UNIVERSITY OF LIÈGE

FACULTY OF APPLIED SCIENCES

Minerals Engineering, Materials & Environment

EFFECT OF GRINDING CHEMISTRY ON THE SULPHIDISATION AND FLOTATION PERFORMANCES OF TRANSITIONAL OXIDE-SULPHIDE COPPER ORE

APPENDIX

By

Simon JACQUES

*Submitted to the "**Collège Doctoral en Sciences de l'Ingénieur et Technologie - Architecture, génie civil et géologie**"*
in partial fulfilment of the requirements for the
degree of doctor in applied sciences

July 2017

TABLE OF CONTENTS

I. EXPERIMENTAL

1.1 Estimated mineral assays from elemental data

By making a few simple assumptions regarding the composition of the minerals present in the ore body, it is possible to estimate mineral assays from the elemental assay data.

The mineral conversions are based on the assumption that each mineral is "pure" (chalcopyrite is $CuFeS_2$ and malachite $CuCO_3Cu(OH)_2$) and that all acid insoluble copper reports to chalcopyrite and all acid soluble copper to malachite. It is also assumed that all iron sulphide minerals are in the form of pyrite only.

1.1.1. Chalcopyrite

Chalcopyrite is made up of copper (63.55 amu), iron (55.85 amu) and sulphur (32.06 amu), therefore the atomic mass of chalcopyrite ($CuFeS_2$) is:

$CuFeS_2$ = Cu + Fe + 2S
$CuFeS_2$ = 63.55 + 55.85 + (2 x 32.06) amu
$CuFeS_2$ = 183.52 amu

Thus, stoichiometric chalcopyrite contains 34.63 percent copper, 30.43 percent iron and 34.94 percent sulphur. Hence, the conversion factor (f_{Ch}) to convert the copper assay to chalcopyrite is given by:

$$f_{CPY} = \frac{CuFeS_2\ amu}{Cu\ amu}$$

$$f_{CPY} = \frac{183.52}{63.55}$$

$$f_{CPY} = 2.88$$

So, an assay of 10 percent in sulphur is equivalent to 28.2 percent chalcopyrite.

1.1.2. Iron sulphide (Pyrite)

Pyrite is made up of iron (55.85 amu) and sulphur (32.06 amu), therefore the atomic mass of chalcopyrite (FeS_2) is:

FeS_2 = Fe + 2S
FeS_2 = 55.85 + (2 x 32.06) amu
FeS_2 = 119.97 amu

Thus, stoichiometric pyrite contains 46.5 percent iron and 53.4 percent sulphur. Hence, the conversion factor (f_{Py}) to convert the sulphur assay to chalcopyrite is given by:

$$f_{Py} = \frac{FeS_2 \ amu}{S \ amu}$$

$$f_{Py} = \frac{119.55}{64.12}$$

$$f_{Py} = 1.87$$

So, an assay of 10 percent of sulphur is equivalent to 18.7 percent pyrite.

The quantity of iron sulphide is then calculating from the sulphur assay by subtracting from the elemental assays the quantity of sulphur attributed to chalcopyrite:

IS= 1.87 x ([S]-[S_{CPY}])
IS= 1.87 x ([S]-0.3206 x [Cu] x 2.88))

1.1.3. Malachite

Malachite is made up of copper (63.55 amu), carbon (12.011 amu), oxygen (15.999 amu) and hydrogen (1.0079 amu), therefore the atomic mass of malachite is:

$CuCO_3Cu(OH)_2$ = 2Cu + C + 5O + 2H
$CuCO_3Cu(OH)_2$ = 2 x 63.55 + 12.011 + 5 x 15.99 + 2 x 1.001 amu
$CuCO_3Cu(OH)_2$ = 221.121 amu

Thus, stoichiometric malachite contains 57.4 percent copper, 5.43 percent carbon, and 36.17 percent oxygen and 0.9 percent hydrogen. Hence the conversion factor (f_{Mal}) to convert the copper assay to malachite is given by:

$$f_{Mal} = \frac{CuCO_3Cu(OH)_2 \ amu}{2Cu \ amu}$$

$$f_{Mal} = \frac{211.121}{127.1}$$

$$f_{Mal} = 1.73$$

So, an assay of 10 percent *acid soluble copper* is equivalent to 17.3 percent malachite.

1.1.4. Non-sulphide gangue

1.1.4.1. Sulphide ore

When both chalcopyrite and iron sulphide are present, the percentage of non sulphide gangue is calculated by subtracting the percent chalcopyrite and iron sulphide from 100 percent. That is:

% NSG = 100 – (%Ch + %IS)

1.1.4.2. Oxide synthetic ore (i.e. Quartz)

When malachite only is present, the percentage of quartz is calculated by subtracting the percent malachite from 100 percent. That is:

% NSG = 100 – %Mal

1.1.4.3. Mixed synthetic ore (i.e. Quartz).

When both chalcopyrite and malachite are present, the percentage of quartz is calculated by subtracting the percent chalcopyrite and malachite from 100 percent. That is:

% NSG = 100 – (%Ch +%Mal)

1.2 Magotteaux Mill® description

A schematic of the Magotteaux Mill® is shown in Figure 1. The Magotteaux Mill® has two chambers: the grinding chamber; and the measuring chamber. The grinding media, ore, water and any reagents, are placed into the grinding chamber. The probes for measuring pulp chemistry during grinding, typically pH, Eh and dissolved oxygen are placed into probe holders in the measuring chamber. The two chambers are separated by the separator plate, which has circular grates covered with 400 micron mesh to allow the flow of slurry containing -400 micron particles from the grinding chamber to the measuring chamber. A peristaltic pump is used to pump the slurry out of the measuring chamber and back into the grinding chamber, such that the slurry being measured is refreshed continuously.

Gases (air or nitrogen) are added into the slurry recirculation line to control the Eh and dissolved oxygen concentration of the slurry during grinding. The pH of slurry in the Magotteaux Mill® is controlled by dosing lime into the measuring chamber of the mill throughout the grind. The Magotteaux Mill® has a heating coil in the mill shell that allows the slurry to be heated to a set temperature. In this way, the Magotteaux Mill® allows the pulp chemistry to be measured and controlled so that the conditions in the plant ball mill discharge can be replicated in the laboratory mill discharge.

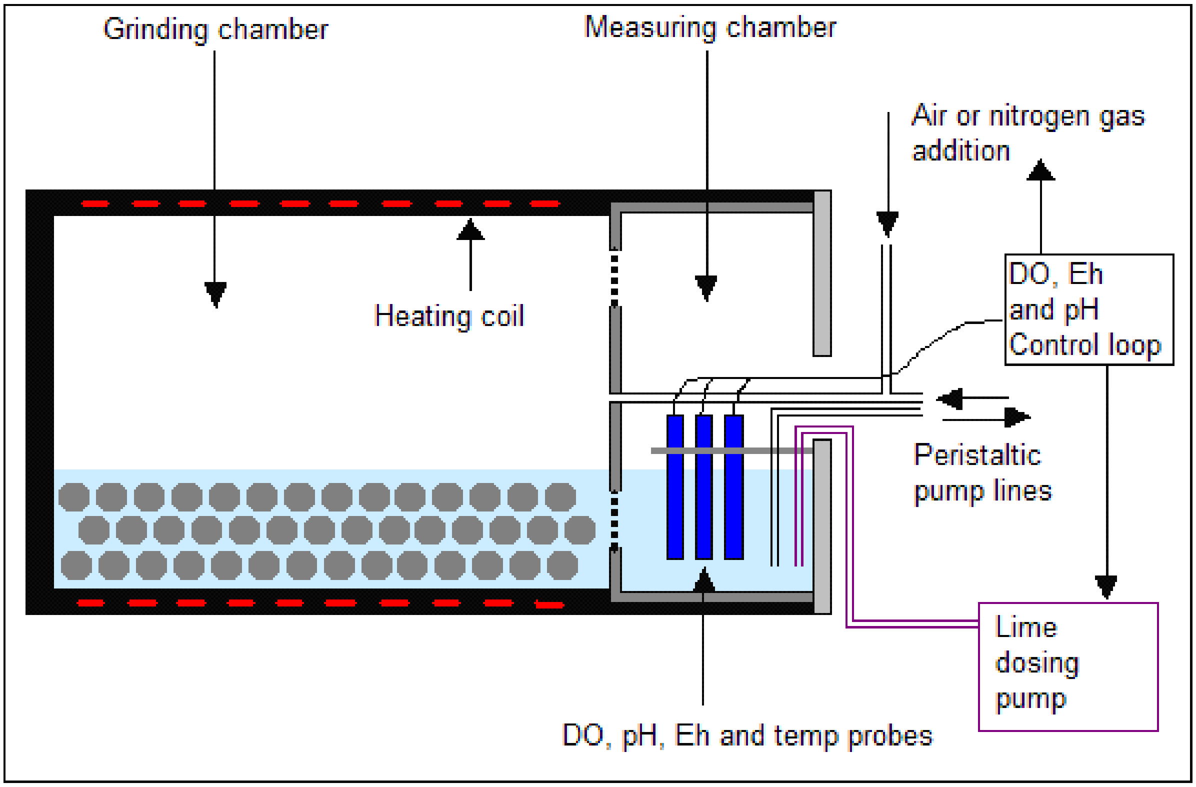

Figure 1: Schematic cross section of the Magotteaux Mill®.

1.3 Magotteaux Flotation Machine description

The Magotteaux Flotation Machine is a bottom driven laboratory flotation machine developed by Magotteaux Australia. A schematic diagram is displayed in Figure 2.

Features of the Magotteaux Flotation Machine are: bottom driven impellor, allowing an unobstructed froth surface; air is introduced into the base of the cell through special one-way valves; pulp chemistry probes can be permanently mounted into the rear of the cell during a test for continuous measurement; and impellor speed is digitally controlled.

All of these features provide for less operator variability and high reproducibility between tests.

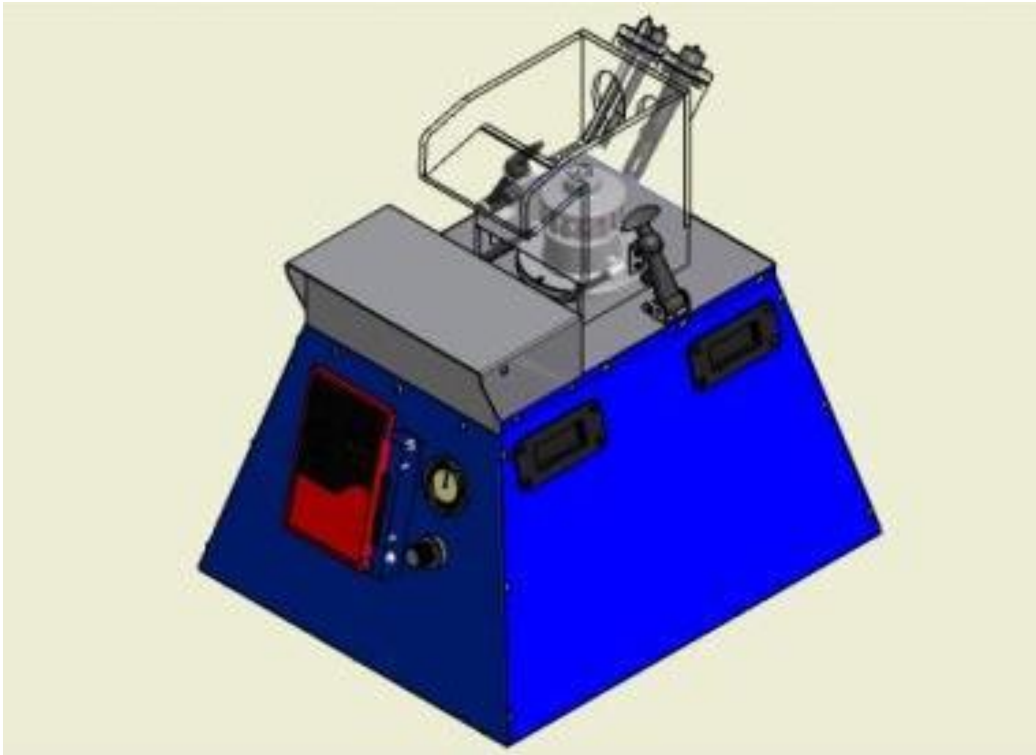

Figure 2: The Magotteaux Flotation Machine.

1.4 Sulphide tarnishing: experimental apparatus

Figure 3: General overview of the oxidation columns: 12 x oxidation columns and associated equipment.

Figure 4: Oxidation column pictures showing the fermenter and pumps alimenting the columns in the middle.

Table 1: Lundgren-Silverman 9K liquor

Chemical	Concentration g/l
$(NH_4)_2SO_4$	3.0
KCl	0.1
K_2HPO_4	0.5
$MgSO_4.7H_20$	0.5
$Ca(NO_3)_2$	0.01
$FeSO_4.7H_2O$	9.0

II. OXIDISED SULPHIDE FLOTATION RESULTS

2.1. Grinding calibration

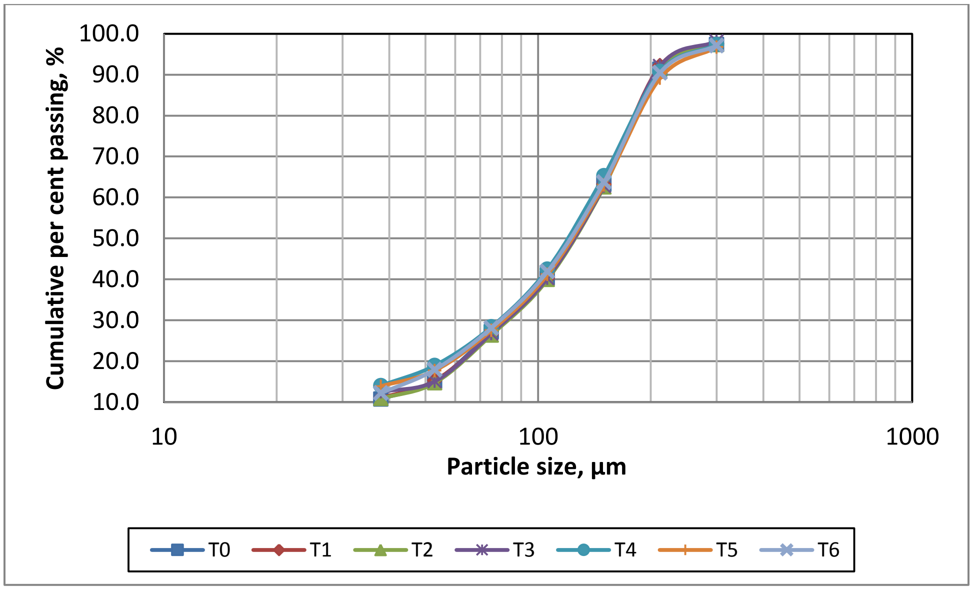

Figure 5: Cumulative size distribution curves for all ore tested with forged steel media only.

Table 2: Magotteaux Mill® parameters and P_{80} for all ore and alloys tested.

Ore	Media Type	Media Charge, Kg	Mill Speed, rpm	P_{80}, microns
T0	Forged	10 x balls	35	187.1
	15%Cr	10 x balls	35	187.5
	21%Cr	10 x balls	37	187.9
	Ceramic	10 x balls	40	186.7
T1	Forged	10 x balls	34	185.9
	15%Cr	10 x balls	34	185.3
	21%Cr	10 x balls	36	187.1
	Ceramic	10 x balls	38	187.6
T2	Forged	10 x balls	33	187.3
	15%Cr	10 x balls	33	188.3
	21%Cr	10 x balls	35	186.1
	Ceramic	10 x balls	37	188.4

T3	Forged	10 x balls	36	186.4
	15%Cr	10 x balls	36	187.5
	21%Cr	10 x balls	39	186.7
	Ceramic	10 x balls	41	189
T4	Forged	10 x balls	32	188.6
	15%Cr	10 x balls	33	188.9
	21%Cr	10 x balls	35	188.1
	Ceramic	10 x balls	37	188.1
T5	Forged	10 x balls	32	189.6
	15%Cr	10 x balls	33	187.8
	21%Cr	10 x balls	35	188.2
	Ceramic	10 x balls	36	187.2
T6	Forged	10 x balls	32	187.7
	15%Cr	10 x balls	33	188.8
	21%Cr	10 x balls	34	185.8
	Ceramic	10 x balls	36	188.8

Table 3: Magotteaux Mill® discharge pulp chemistry for each alloy tested.

Ore	Media	pH	Eh, mV (SHE)	DO, ppm	Temp, °C
T0	Forged	9.3	155	1.72	27.5
	15%Cr	9.4	195	2.34	26.9
	21%Cr	9.3	171	2.38	26.8
	Ceramic	9.3	178	2.55	26.1
T1	Forged	9.3	264	2.2	27.4
	15%Cr	9.3	250	1.9	26.9
	21%Cr	9.3	263	2.5	26.7
	Ceramic	9.3	239	2.6	26.4

II. Oxidised sulphide flotation results

T2	Forged	9.4	239	2.04	28.9
	15%Cr	9.4	250	2.53	27.9
	21%Cr	9.3	263	2.32	27.3
	Ceramic	9.3	239	2.52	27.0
T3	Forged	9.4	34	1.53	26.7
	15%Cr	9.4	184	2.71	26.5
	21%Cr	9.3	177	2.94	26.7
	Ceramic	9.4	186	2.98	28.1
T4	Forged	9.4	26	1.20	28.8
	15%Cr	9.4	122	1.80	27.9
	21%Cr	9.4	157	1.83	29.9
	Ceramic	9.4	169	2.23	28.3
T5	Forged	9.4	122	1.00	27.8
	15%Cr	9.4	210	1.88	27.5
	21%Cr	9.4	220	2.20	26.4
	Ceramic	9.5	224	2.40	28.3
T6	Forged	9.4	224	2.30	27.7
	15%Cr	9.6	228	2.26	26.2
	21%Cr	9.4	243	2.55	26.2
	Ceramic	9.4	202	2.75	27.7

II. Oxidised sulphide flotation results

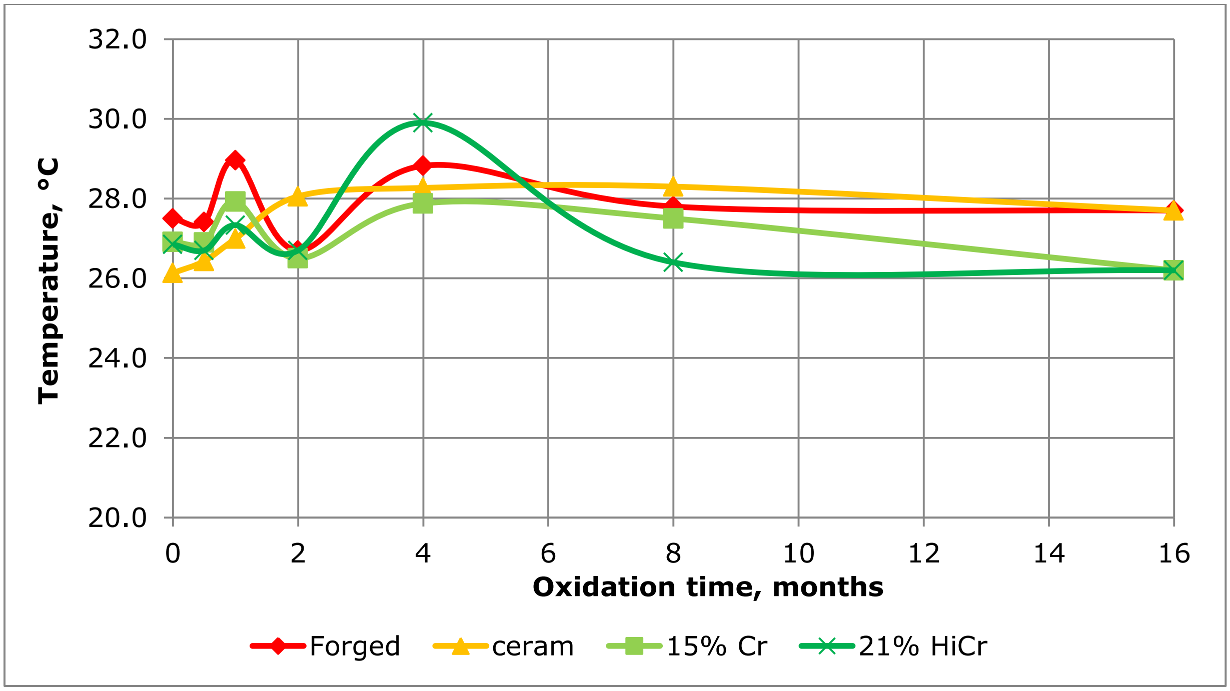

Figure 6: Mill discharge temperature values versus oxidation time (i.e. for each ore tested) for all media.

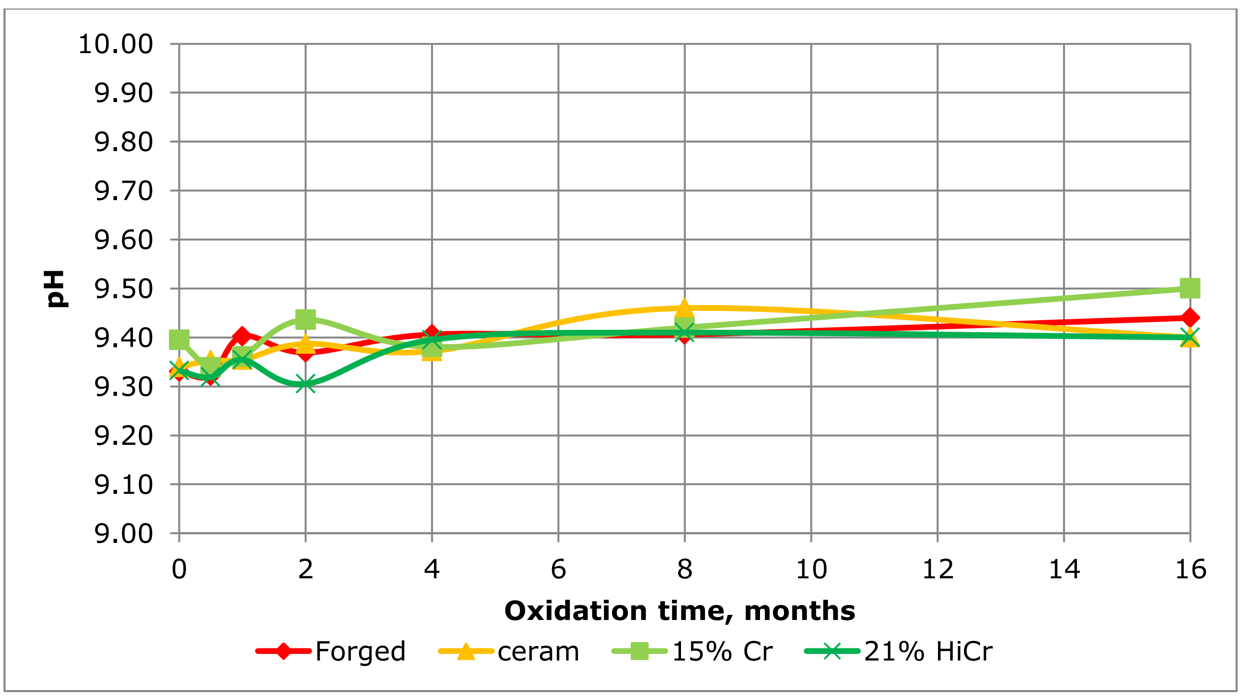

Figure 7: Mill discharge pH values versus oxidation time (i.e. for each ore tested) for all media.

II. Oxidised sulphide flotation results

2.2. Flotation chemistry

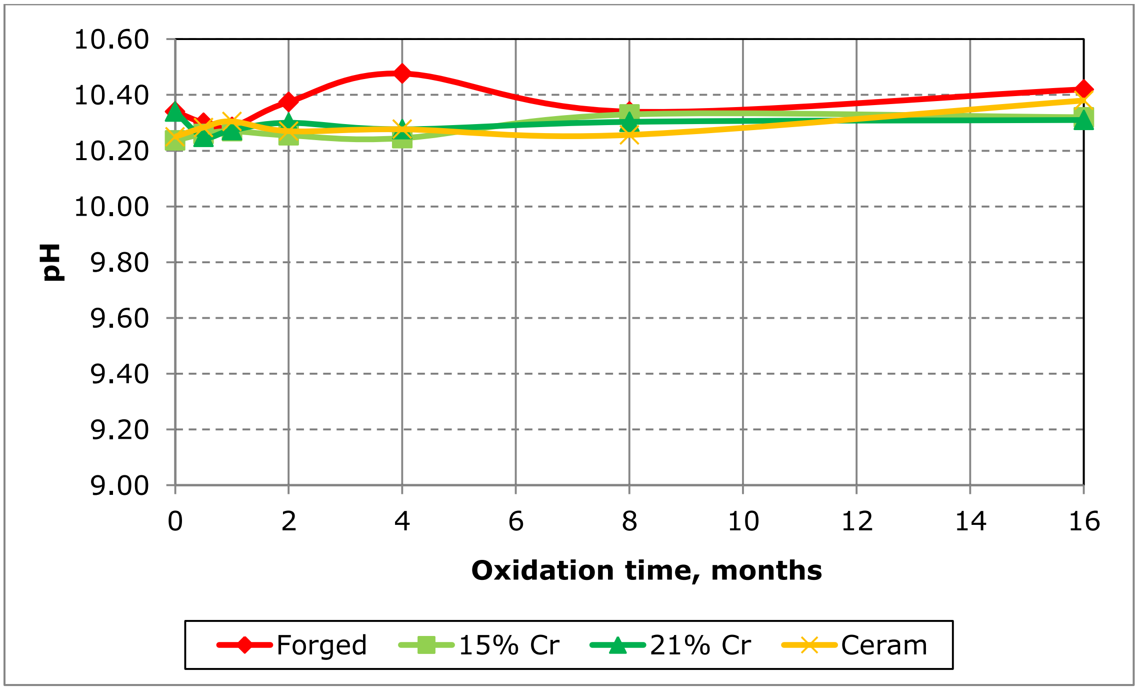

Figure 8: Flotation feed pH values versus oxidation time (i.e. for each ore tested) for all media tested.

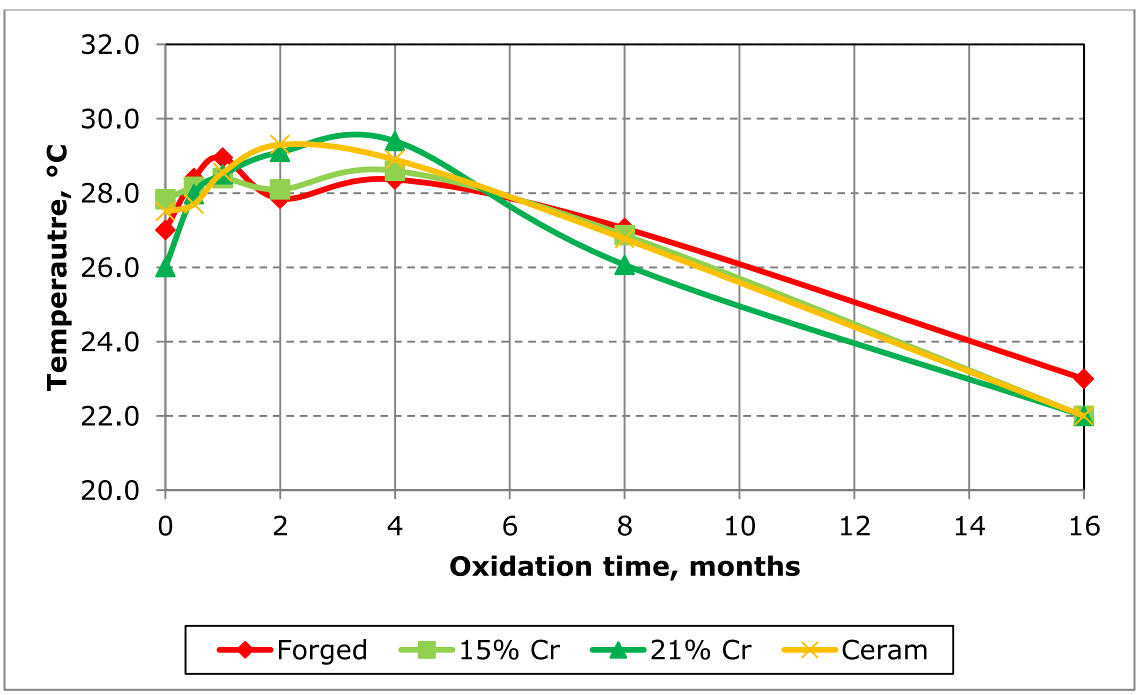

Figure 9: Flotation feed temperature values versus oxidation time (i.e. for each ore tested) for all media tested.

Table 4: Flotation feed chemistry for each alloy tested.

Ore	Media	pH	Eh, mV (SHE)	DO, ppm	Temp, °C
TO	Forged	10.3	93	3.5	27.0
	15%Cr	10.3	164	7.1	27.8
	21%Cr	10.3	166	7.0	16.0
	Ceramic	10.2	167	7.3	27.5
T1	Forged	10.3	106	5.1	28.4
	15%Cr	10.3	170	7.0	28.2
	21%Cr	10.2	170	7.2	28.0
	Ceramic	10.3	174	7.2	27.7
T2	Forged	10.3	155	6.3	28.9
	15%Cr	10.3	172	7.0	28.4
	21%Cr	10.3	166	7.3	28.5
	Ceramic	10.3	172	7.3	28.5
T3	Forged	10.4	145	5.7	27.8
	15%Cr	10.3	173	6.3	28.1
	21%Cr	10.3	174	6.8	29.1
	Ceramic	10.3	172	6.6	29.3
T4	Forged	10.5	130	5.7	27.0
	15%Cr	10.3	165	6.5	26.9
	21%Cr	10.3	159	6.6	26.1
	Ceramic	10.3	167	7.0	26.8
T5	Forged	6.6	183	6.6	27.0
	15%Cr	7.4	225	7.4	26.9
	21%Cr	7.4	221	7.4	26.1
	Ceramic	7.5	243	7.5	26.8
T6	Forged	10.4	185	7.5	23.0
	15%Cr	10.3	187	7.5	22.0
	21%Cr	10.3	191	8.1	22.0
	Ceramic	10.4	187	7.8	22.0

II. Oxidised sulphide flotation results

2.3. Oxygen demand data

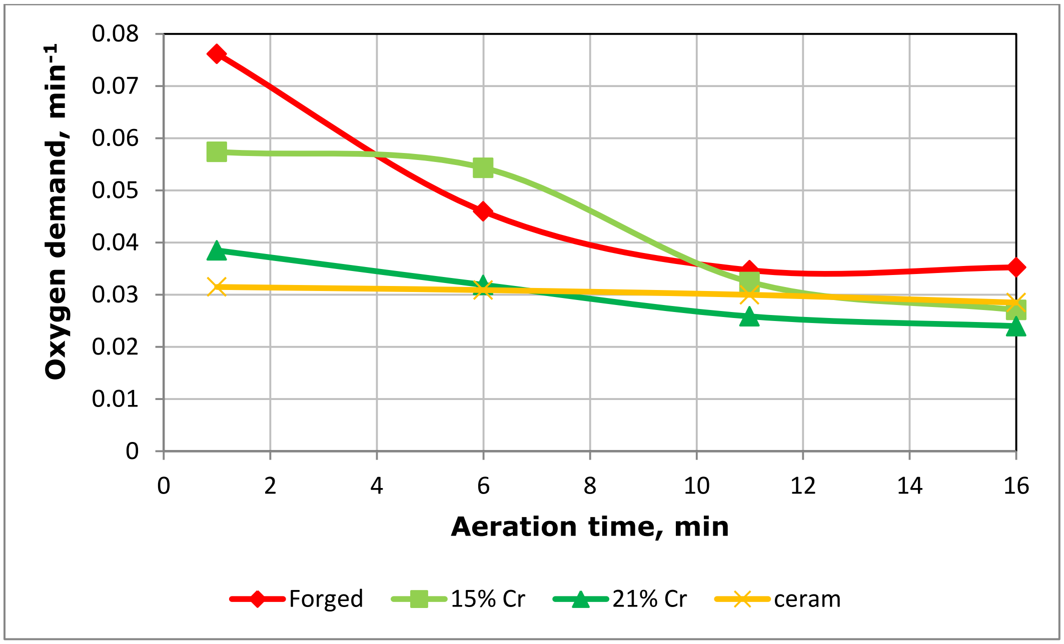

Figure 10: Oxygen demand versus aeration time for tests completed on T0 ore samples with all media.

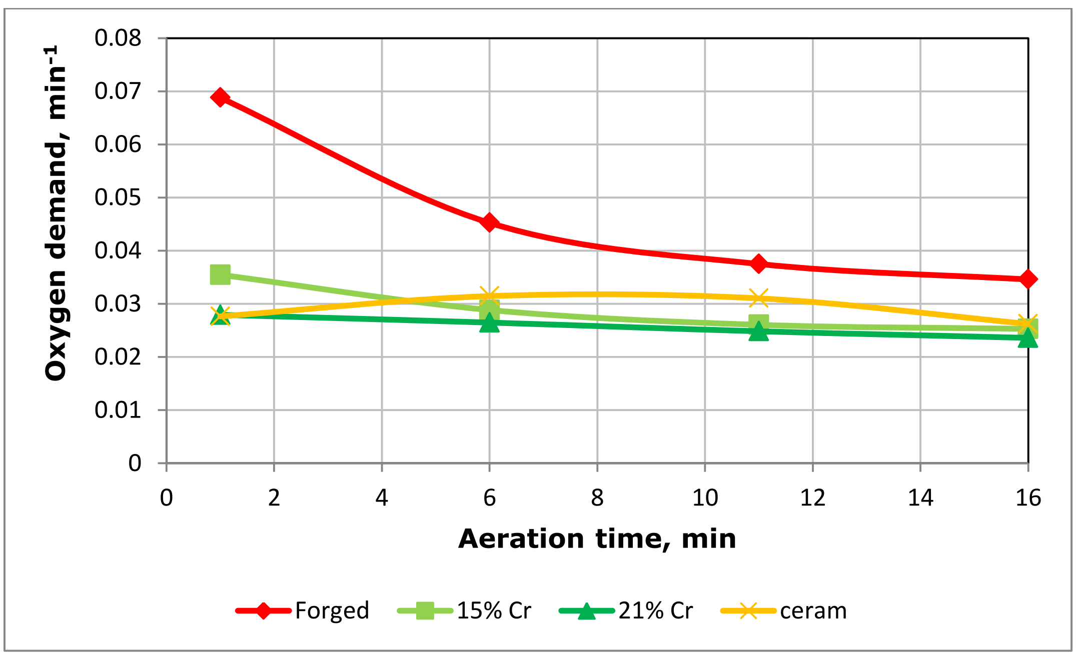

Figure 11: Oxygen demand versus aeration time for tests completed on T1 ore samples with all media.

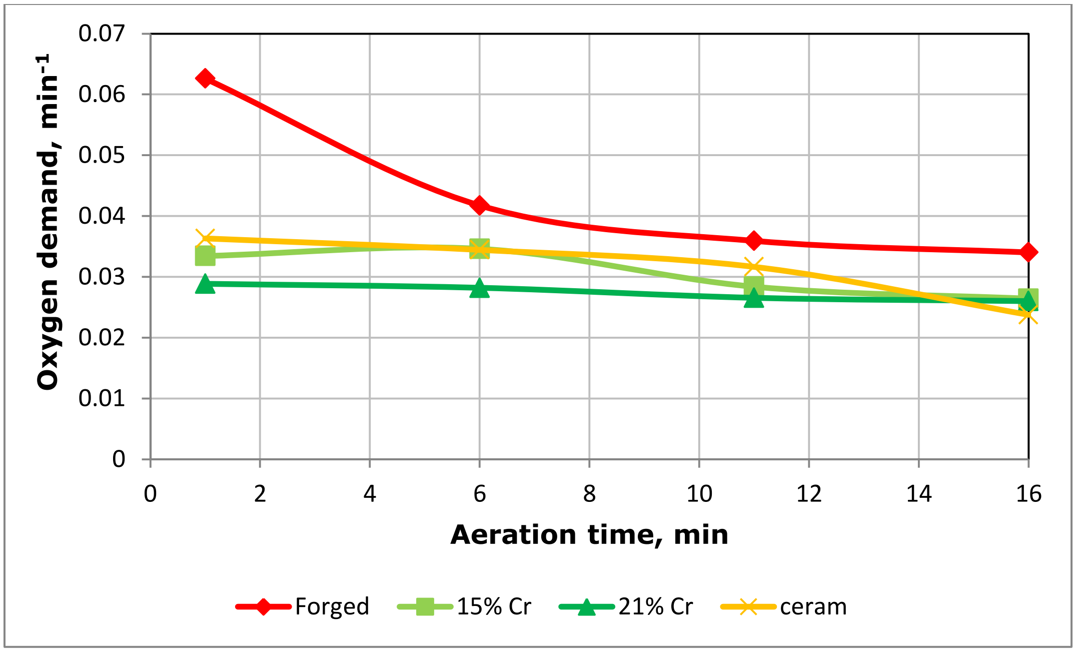

Figure 12: Oxygen demand versus aeration time for tests completed on T2 ore samples with all media.

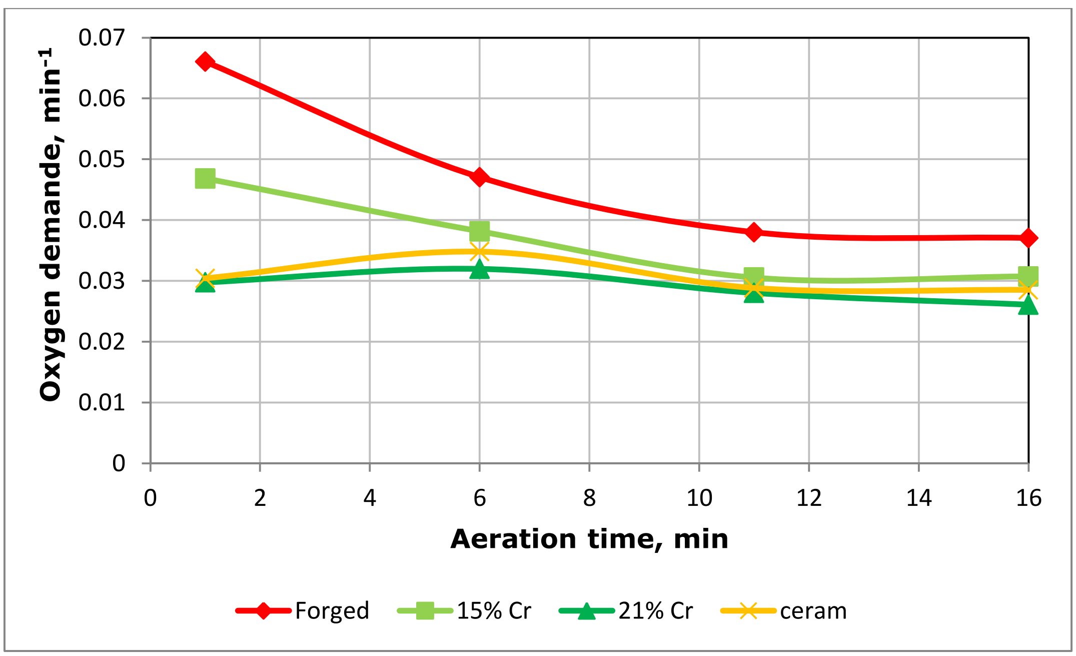

Figure 13: Oxygen demand versus aeration time for tests completed on T3 ore samples with all media.

II. Oxidised sulphide flotation results

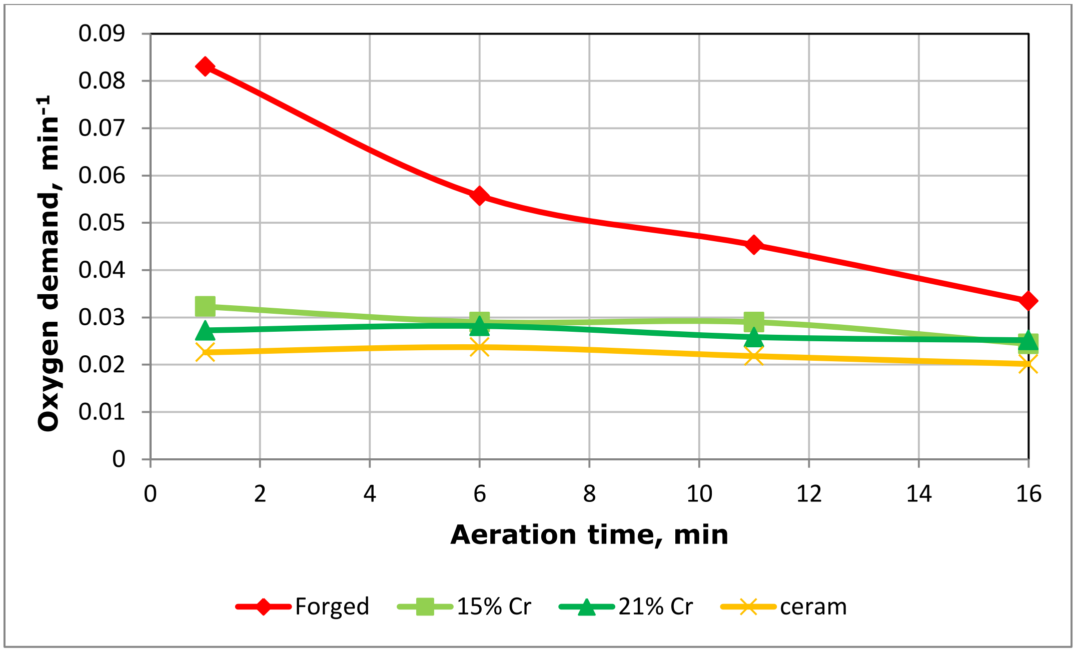

Figure 14: Oxygen demand versus aeration time for tests completed on T4 ore samples with all media.

