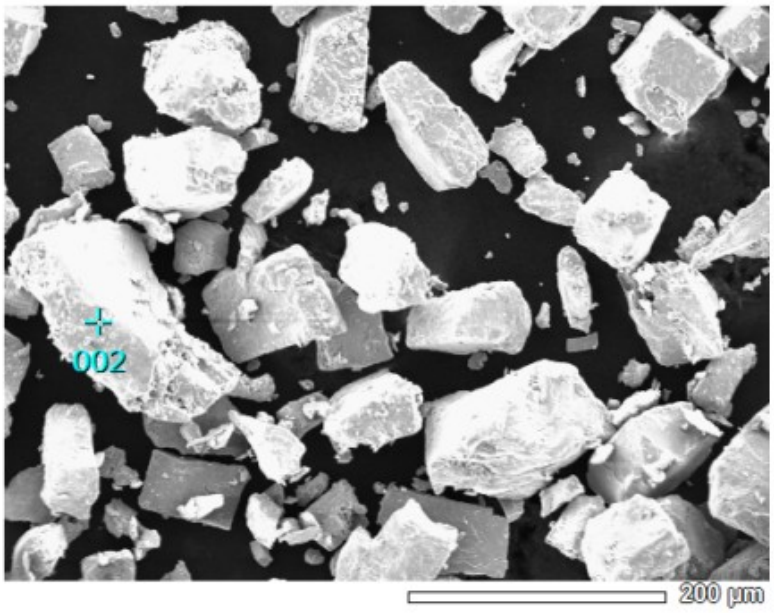
ZAF Method Standardless Quantitative Analysis
Fitting Coefficient : 0.0485

Element	(keV)	Mass%	Sigma	Atom%	Compound	Mass%	Cation	K
C K	0.277	3.99	0.06	14.62				1.0784
O K								
Mg K	1.253	0.55	0.03	0.99				0.1989
Al K	1.486	2.97	0.05	4.84				1.7402
Si K	1.739	1.10	0.03	1.72				0.7000
V K	4.949	75.87	0.26	65.53				81.6971
Cr K	5.411	0.92	0.07	0.78				1.0154
Mn K	5.894	1.57	0.06	1.26				1.3871
Fe K	6.398	13.03	0.14	10.27				12.1828

JED-2300 AnalysisStation JEOL

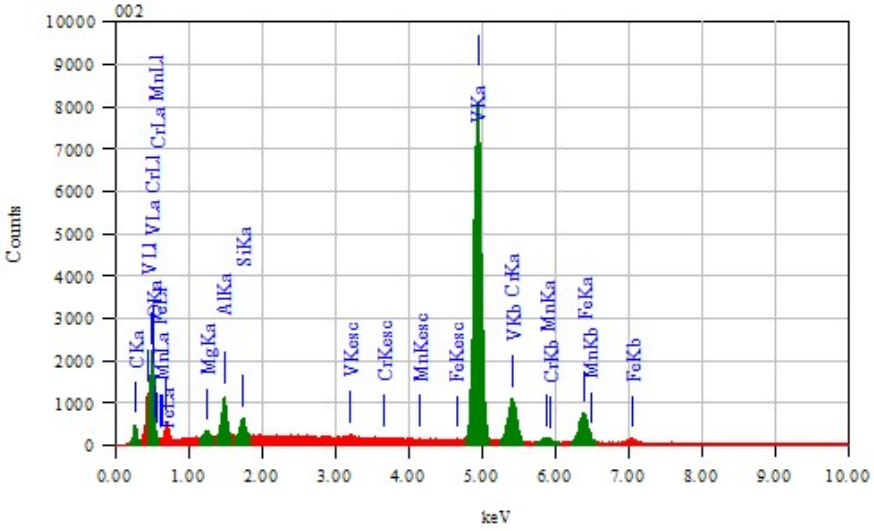
View003

JEOL 1/1



Title : IMG1

Instrument : JCM-6000
Volt : 15.00 kV
Mag. : x 200
Date : 2017/09/25



Acquisition Parameter
Instrument : JCM-6000
Acc. Voltage : 15.0 kV
Probe Current: 1.00000 nA
PHA mode : T3
Real Time : 62.98 sec
Live Time : 60.00 sec
Dead Time : 4 %
Counting Rate: 4719 cps
Energy Range : 0 - 20 ke

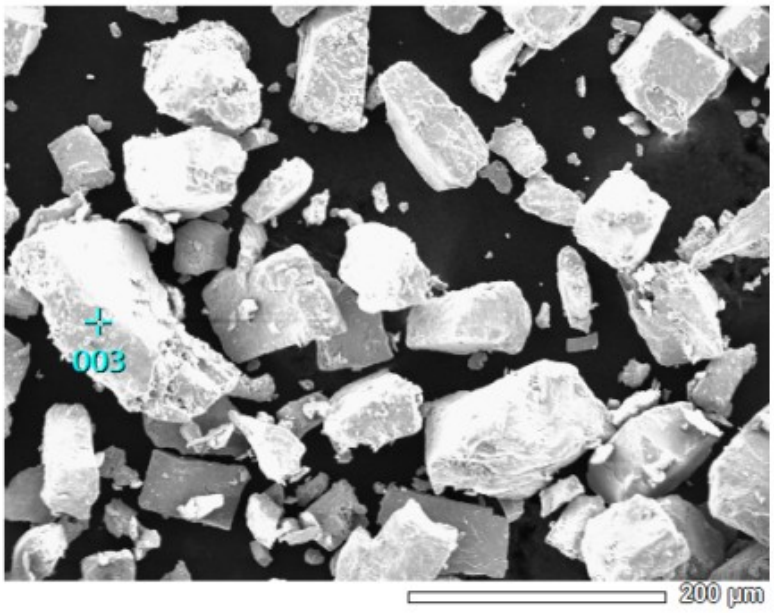
ZAF Method Standardless Quantitative Analysis
Fitting Coefficient : 0.0472