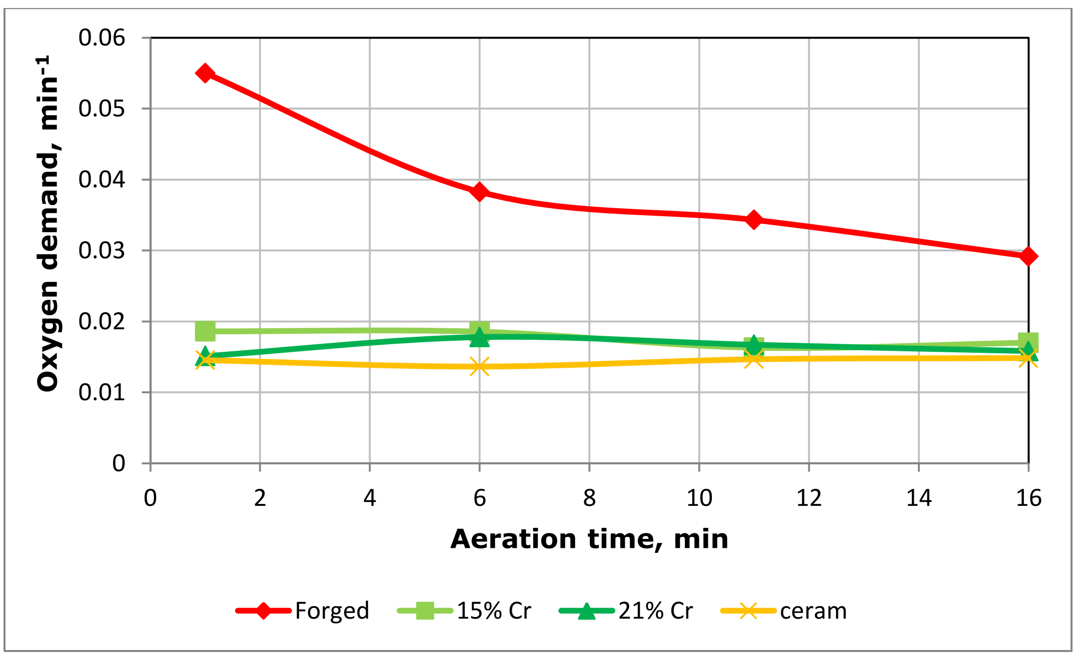

Figure 15: Oxygen demand versus aeration time for tests completed on T5 ore samples with all media tested.

II. Oxidised sulphide flotation results

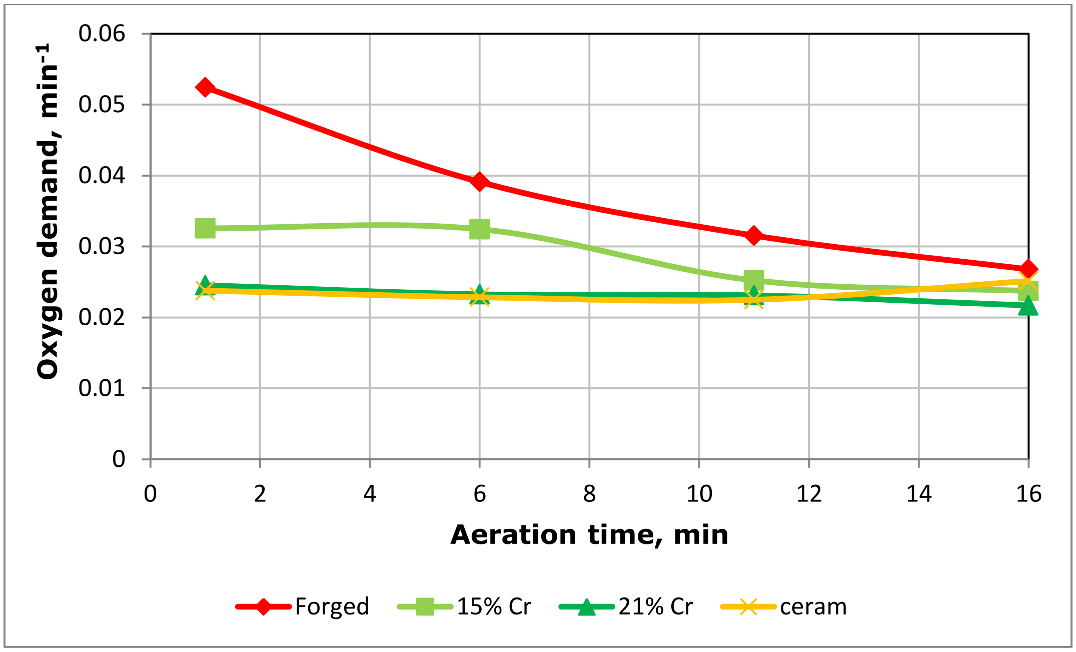

Figure 16: Oxygen demand versus aeration time for tests completed on T6 ore samples with all media tested.

2.4. Flotation results

2.4.1. Reproducibility

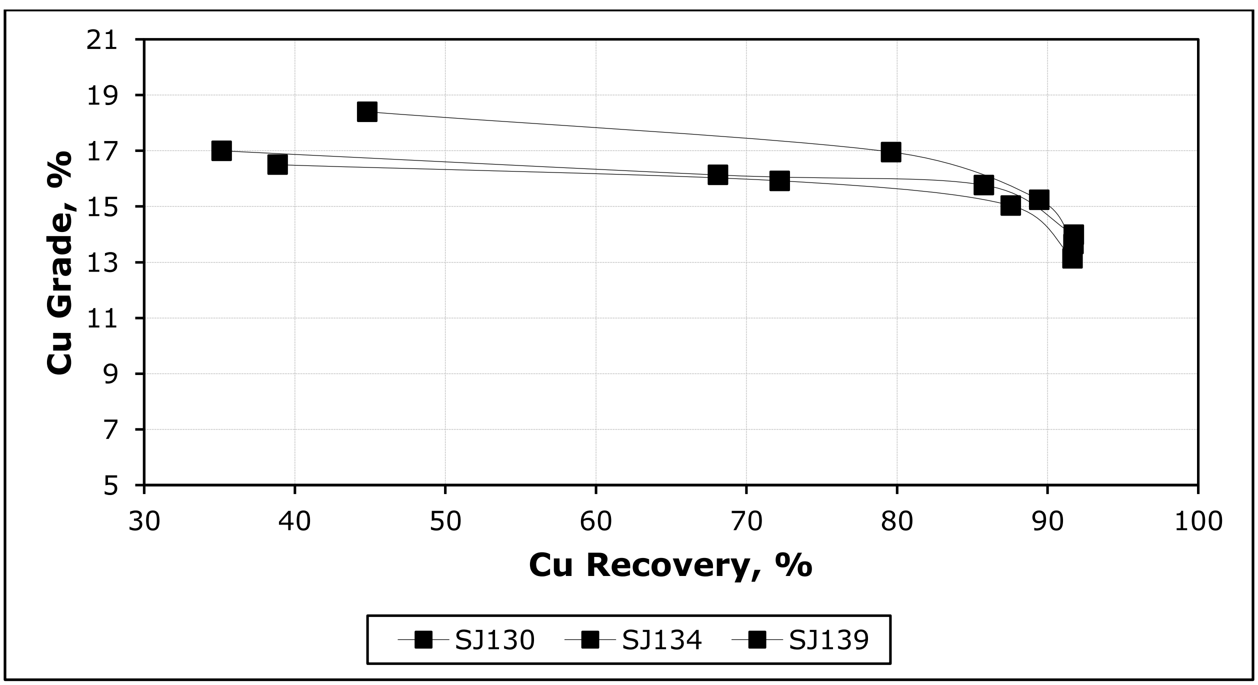

Figure 17: Copper grade recovery curves for flotation tests completed with forged steel media on T0 ore sample.

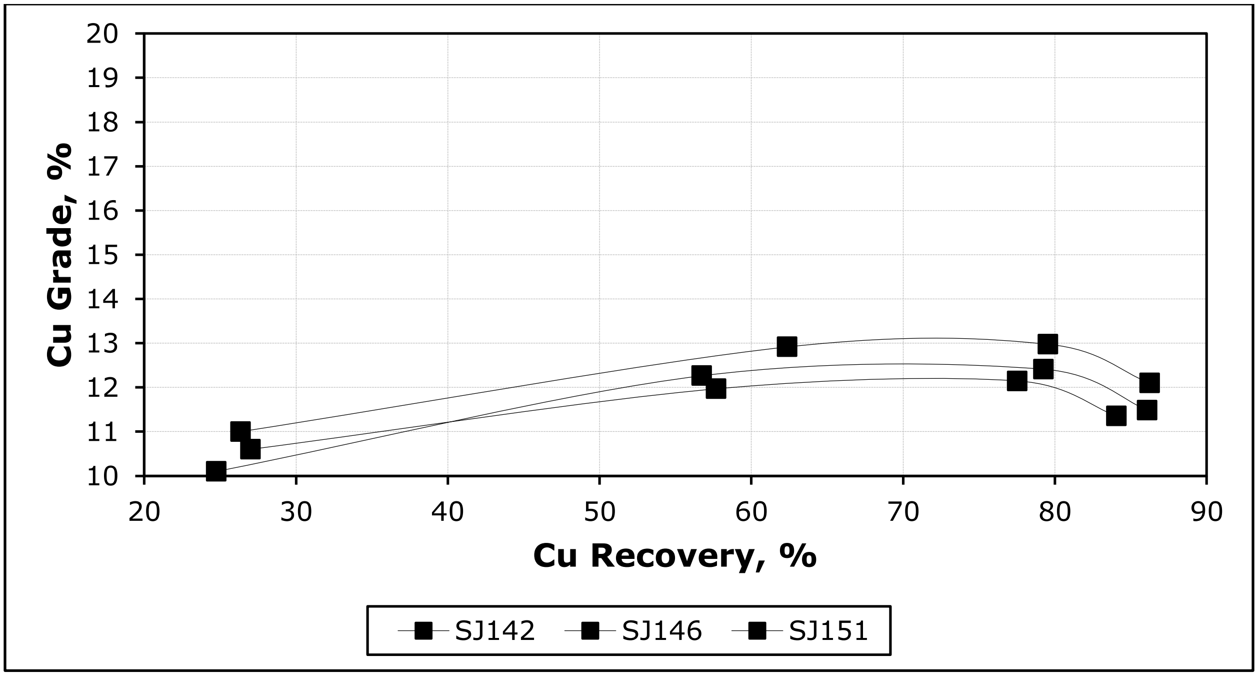

Figure 18: Copper grade recovery curves for flotation tests completed with forged steel media on T1 ore sample.

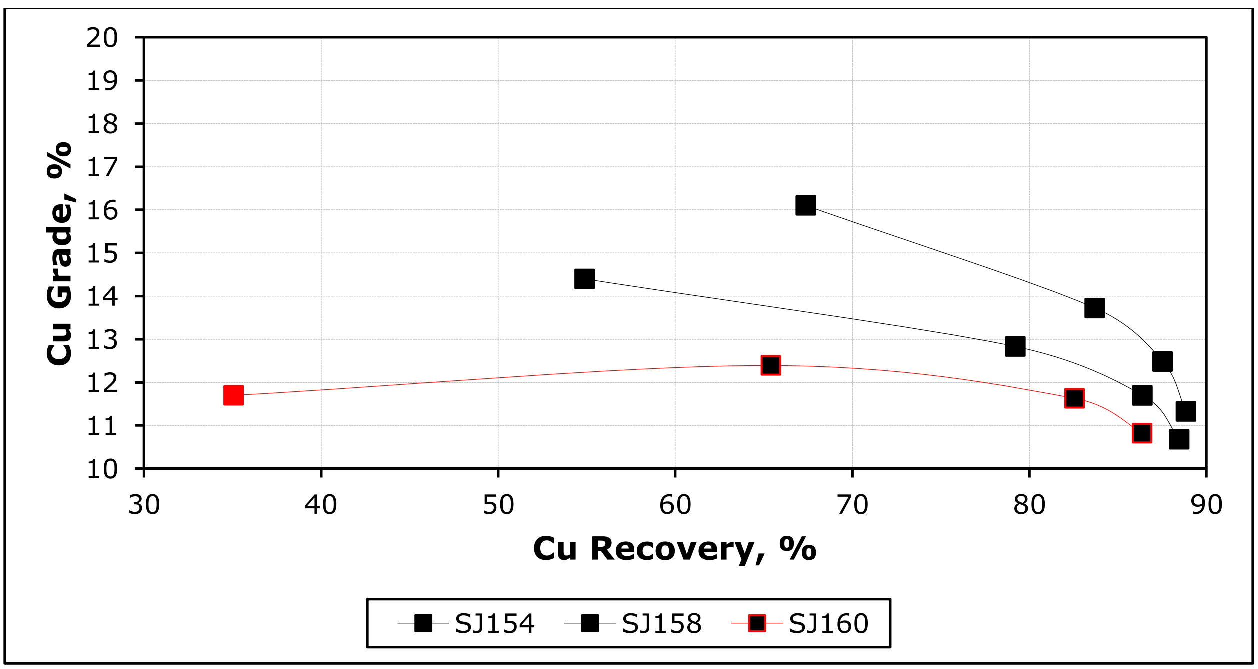

Figure 19: Copper grade recovery curves for flotation tests completed with forged steel media on T2 ore sample, SJ160 test was discarded.

II. Oxidised sulphide flotation results

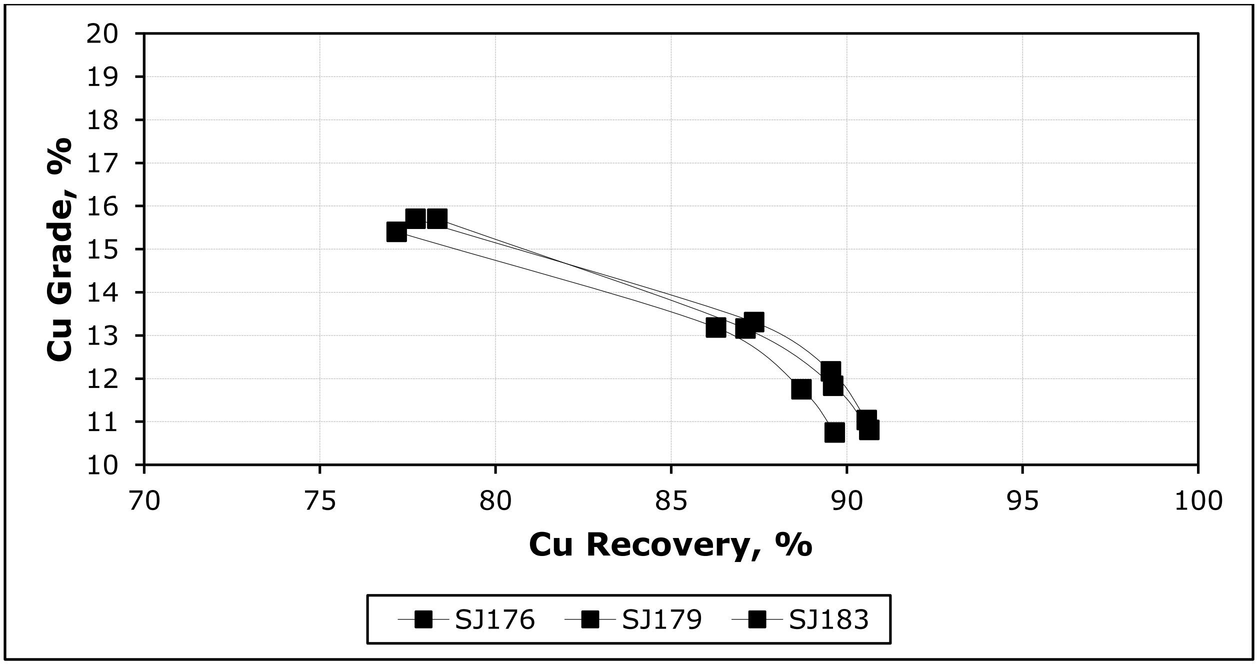

Figure 20: Copper grade recovery curves for flotation tests completed with forged steel media on T3 ore sample.

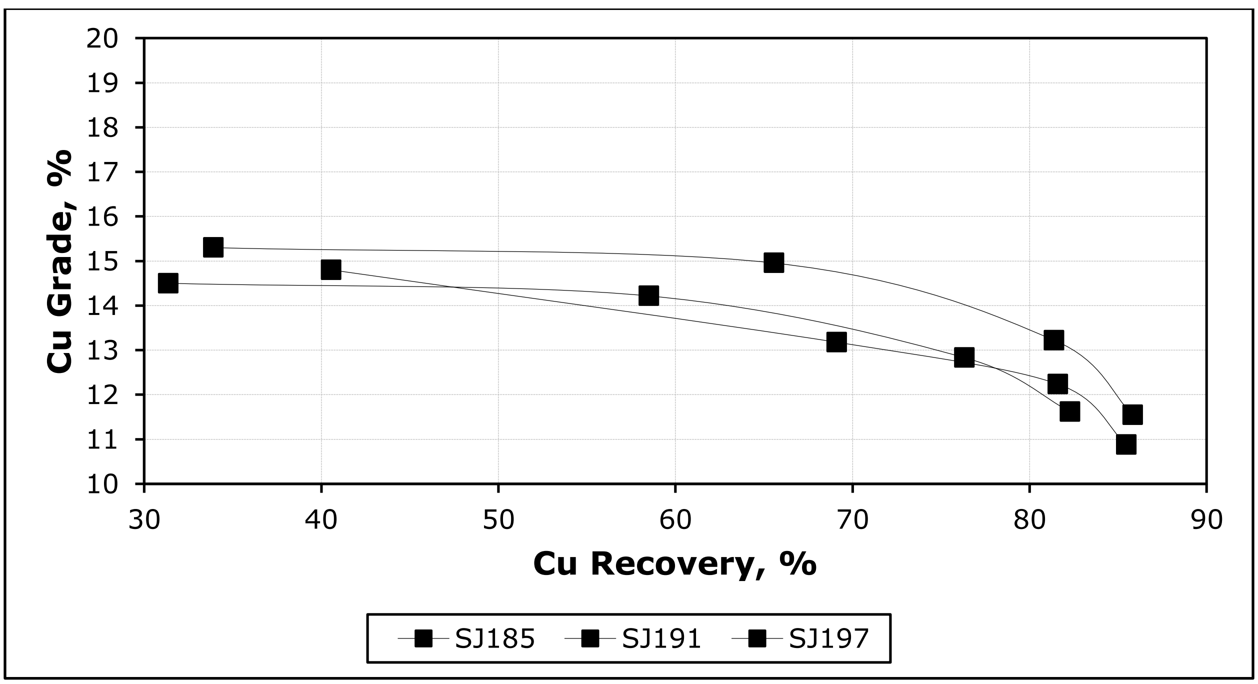

Figure 21: Copper grade recovery curves for flotation tests completed with forged steel media on T4 ore sample.

II. Oxidised sulphide flotation results

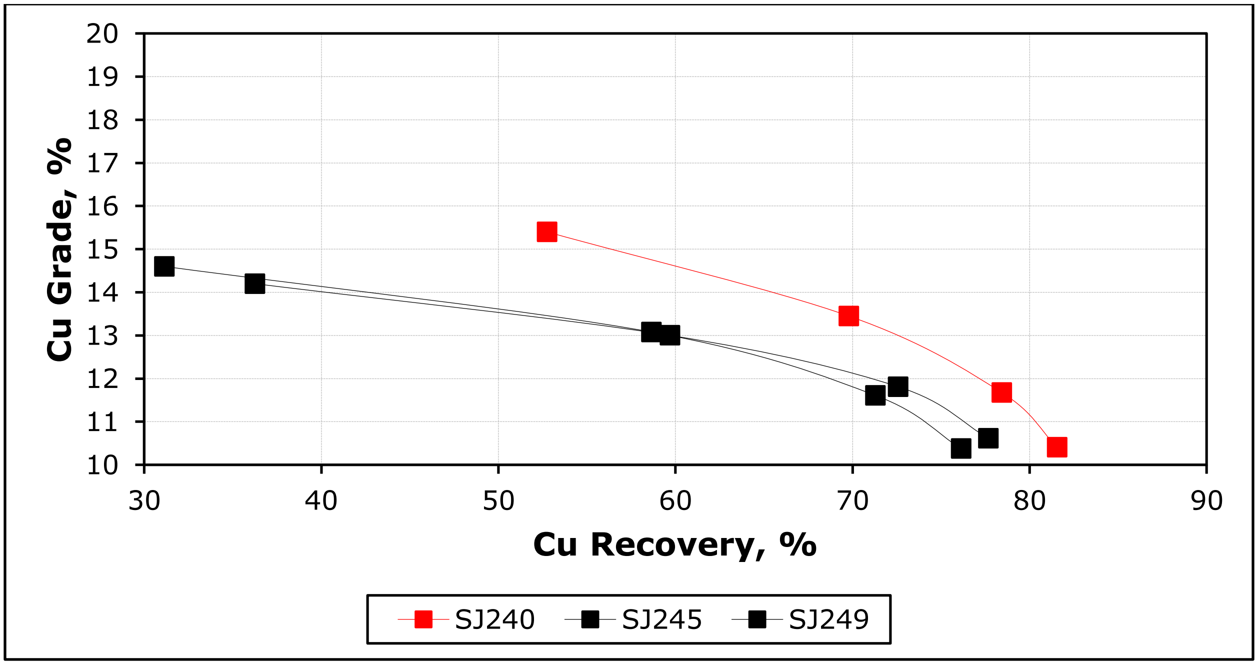

Figure 22: Copper grade recovery curves for flotation tests completed with forged steel media on T5 ore sample, SJ240 was discarded.

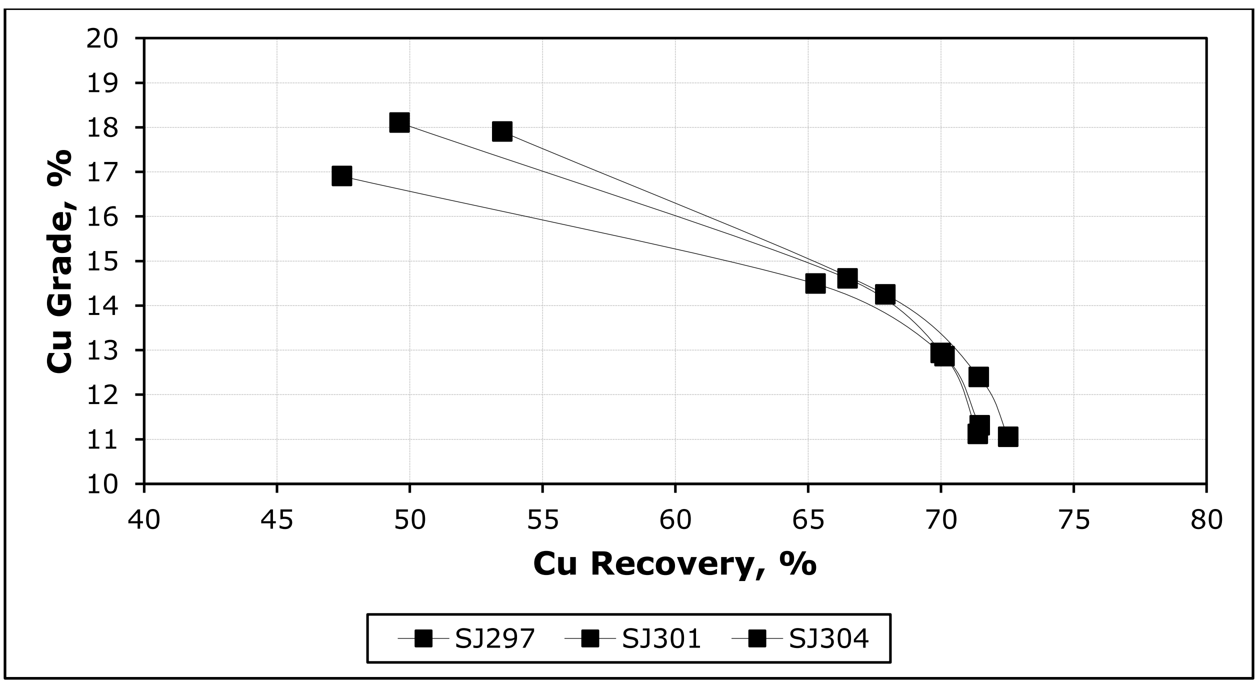

Figure 23: Copper grade recovery curves for flotation tests completed with forged steel media on T6 ore sample.

II. Oxidised sulphide flotation results

2.4.2. *Alloy scoping*

- Copper grade-recovery curves for each alloy tested.

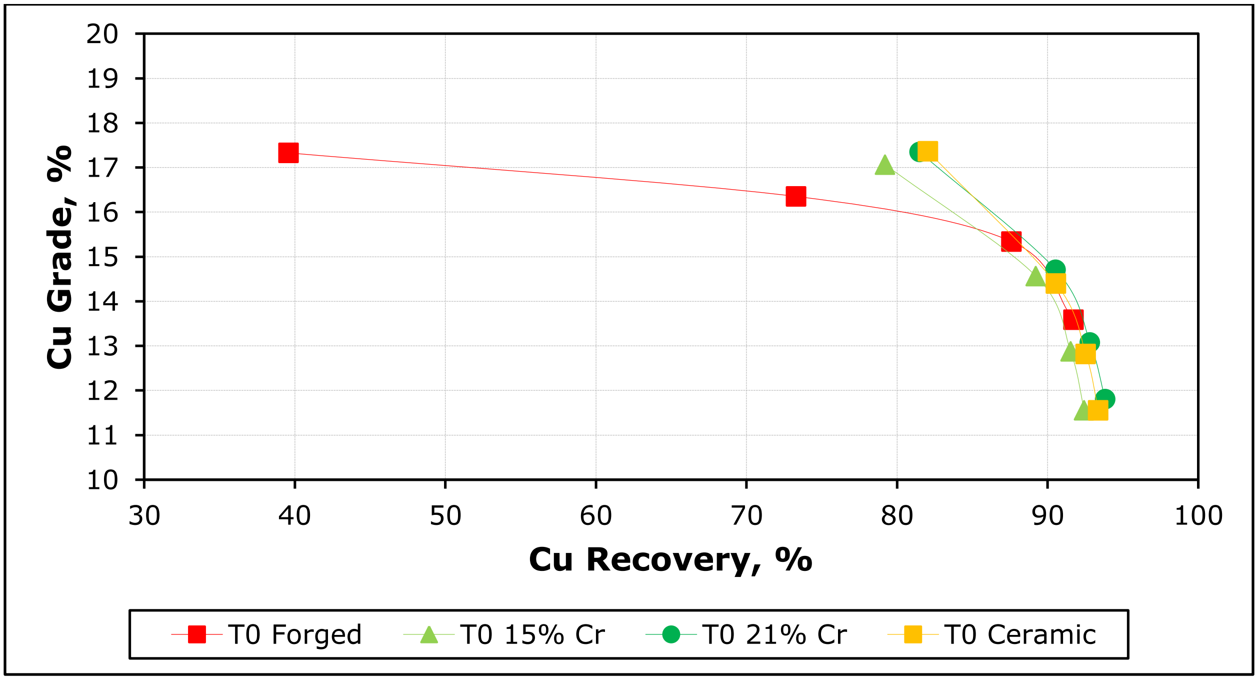

Figure 24: Copper grade recovery curves for flotation tests completed on T0 ore sample comparing all media tested.

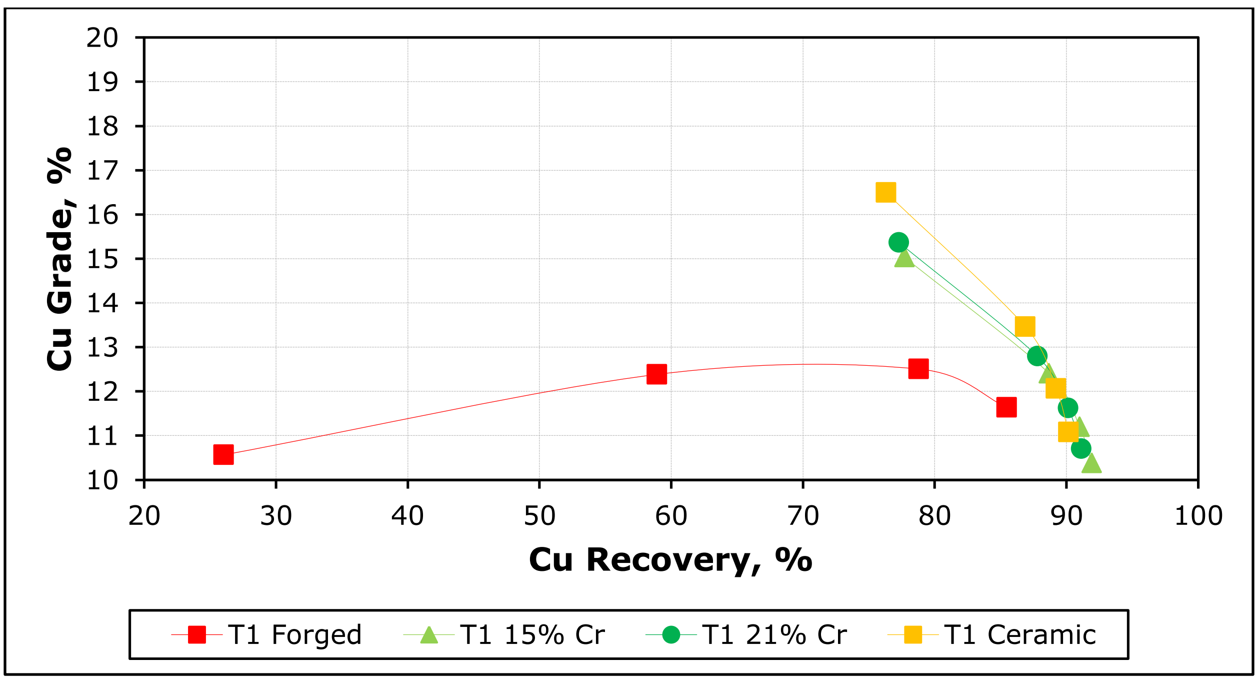

Figure 25: Copper grade recovery curves for flotation tests completed on T1 ore sample comparing all media tested.

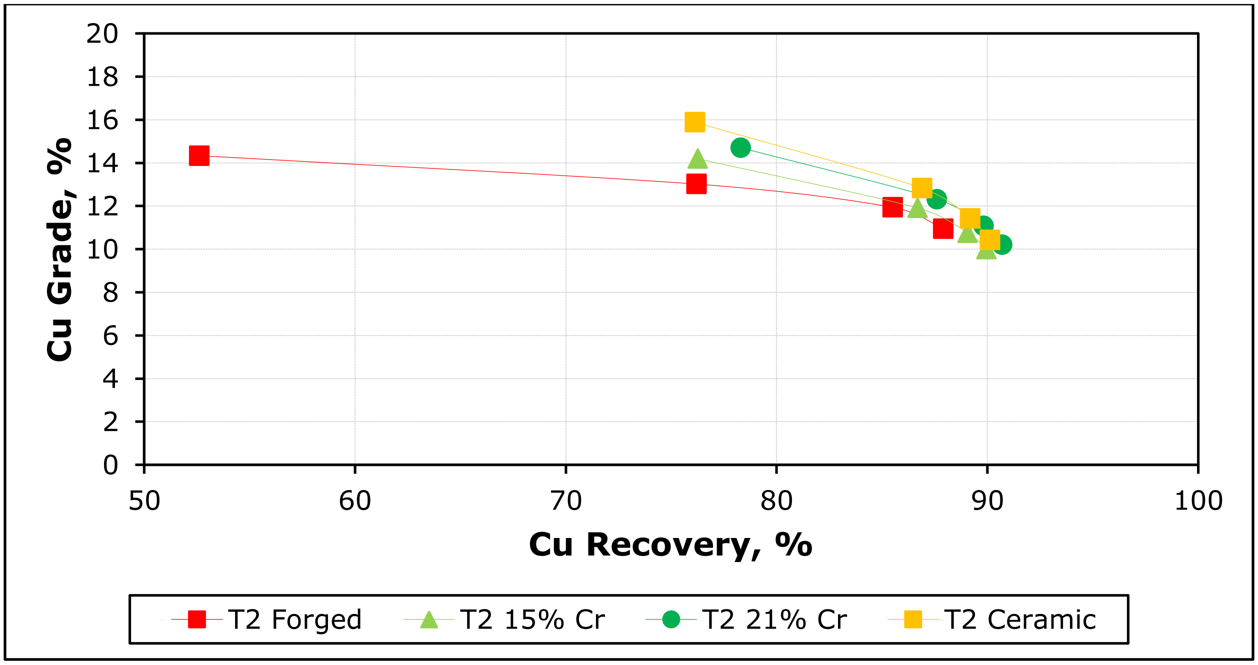

Figure 26: Copper grade recovery curves for flotation tests completed on T2 ore sample comparing all media tested.

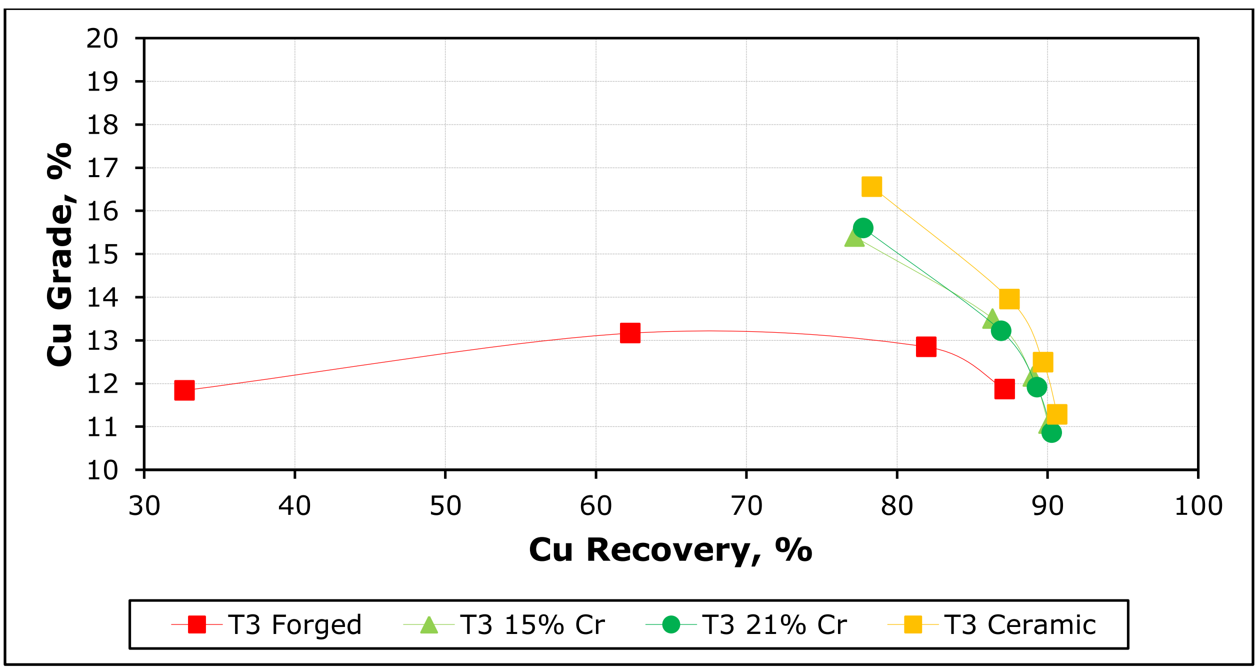

Figure 27: Copper grade recovery curves for flotation tests completed on T3 ore sample comparing all media tested.

II. Oxidised sulphide flotation results

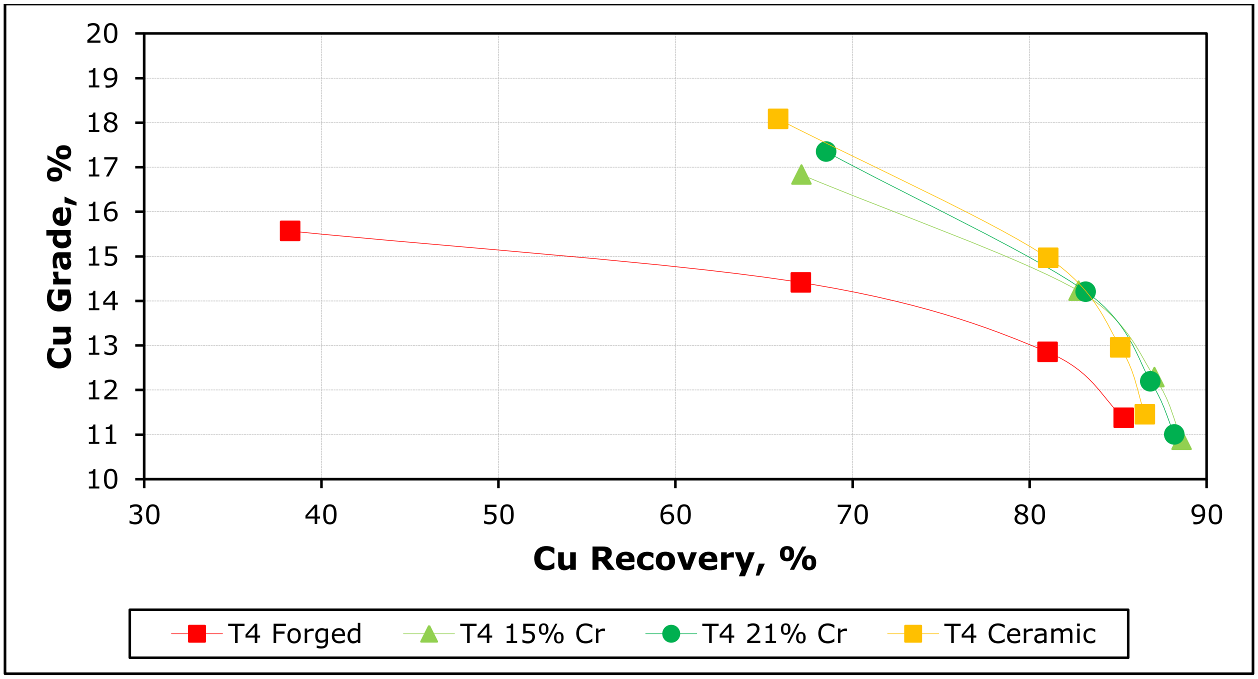

Figure 28: Copper grade recovery curves for flotation tests completed on T4 ore sample comparing all media tested.

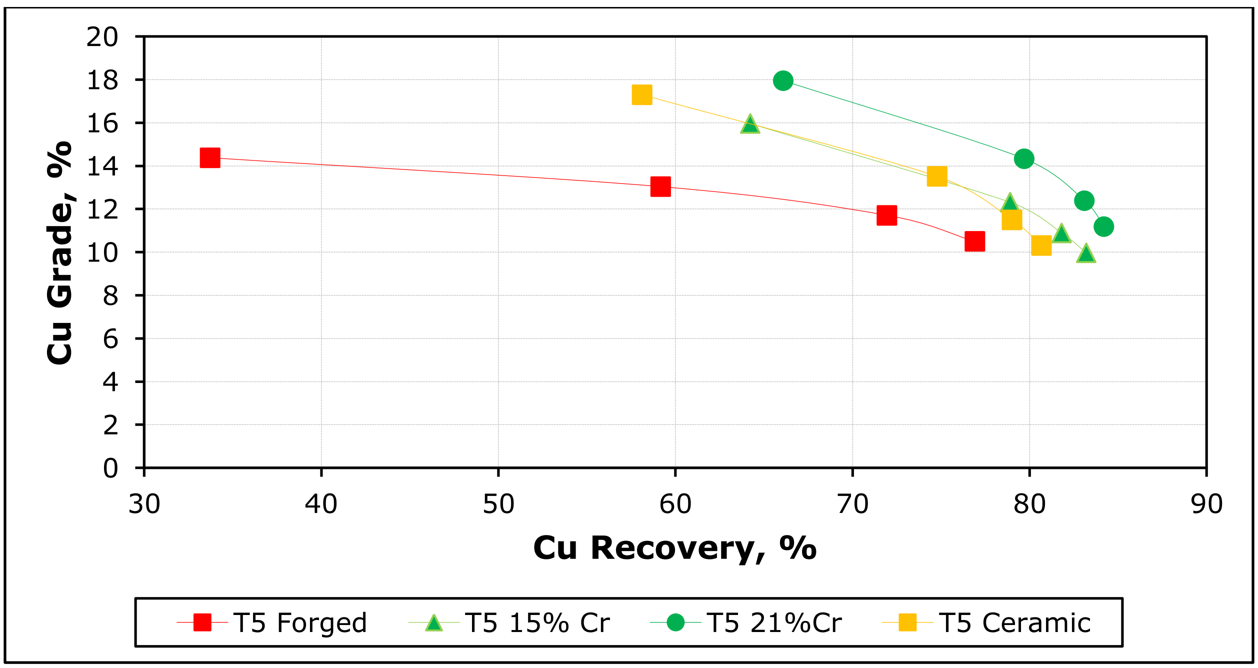

Figure 29: Copper grade recovery curves for flotation tests completed on T5 ore samples comparing all media tested.

II. Oxidised sulphide flotation results

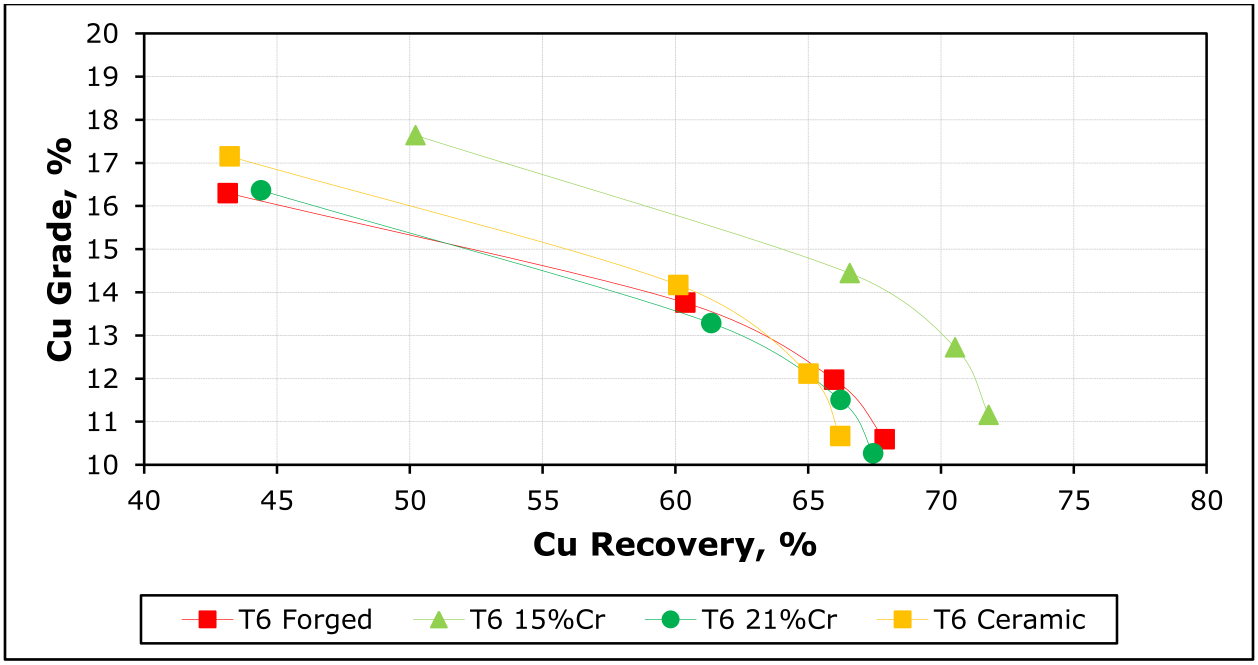

Figure 30: Copper grade recovery curves for flotation tests completed on T6 ore sample comparing all media tested.

- Iron sulphide selectivity curves.

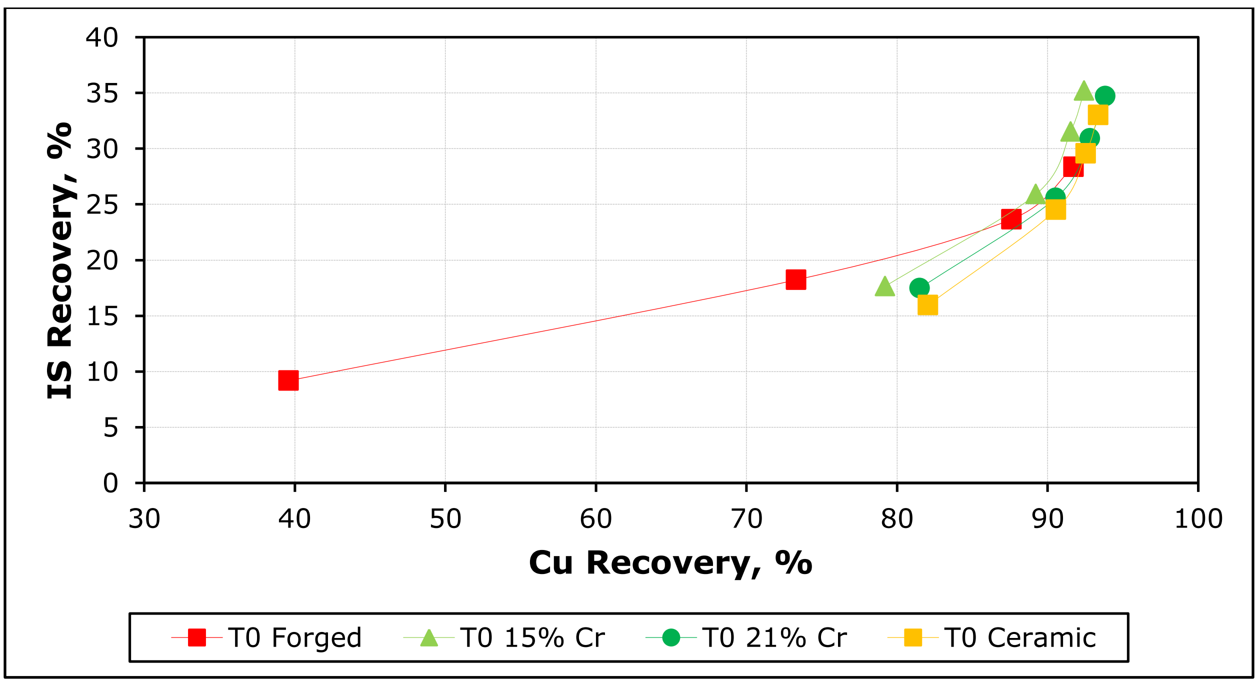

Figure 31: Iron sulphide selectivity curves against copper for flotation tests completed on T0 samples.

II. Oxidised sulphide flotation results

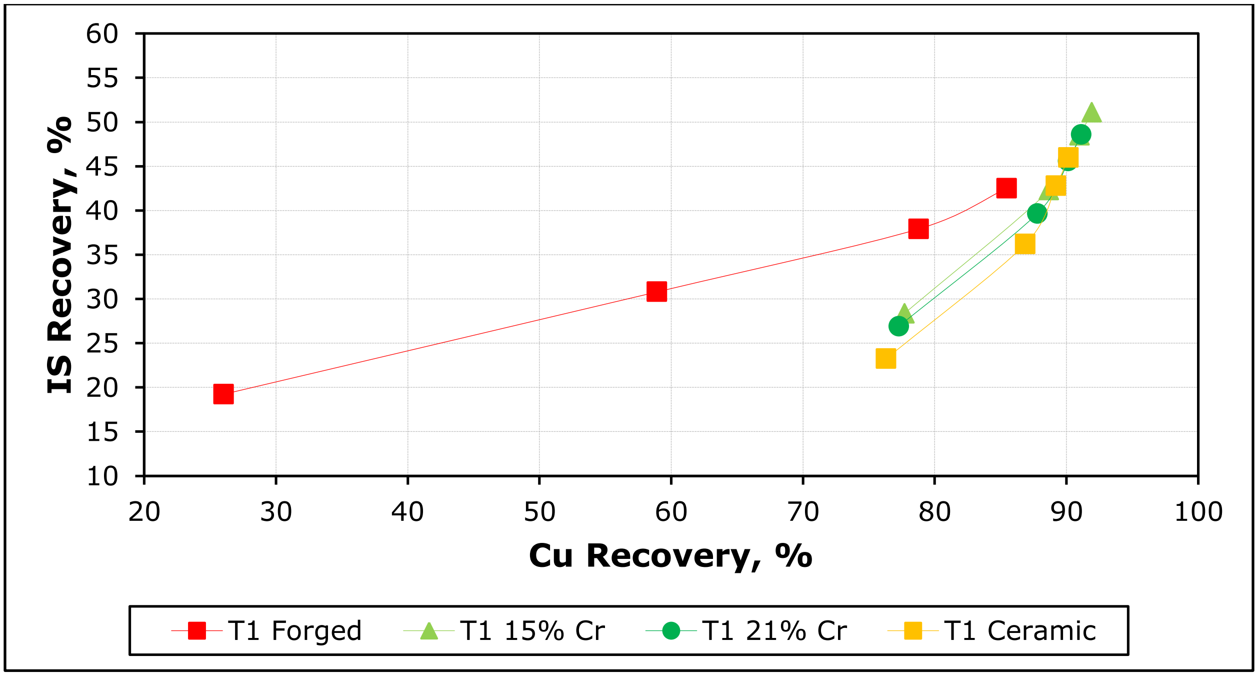

Figure 32: Iron sulphide selectivity curves against copper for flotation tests completed on T1 samples.

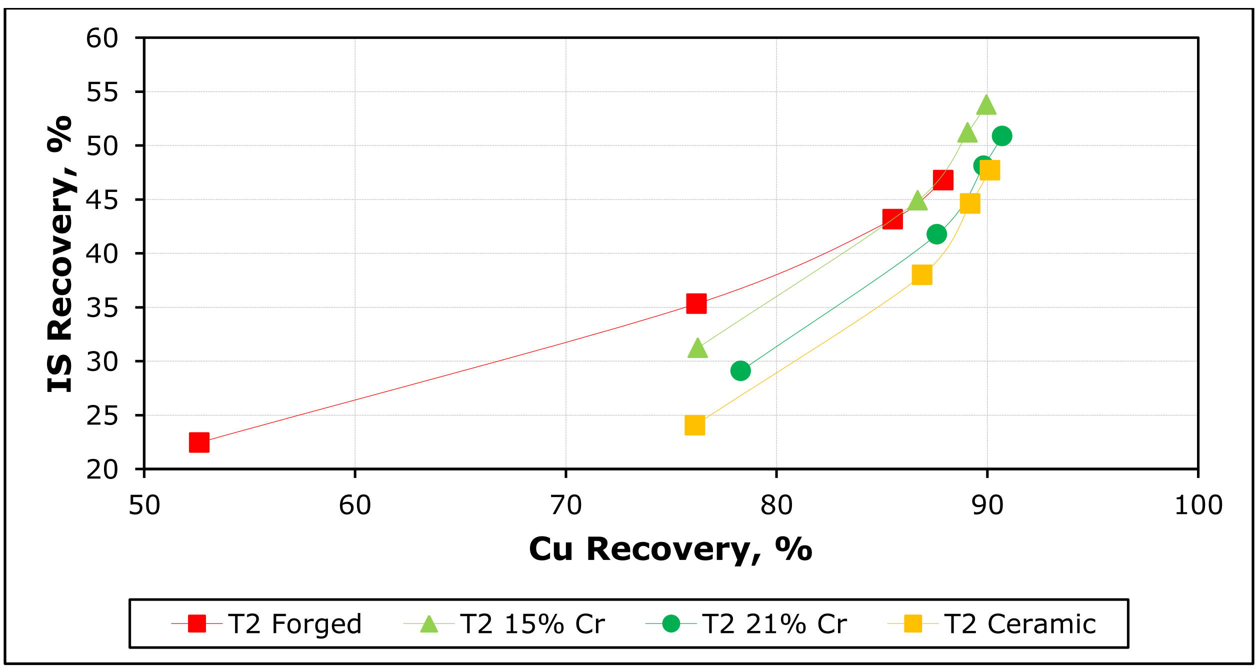

Figure 33: Iron sulphide selectivity curves against copper for flotation tests completed on T2 samples.

II. Oxidised sulphide flotation results

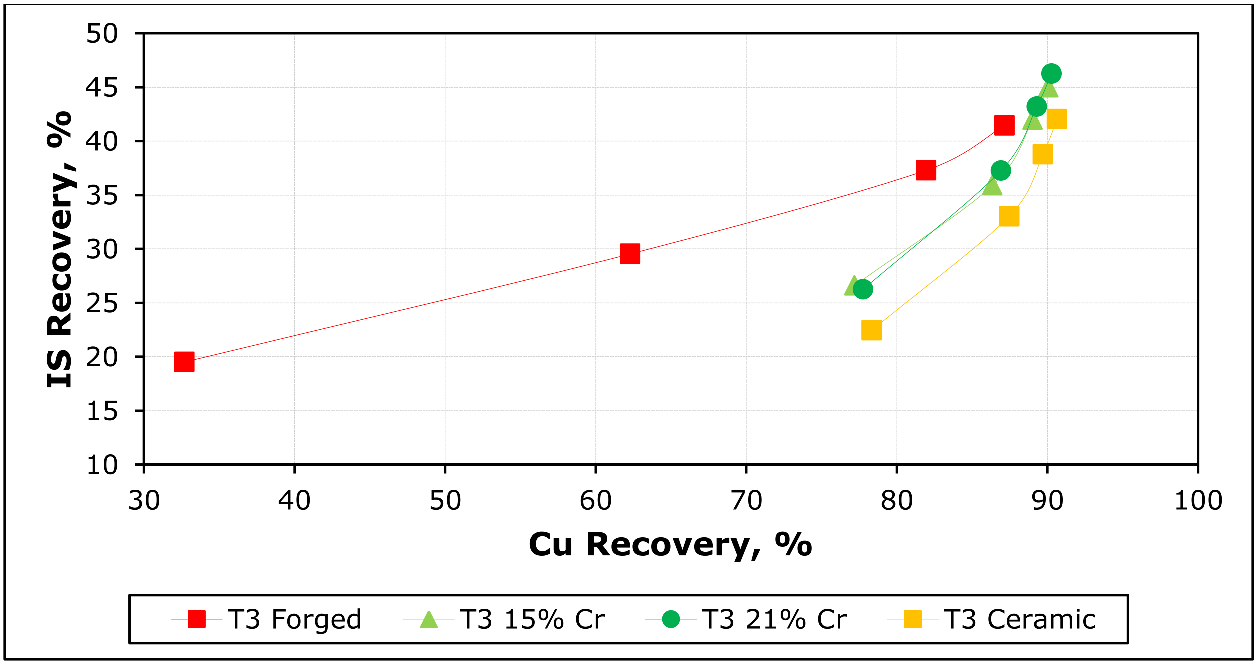

Figure 34: Iron sulphide selectivity curves against copper for flotation tests completed on T3 samples.

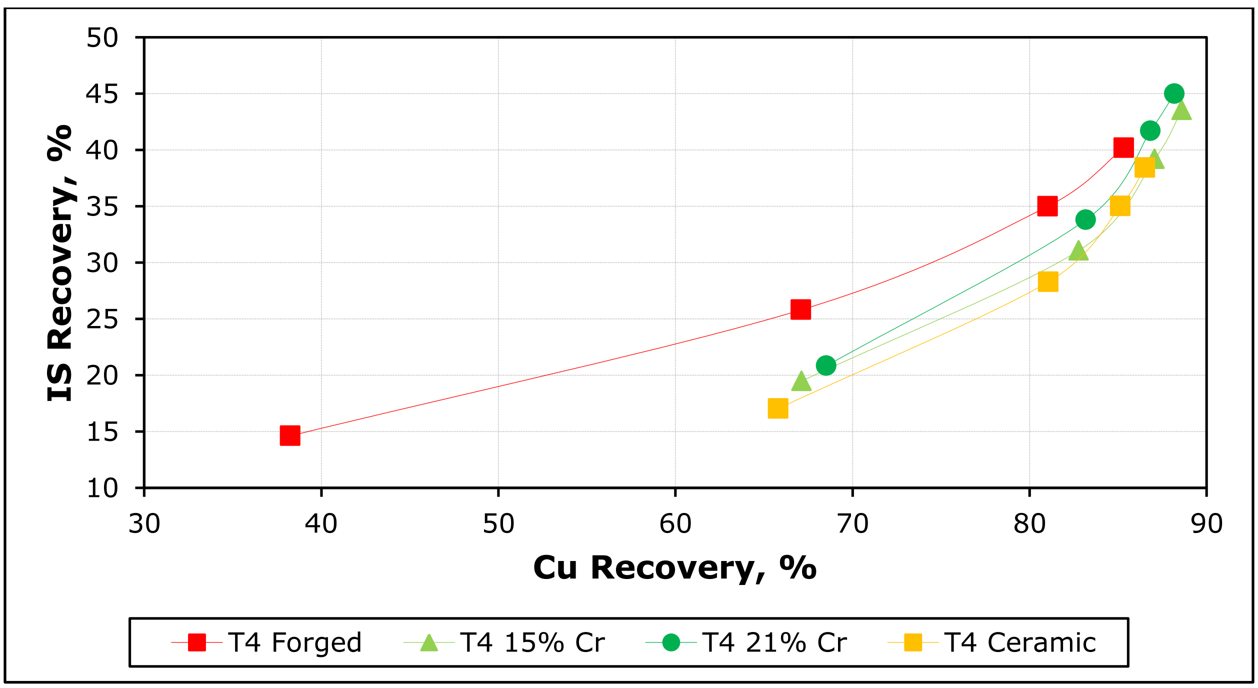

Figure 35: Iron sulphide selectivity curves against copper for flotation tests completed on T4 samples.

II. Oxidised sulphide flotation results

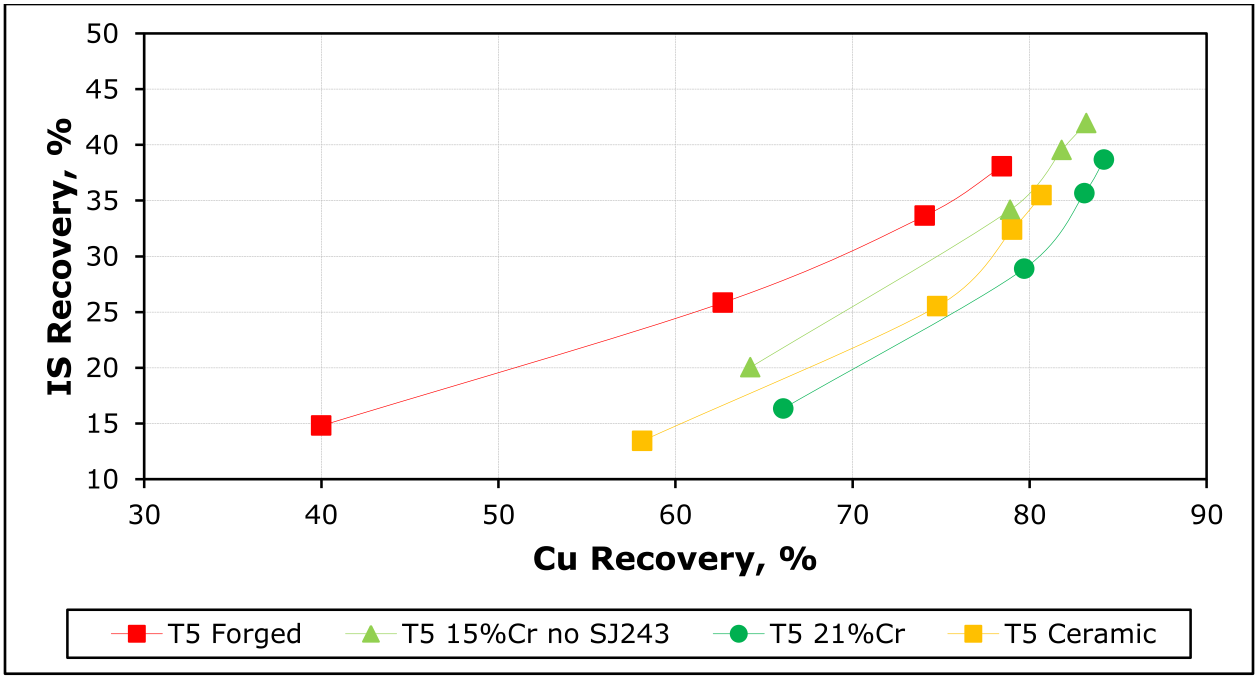

Figure 36: Iron sulphide selectivity curves against copper for flotation tests completed on T5 samples.

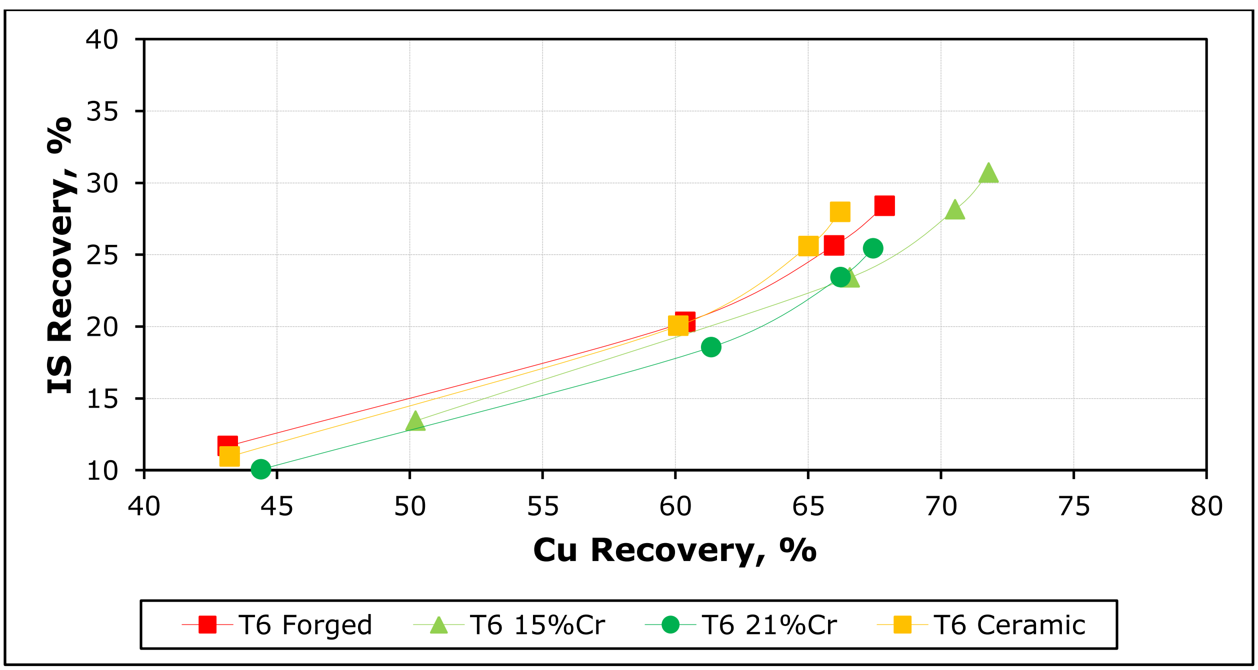

Figure 37: Iron sulphide selectivity curves against copper for flotation tests completed on T6 samples.

- Non-sulphide gangue selectivity curves.

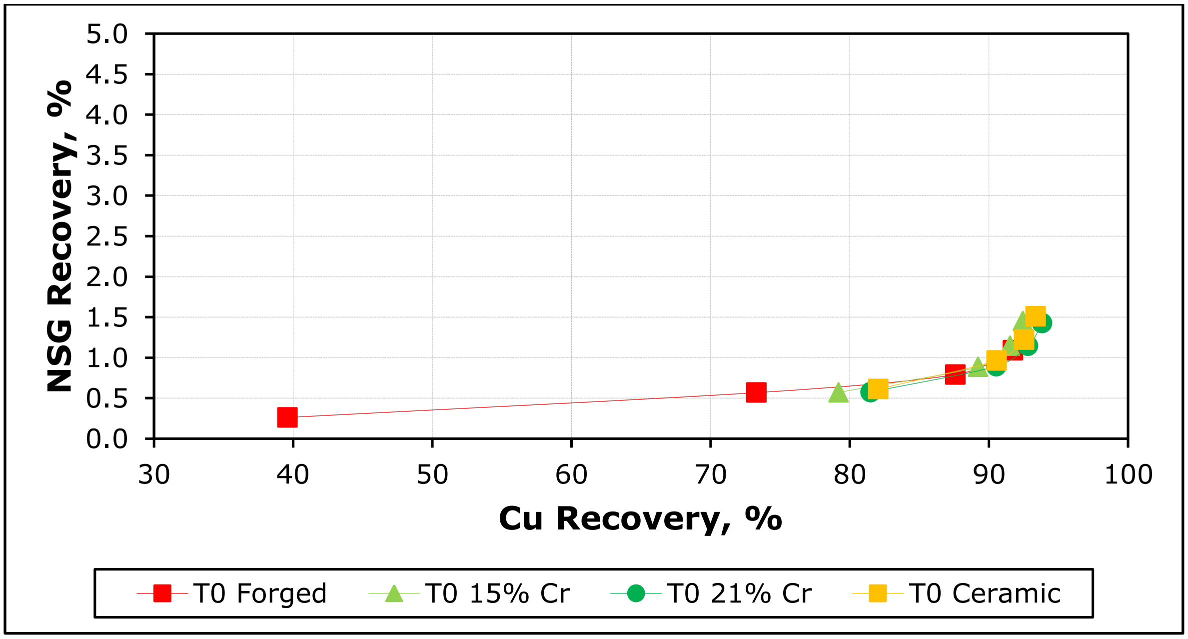

Figure 38: Non-sulphide gangue selectivity curves against copper for flotation tests completed on T0 samples.

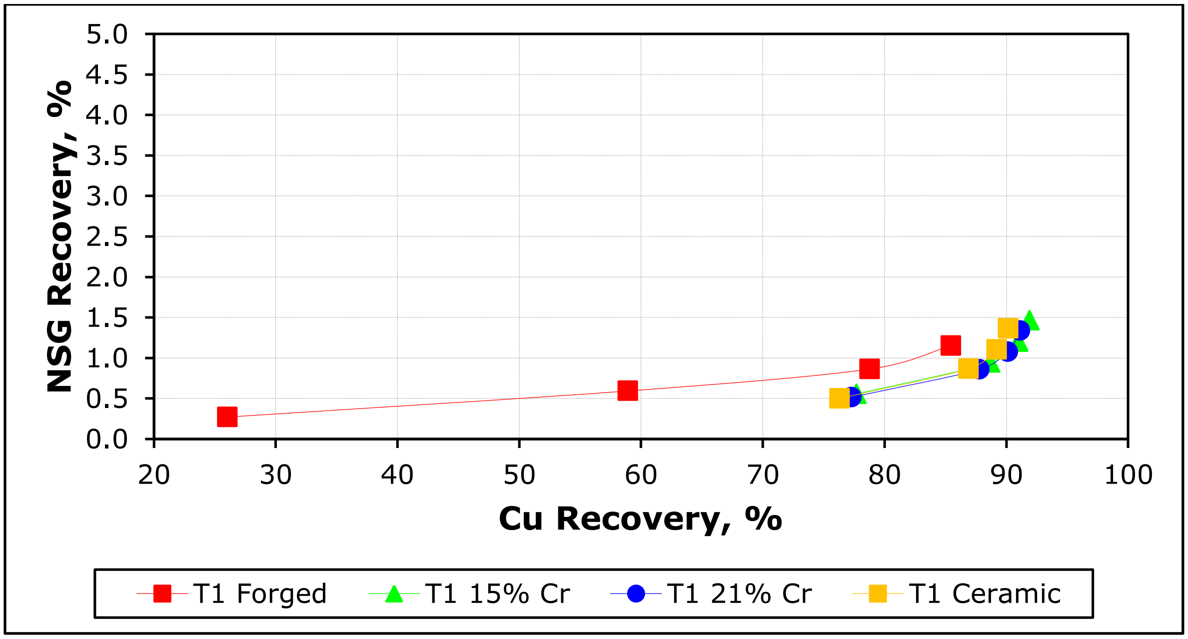

Figure 39: Non-sulphide gangue selectivity curves against copper for flotation tests completed on T1 samples.

II. Oxidised sulphide flotation results

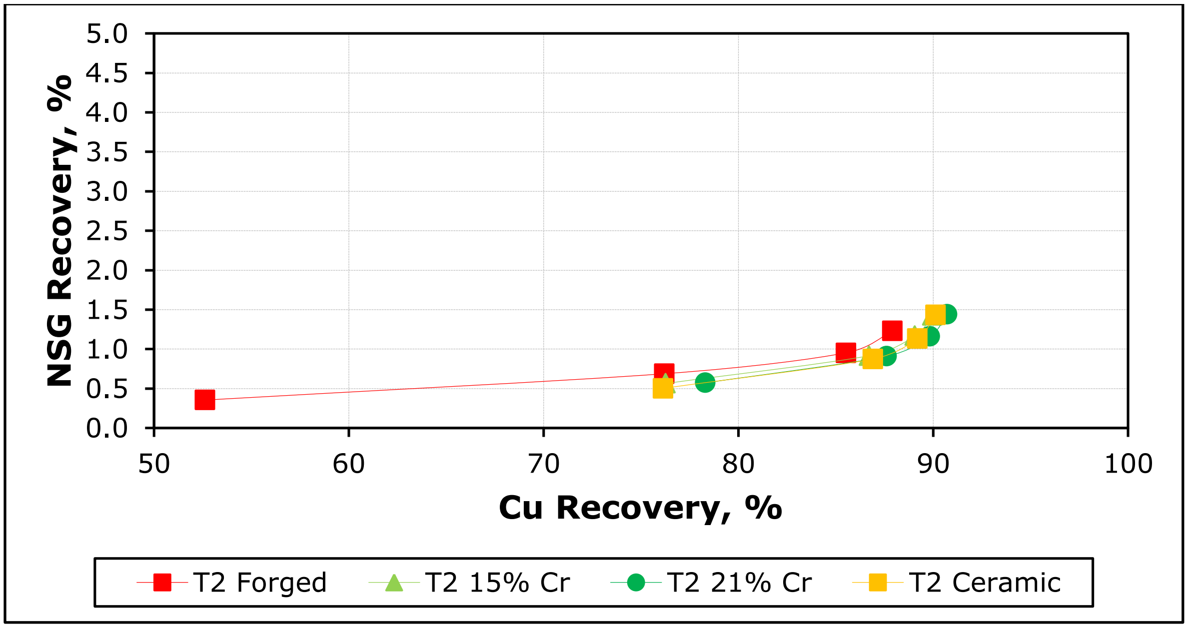

Figure 40: Non-sulphide gangue selectivity curves against copper for flotation tests completed on T2 samples.

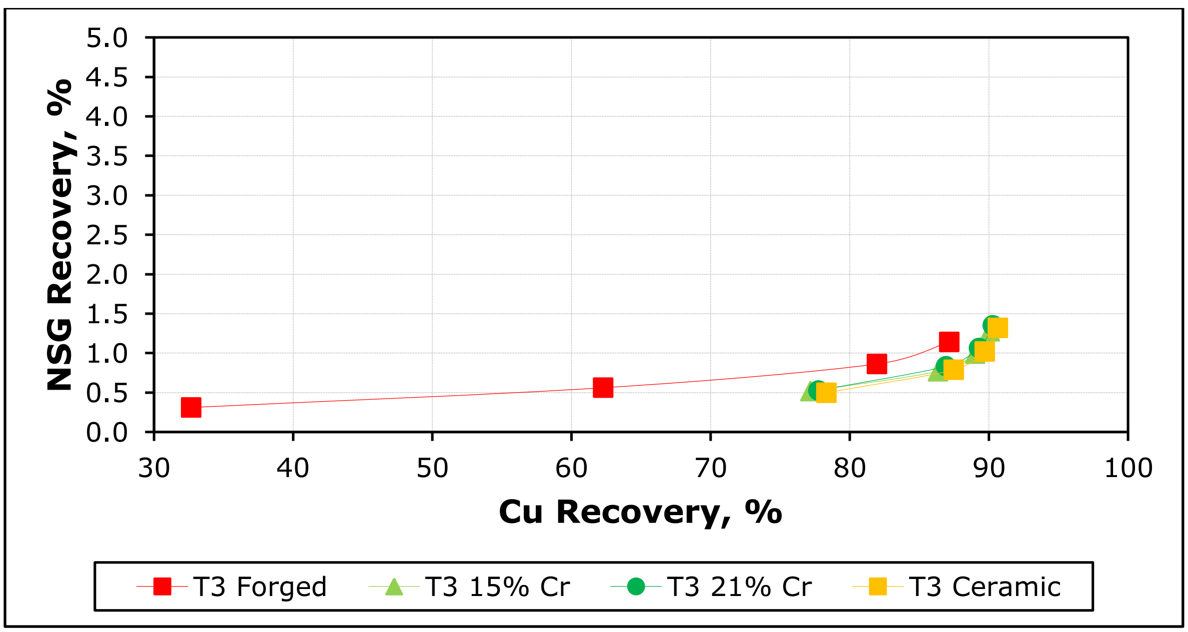

Figure 41: Non-sulphide gangue selectivity curves against copper for flotation tests completed on T3 samples.

II. Oxidised sulphide flotation results

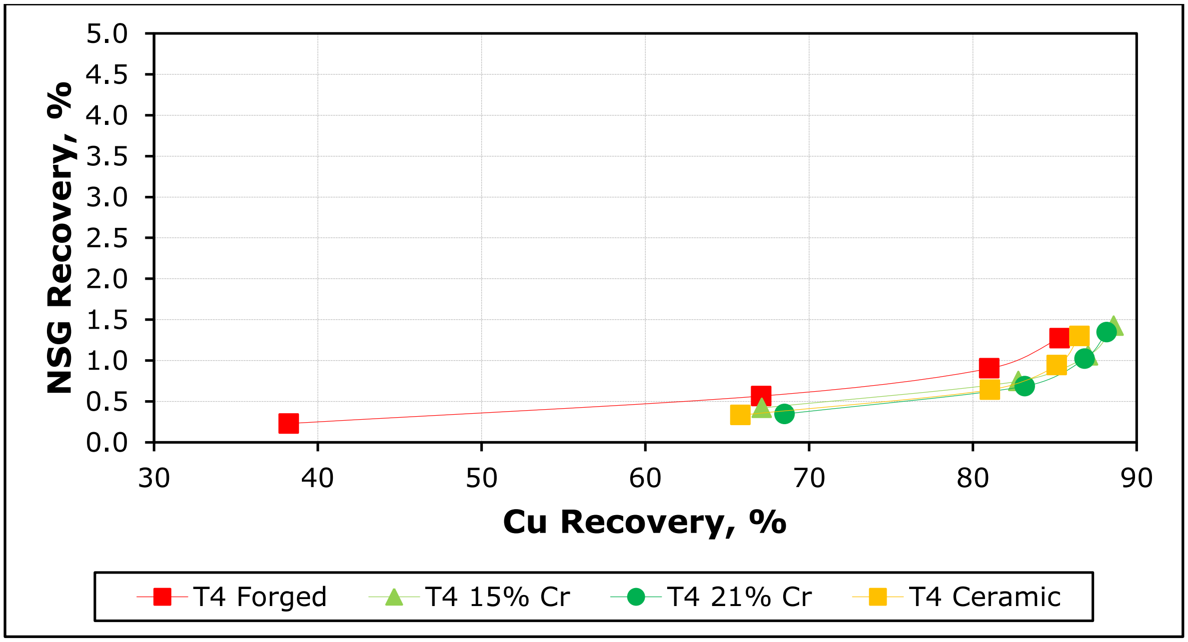

Figure 42: Non-sulphide gangue selectivity curves against copper for flotation tests completed on T4 samples.

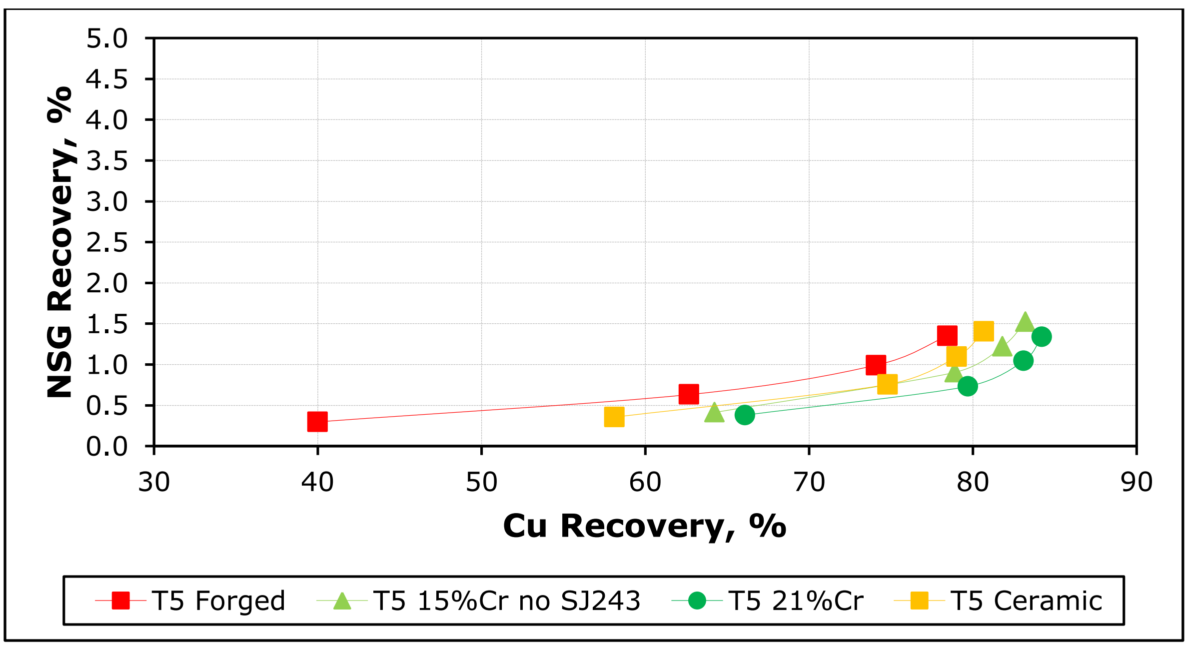

Figure 43: Non-sulphide gangue selectivity curves against copper for flotation tests completed on T5 samples.

II. Oxidised sulphide flotation results

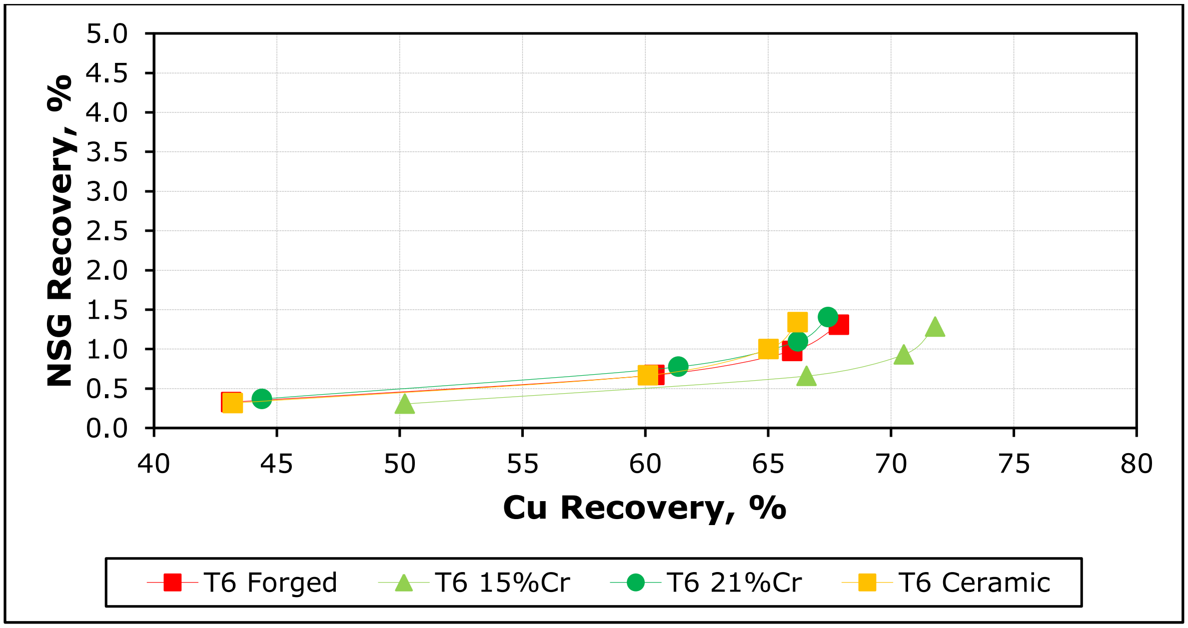

Figure 44: Non-sulphide gangue selectivity curves against copper for flotation tests completed on T6 samples.

2.4.3. Size by size recovery

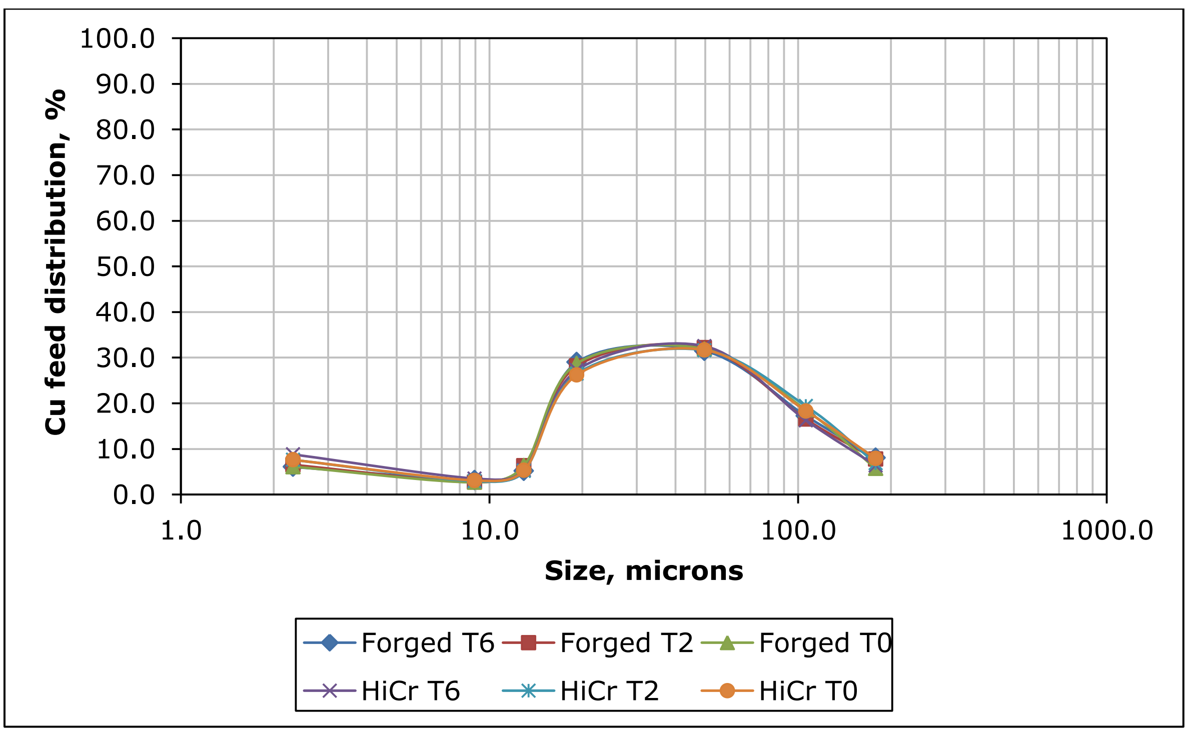

Figure 45: Copper feed distribution by size for T0, T2 and T6 samples ground with forged steel and 21% high chrome media.

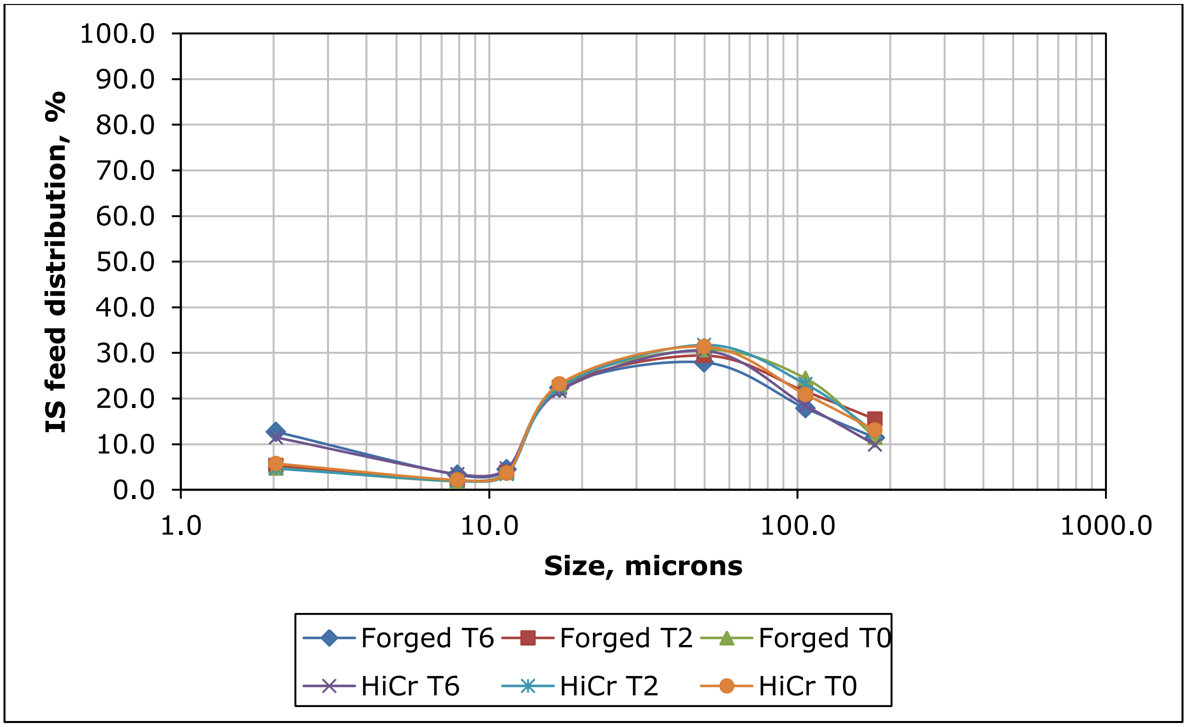

Figure 46: Iron sulphide feed distribution by size for T0, T2 and T6 samples ground with forged steel and 21% high chrome media.