Element	(keV)	Mass%	Sigma	Atom%	Compound	Mass%	Cation	K
C K	0.277	9.02	0.09	28.78				2.5053
O K								
Mg K	1.253	0.62	0.04	0.98				0.2423
Al K	1.486	3.52	0.06	5.00				2.2065
Si K	1.739	1.34	0.03	1.83				0.9018
V K	4.949	71.29	0.25	53.61				80.1439
Cr K	5.411	0.72	0.07	0.53				0.8338
Mn K	5.894	1.47	0.06	1.02				1.3635
Fe K	6.398	12.02	0.13	8.24				11.8029

JED-2300 AnalysisStation JEOL

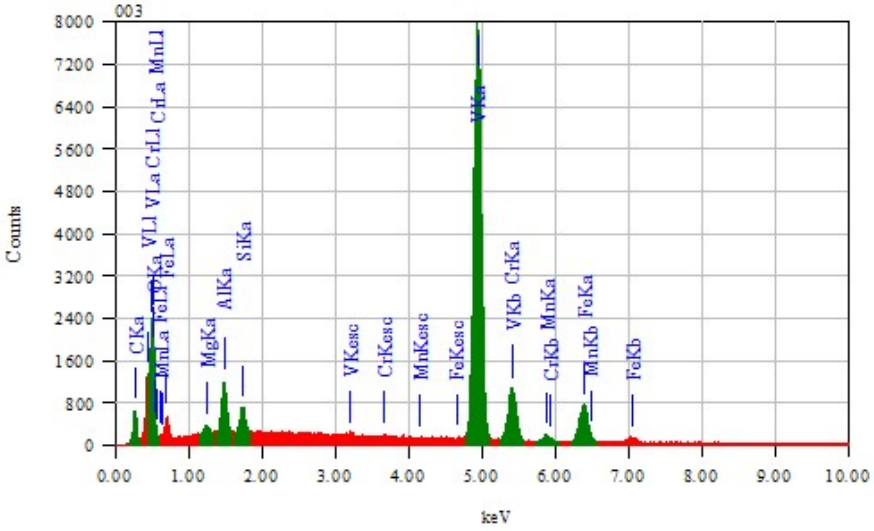
View003

JEOL 1/1



Title : IMG1

Instrument : JCM-6000
Volt : 15.00 kV
Mag. : x 200
Date : 2017/09/25



Acquisition Parameter
Instrument : JCM-6000
Acc. Voltage : 15.0 kV
Probe Current: 1.00000 nA
PHA mode : T3
Real Time : 63.00 sec
Live Time : 60.00 sec
Dead Time : 4 %
Counting Rate: 4817 cps
Energy Range : 0 - 20 ke

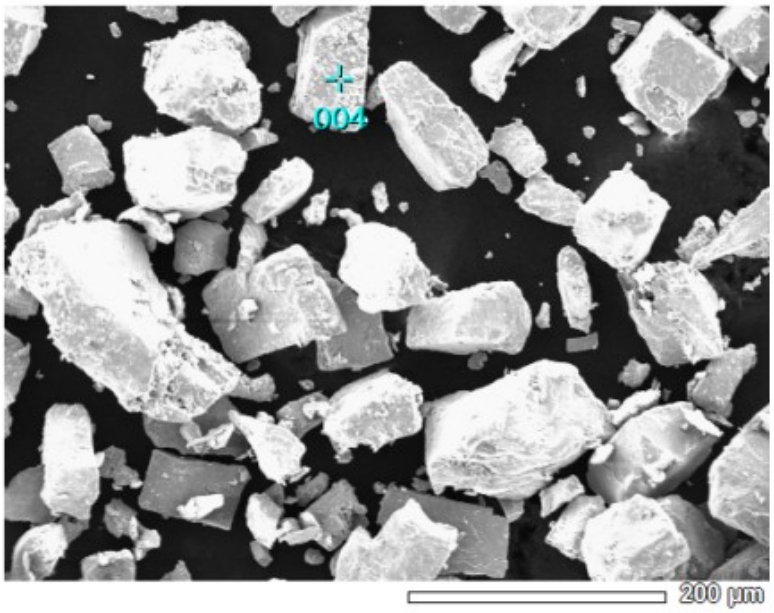
ZAF Method Standardless Quantitative Analysis
Fitting Coefficient : 0.0514

Element	(keV)	Mass%	Sigma	Atom%	Compound	Mass%	Cation	K
C K	0.277	12.09	0.10	35.83				3.4401
O K								
Mg K	1.253	0.65	0.04	0.95				0.2639
Al K	1.486	3.55	0.06	4.69				2.3100
Si K	1.739	1.48	0.04	1.88				1.0338
V K	4.949	68.19	0.24	47.65				78.6815
Cr K	5.411	0.84	0.07	0.58				0.9968
Mn K	5.894	1.46	0.06	0.95				1.3993
Fe K	6.398	11.73	0.13	7.48				11.8746