II. Oxidised sulphide flotation results

III. SINGLE MALACHITE ORE

3.1. Grinding calibration

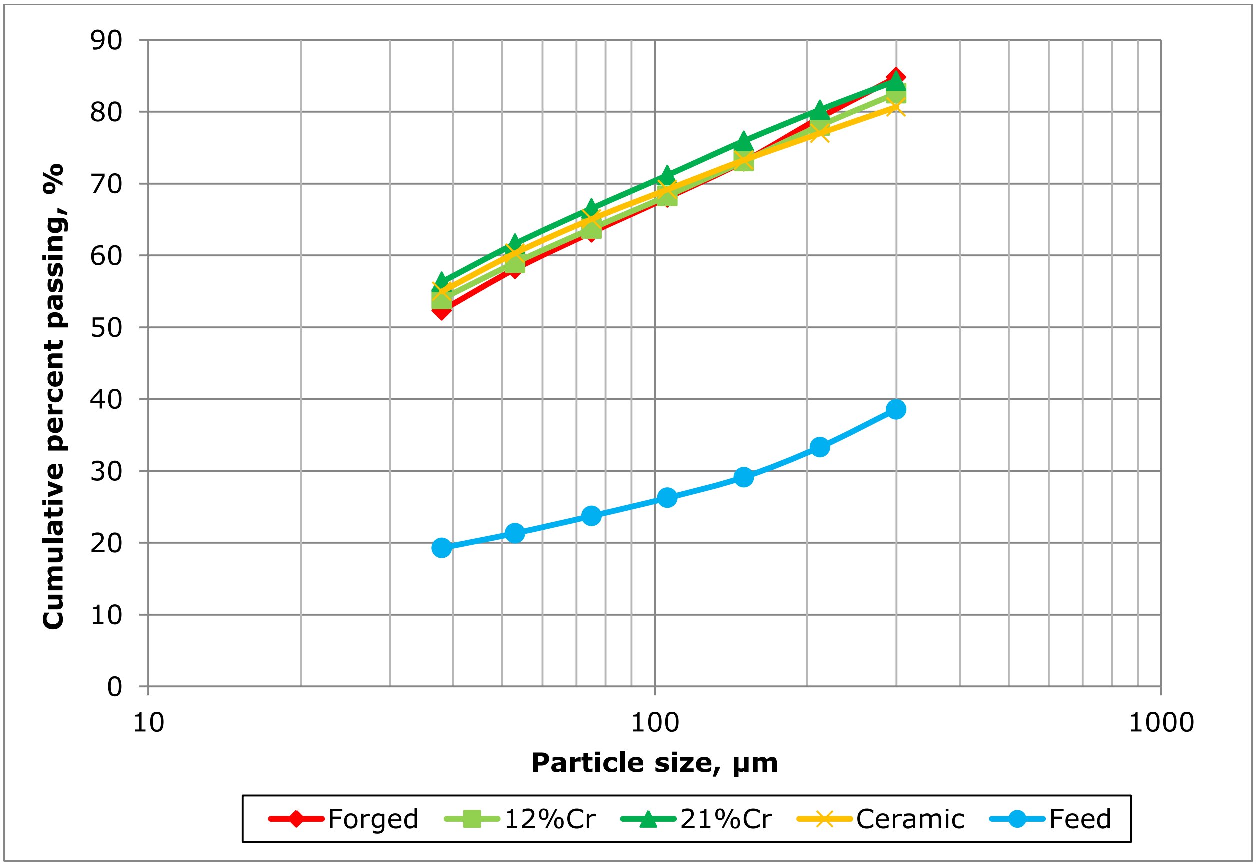

Figure 47: Size distribution curves for the bulk malachite ore ground in the Magotteaux Mill® with forged steel, 12% Cr, 21% Cr and ceramic grinding media. Unground feed sample shown for comparison.

Table 5: Magotteaux Mill® parameters and P_{65} for forged steel, 12%Cr, 21% Cr and ceramic grinding media.

Media Type	Media Charge, Kg	Mill Speed, rpm	Passing 65 µm, %
Forged	10 x balls	20	63.2
12%Cr	10 x balls	21	63.7
21%Cr	10 x balls	21	66.5
Ceramic	10 x balls	19	65.1

3.2. Grinding chemistry

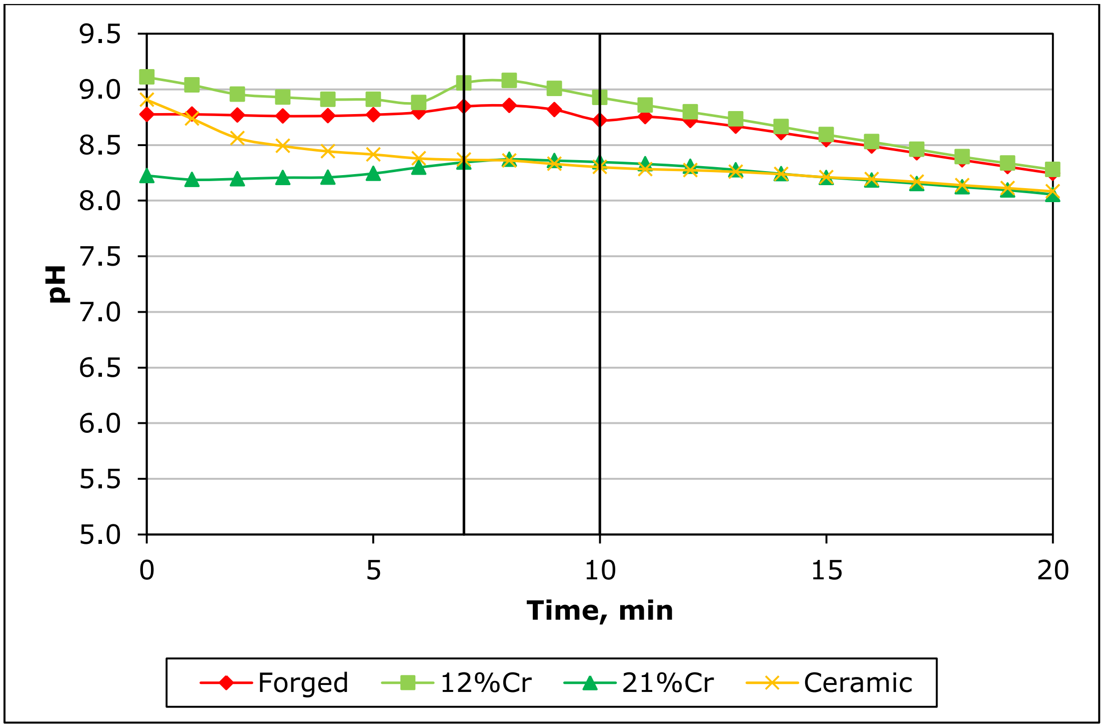

Figure 48: pH trends during grinding using various grinding media, bulk malachite ore.

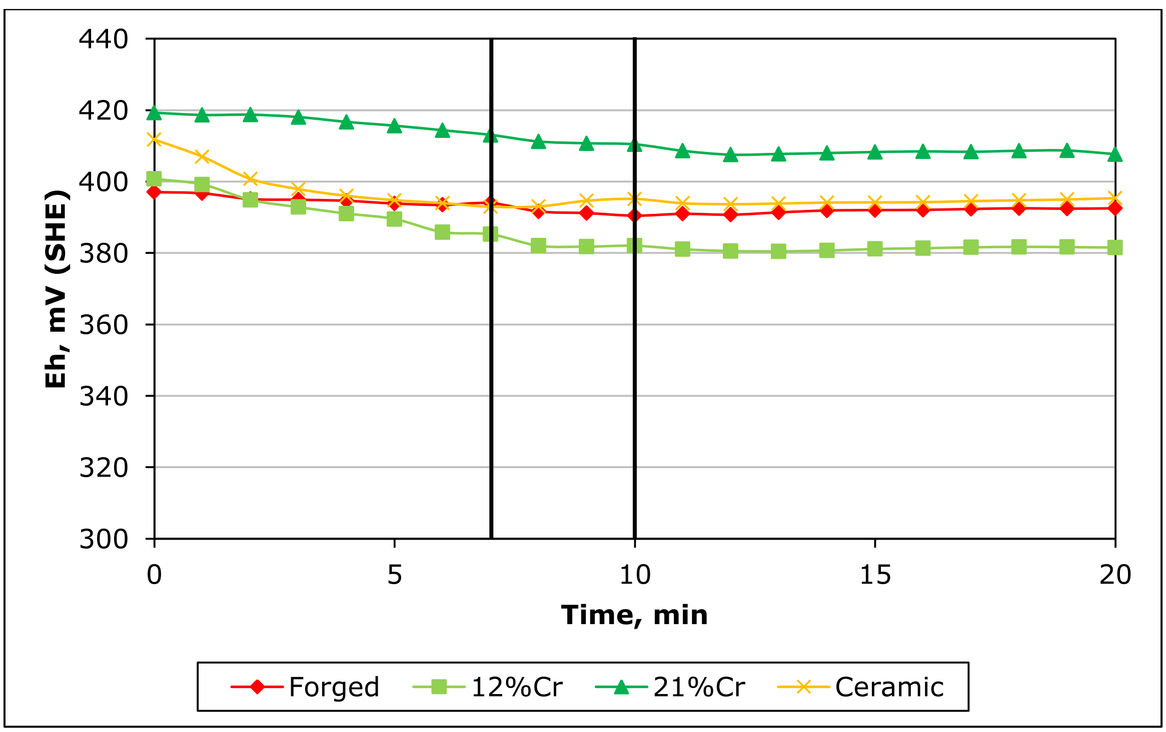

Figure 49: Eh trends during grinding using various grinding media, bulk malachite ore.

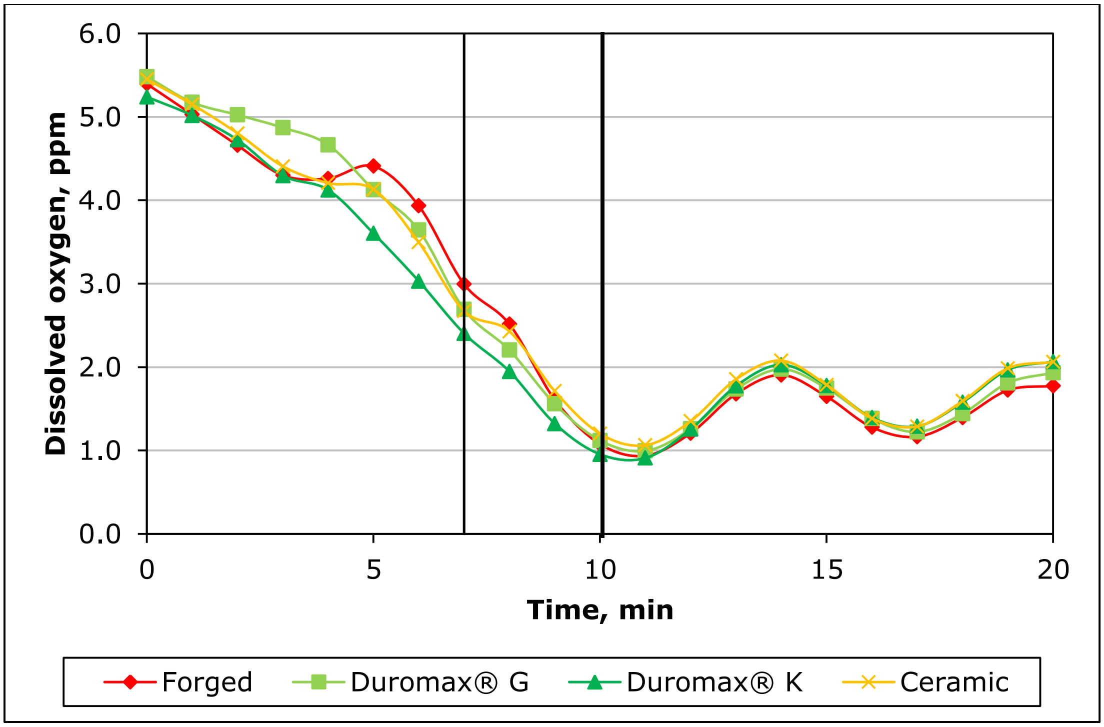

Figure 50: Dissolved oxygen trends during grinding using various grinding media, bulk malachite ore.

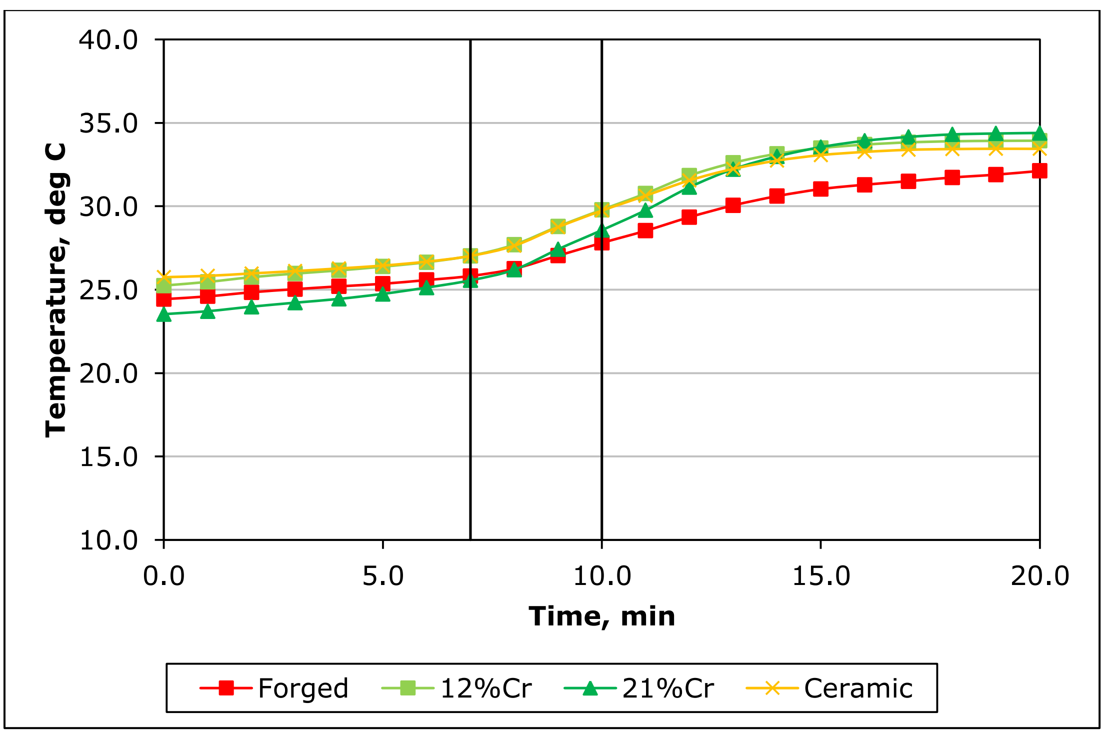

Figure 51: Temperature trends during grinding using various grinding media, bulk malachite ore.

III. Single malachite ore

3.3. Flotation chemistry

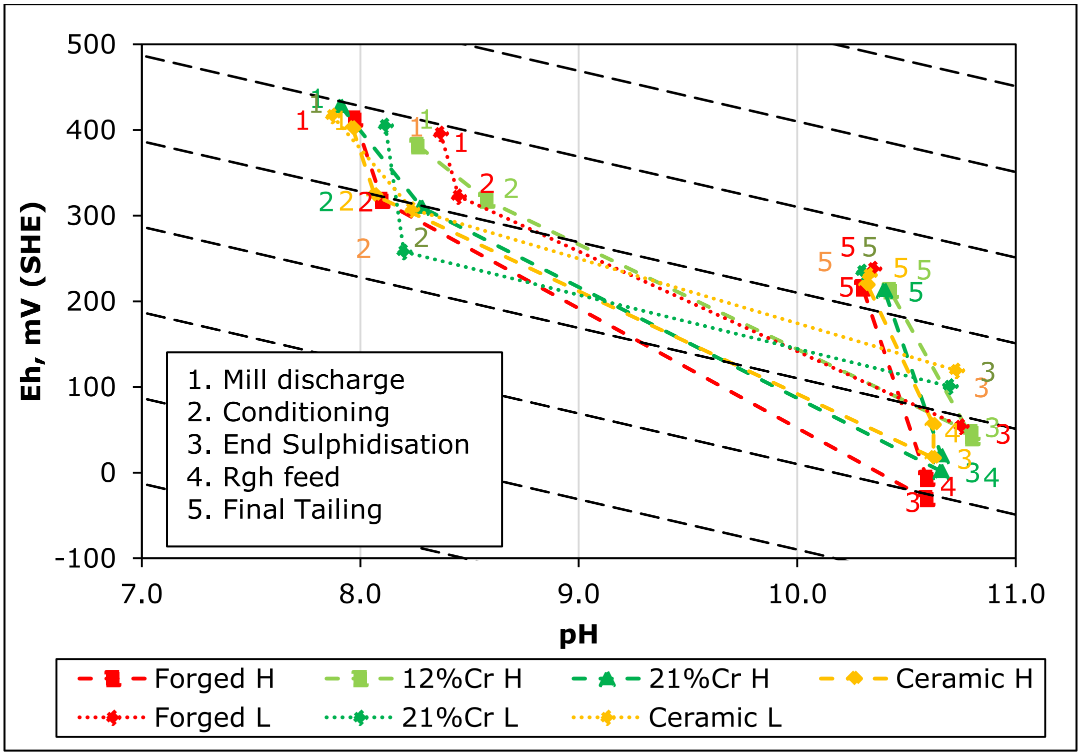

Figure 52: Eh-pH profiles during flotation tests of the bulk malachite ore comparing various grinding media.

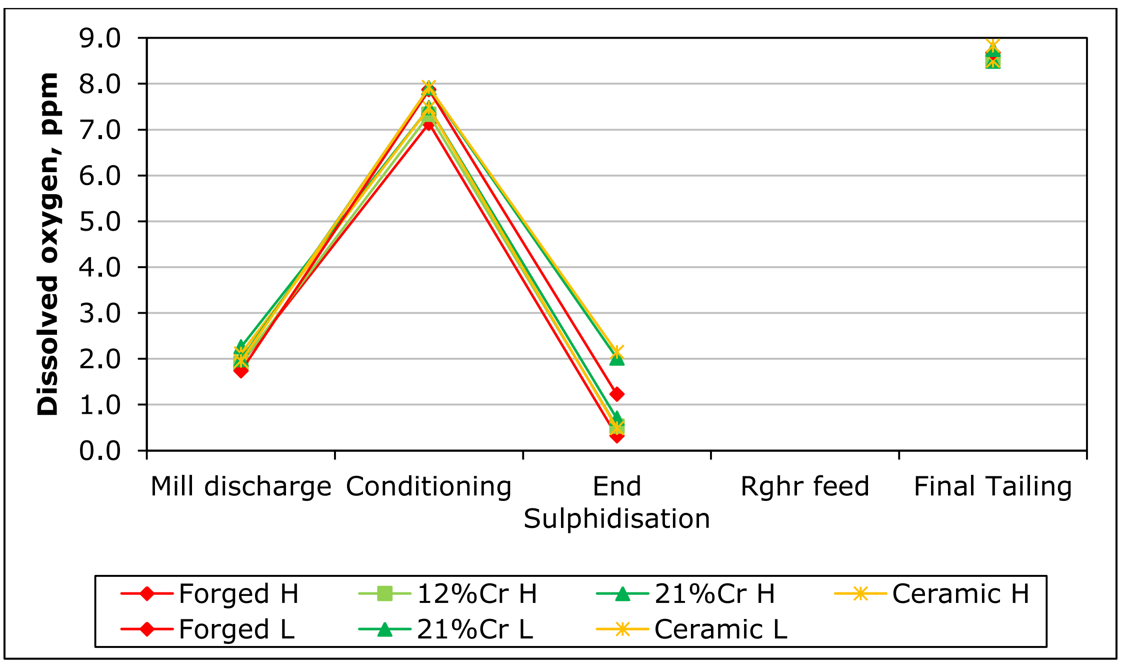

Figure 53: Dissolved oxygen profiles during flotation tests of the bulk malachite ore comparing various grinding media.

3.4. Flotation results:

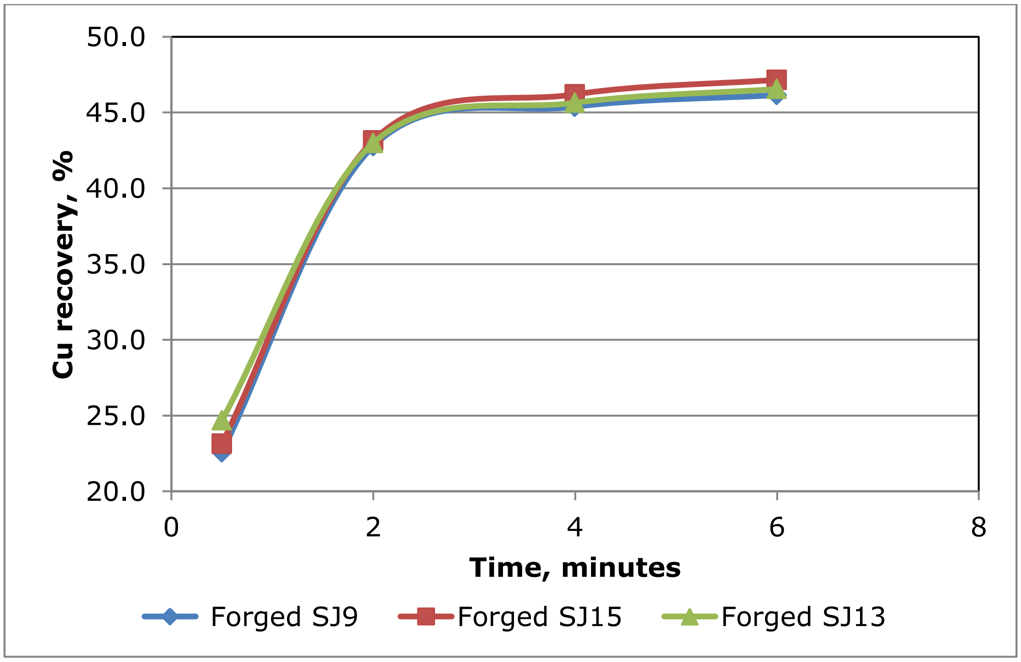

Figure 54: Copper recovery versus time curves for tests completed with forged steel media, bulk malachite ore sample.

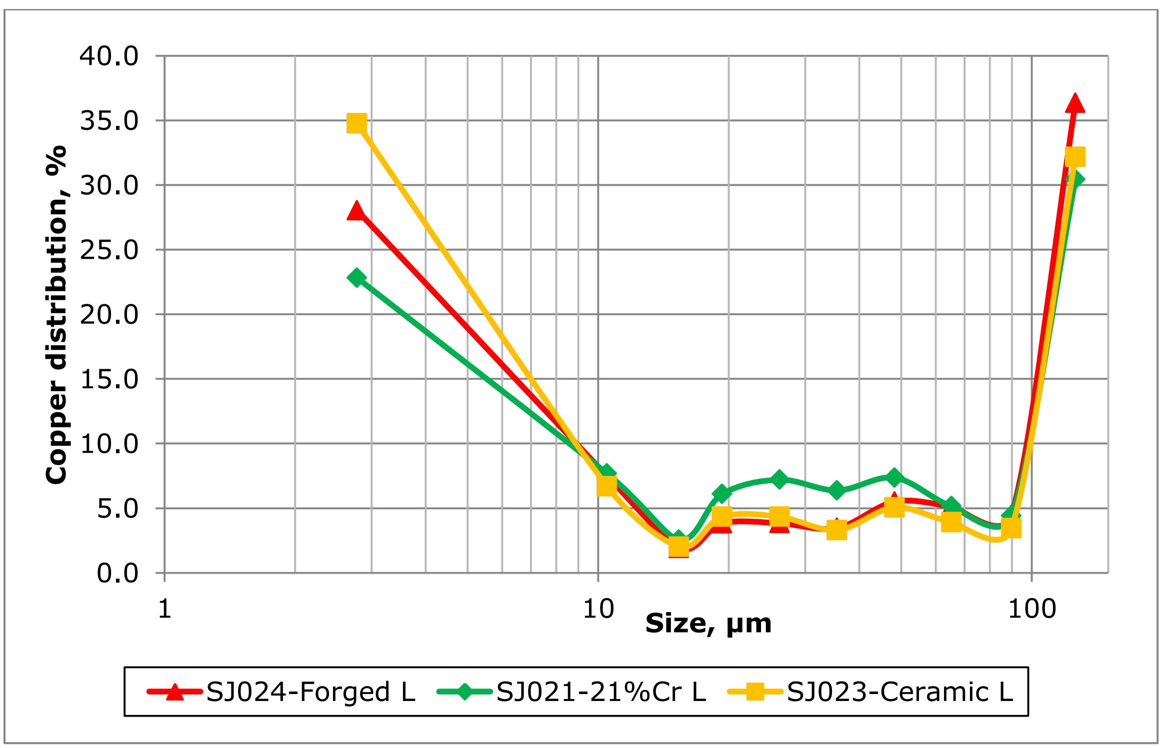

Figure 55: Copper feed distribution by size for all media tested.

III. Single malachite ore

3.5. XPS analysis

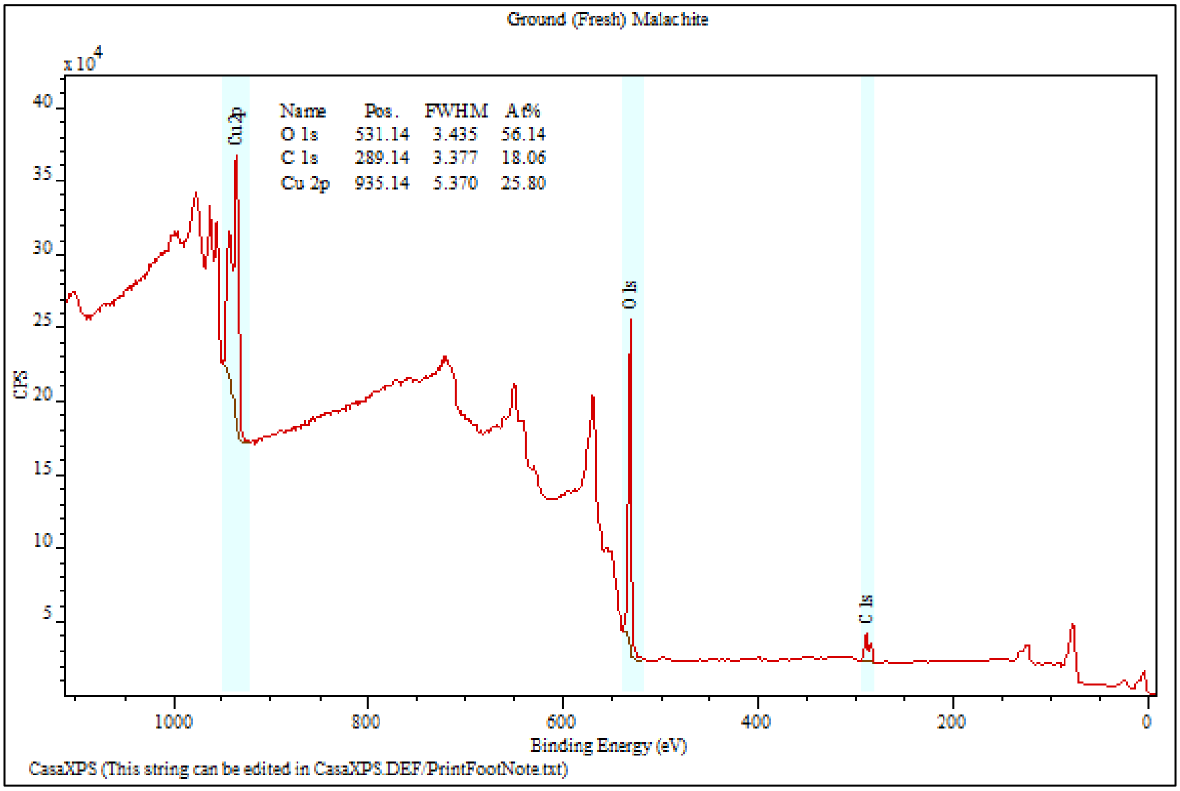

Figure 56: XPS general spectrum of malachite before sulphidisation.

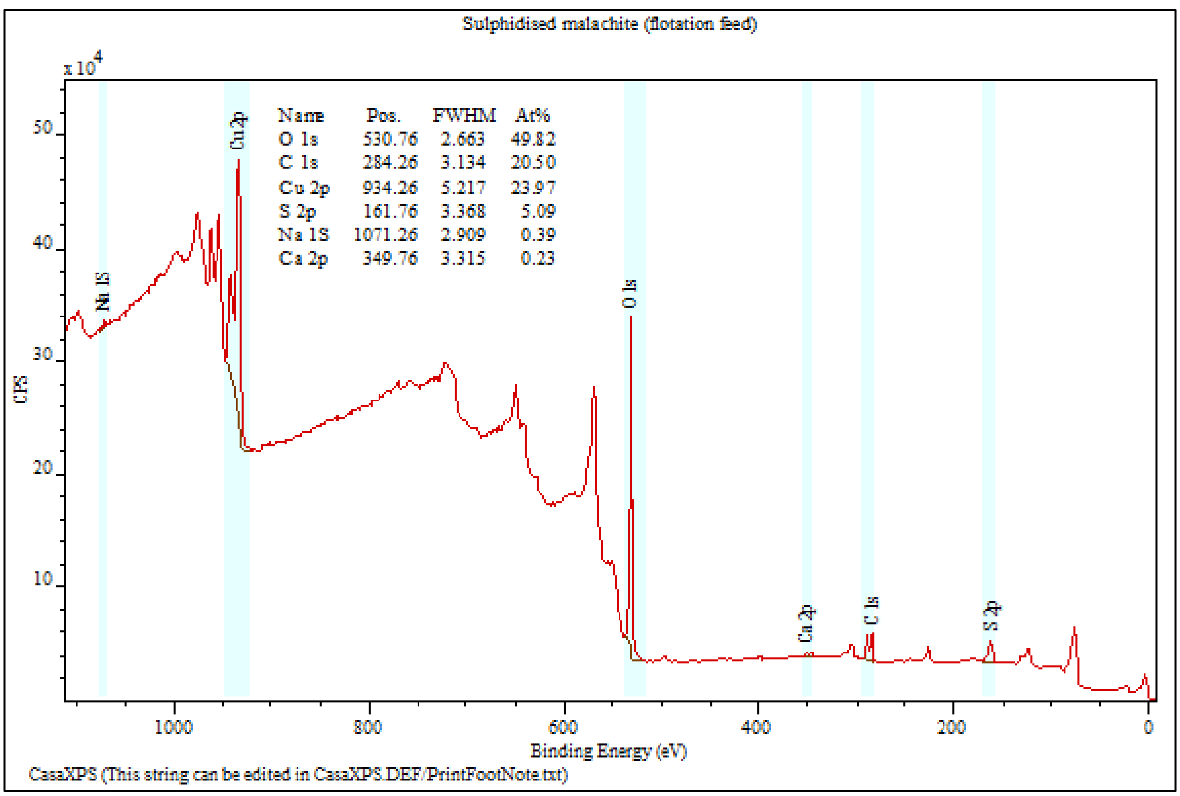

Figure 57: XPS general spectrum of malachite after sulphidisation.

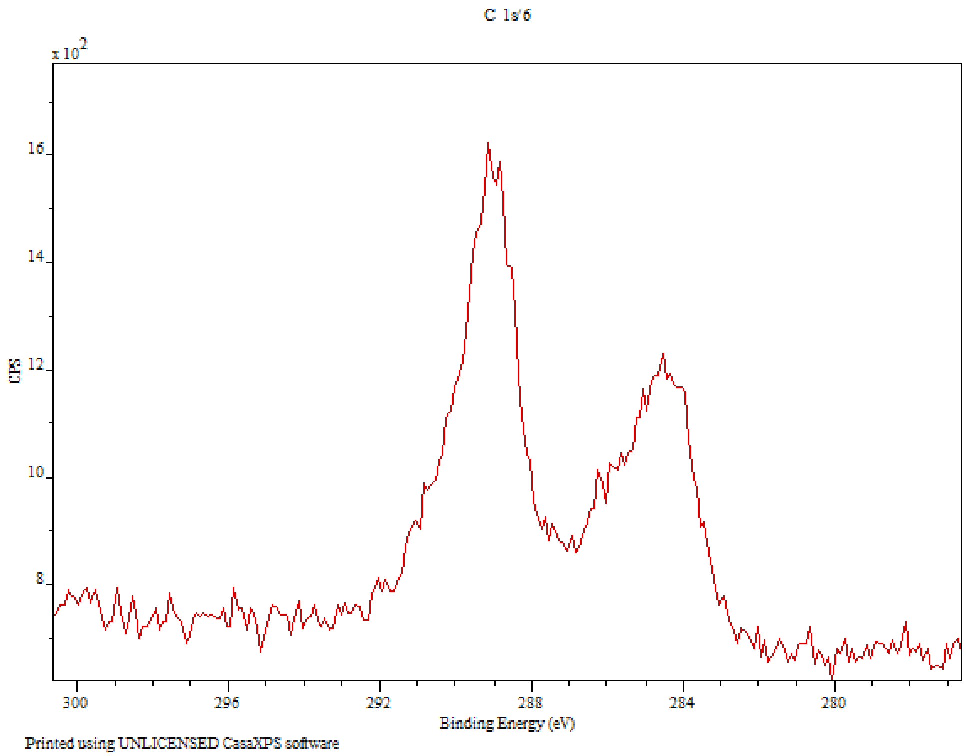

Figure 58: C 1S high resolution spectrum of malachite before sulphidisation.

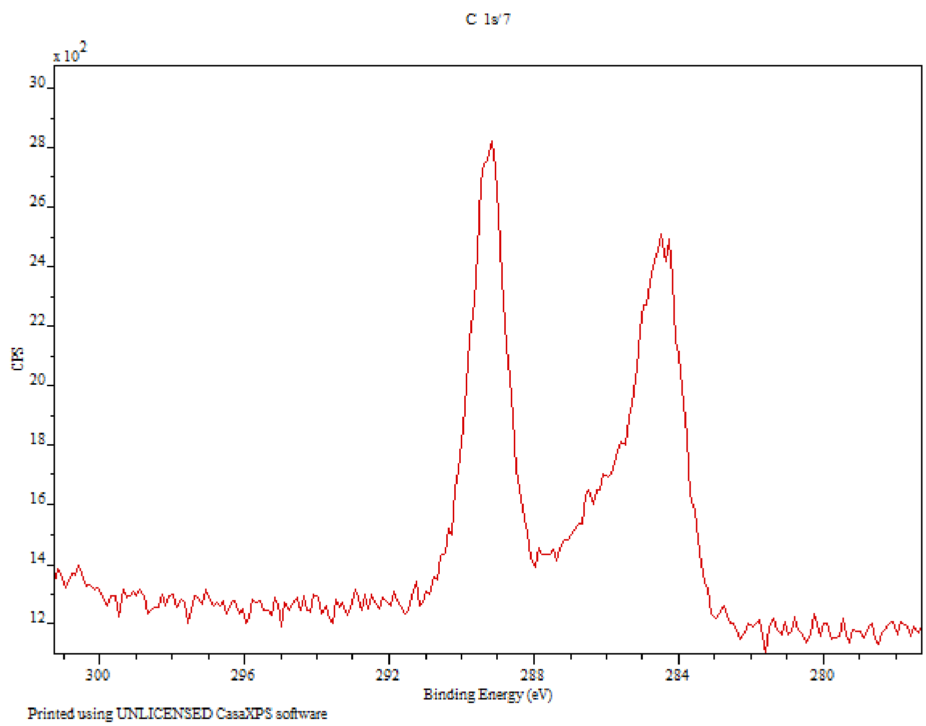

Figure 59: C 1S high resolution spectrum of malachite after sulphidisation.

III. Single malachite ore

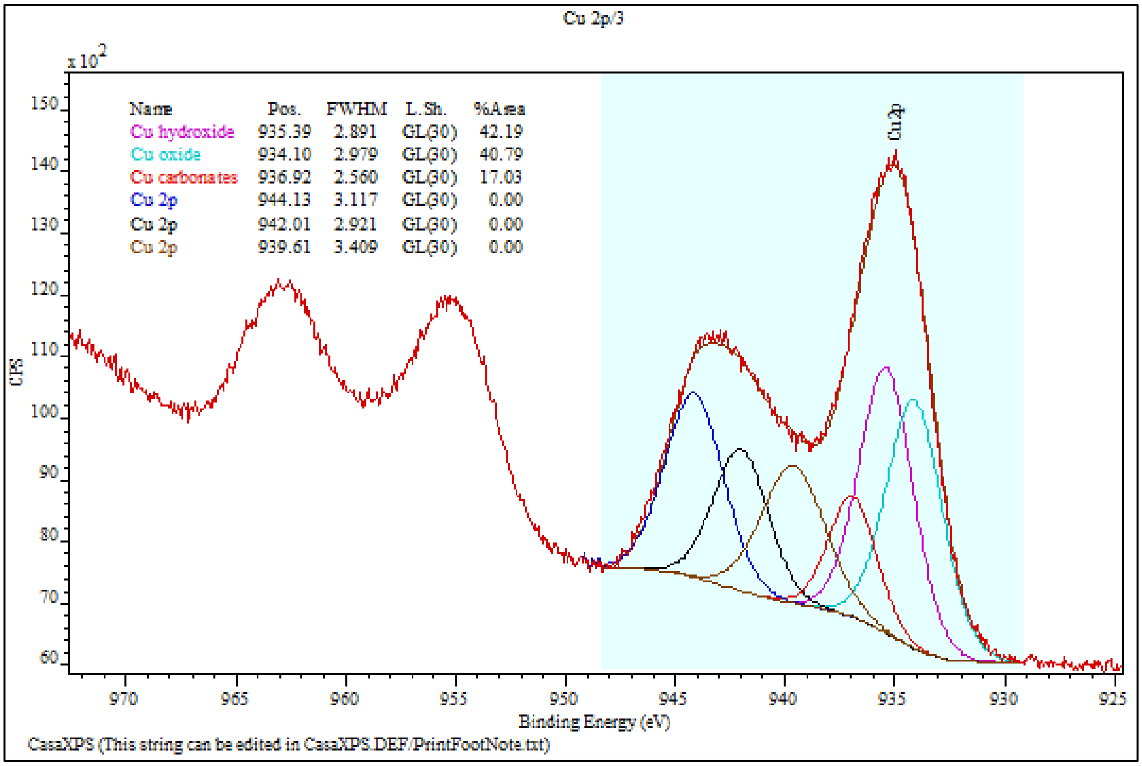

Figure 60: Cu 2p$_{3/2}$ high resolution spectrum using peak fitting of malachite before sulphidisation.

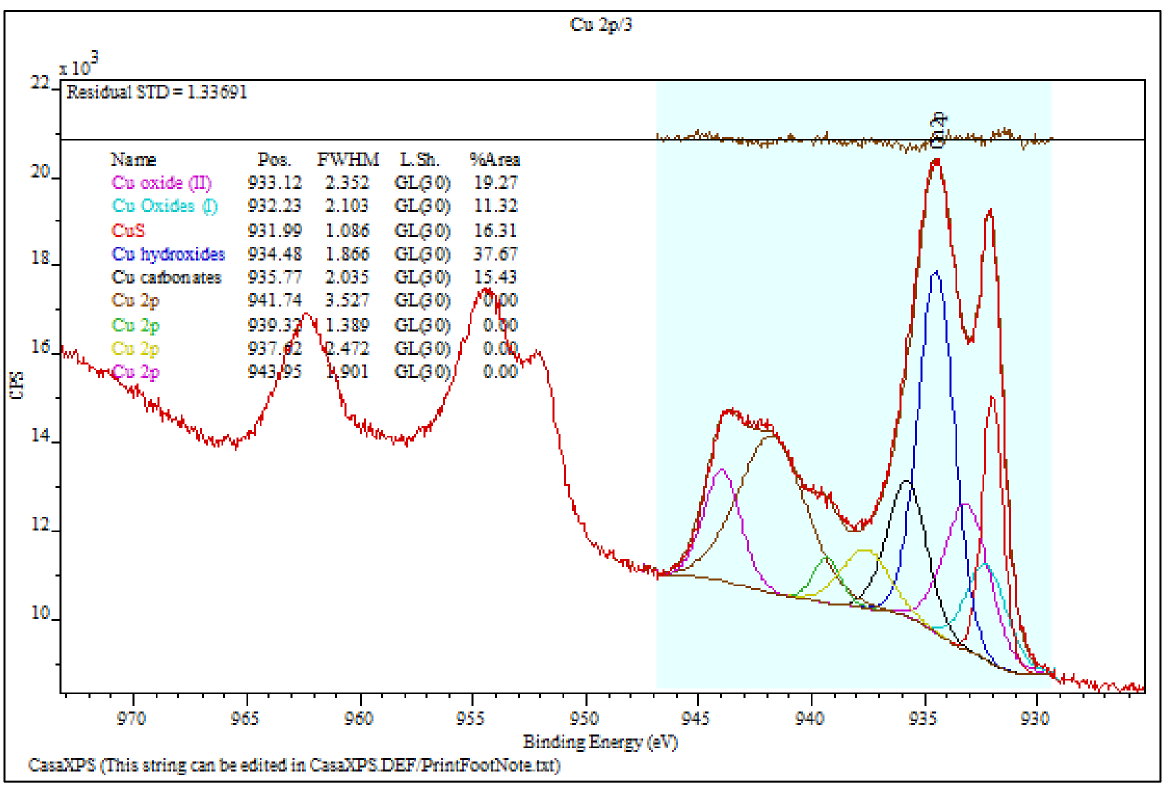

Figure 61: Cu 2p$_{3/2}$ high resolution spectrum using peak fitting of malachite after sulphidisation.

III. Single malachite ore

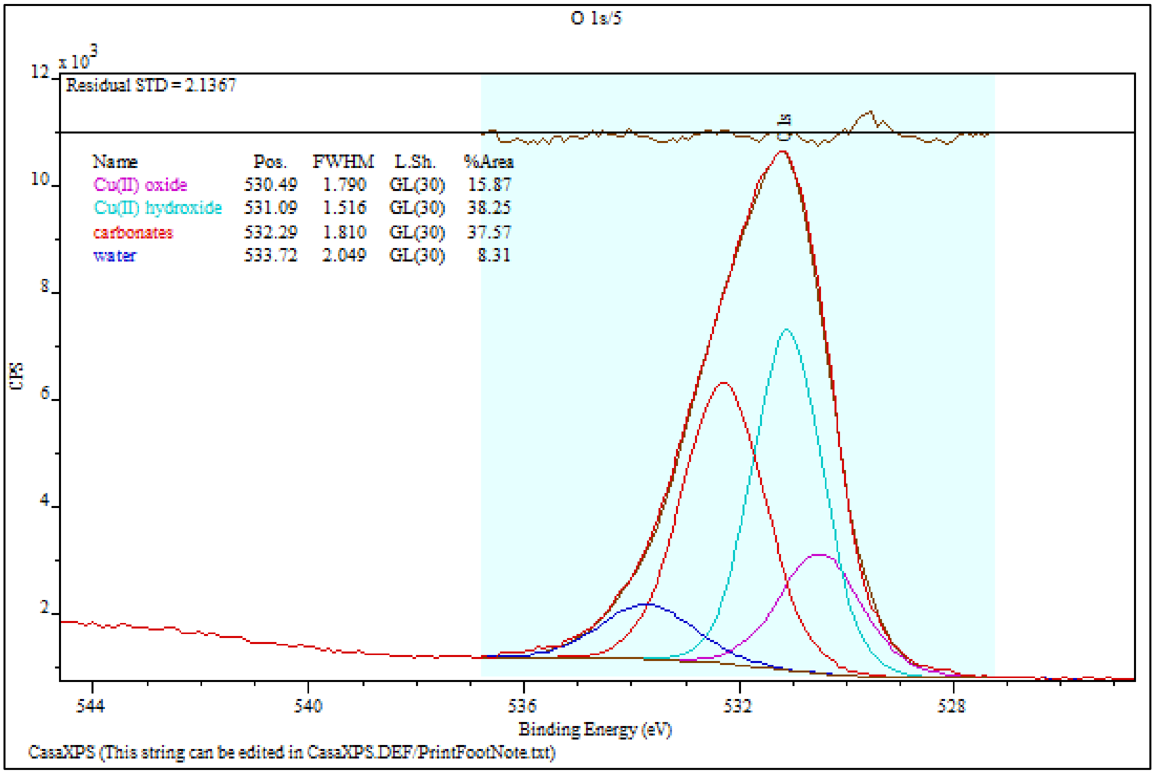

Figure 62: O 1S high resolution spectrum using peak fitting of malachite before sulphidisation.

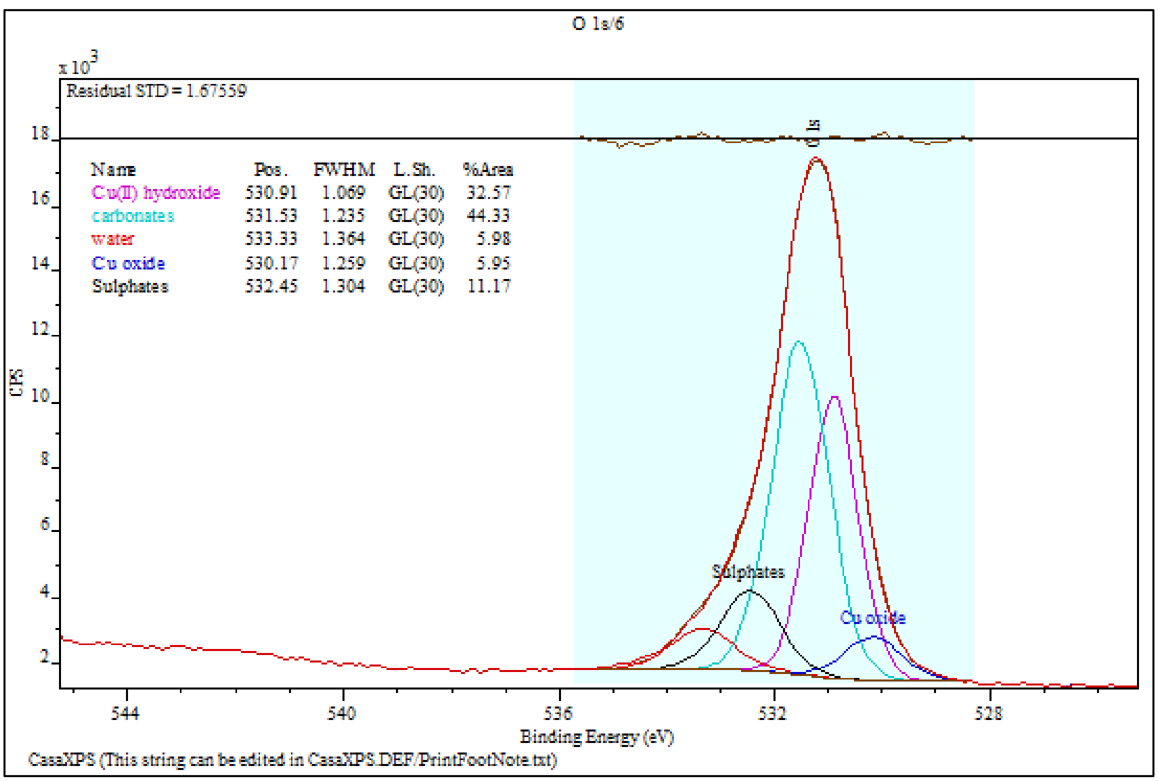

Figure 63: O 1s high resolution spectrum using peak fitting of malachite after sulphidisation.

III. Single malachite ore

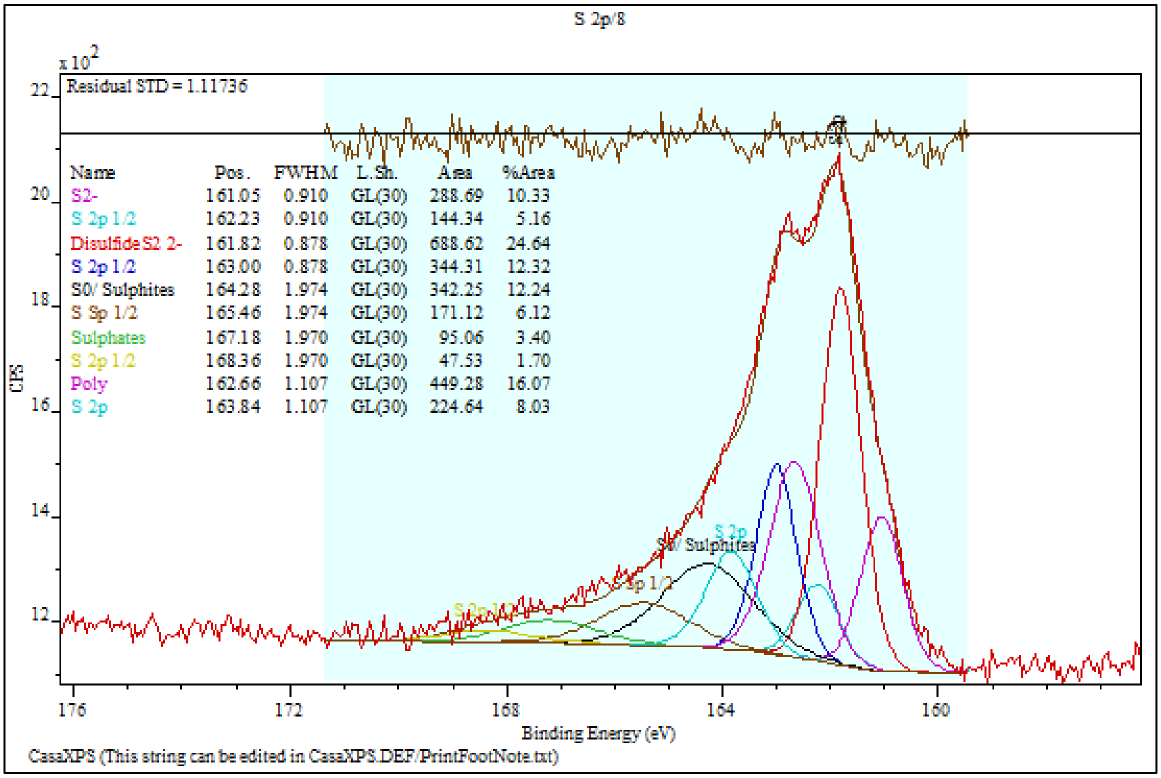

Figure 64: S $2p_{3/2}$ high resolution spectrum using peak fitting of sulphidised malachite.

IV. SYNTHETIC OXIDE ORE

4.1. Grinding calibration

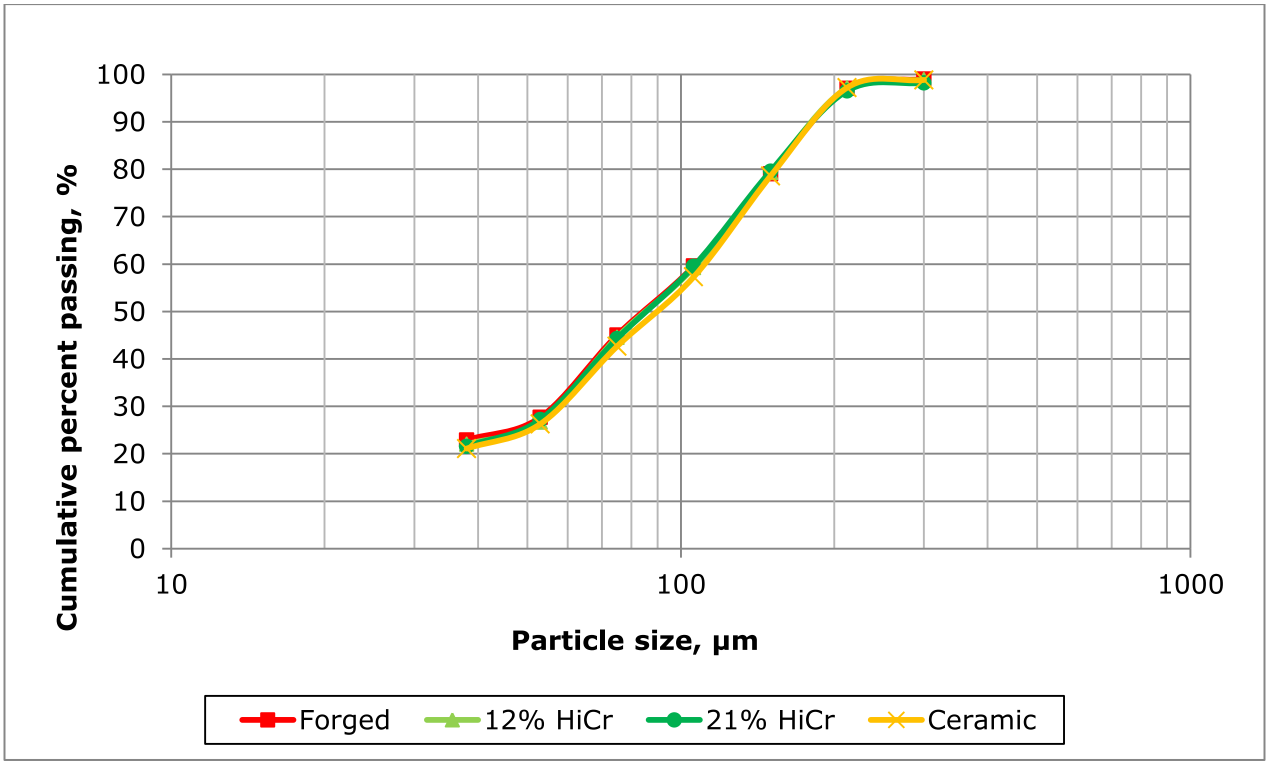

Figure 65: Cumulative size distribution curves for all media tested, oxide synthetic ore.

Table 6: Magotteaux Mill® parameters and P_{80} for forged steel, 12% HiCr, 21% HiCr and ceramic grinding media.

Media Type	Media Charge, Kg	Mill Speed, rpm	P_{80}, µm
Forged	20 x balls	41	153.3
12%Cr	20 x balls	43	152.1
21%Cr	20 x balls	45	151.5
Ceramic	20 x balls	47	151.8

4.2. Grinding chemistry

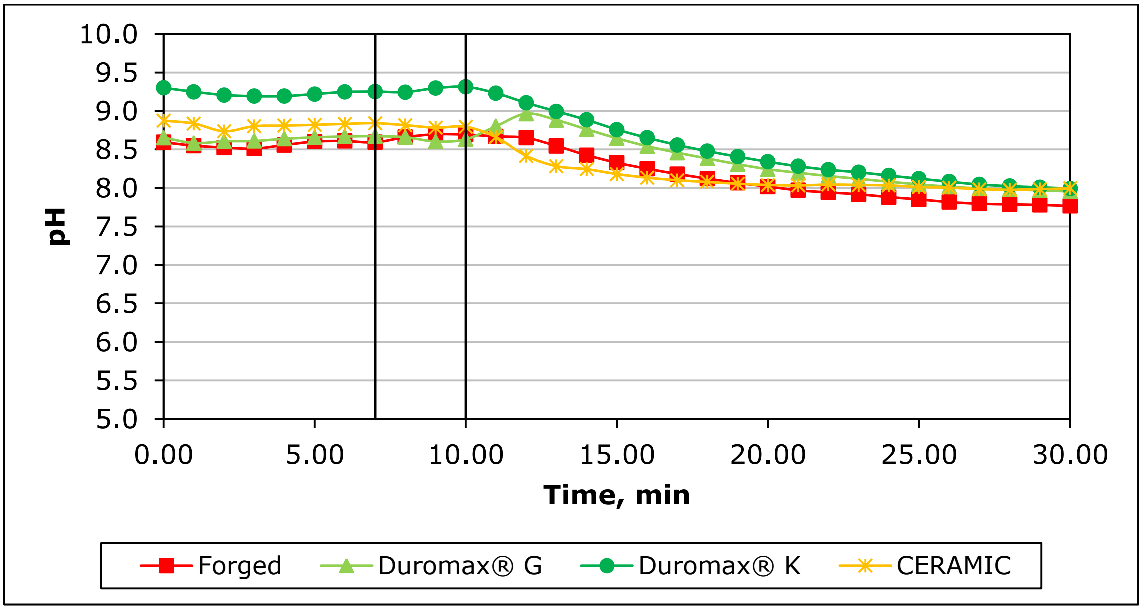

Figure 66: pH trends during grinding using various grinding media, oxide synthetic ore.

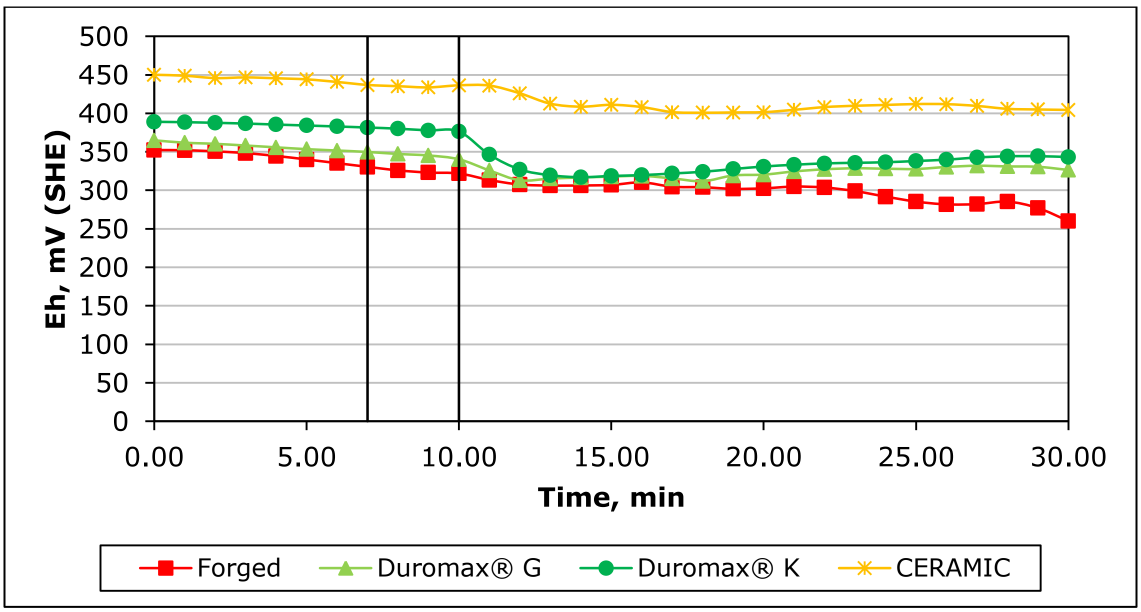

Figure 67: Eh (vs SHE) trends during grinding using various grinding media, oxide synthetic ore.

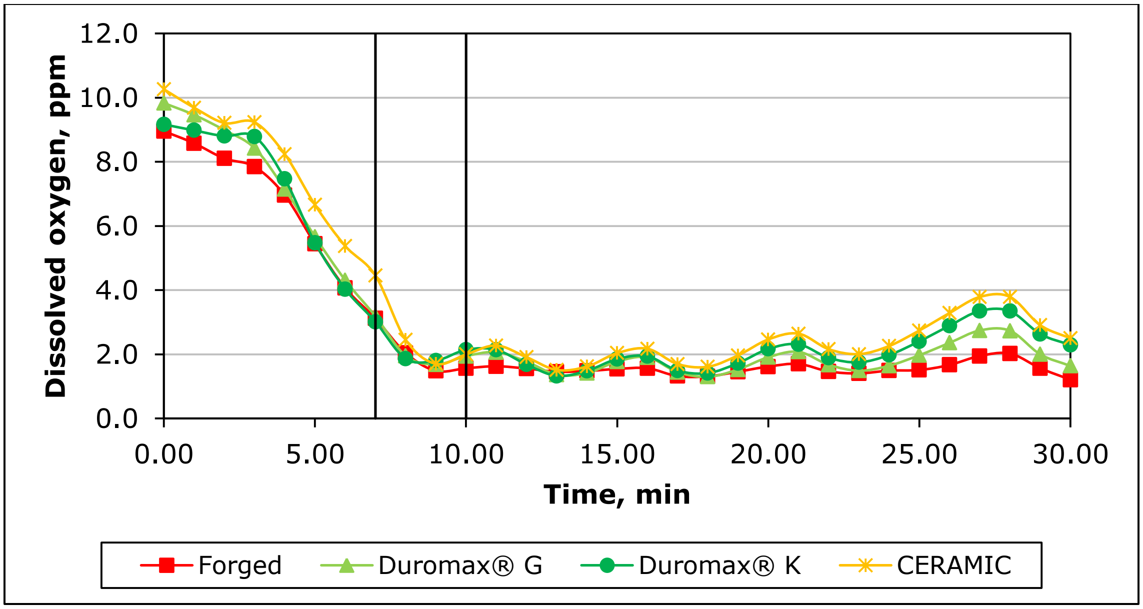

Figure 68: Dissolved oxygen (ppm) trends during grinding using various grinding media, oxide synthetic ore.

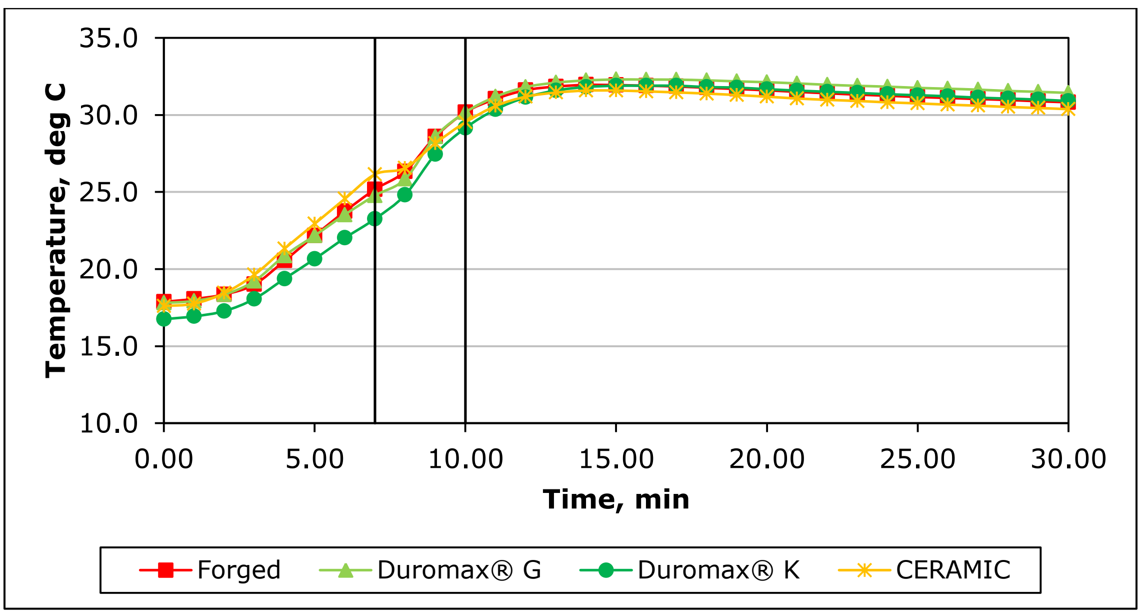

Figure 69: Temperature trends during grinding using various grinding media, oxide synthetic ore.

4.3. Flotation chemistry

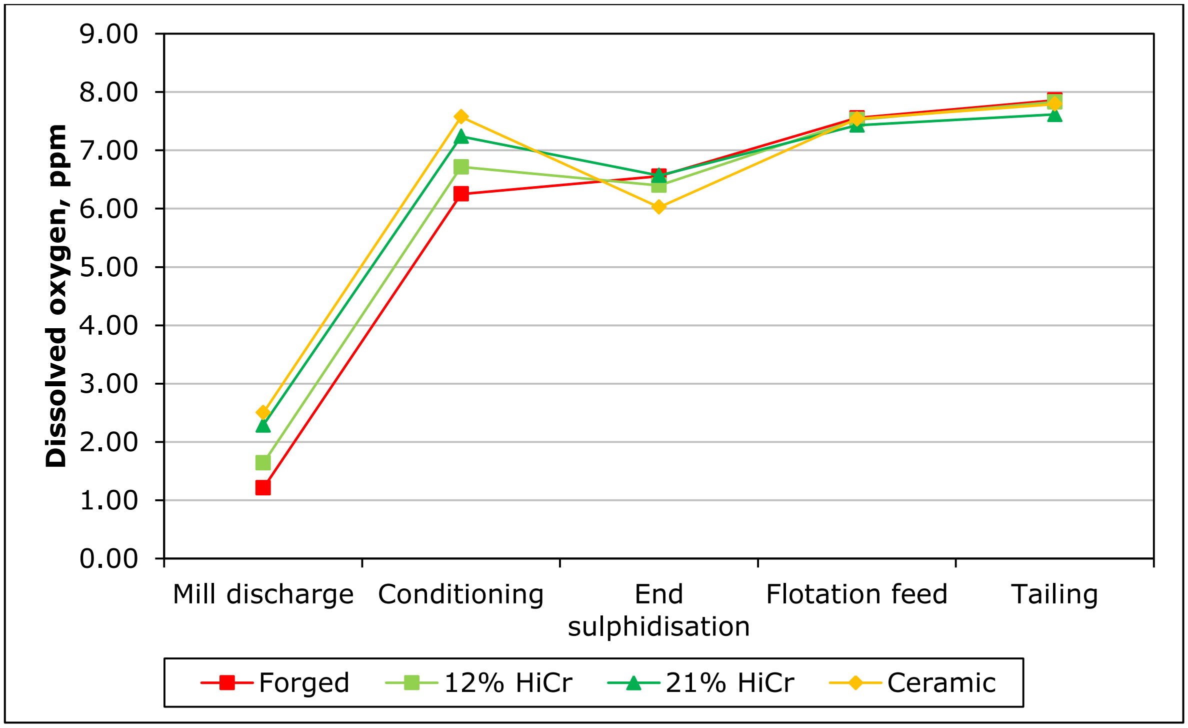

Figure 70: Dissolved oxygen profiles during flotation tests of the oxide synthetic ore comparing various grinding media.

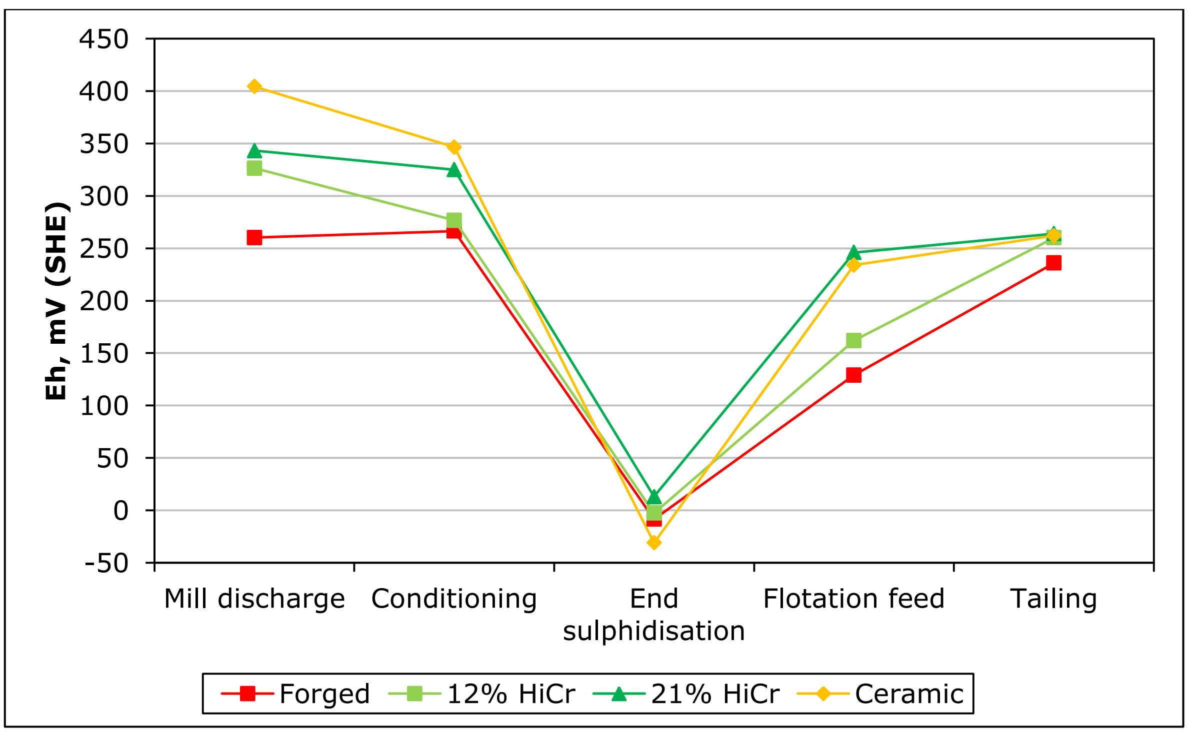

Figure 71: Eh (SHE) profiles during flotation tests of the oxide synthetic ore comparing various grinding media.

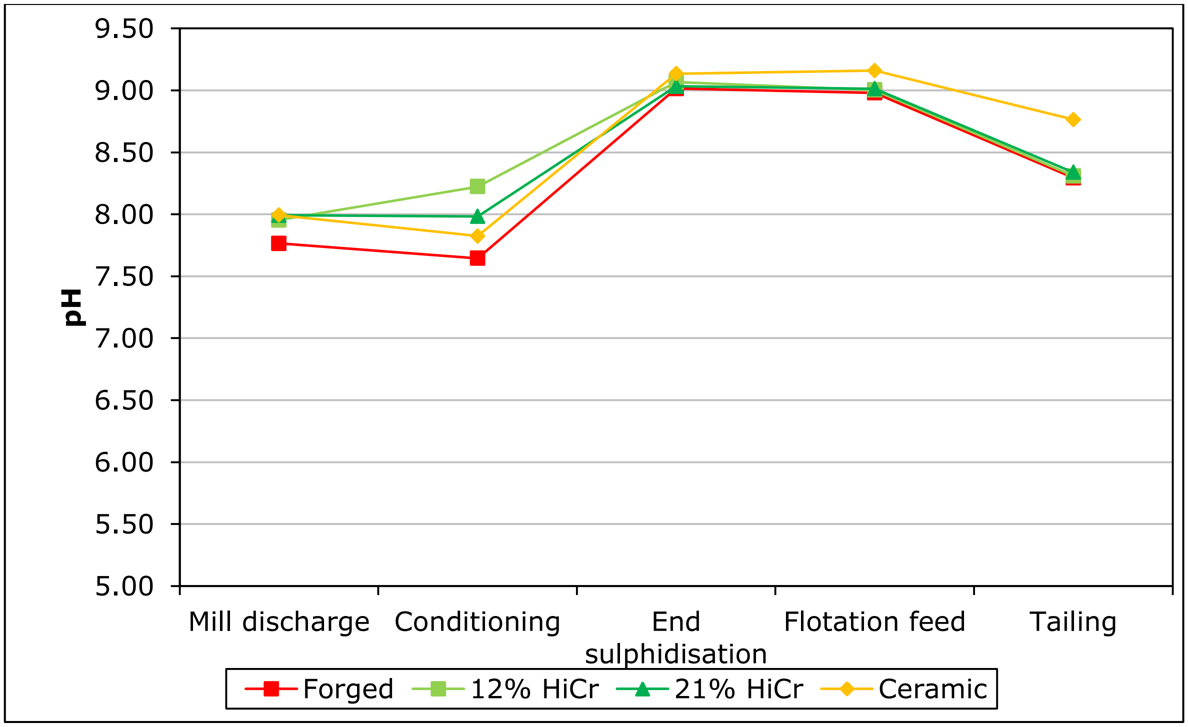

Figure 72 pH profiles during flotation tests of the oxide synthetic ore comparing various grinding media.

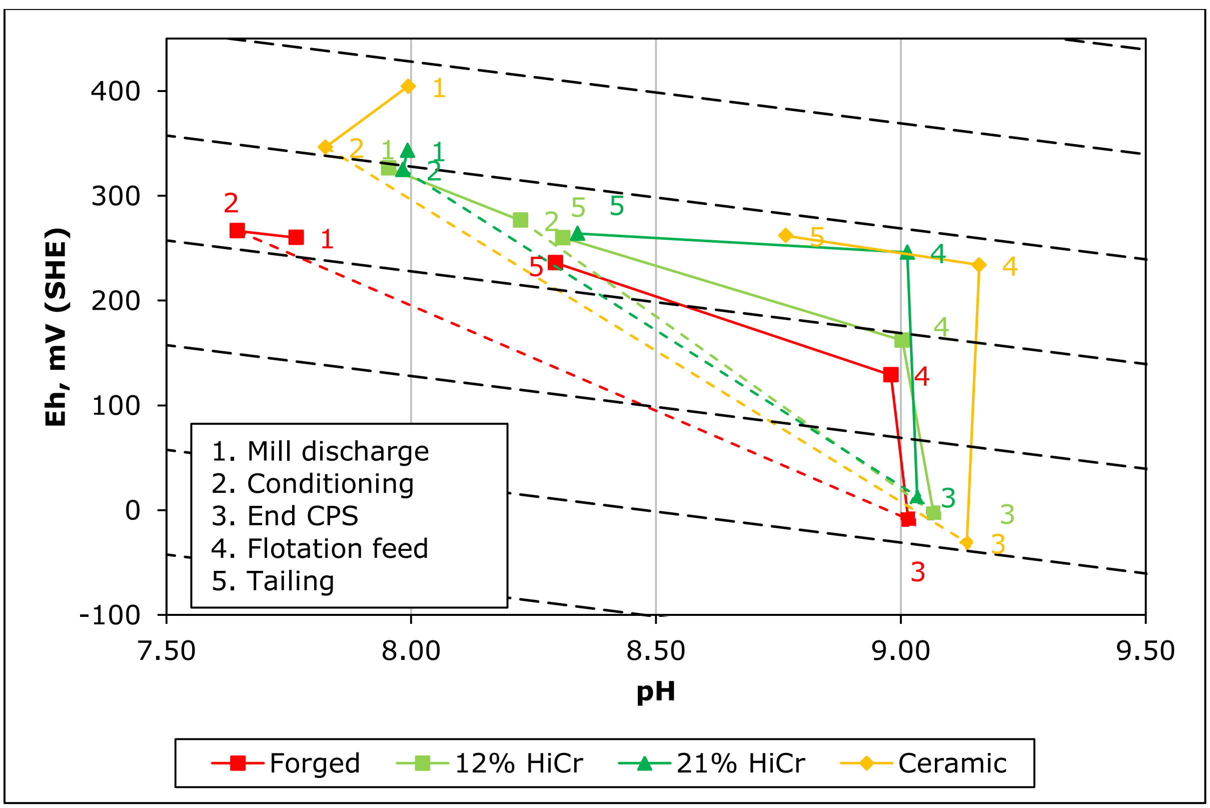

Figure 73 Eh-pH profiles during flotation tests of the oxide synthetic ore comparing various grinding media.

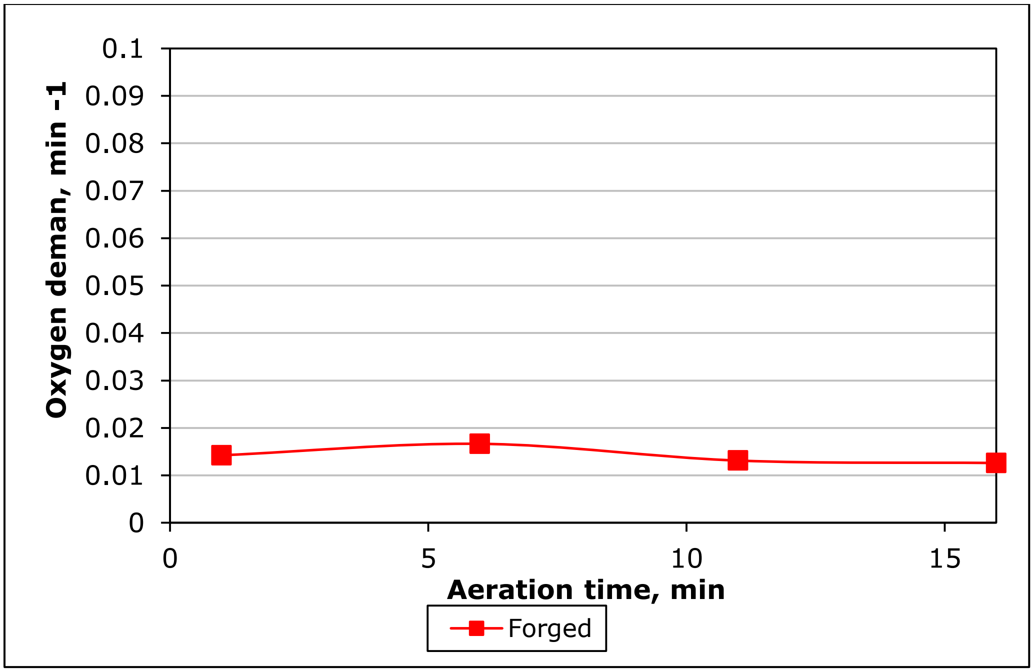

Figure 74: Oxygen demand versus aeration time for the test completed with forged steel media on the oxide synthetic ore.

4.4. Flotation results

4.4.1 Reproducibility

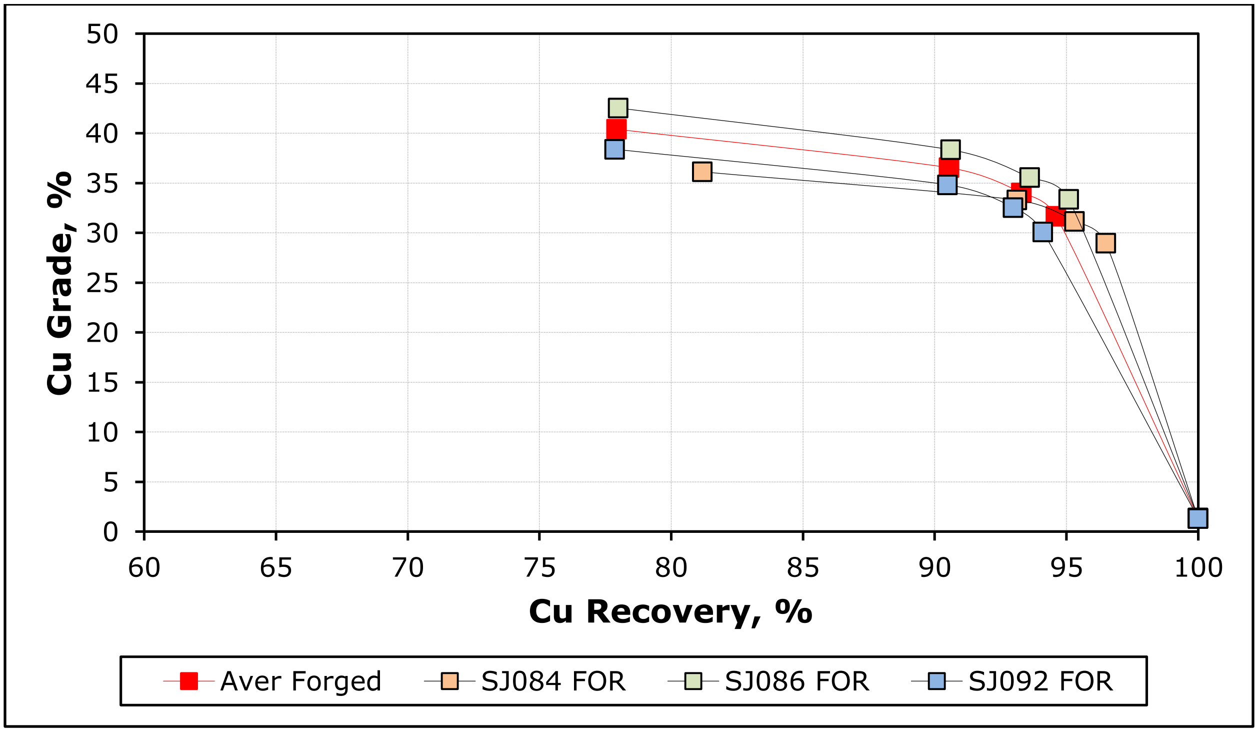

Figure 75 Copper grade recovery curves for flotation tests completed with forged steel media on oxide ore sample.

4.4.2 Selectivity curves

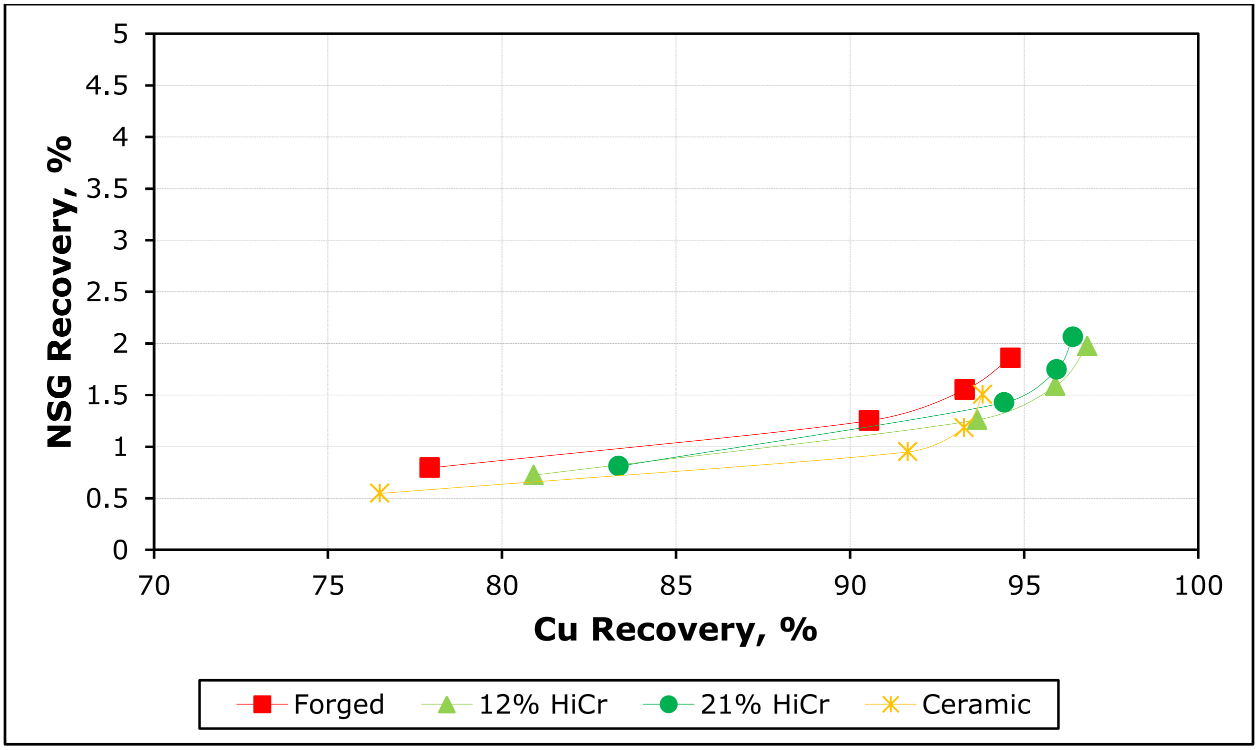

Figure 76: Non-sulphide gangue versus copper recovery curves for flotation tests completed with several grinding media on oxide ore.