JED-2300 AnalysisStation JEOL

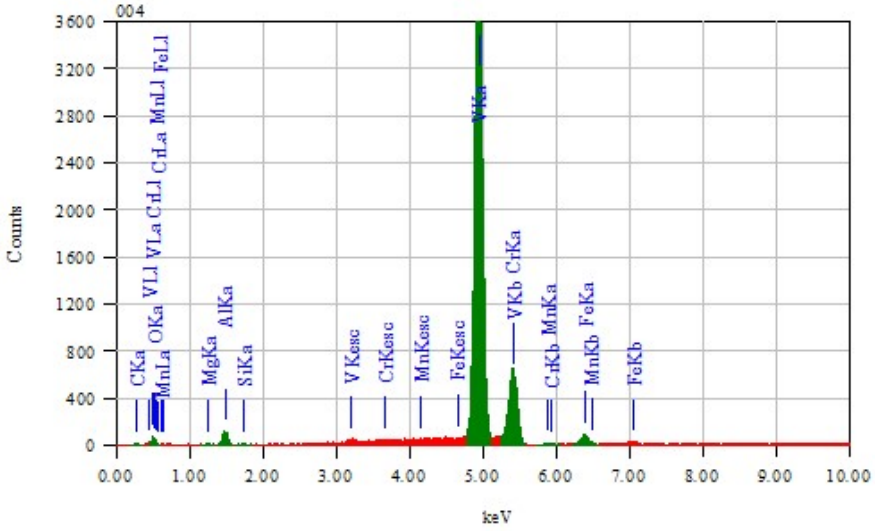
View003

JEOL 1/1



Title : IMG1

Instrument : JCM-6000
Volt : 15.00 kV
Mag. : x 200
Date : 2017/09/25



Acquisition Parameter
Instrument : JCM-6000
Acc. Voltage : 15.0 kV
Probe Current: 1.00000 nA
PHA mode : T3
Real Time : 61.34 sec
Live Time : 60.00 sec
Dead Time : 2 %
Counting Rate: 1508 cps
Energy Range : 0 - 20 ke

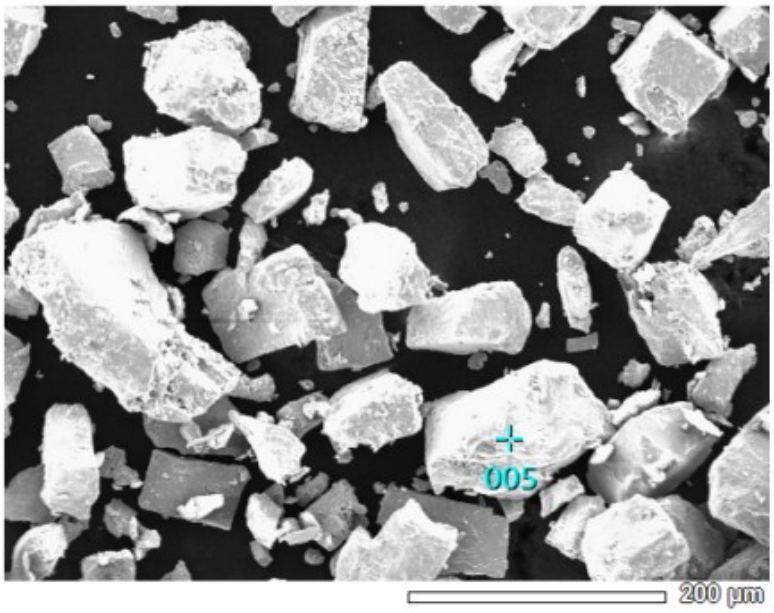
ZAF Method Standardless Quantitative Analysis
Fitting Coefficient : 0.0570

Element	(keV)	Mass%	Sigma	Atom%	Compound	Mass%	Cation	K
C K	0.277	0.41	0.03	1.65				0.1235
O K	0.525	1.66	0.45	4.98				0.2755
Mg K	1.253	0.16	0.03	0.31				0.0552
Al K	1.486	1.13	0.06	2.01				0.6407
Si K	1.739	0.03	0.01	0.04				0.0162
V K	4.949	90.68	0.55	85.73				93.3078
Cr K	5.411	2.31	0.15	2.14				2.4334
Mn K	5.894	0.07	0.05	0.07				0.0609
Fe K	6.398	3.55	0.14	3.06				3.0867

JED-2300 AnalysisStation JEOL

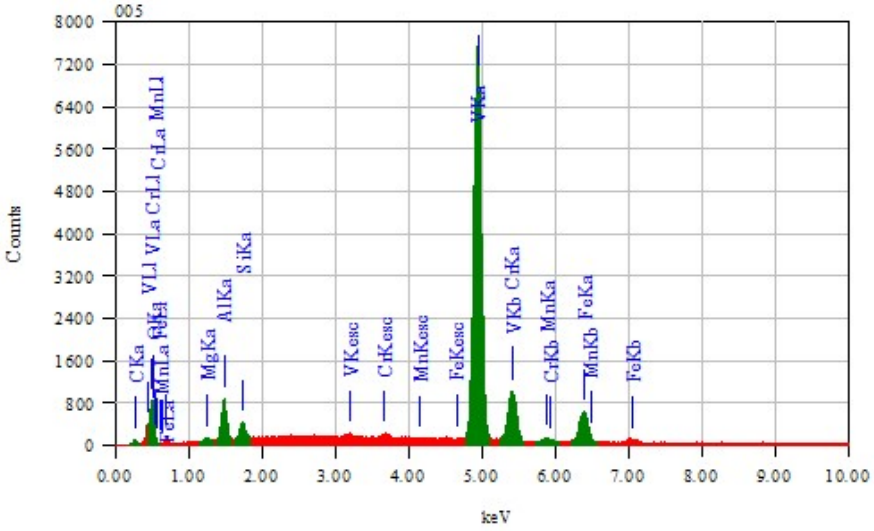
View003

JEOL 1/1



Title : IMG1

Instrument : JCM-6000
Volt : 15.00 kV
Mag. : x 200
Date : 2017/09/25



Acquisition Parameter
Instrument : JCM-6000
Acc. Voltage : 15.0 kV
Probe Current: 1.00000 nA
PHA mode : T3
Real Time : 62.55 sec
Live Time : 60.00 sec
Dead Time : 4 %
Counting Rate: 3764 cps
Energy Range : 0 - 20 ke