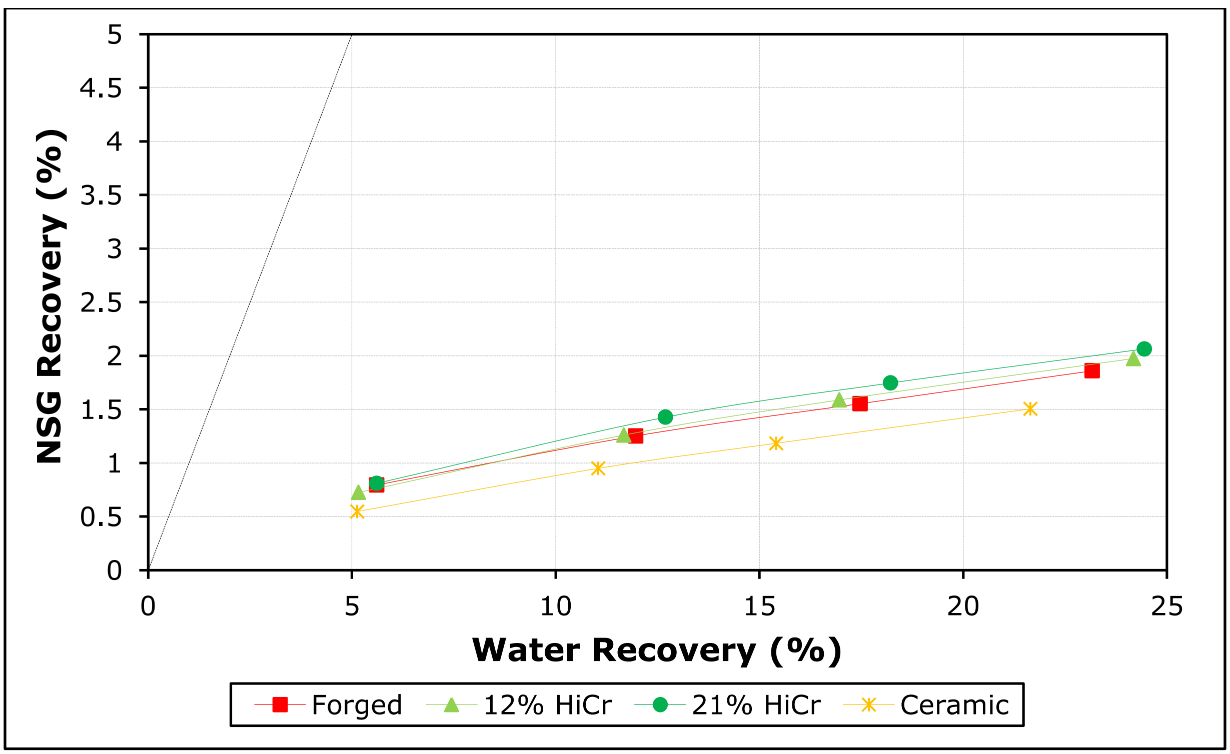

Figure 77: Non-sulphide gangue recovery versus water recovery curves for flotation tests completed with several grinding media on oxide ore.

4.4.3 Feed size distribution

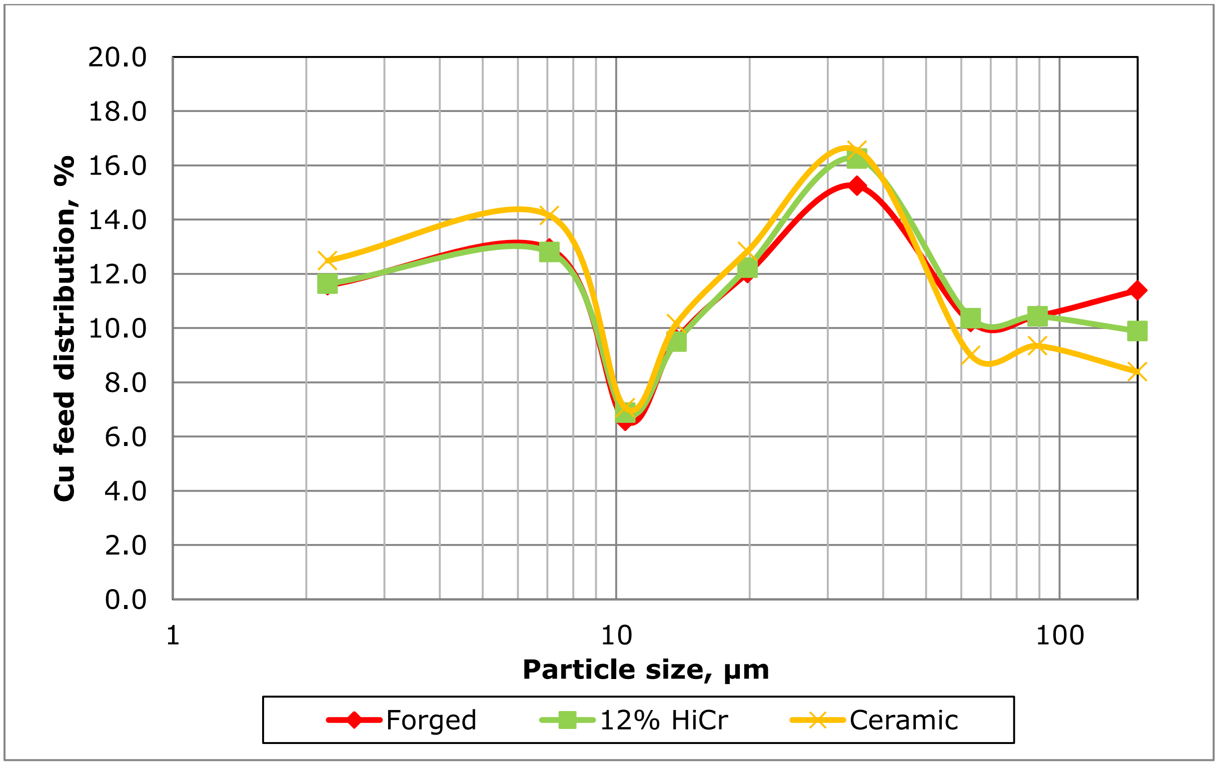

Figure 78: Copper feed distribution by size for all media tested, oxide ore.

V. SYNTHETIC MIXED ORE

5.1. Grinding calibration

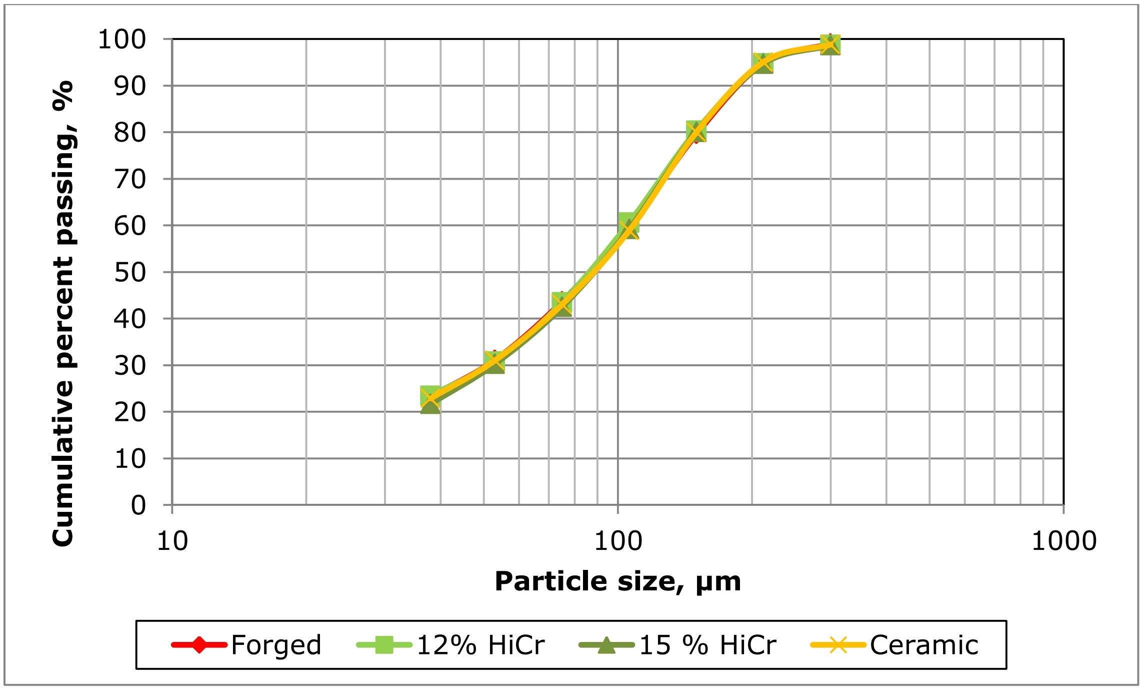

Figure 79: Cumulative size distribution curves for all media tested, mixed synthetic ore.

Table 7: Magotteaux Mill® parameters and P_{80} for forged steel, 12% HiCr, 21% HiCr and ceramic grinding media.

Media Type	Media Charge, Kg	Mill Speed, rpm	P_{80}, µm
Forged	20 x balls	43	151.6
12%Cr	20 x balls	46	149.2
21%Cr	20 x balls	44	149.9
Ceramic	20 x balls	49	150.2

5.2. Grinding chemistry

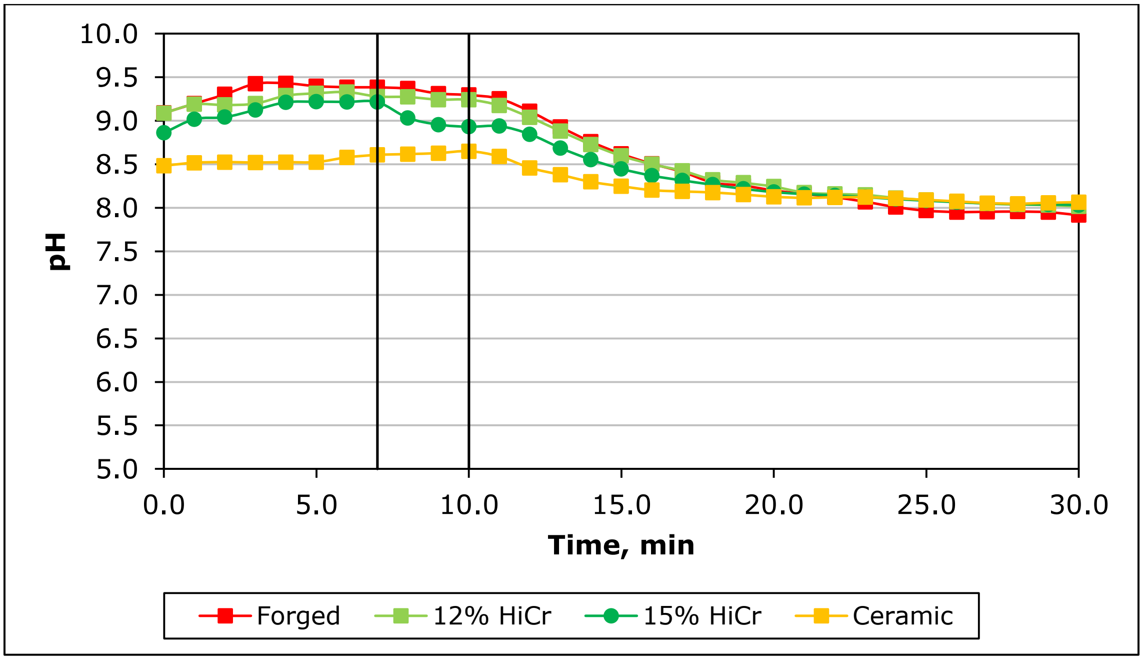

Figure 80: pH trends during grinding using various grinding media, mixed synthetic ore.

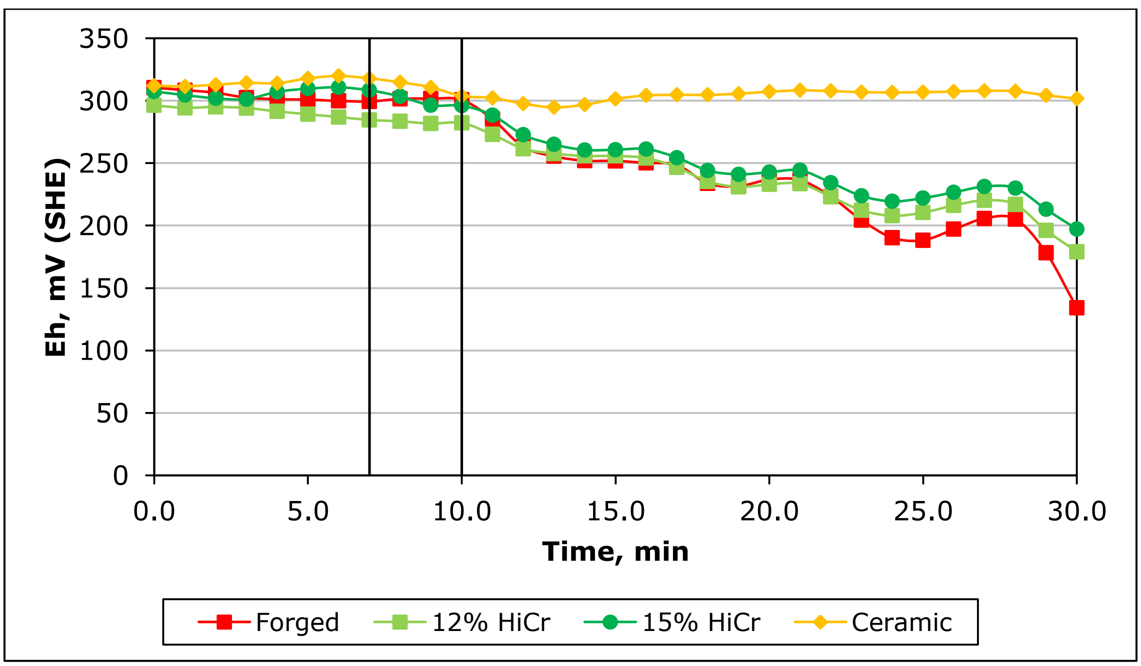

Figure 81: Eh trends during grinding using various grinding media, mixed synthetic ore.

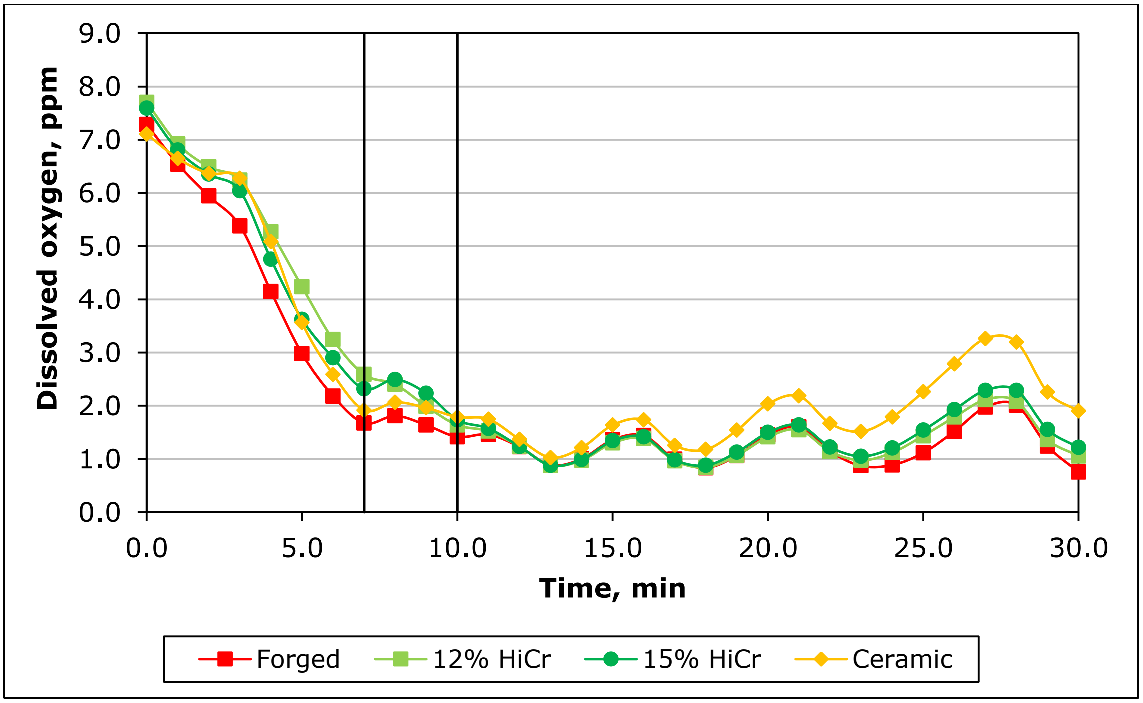

Figure 82: Dissolved oxygen trends during grinding using various grinding media, mixed synthetic ore.

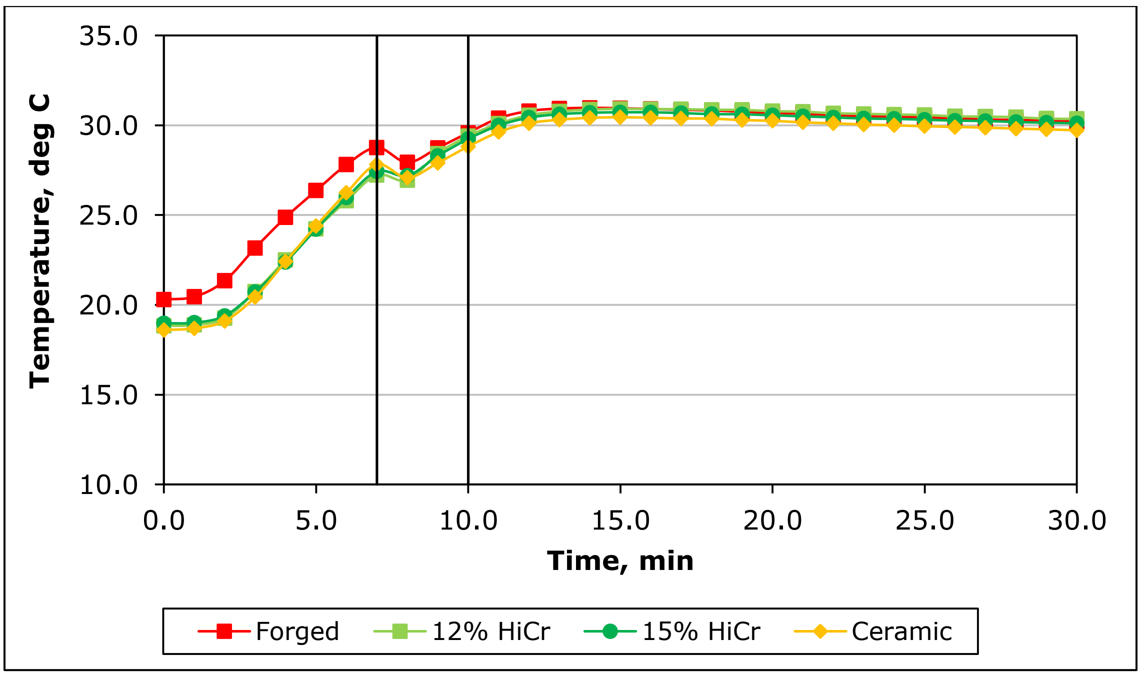

Figure 83: Temperature trends during grinding using various grinding media, mixed synthetic ore.

5.3. *Flotation chemistry*

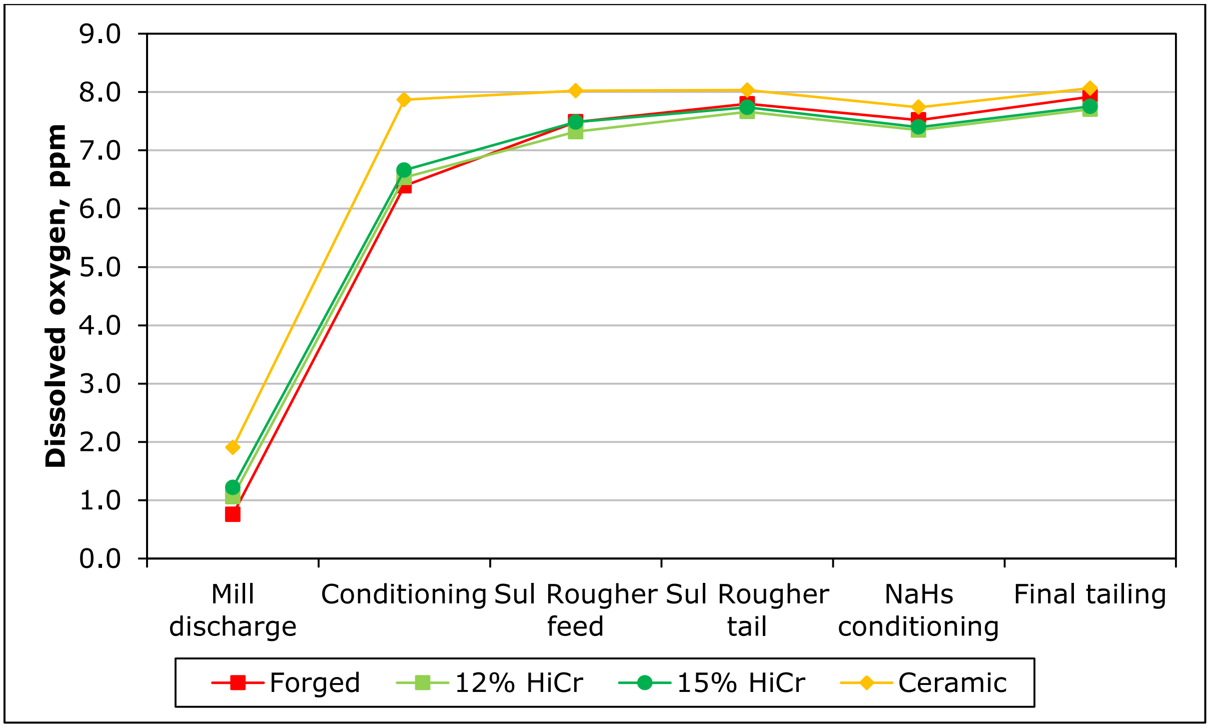

Figure 84: Dissolved oxygen profiles during flotation tests of mixed synthetic ore with all media tested.

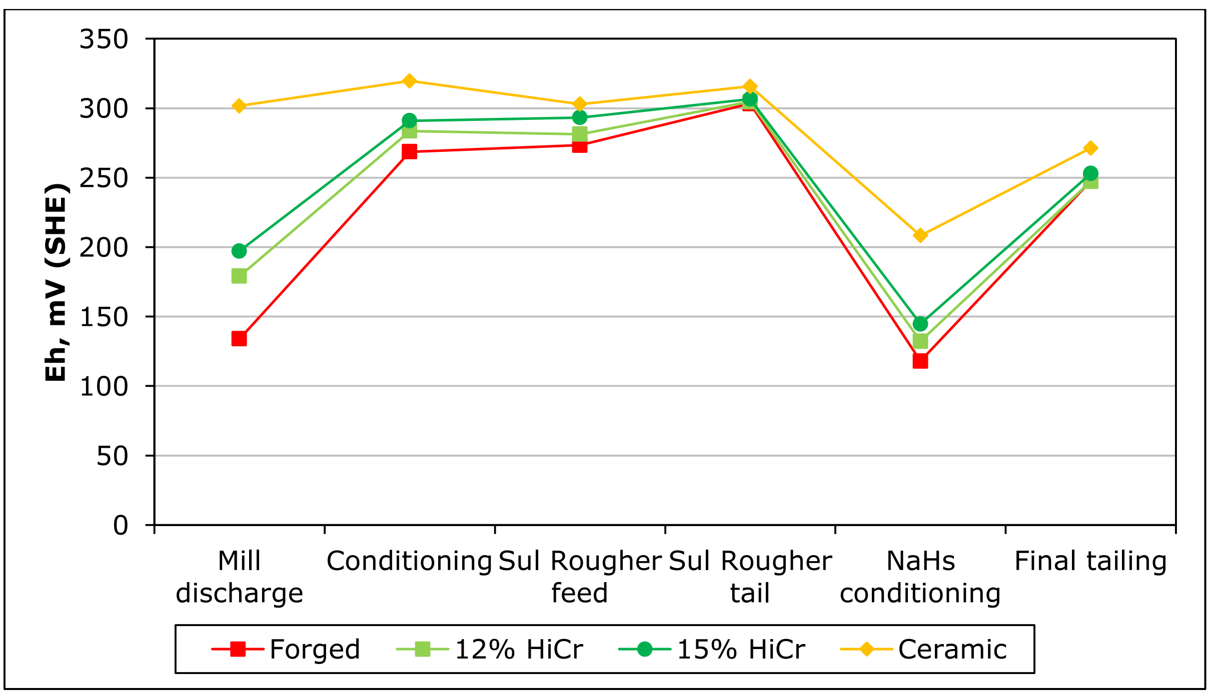

Figure 85: Eh profiles during flotation tests of mixed synthetic ore with all media tested.

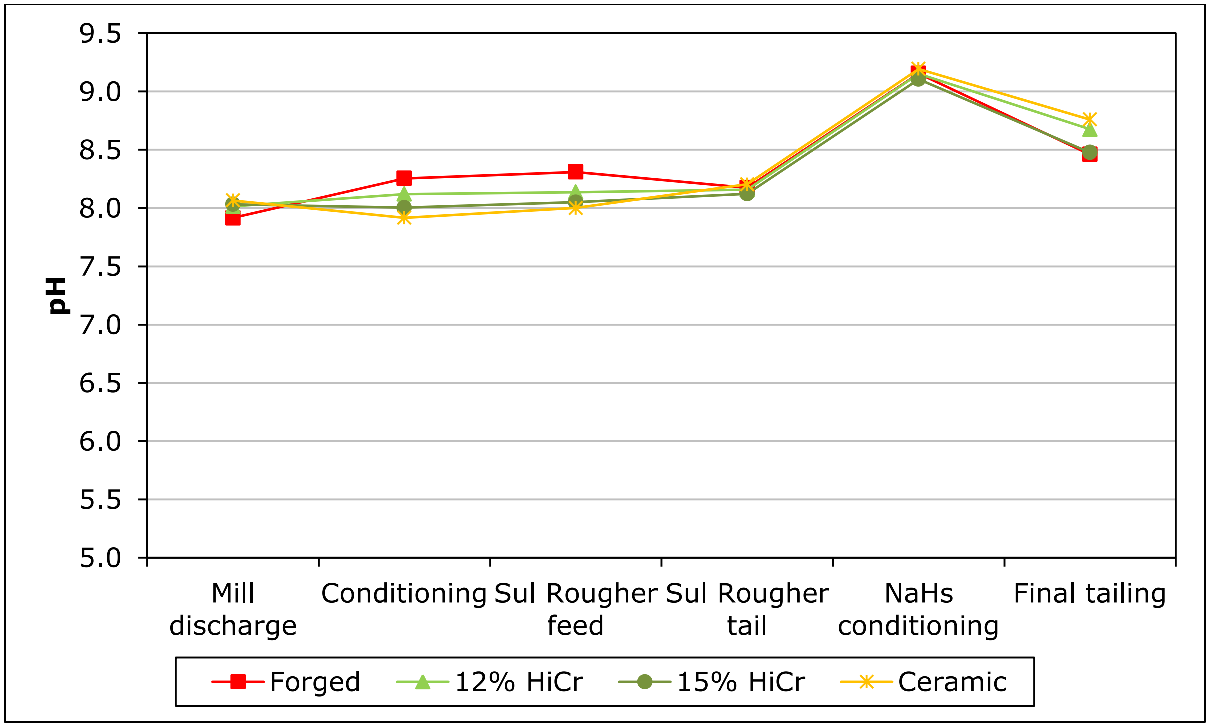

Figure 86: pH profiles during flotation tests of mixed synthetic ore with all media tested.

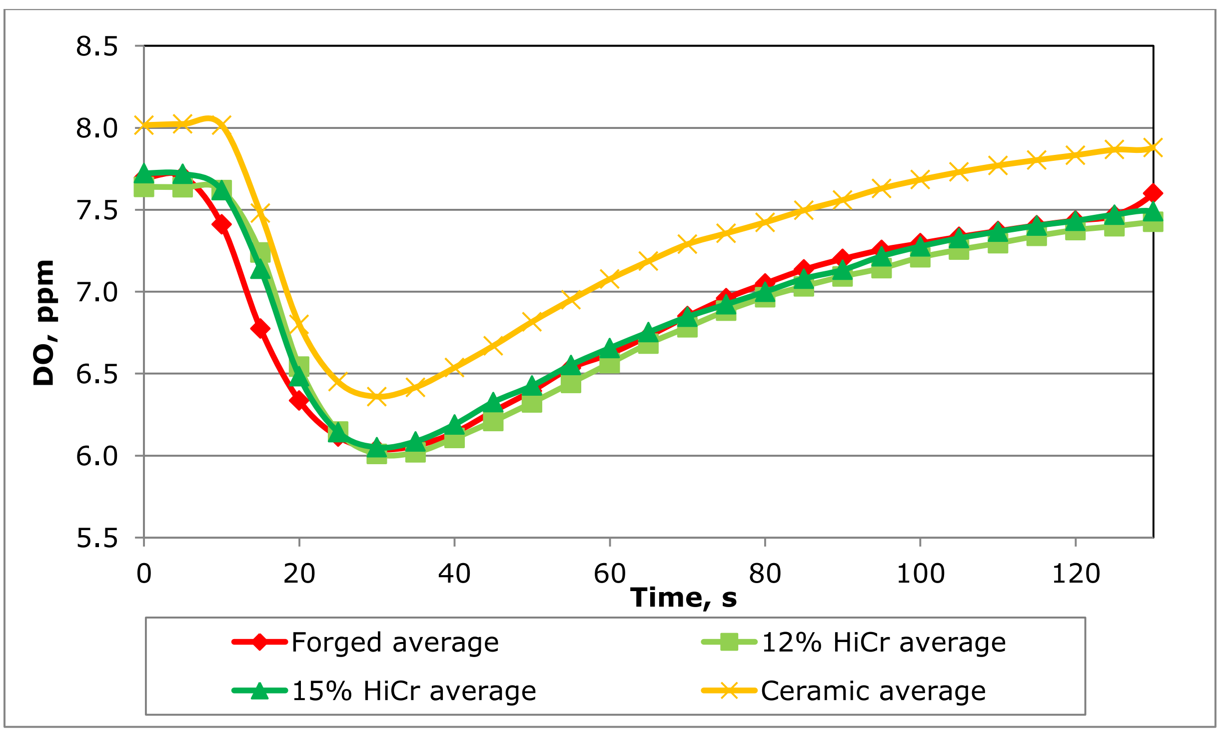

Figure 87: Dissolved oxygen profiles during NaHS conditioning of mixed synthetic ore with all media tested.

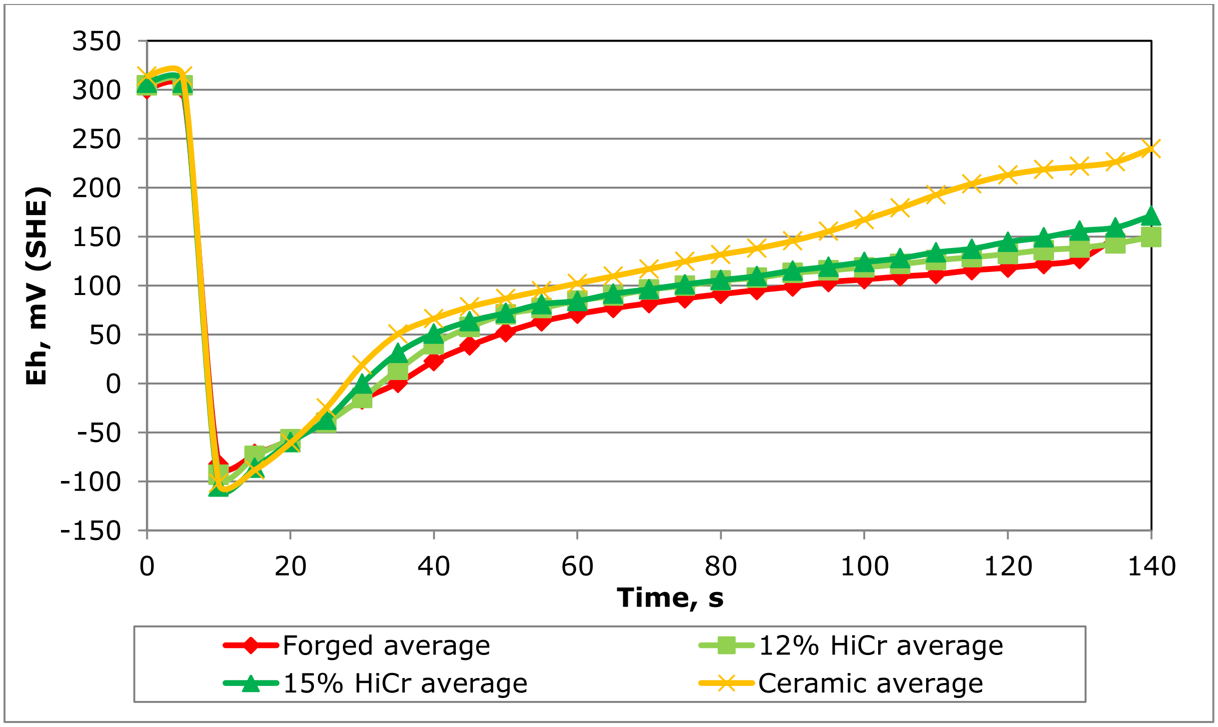

Figure 88: Eh profiles during NaHS conditioning of mixed synthetic ore with all media tested.

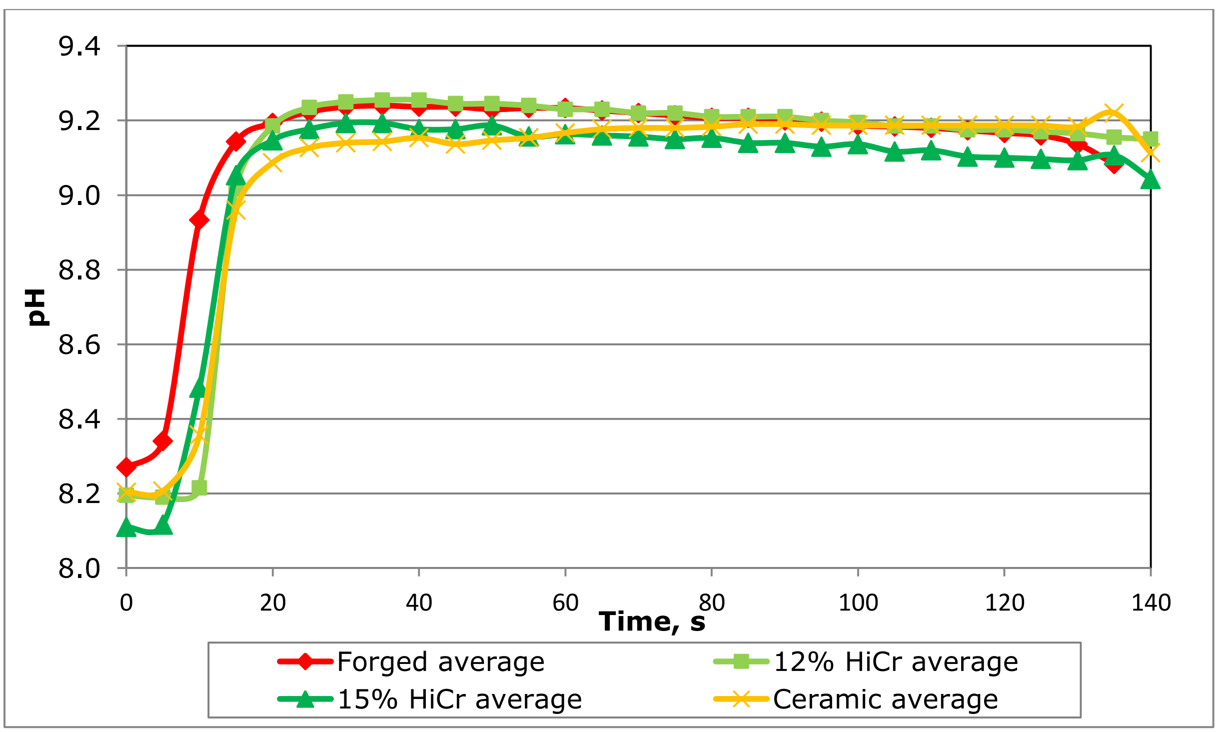

Figure 89: pH profiles during NaHS conditioning of mixed synthetic ore with all media tested.

5.4. Flotation results

5.4.1 Reproducibility

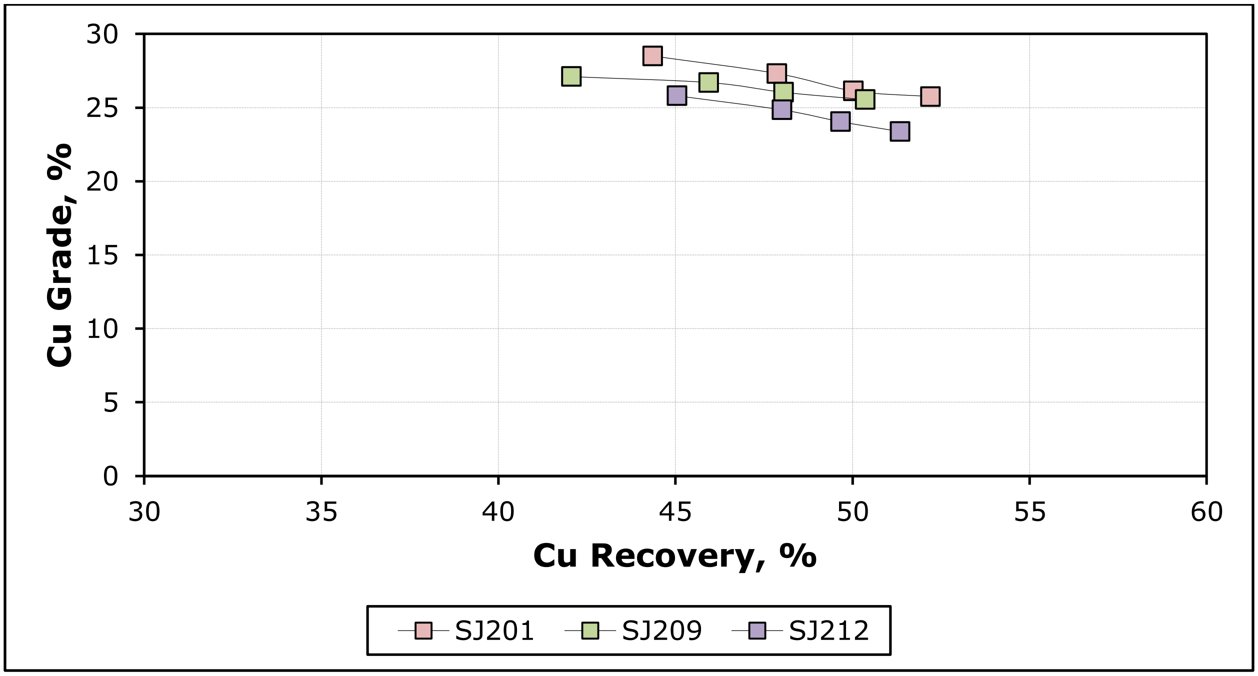

Figure 90: Copper grade recovery curves during the sulphide flotation stage on mixed ore completed with forged steel media.

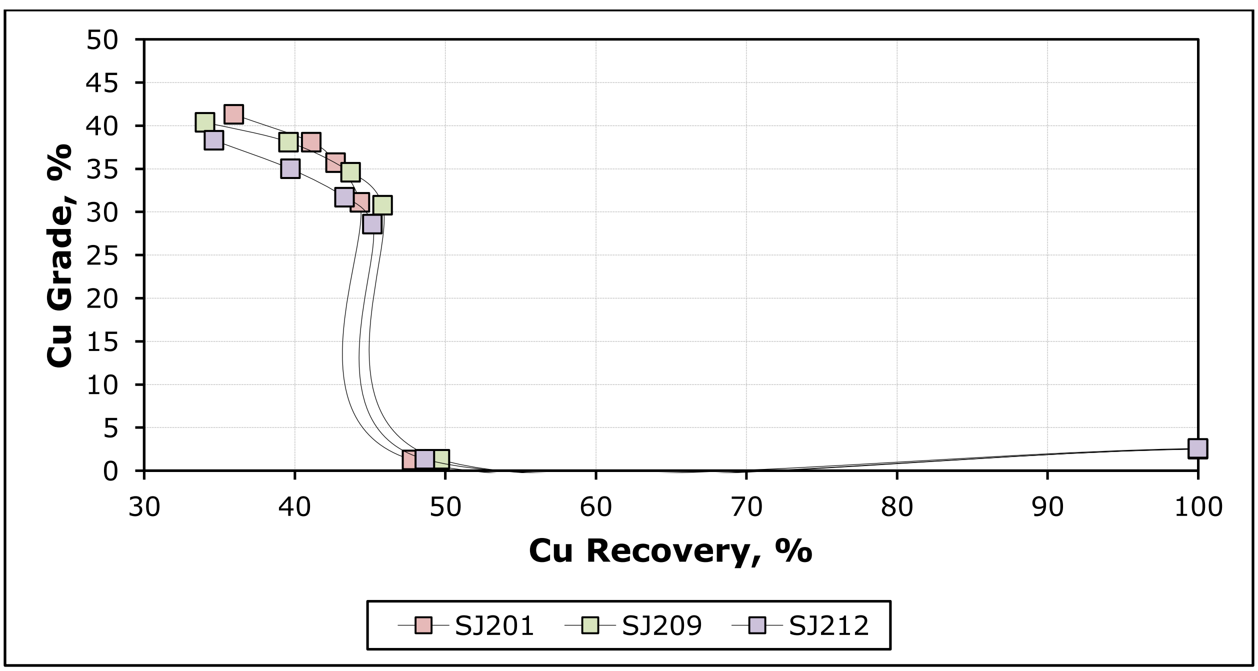

Figure 91: Copper grade recovery curves during the oxide flotation stage on mixed ore completed with forged steel media.

5.4.2 Standard flotation tests

- Sulphide flotation stage

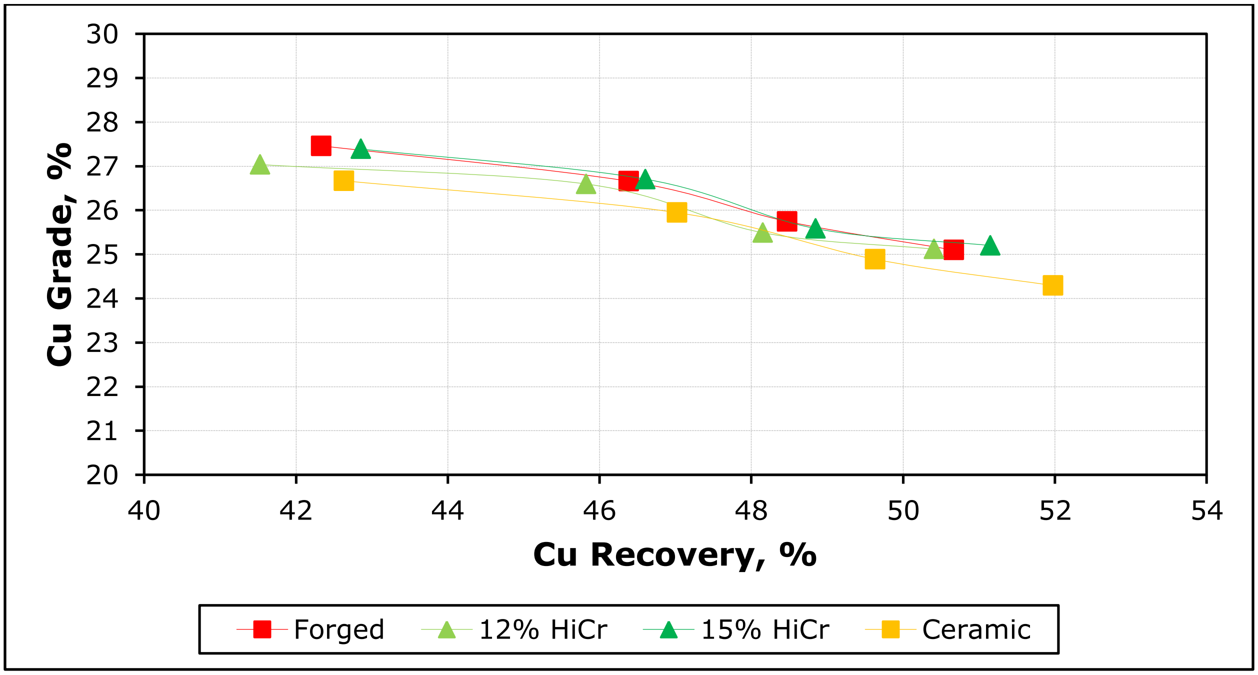

Figure 92: Copper grade-recovery curve for the sulphide flotation stage of synthetic ore ground with several grinding media.