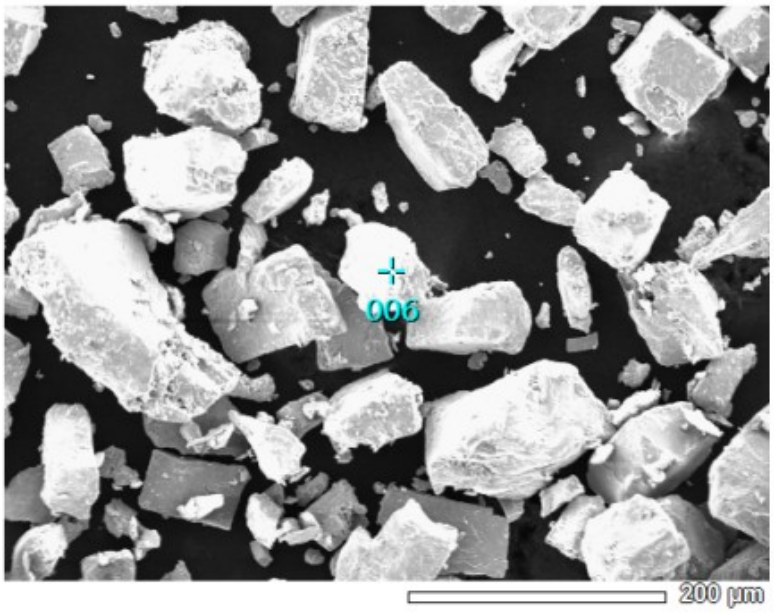
ZAF Method Standardless Quantitative Analysis
Fitting Coefficient : 0.0507

Element	(keV)	Mass%	Sigma	Atom%	Compound	Mass%	Cation	K
C K	0.277	2.41	0.05	9.22				0.6423
O K								
Mg K	1.253	0.32	0.03	0.59				0.1139
Al K	1.486	3.52	0.06	5.99				2.0532
Si K	1.739	1.09	0.03	1.78				0.6835
V K	4.949	78.25	0.29	70.49				83.1278
Cr K	5.411	1.18	0.08	1.05				1.2920
Mn K	5.894	1.19	0.06	0.99				1.0300
Fe K	6.398	12.03	0.15	9.89				11.0571

JED-2300 AnalysisStation JEOL

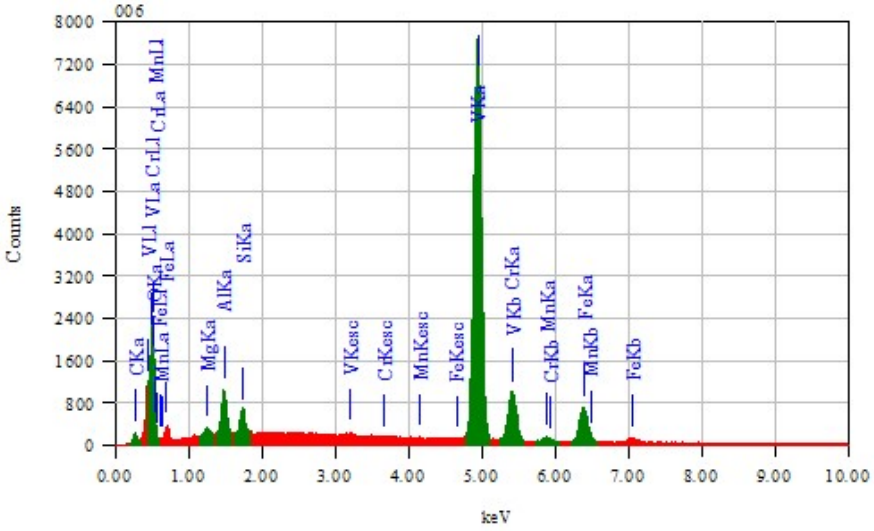
View003

JEOL 1/1



Title : IMG1

Instrument : JCM-6000
Volt : 15.00 kV
Mag. : x 200
Date : 2017/09/25



Acquisition Parameter
Instrument : JCM-6000
Acc. Voltage : 15.0 kV
Probe Current: 1.00000 nA
PHA mode : T3
Real Time : 62.83 sec
Live Time : 60.00 sec
Dead Time : 4 %
Counting Rate: 4512 cps
Energy Range : 0 - 20 ke

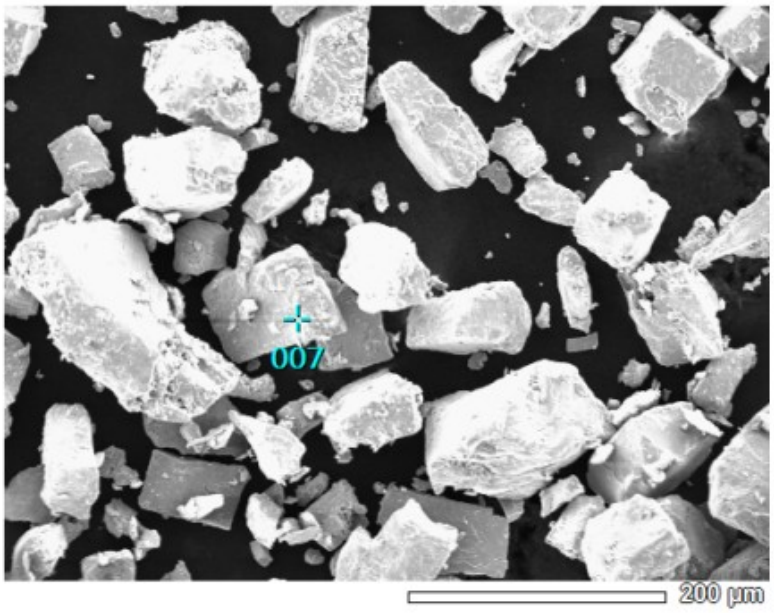
ZAF Method Standardless Quantitative Analysis
Fitting Coefficient : 0.0555

Element	(keV)	Mass%	Sigma	Atom%	Compound	Mass%	Cation	K
C K	0.277	4.10	0.06	14.83				1.0825
O K								
Mg K	1.253	0.72	0.04	1.29				0.2682
Al K	1.486	3.50	0.06	5.64				2.0862
Si K	1.739	1.57	0.04	2.43				1.0044
V K	4.949	75.56	0.27	64.44				81.8145
Cr K	5.411	0.67	0.07	0.56				0.7415
Mn K	5.894	1.35	0.06	1.07				1.1968
Fe K	6.398	12.54	0.15	9.75				11.8059

JED-2300 AnalysisStation JEOL

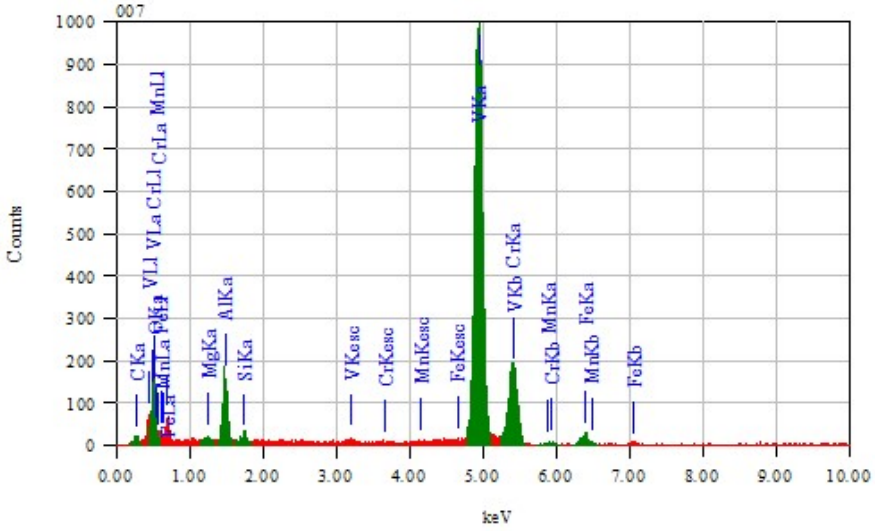
View003

JEOL 1/1



Title : IMG1

Instrument : JCM-6000
Volt : 15.00 kV
Mag. : x 200
Date : 2017/09/25



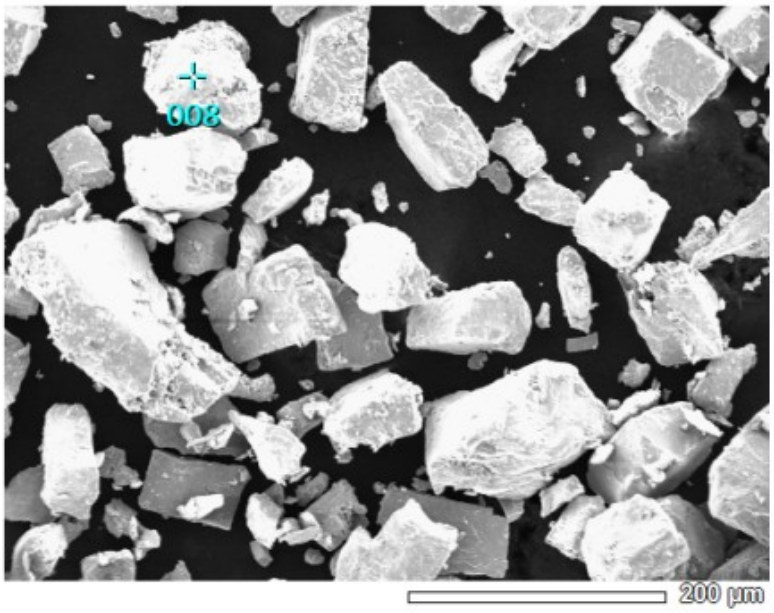
Acquisition Parameter
Instrument : JCM-6000
Acc. Voltage : 15.0 kV
Probe Current: 1.00000 nA
PHA mode : T3
Real Time : 60.82 sec
Live Time : 60.00 sec
Dead Time : 1 %
Counting Rate: 479 cps
Energy Range : 0 - 20 ke

ZAF Method Standardless Quantitative Analysis
Fitting Coefficient : 0.0772

Element	(keV)	Mass%	Sigma	Atom%	Compound	Mass%	Cation	K
C K	0.277	2.91	0.14	9.53				0.8350
O K	0.525	7.01	1.71	17.23				1.4418
Mg K	1.253	0.27	0.07	0.43				0.1074
Al K	1.486	5.08	0.17	7.41				3.2737
Si K	1.739	0.42	0.06	0.58				0.2803
V K	4.949	76.41	0.76	59.01				85.6245
Cr K	5.411	4.50	0.25	3.40				5.1579
Mn K	5.894	0.20	0.08	0.15				0.1876
Fe K	6.398	3.20	0.20	2.26				3.0917