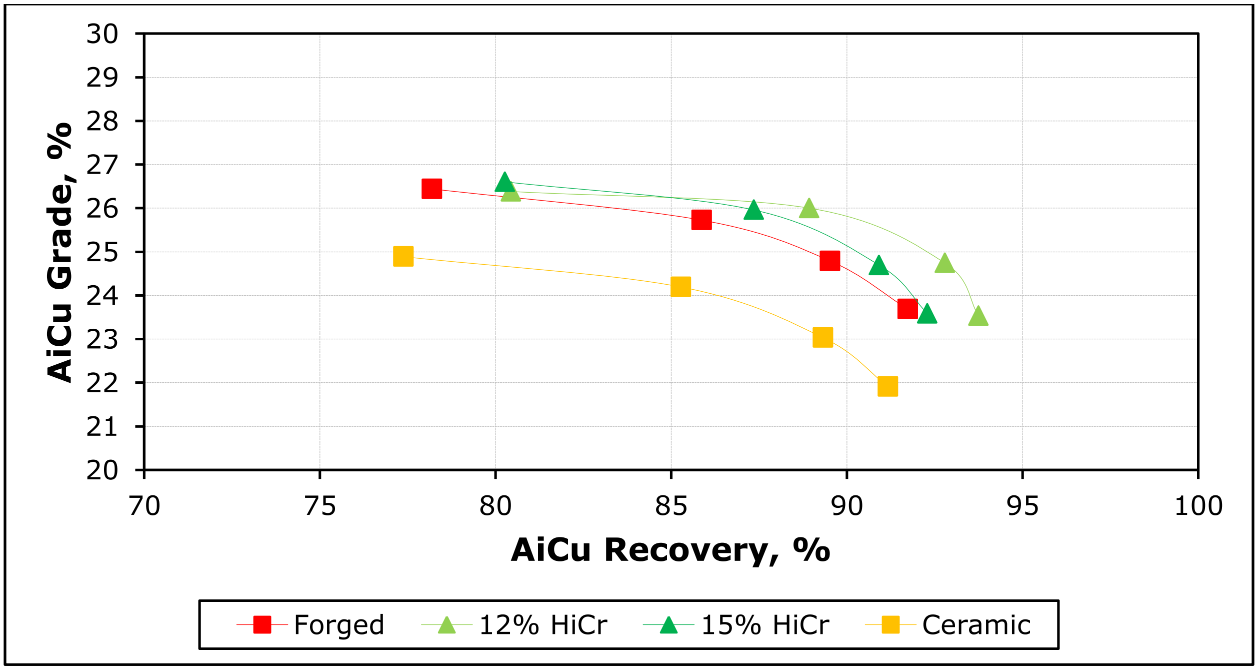

Figure 93 Acid insoluble copper grade-recovery curve for the sulphide flotation stage of synthetic ore ground with several media.

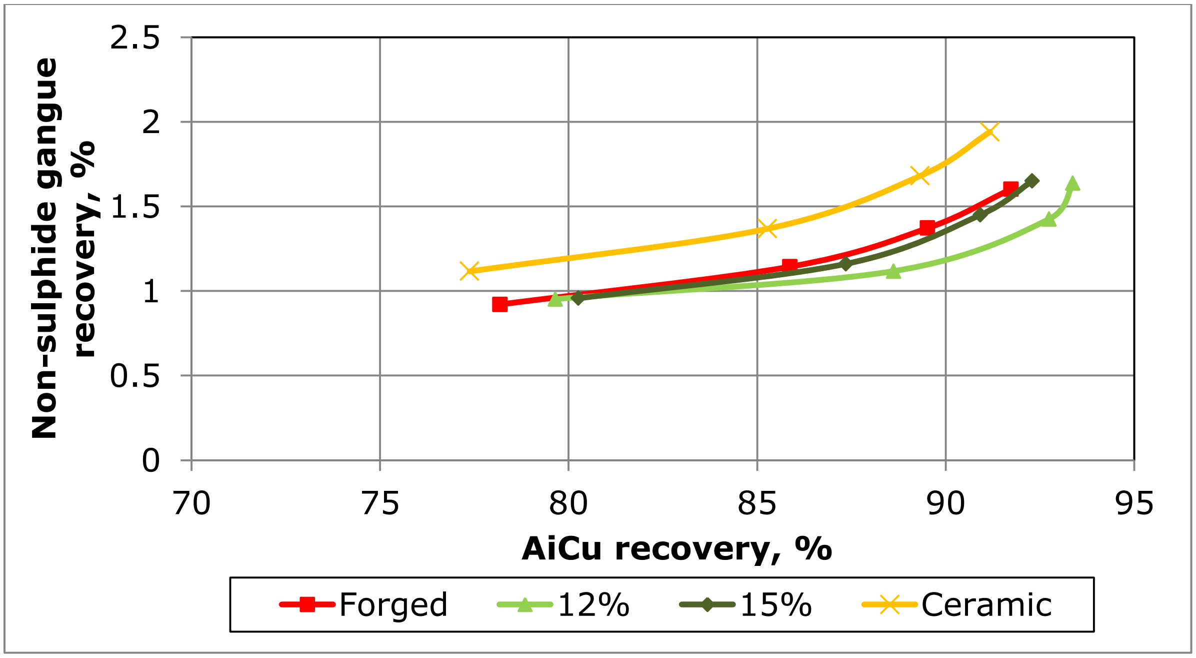

Figure 94: Non-sulphide gangue versus acid insoluble copper recovery curves during the sulphide flotation stage of mixed ore comparing several grinding media.

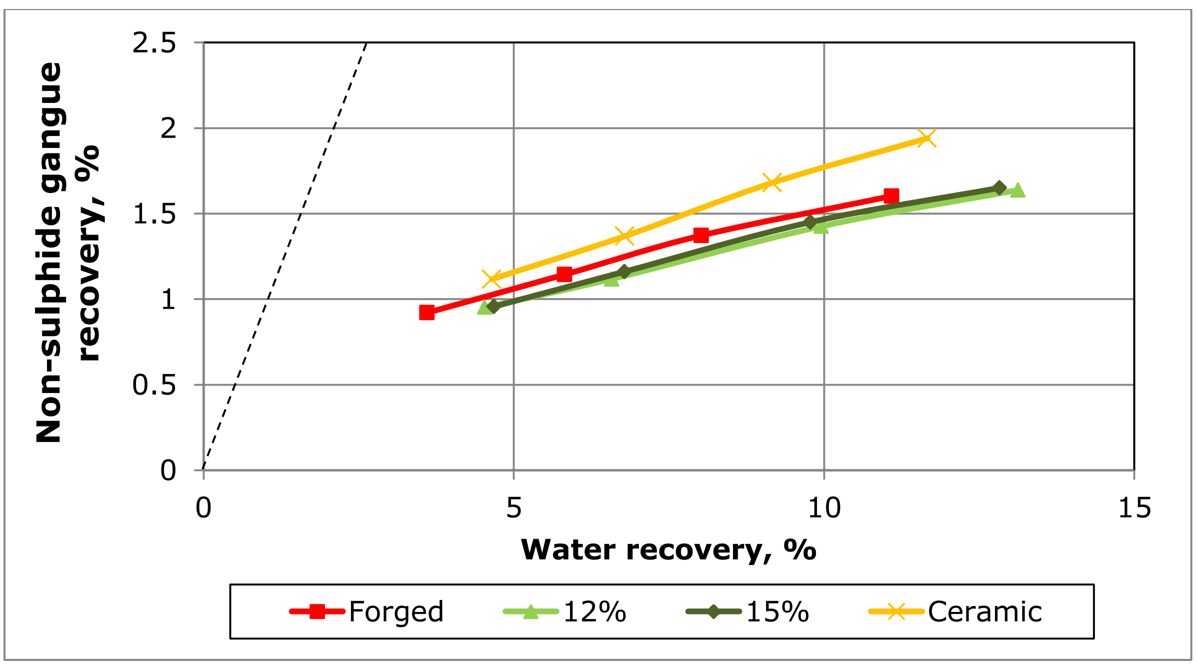

Figure 95: Non-sulphide gangue versus water recovery curves during the sulphide flotation stage of mixed ore comparing several grinding media.

- <u>Oxide flotation stage</u>

Table 8: Oxide flotation stage feed head grade of mixed ore comparing several grinding media.

Media	Cu, %	AiCu, %	AsCu, %	SiO2, %
Forged	1.32	0.12	1.22	97.55
12% HiCr	1.32	0.08	1.26	97.57
15% HiCr	1.35	0.11	1.24	97.52
Ceramic	1.32	0.12	1.20	97.56

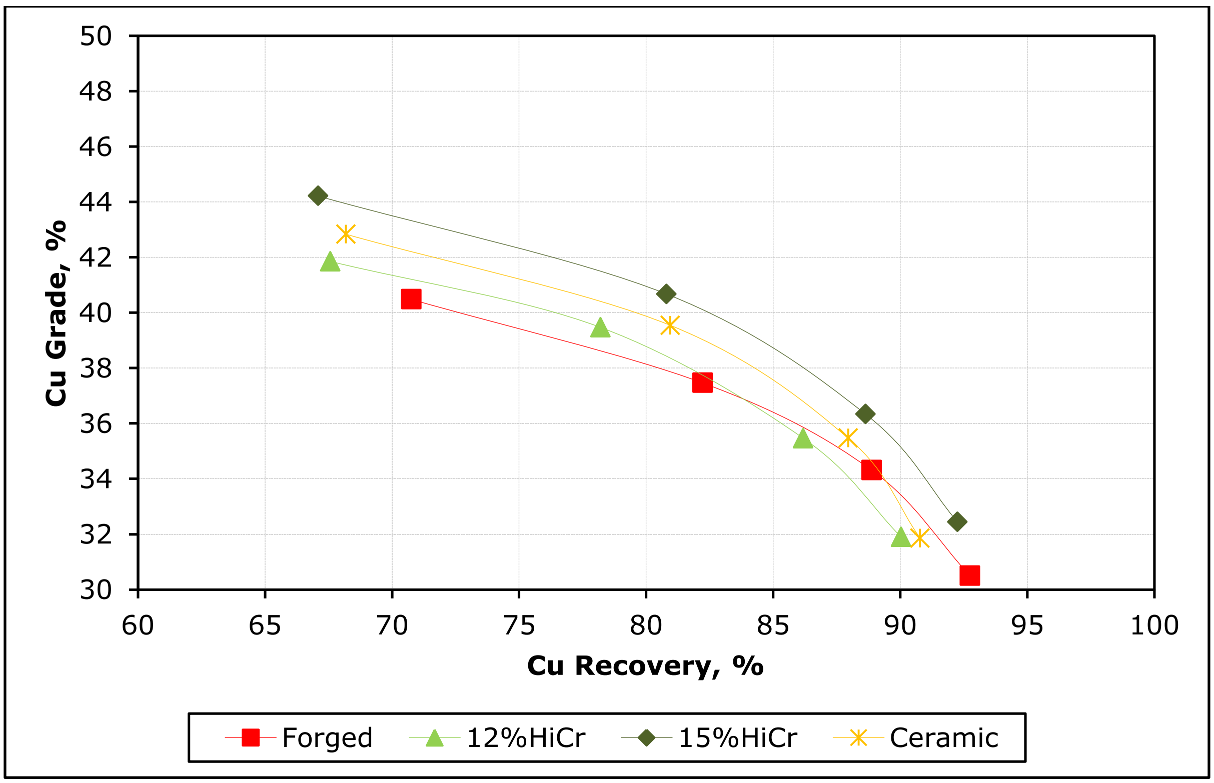

Figure 96: Copper grade recovery curves, with respect to oxide flotation feed, for mixed ore ground with several grinding media.

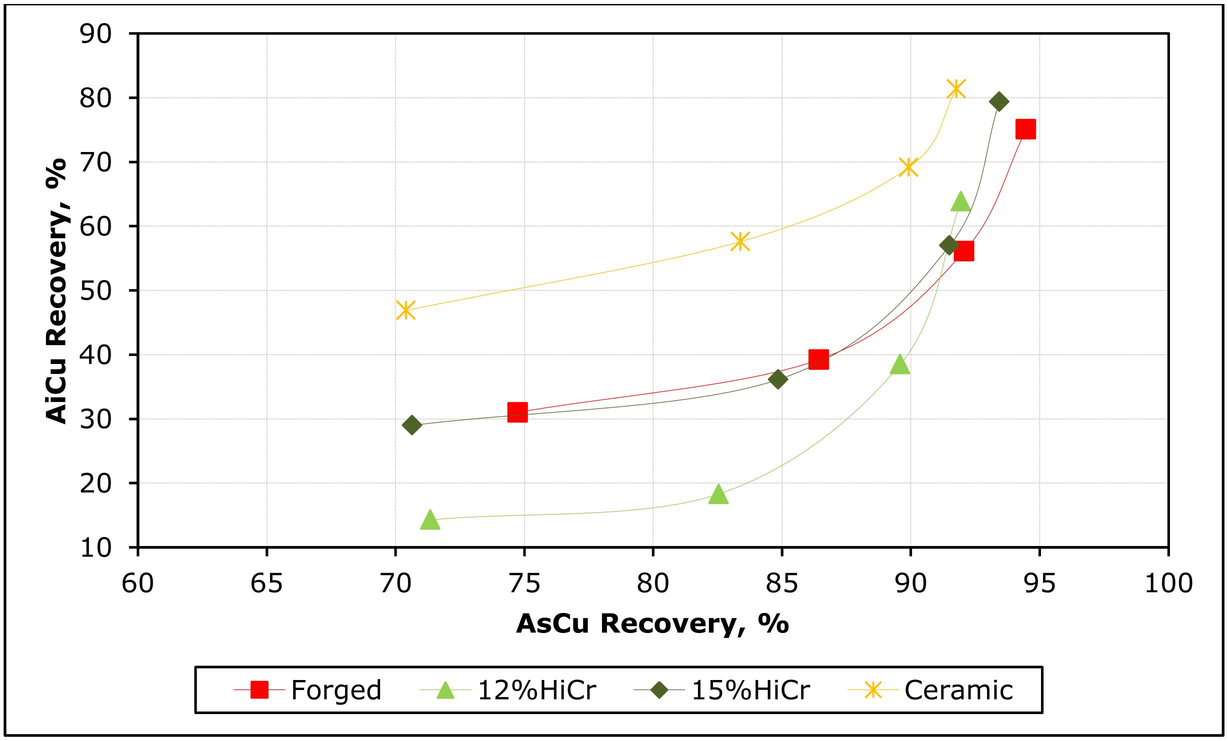

Figure 97: Acid insoluble copper versus acid soluble copper recovery curves, with respect to oxide flotation feed, for flotation tests completed with several grinding media on oxide ore.

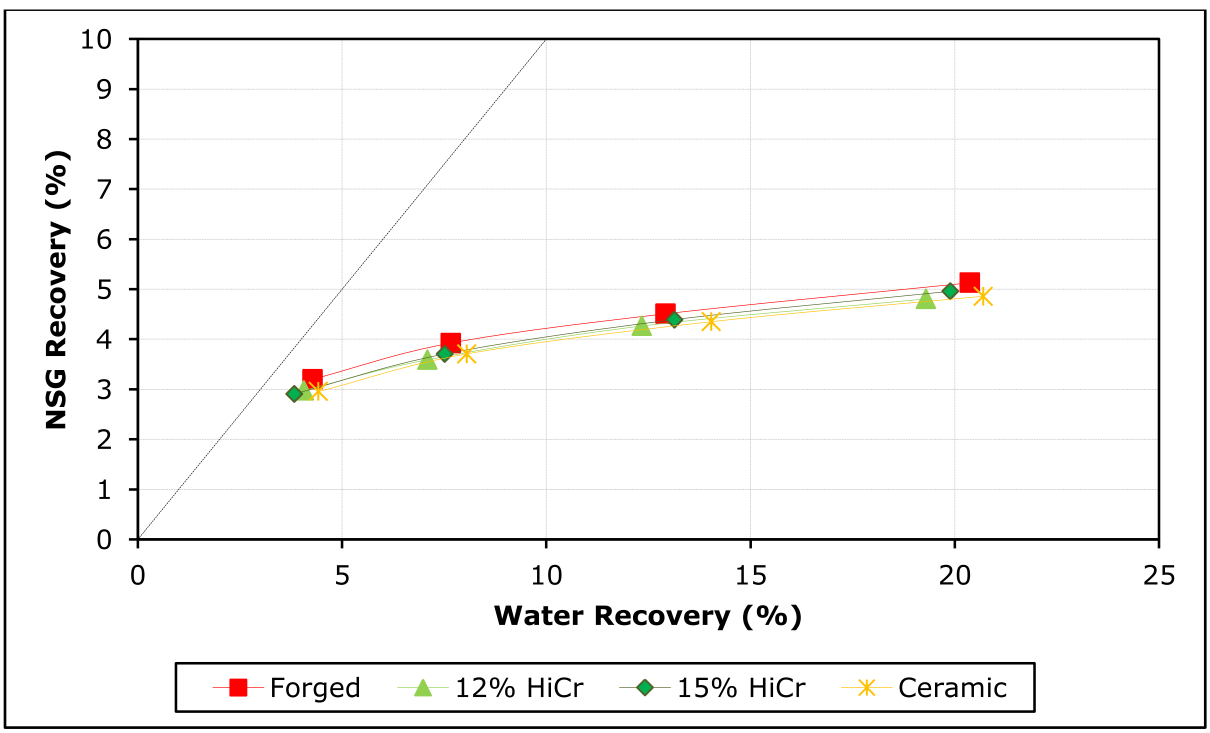

Figure 98: Non-sulphide gangue versus water recovery curves, with respect to oxide flotation feed, for flotation tests completed with several grinding media on oxide ore.

V. Synthetic mixed ore

5.4.3 Size by size

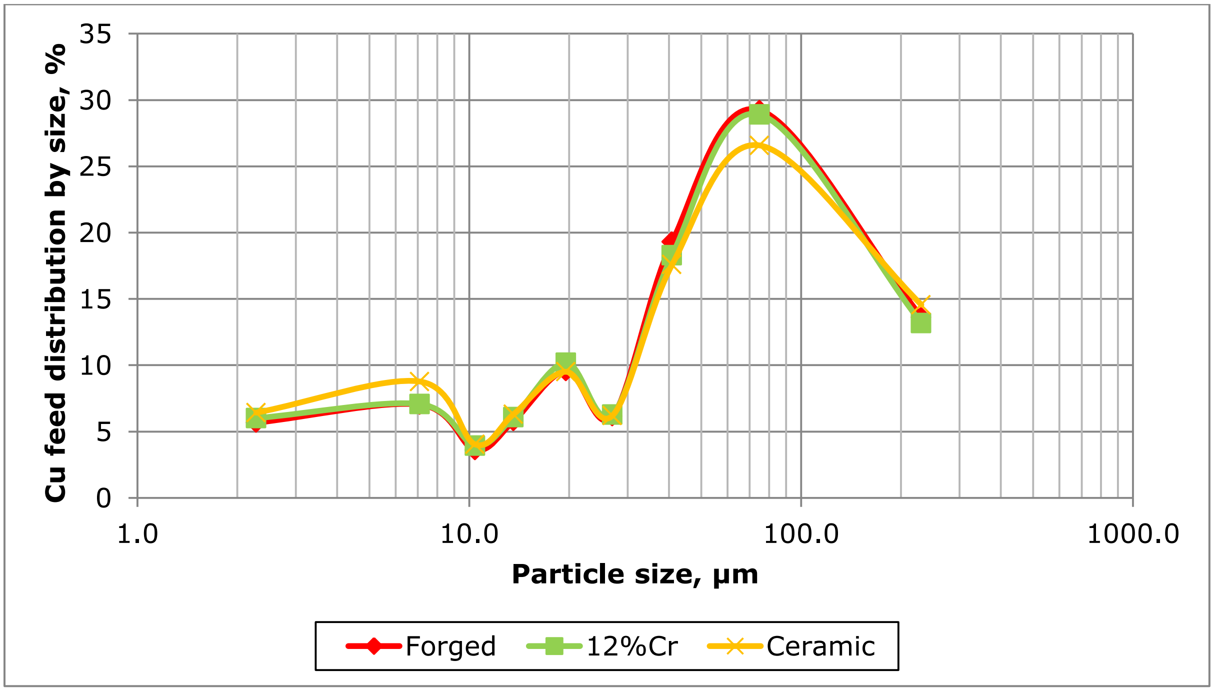

Figure 99: Copper feed distribution for all media tested, mixed ore.

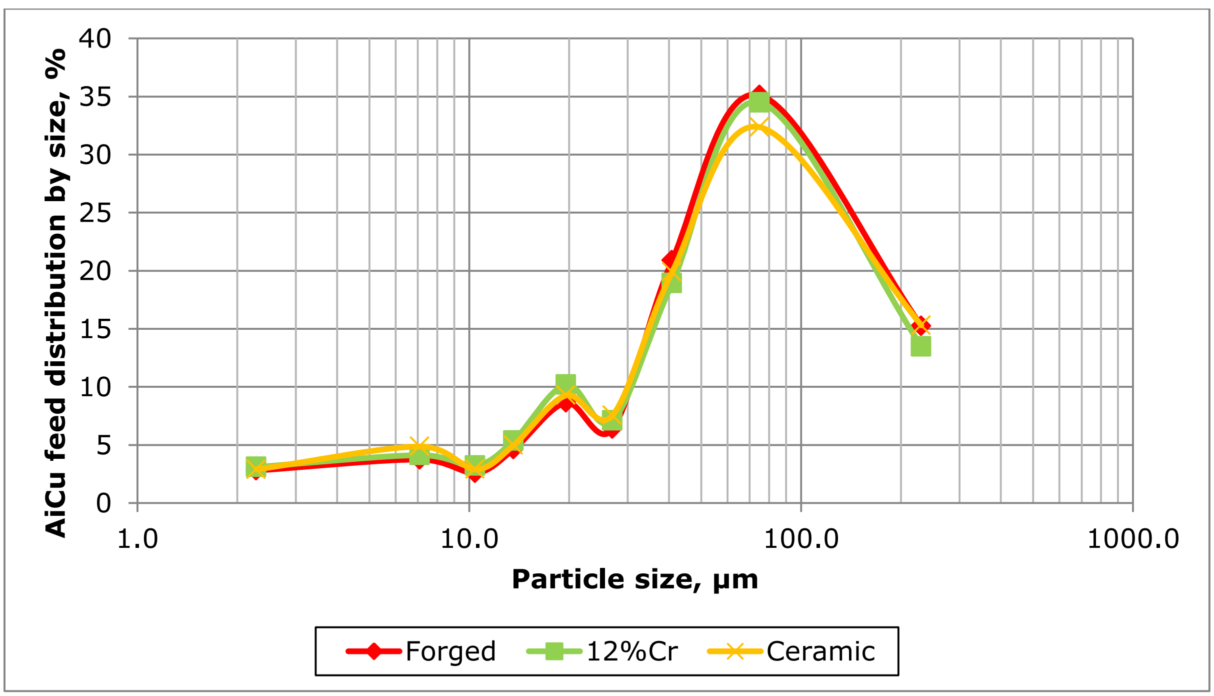

Figure 100: Acid insoluble copper feed distribution for all media tested, mixed ore.

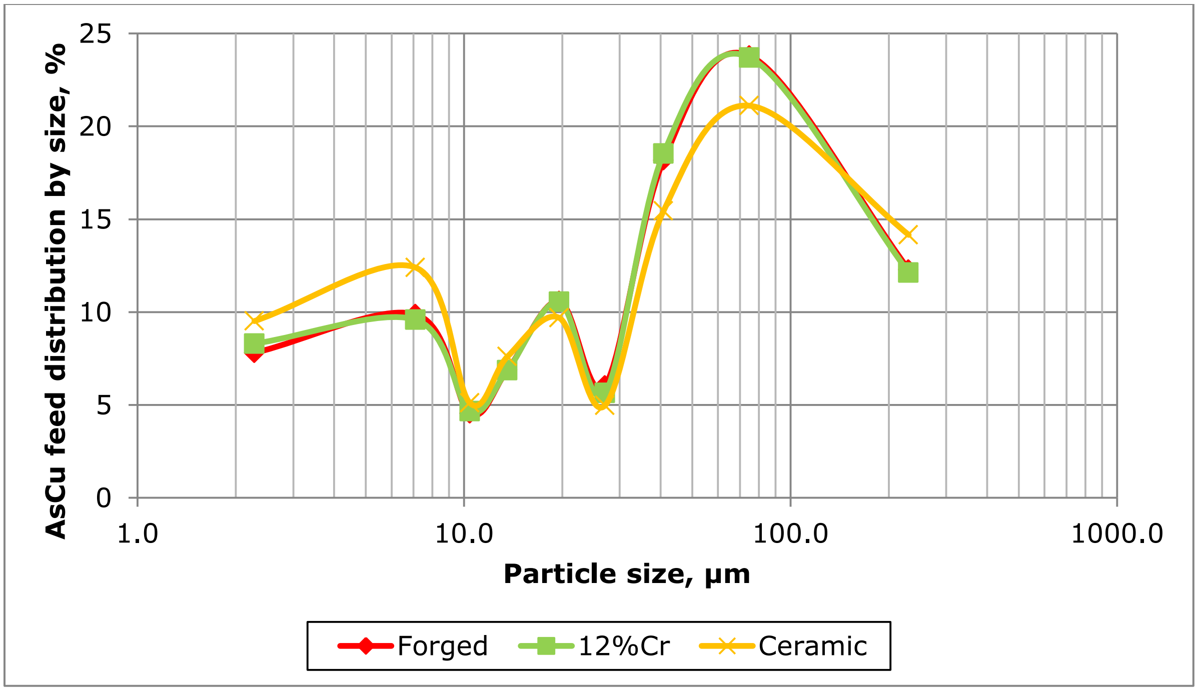

Figure 101: Acid soluble copper feed distribution for all media tested, mixed ore.

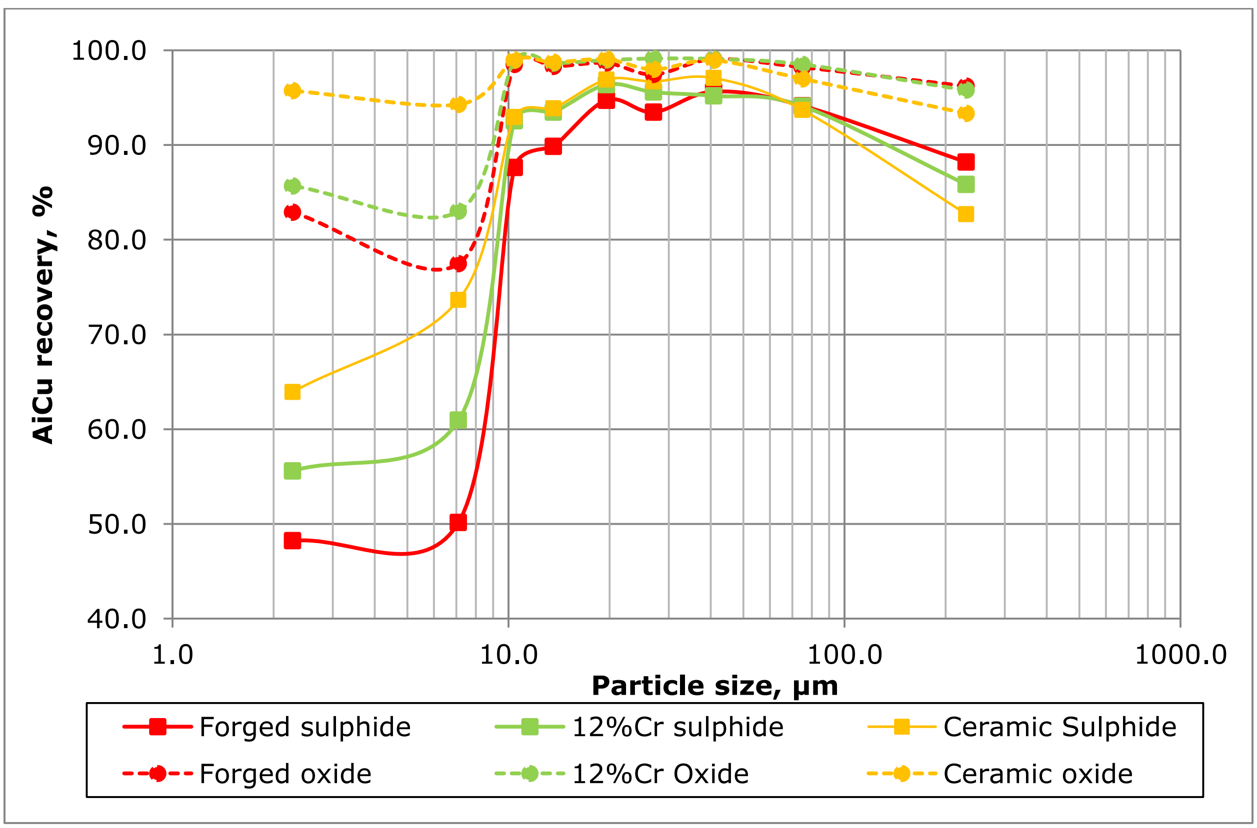

Figure 102: Cumulative acid insoluble copper recovery by size after the sulphide and oxide flotation stages, mixed ore.

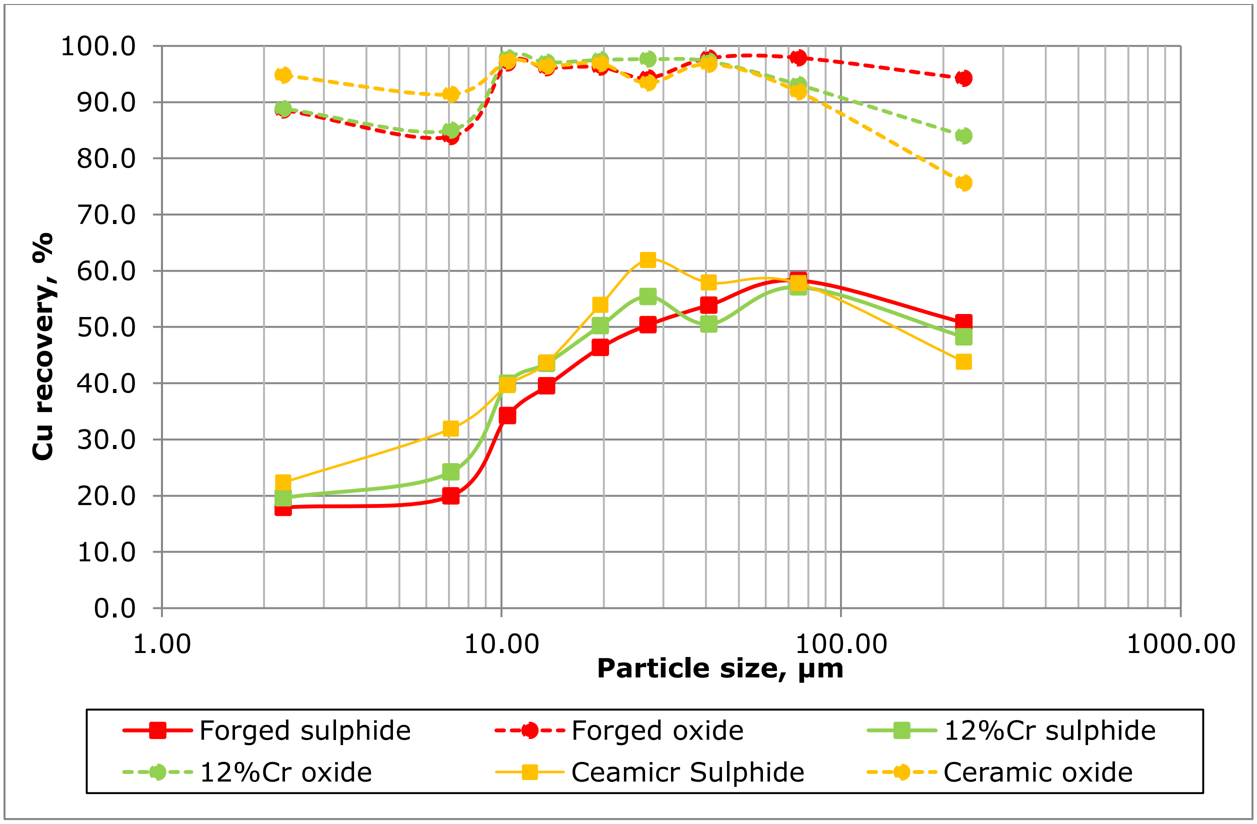

Figure 103: Cumulative copper recovery by size after the sulphide and oxide flotation stages, mixed ore.

5.4.4 Eh-pH diagram:

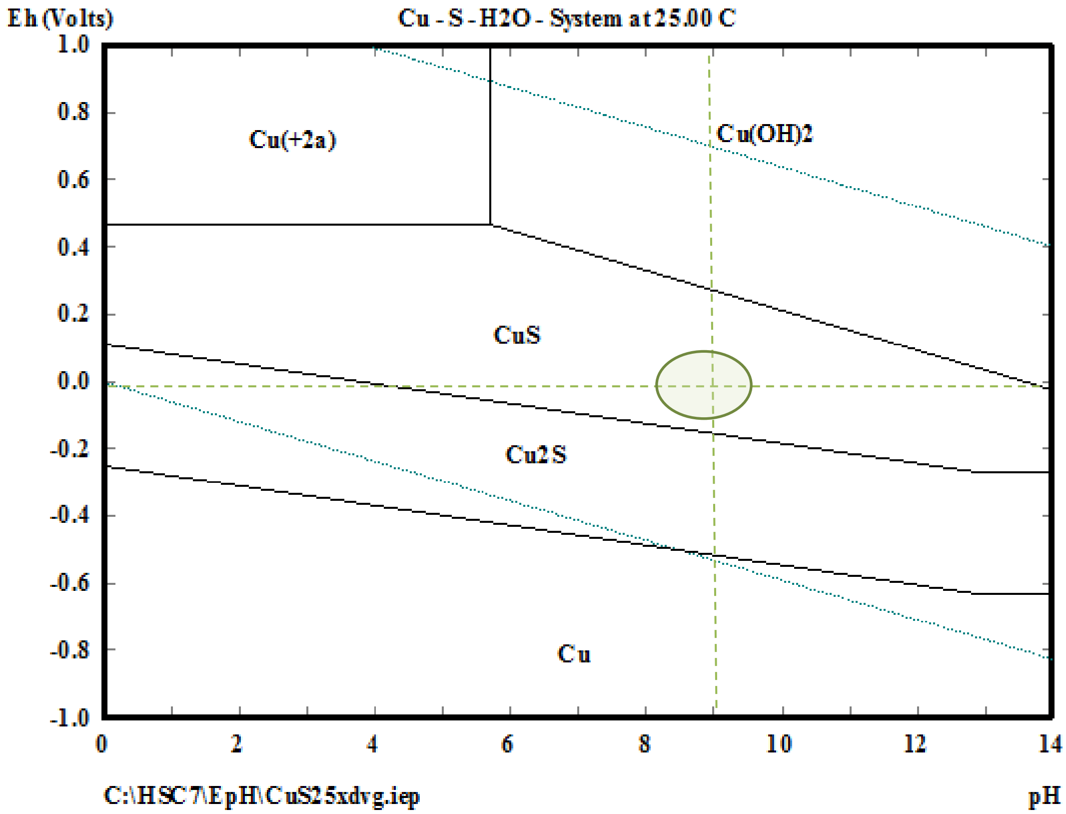

Figure 104: Eh-pH diagram for the Cu-S-H_2O system. [Cu]=10^{-6} M and [S] =10^{-11} M. Sulphur oxidation state: -II. Green circle corresponds to the Eh-pH window measured during the sulphidisation stage. Blue lines correspond to water dissociation limits (generated by HSC Chemistry®).

Table 9: Thermodynamic data for the construction of the Eh-pH diagrams.

Species	ΔG_{298} (Kcal/M)	Species	ΔG_{298} (Kcal/M)
Cu	0	*S*	0
CuO	-30.97	HS^-	2.97
Cu_2O	-34.86	HS_2^-	2.75
$Cu(OH)_2$	-89.07	S^{2-}	20.54
CuS	-12.84	SO_3^{2-}	-116.28
Cu_2S	21.18	SO_4^{2-}	-177.9
Cu^{2+}	15.54	$S2O_3^{2-}$	-123.96
Cu^+	11.94		

5.4.5 Capillary electrophoresis

Pulp samples from the forged steel and ceramic systems were collected after the sulphidisation stage when the Es was equal to -360 mV. The samples were directly filtered through a 0.45 μm filter and the liquid was analysed by capillary electrophoresis at the University of Liège.

In addition to tests completed on the forged steel and ceramic system, a blank sample with Liège tap water (the water used in the experiments) was carried out. The results for the water, forged and ceramic systems are presented here below:

- Water

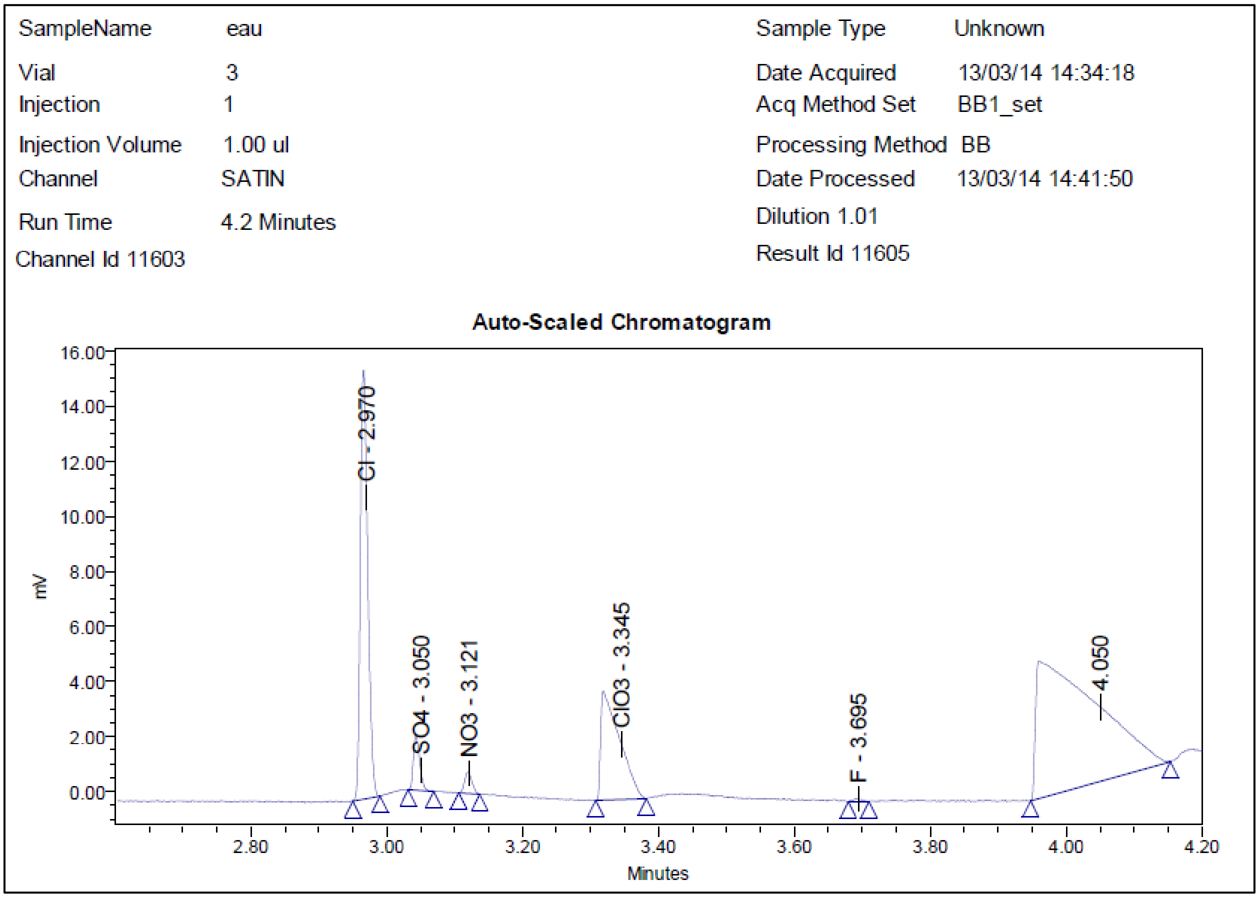

Figure 105: Capillary electrophoresis spectrum and conditions, water samples.

Table 10: Peak results from capillary electrophoresis, water sample.