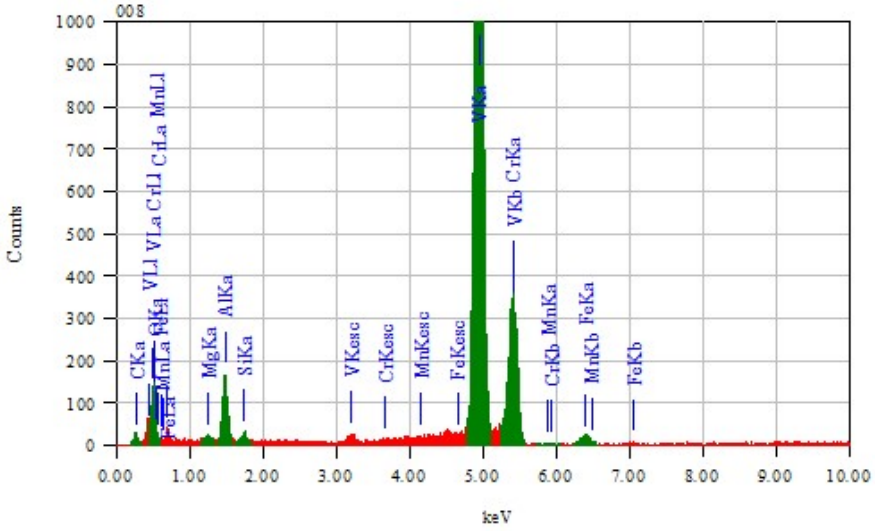
View003

JEOL 1/1



Title : IMG1

Instrument : JCM-6000
Volt : 15.00 kV
Mag. : x 200
Date : 2017/09/25



Acquisition Parameter
Instrument : JCM-6000
Acc. Voltage : 15.0 kV
Probe Current: 1.00000 nA
PHA mode : T3
Real Time : 61.06 sec
Live Time : 60.00 sec
Dead Time : 1 %
Counting Rate: 800 cps
Energy Range : 0 - 20 ke

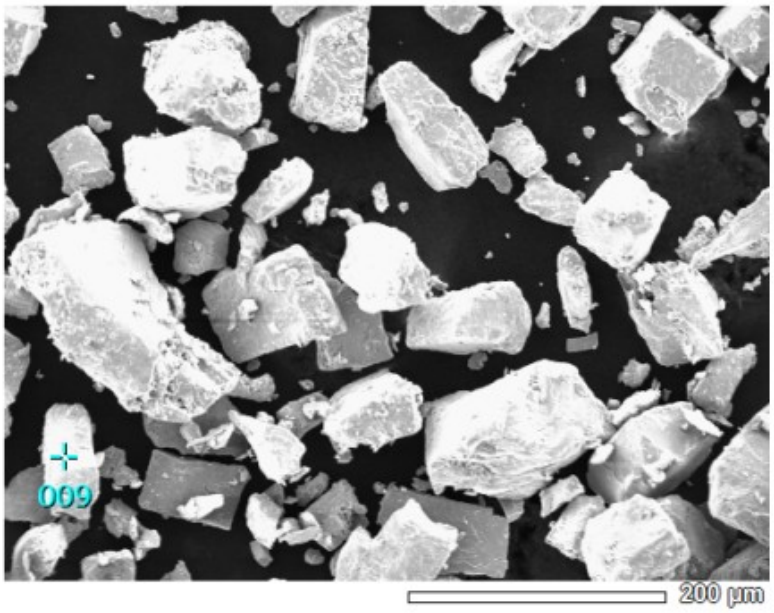
ZAF Method Standardless Quantitative Analysis
Fitting Coefficient : 0.0643

Element	(keV)	Mass%	Sigma	Atom%	Compound	Mass%	Cation	K
C K	0.277	1.74	0.07	5.74				0.5390
O K	0.525	9.13	0.99	22.68				1.7343
Mg K	1.253	0.26	0.04	0.42				0.1004
Al K	1.486	2.61	0.09	3.84				1.6531
Si K	1.739	0.26	0.03	0.37				0.1781
V K	4.949	82.06	0.56	64.02				91.5875
Cr K	5.411	2.36	0.14	1.80				2.6925
Mn K								
Fe K	6.398	1.59	0.10	1.13				1.5151

JED-2300 AnalysisStation JEOL

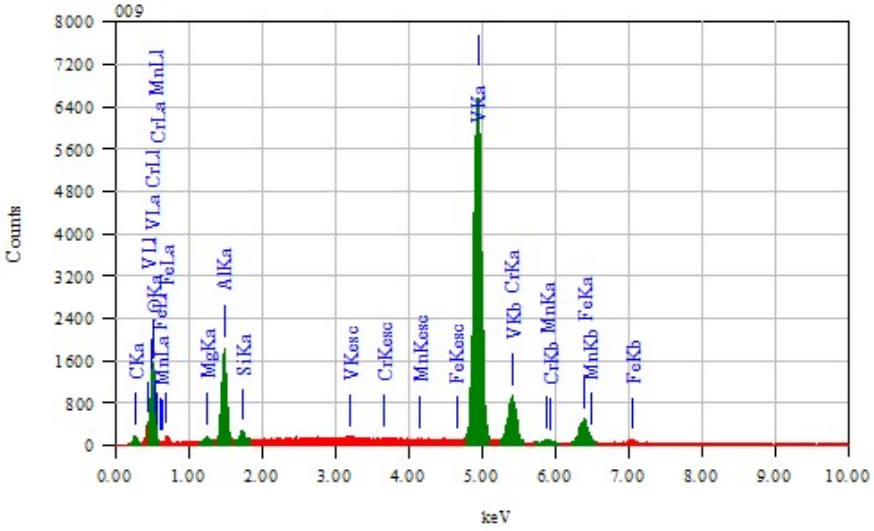
View003

JEOL 1/1



Title : IMG1

Instrument : JCM-6000
Volt : 15.00 kV
Mag. : x 200
Date : 2017/09/25



Acquisition Parameter
Instrument : JCM-6000
Acc. Voltage : 15.0 kV
Probe Current: 1.00000 nA
PHA mode : T3
Real Time : 62.30 sec
Live Time : 60.00 sec
Dead Time : 3 %
Counting Rate: 3434 cps
Energy Range : 0 - 20 ke

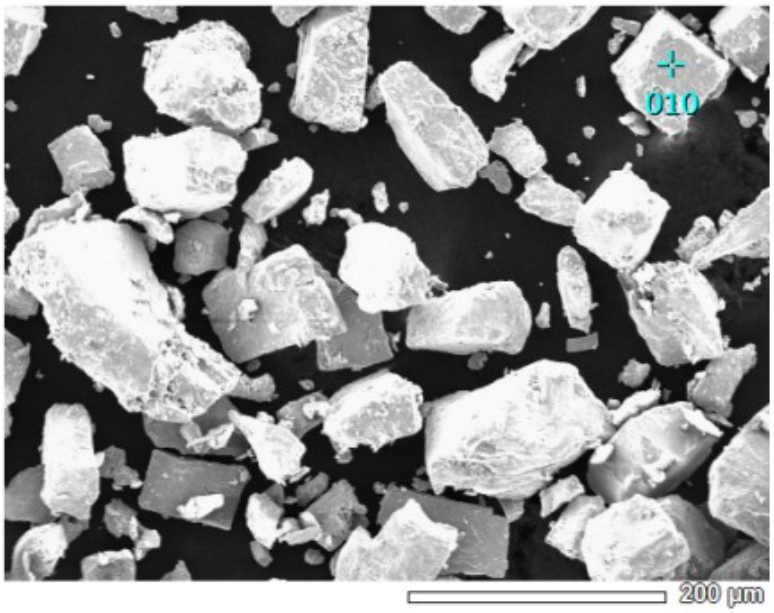
ZAF Method Standardless Quantitative Analysis
Fitting Coefficient : 0.0502

Element	(keV)	Mass%	Sigma	Atom%	Compound	Mass%	Cation	K
C K	0.277	2.99	0.05	7.14				1.0046
O K	0.525	28.78	0.49	51.56				10.2086
Mg K	1.253	0.33	0.02	0.39				0.1664
Al K	1.486	5.69	0.06	6.04				4.5792
Si K	1.739	0.48	0.02	0.49				0.3994
V K	4.949	53.66	0.21	30.20				73.5882
Cr K	5.411	0.76	0.06	0.42				1.0614
Mn K	5.894	0.57	0.04	0.30				0.6754
Fe K	6.398	6.74	0.10	3.46				8.3168

JED-2300 AnalysisStation JEOL

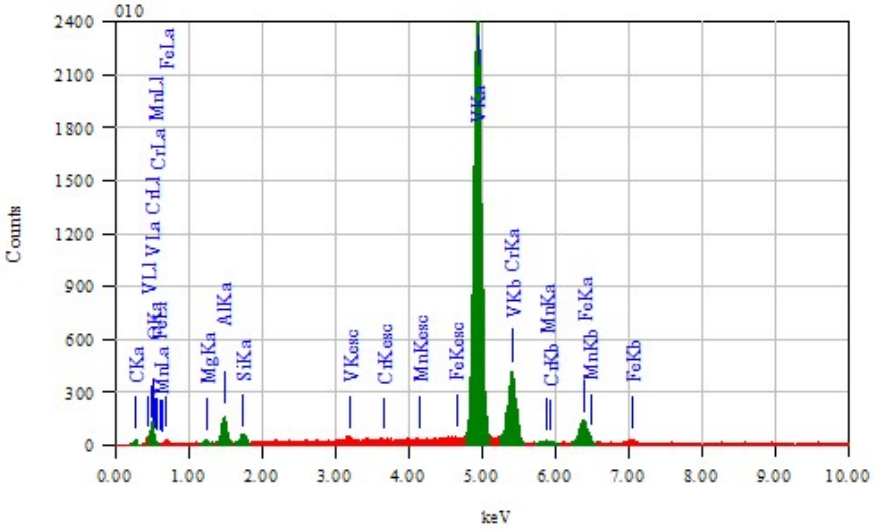
View003

JEOL 1/1



Title : IMG1

Instrument : JCM-6000
Volt : 15.00 kV
Mag. : x 200
Date : 2017/09/25



Acquisition Parameter
Instrument : JCM-6000
Acc. Voltage : 15.0 kV
Probe Current: 1.00000 nA
PHA mode : T3
Real Time : 61.18 sec
Live Time : 60.00 sec
Dead Time : 1 %
Counting Rate: 1081 cps
Energy Range : 0 - 20 ke