Name	Migration time	Area	Height	Amount	R^2
Br	2.771	-	-	-	0.99
NO_2	2.904	-	-	-	0.99
Cl	2.970	11630	15611	24.090	0.99
SO_4	3.050	1289	2055	3.666	0.99
NO_3	3.121	588	769	1.880	0.99
ClO_3	3.345	7630	3969	1.000	1.00
H_2PO_4	3.651	-	-	-	0.99
F	3.695	103	113	0.71	0.99
HCO_3	4.276	-	-	-	-
S_2O_3		-	-		-

V. Synthetic mixed ore

- <u>Forged steel</u>

SampleName	FG	Sample Type	Unknown
Vial	2	Date Acquired	13/03/14 14:09:58
Injection	1	Acq Method Set	BB1_set
Injection Volume	1.00 ul	Processing Method	BB
Channel	SATIN	Date Processed	13/03/14 14:15:27
Run Time	4.2 Minutes	Dilution 5.00	
Channel Id 11594		Result Id 11598	

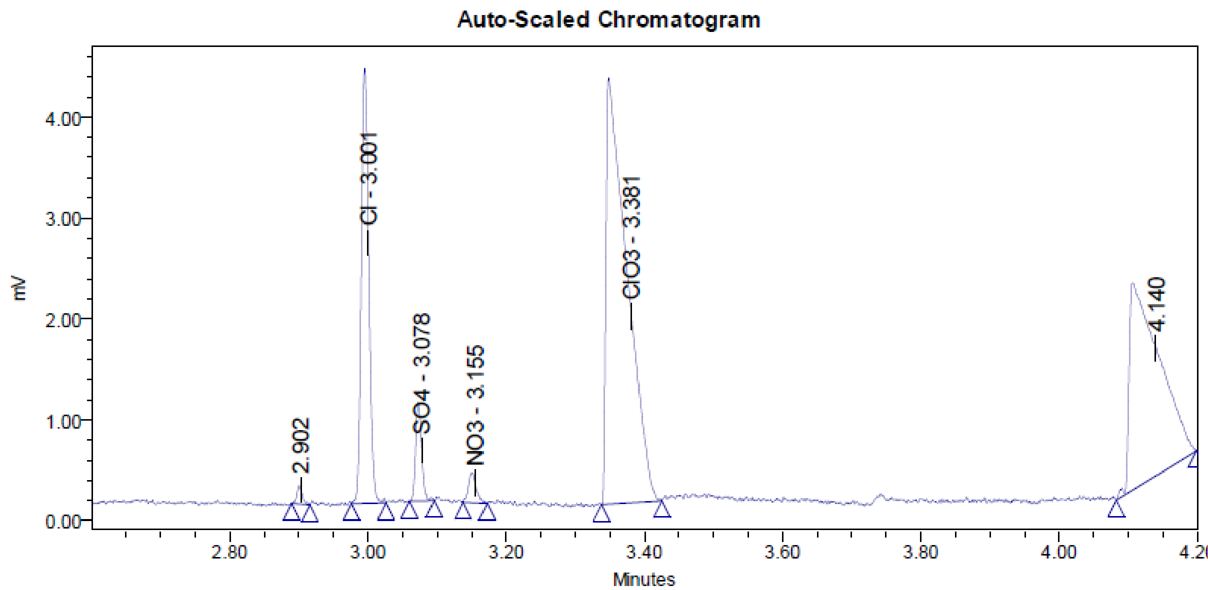

Figure 106: Capillary electrophoresis spectrum and conditions, forged steel samples.

Table 11: Peak results from capillary electrophoresis, forged steel sample.

Name	Migration time	Area	Height	Amount	R^2
Br	2.771	-	-	-	0.99
NO_2	2.904	-	-	-	0.99
Cl^-	3.001	3282	4321	28.887	0.99
SO_4^{2-}	3.078	597	986	7.231	0.99
NO_3	3.155	239	293	3.253	0.99
ClO_3	3.381	8893	4230	1.000	1.00
H_2PO_4	3.465	-	-	-	0.99
F	3.651	-	-	-	0.99
HCO_3	4.276	-	-	-	-
$S_2O_3^{2-}$		103			-

V. Synthetic mixed ore

- <u>Ceramic</u>

SampleName	CER	Sample Type	Unknown
Vial	1	Date Acquired	13/03/14 11:26:56
Injection	1	Acq Method Set	BB1_set
Injection Volume	1.00 ul	Processing Method	BB
Channel	SATIN	Date Processed	13/03/14 14:35:17
Run Time	4.2 Minutes	Dilution 5.00	
Channel Id 11589		Result Id 11604	

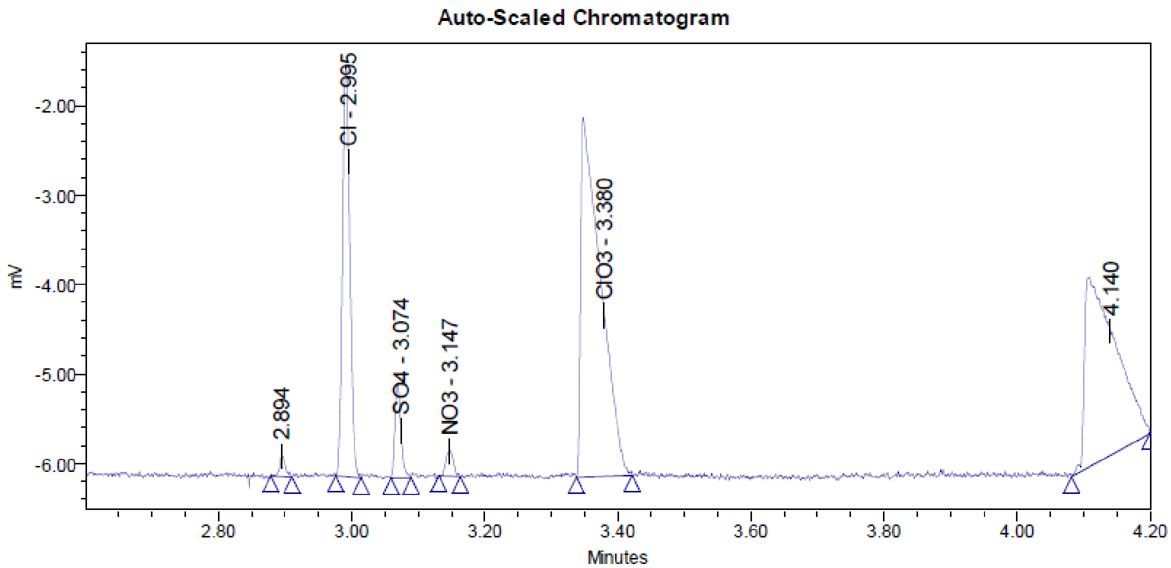

Figure 107: Capillary electrophoresis spectrum and conditions, ceramic samples.

Table 12: Peak results from capillary electrophoresis, ceramic sample.

Name	Migration time	Area	Height	Amount	R^2
Br	2.771	-	-	-	0.99
NO_2	2.904	-	-	-	0.99
Cl	2.995	3516	4612	33.085	0.99
SO_4	3.074	654	1069	8.459	0.99
NO_3	3.147	220	285	3.194	0.99
ClO_3	3.38	8330	4040	1.000	1.00
F	3.465	-	-	-	0.99
H_2PO_4	3.651	-	-	-	0.99
HCO_3	4.276	-	-	-	0.99
S_2O_3		146			

V. Synthetic mixed ore

5.4.6 Higher NaHS

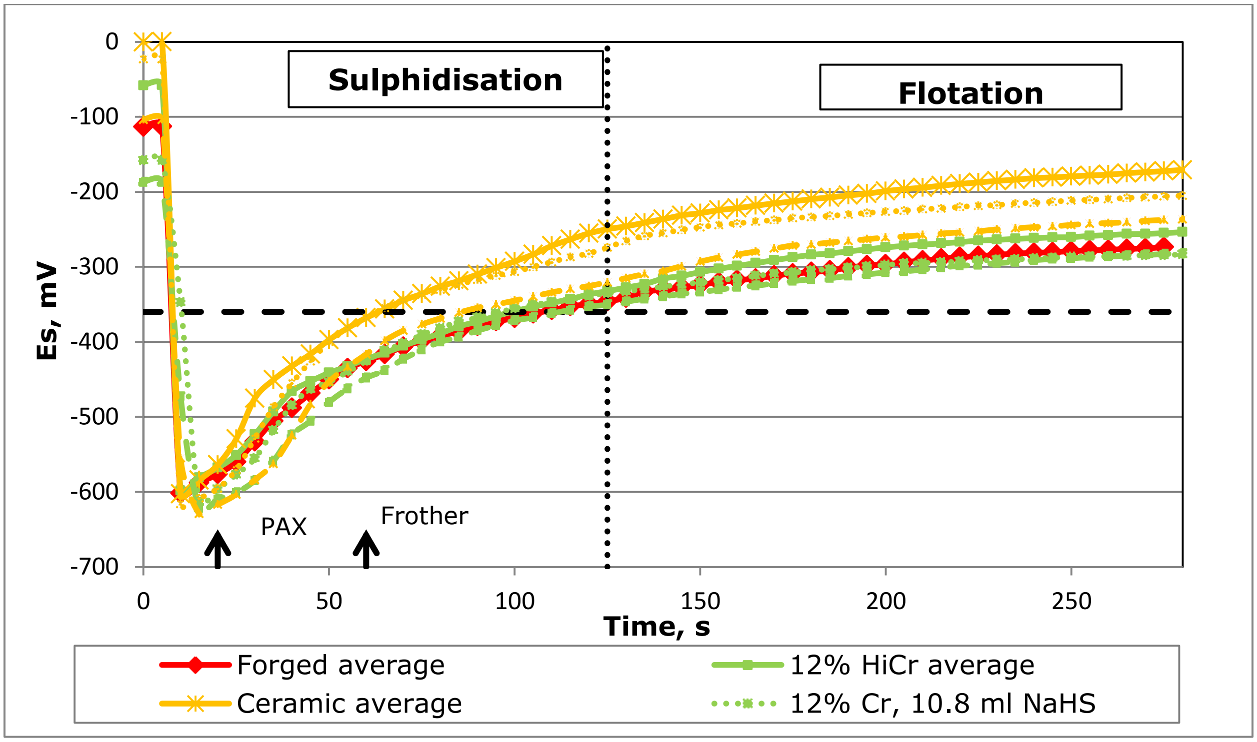

Figure 108: Es profiles during the sulphidisation stages on a mixed ore ground with several grinding media and for various NaHS dosages.

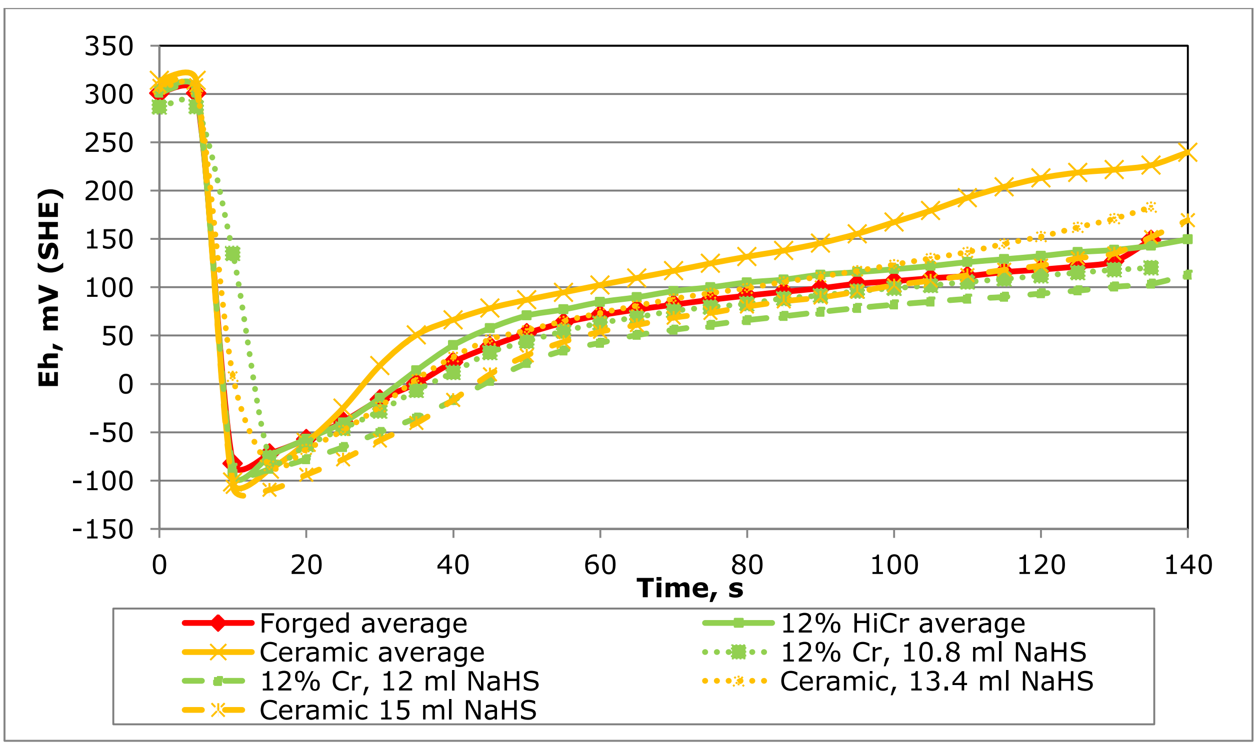

Figure 109: Eh profiles during the sulphidisation stages on a mixed ore ground with several grinding media and for various NaHS dosages.

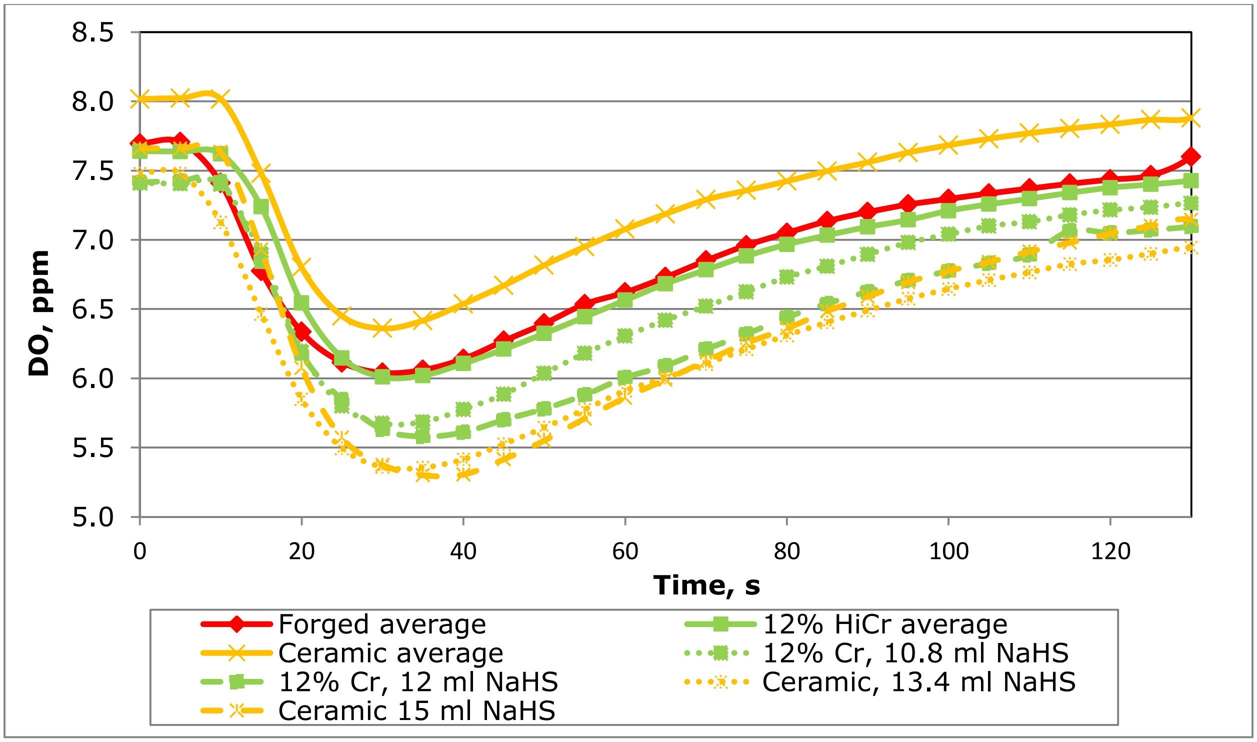

Figure 110: DO profiles during the sulphidisation stages on a mixed ore ground with several grinding media and for various NaHS dosages.

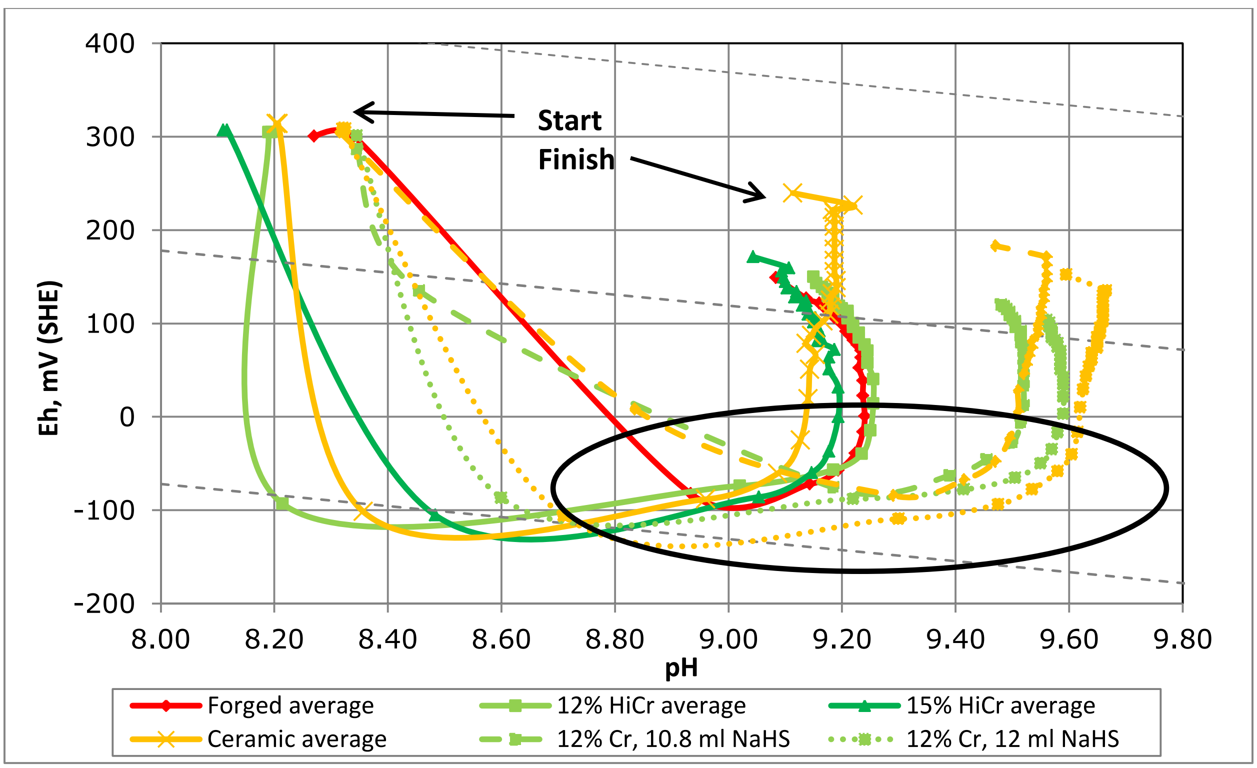

Figure 111: Eh-pH profiles during the sulphidisation stages of a mixed ore ground with several grinding media and for various NaHS dosages.

5.4.7 $FeSO_4$

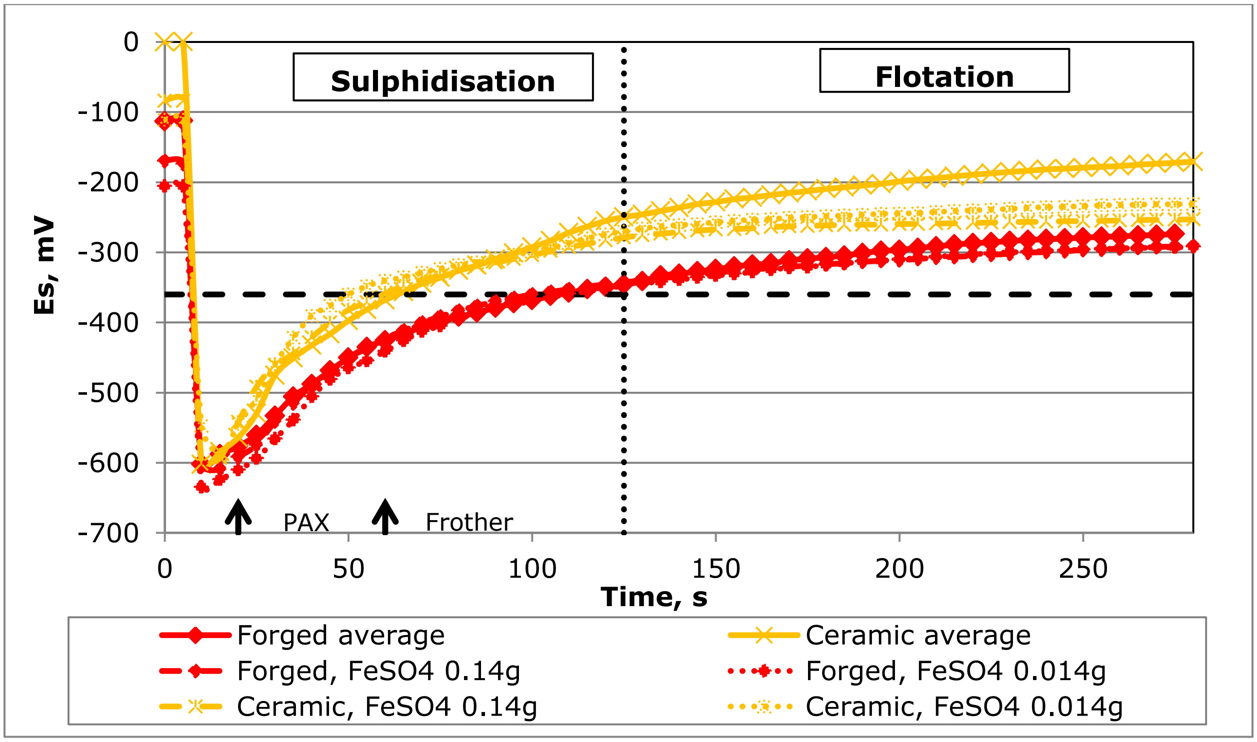

Figure 112: Es profiles during the sulphidisation stages of a mixed ore ground with forged steel and ceramic media and for various $FeSO_4$ dosages.

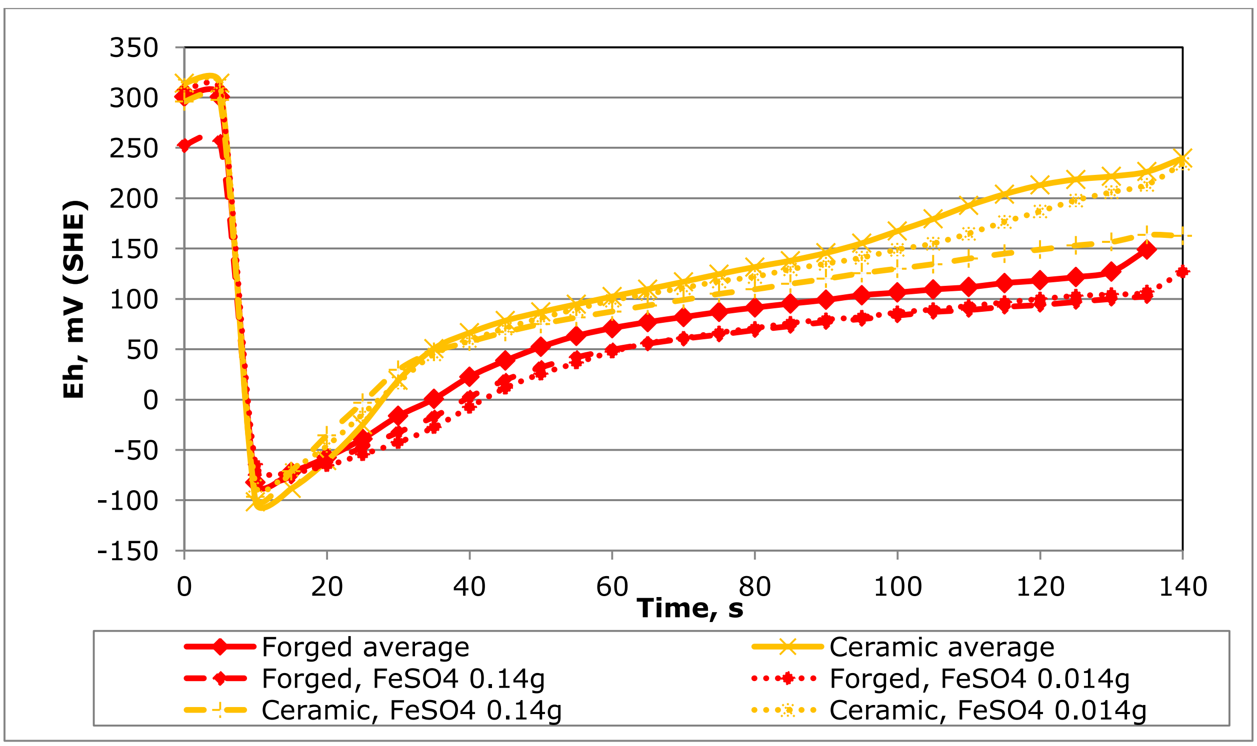

Figure 113: Eh profiles during the sulphidisation stages of a mixed ore ground with forged steel and ceramic media and for various $FeSO_4$ dosages.

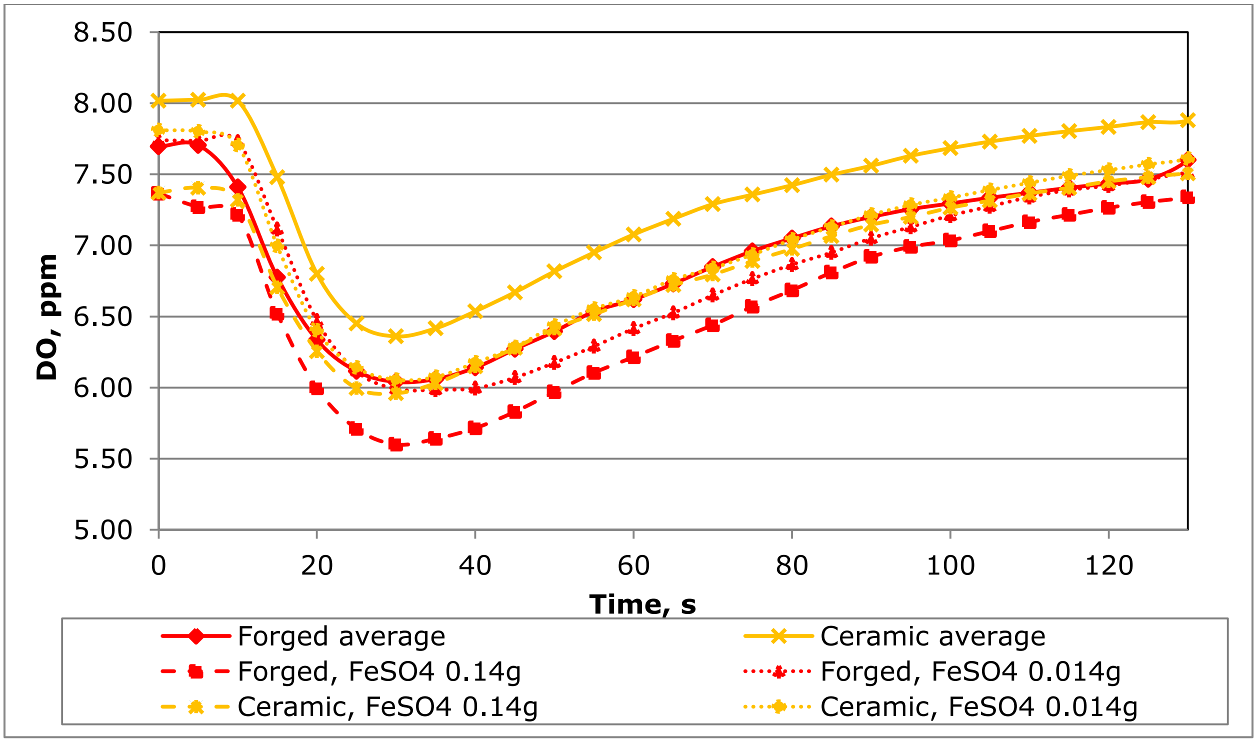

Figure 114: DO profiles during the sulphidisation stages of a mixed ore ground with forged steel and ceramic media and for various $FeSO_4$ dosages.

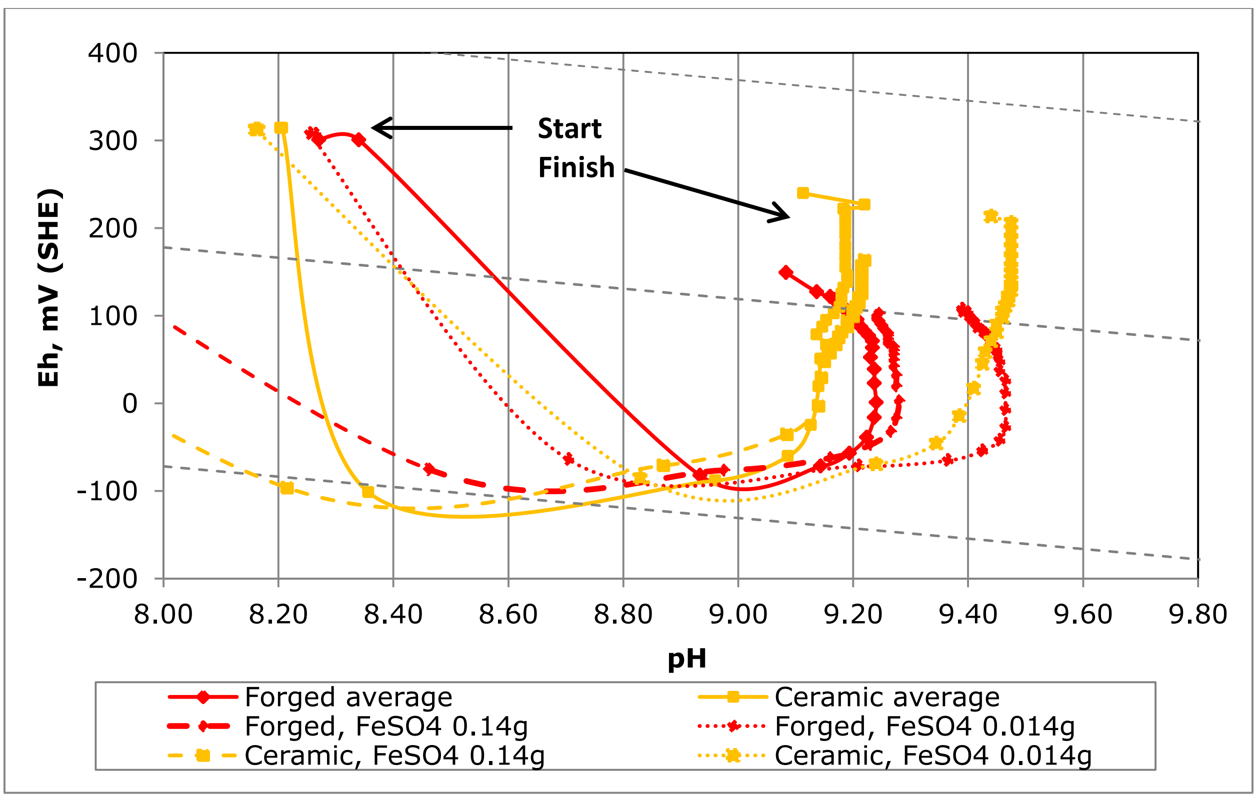

Figure 115: Eh-pH profiles during the sulphidisation stages of a mixed ore ground with forged steel and ceramic media and for various $FeSO_4$ dosages.

5.4.8 N_2 purging

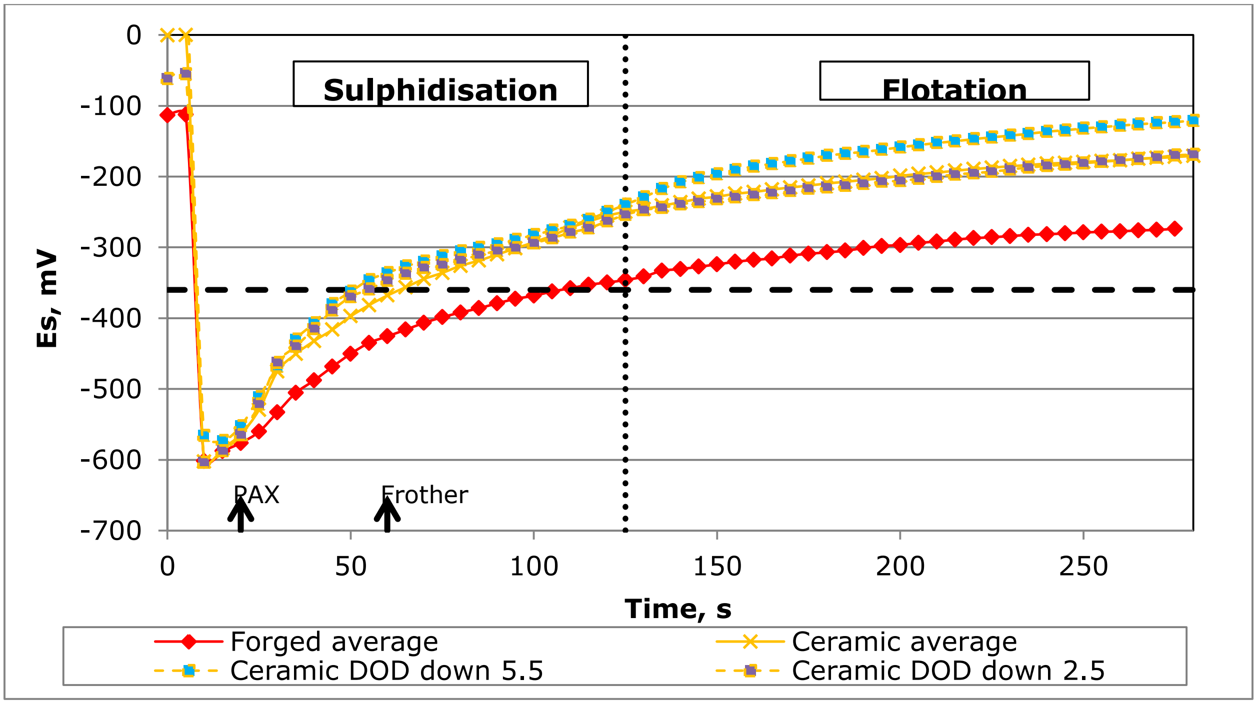

Figure 116: Es profiles during the sulphidisation stages of a mixed ore ground with forged steel and ceramic media under two different nitrogen purging conditions.

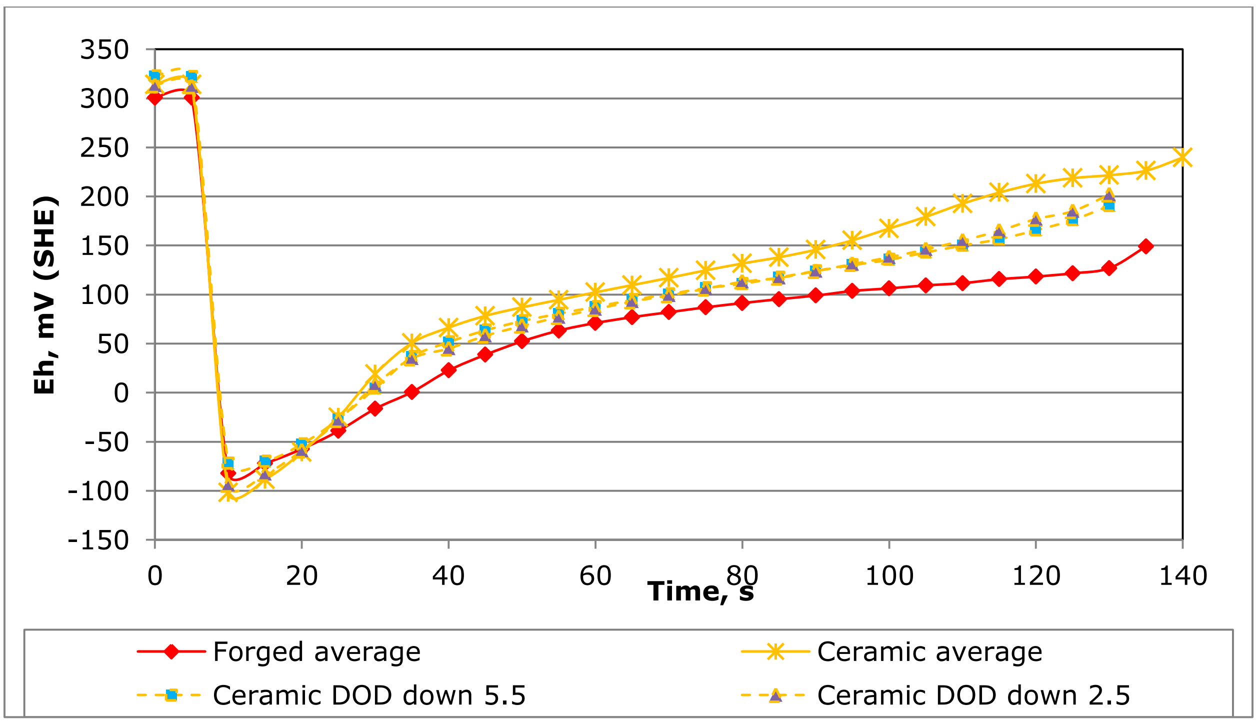

Figure 117: Eh profiles during the sulphidisation stages of a mixed ore ground with forged steel and ceramic media under two different nitrogen purging conditions.

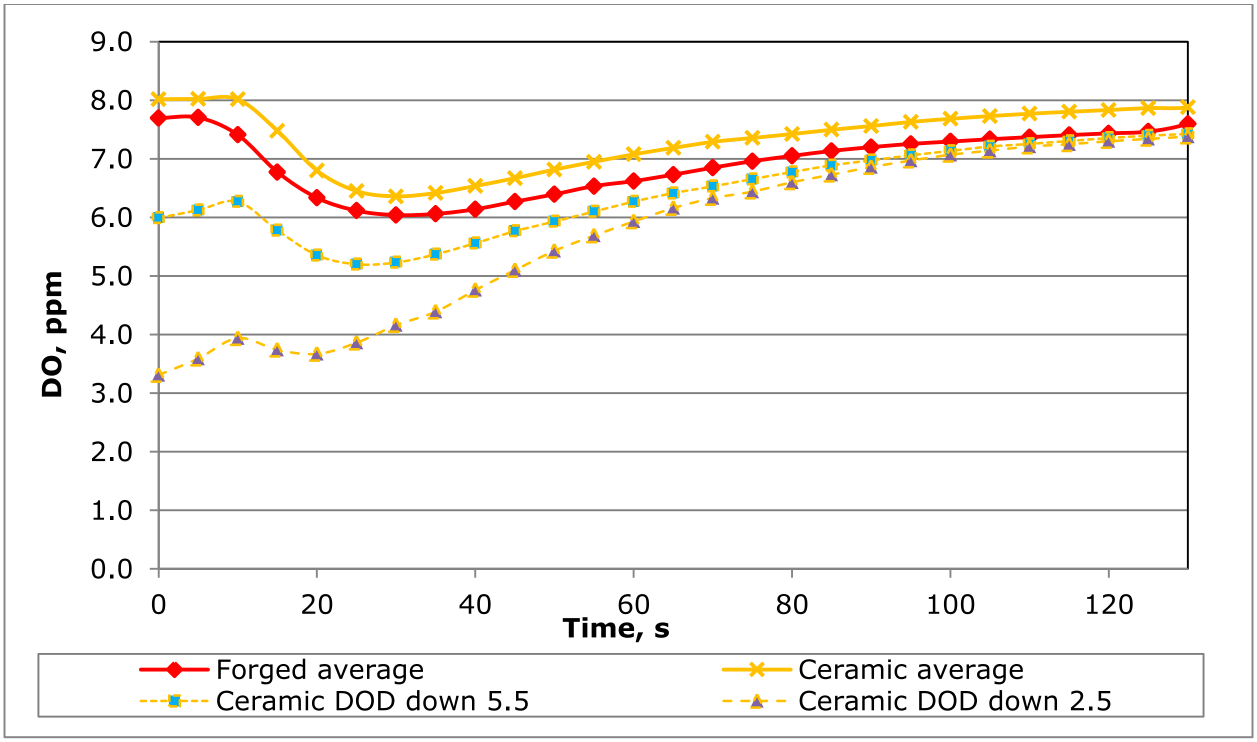

Figure 118: DO profiles during the sulphidisation stages of a mixed ore ground with forged steel and ceramic media under two different nitrogen purging conditions.

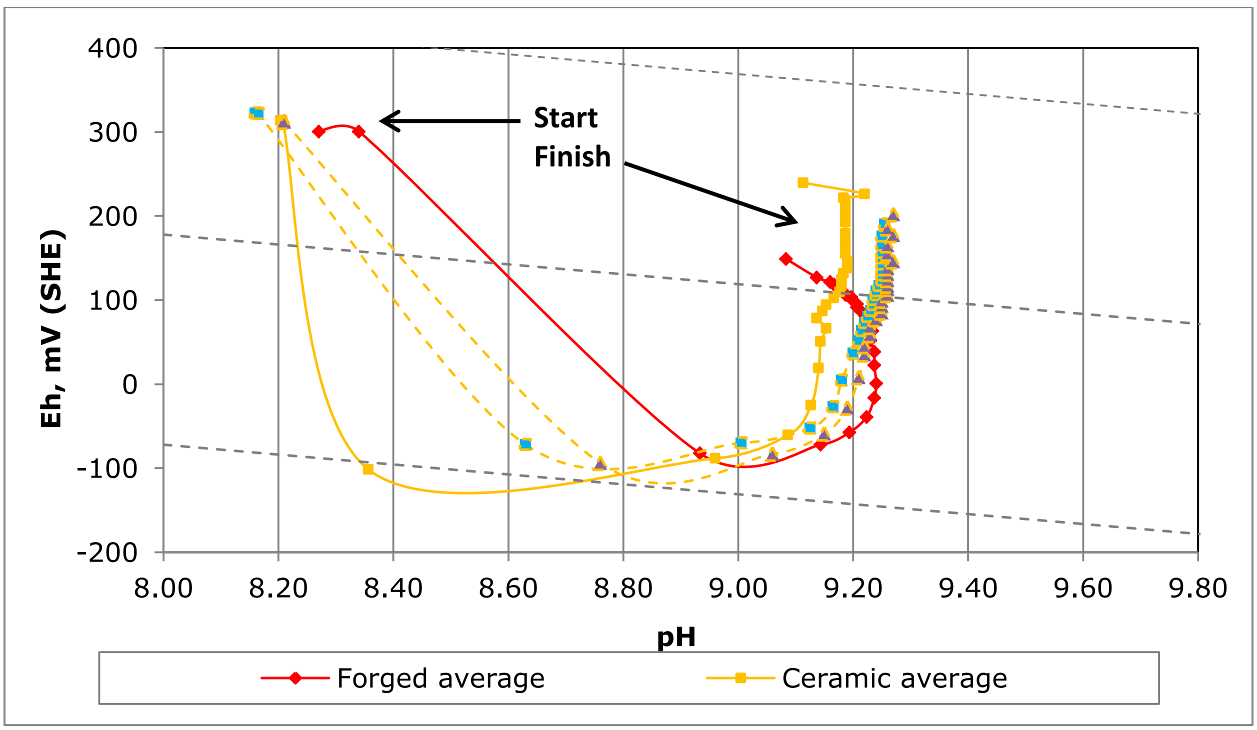

Figure 119: Eh-pH profiles during the sulphidisation stages of a mixed ore ground with forged steel and ceramic media under two different nitrogen purging conditions.