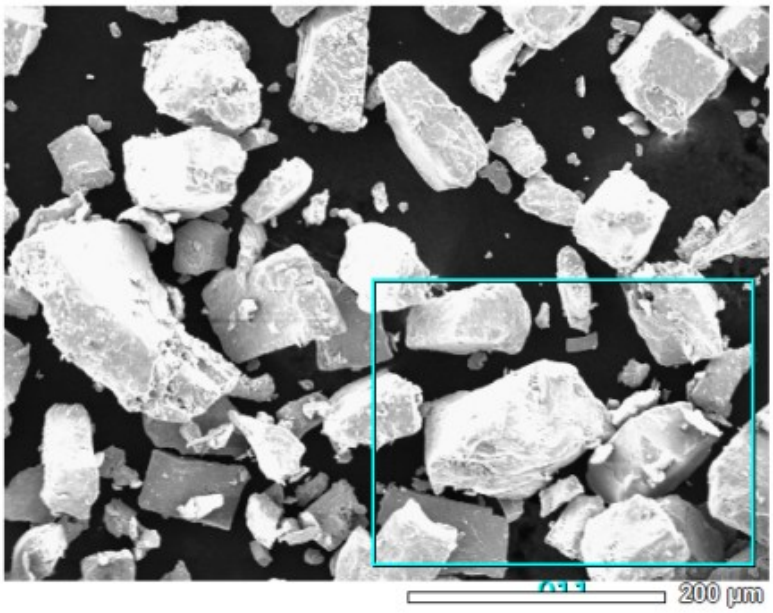
ZAF Method Standardless Quantitative Analysis
Fitting Coefficient : 0.0586

Element	(keV)	Mass%	Sigma	Atom%	Compound	Mass%	Cation	K
C K	0.277	2.27	0.08	8.83				0.6405
O K								
Mg K	1.253	0.26	0.04	0.50				0.0931
Al K	1.486	2.13	0.08	3.69				1.2180
Si K	1.739	0.47	0.04	0.78				0.2928
V K	4.949	82.91	0.52	76.03				86.6125
Cr K	5.411	2.69	0.16	2.41				2.8767
Mn K	5.894	0.43	0.08	0.37				0.3638
Fe K	6.398	8.85	0.22	7.40				7.9026

JED-2300 AnalysisStation JEOL

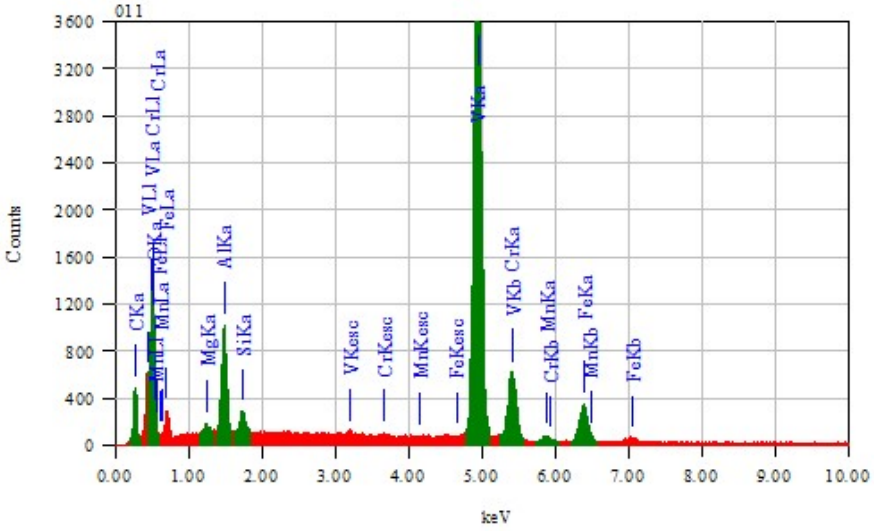
View003

JEOL 1/1



Title : IMG1

Instrument : JCM-6000
Volt : 15.00 kV
Mag. : x 200
Date : 2017/09/25



Acquisition Parameter
Instrument : JCM-6000
Acc. Voltage : 15.0 kV
Probe Current: 1.00000 nA
PHA mode : T3
Real Time : 123.11 sec
Live Time : 120.00 sec
Dead Time : 2 %
Counting Rate: 1260 cps
Energy Range : 0 - 20 ke

ZAF Method Standardless Quantitative Analysis
Fitting Coefficient : 0.0500

Element	(keV)	Mass%	Sigma	Atom%	Compound	Mass%	Cation	K
C K	0.277	15.30	0.14	37.73				4.8996
O K	0.525	8.01	0.83	14.83				2.0038
Mg K	1.253	0.47	0.04	0.58				0.2223
Al K	1.486	5.04	0.08	5.53				3.7985
Si K	1.739	0.87	0.04	0.91				0.6796
V K	4.949	60.42	0.29	35.13				77.0729
Cr K	5.411	0.99	0.08	0.57				1.2974
Mn K	5.894	0.75	0.06	0.40				0.8080
Fe K	6.398	8.15	0.14	4.32				9.2179

JED-2300 AnalysisStation JEOL

Point	V, %	Cr, %	Fe, %	Mn, %	Al, %
1	75.87	0.92	13.03	1.57	2.97
2	71.29	0.72	12.02	1.47	3.52
3	68.19	0.84	11.73	1.46	3.55
4	90.68	2.31	3.55	0.07	1.13
5	78.25	1.18	12.03	1.19	3.52
6	75.65	0.67	12.54	1.35	3.50
7	76.41	4.50	3.20	0.20	5.08
8	82.06	2.36	1.59	-	2.61
9	54.66	0.76	6.74	0.57	5.69
10	82.91	2.69	8.85	0.43	2.13
area	60.42	0.99	8.15	0.75	5.054
mean	73.85	1.63	8.5	0.91	3.52
σ	11.84	1.15	3.98	0.53	1.29