

Reduction rate of MnO from two different manganese ores producing ferromanganese

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Abstract – The present work examines the reduction rate of MnO by solid carbon from two different charges during the ferromanganese production process. Charge composition and temperature were used as experimental variables in this investigation. The emphasis was on how different charge compositions affect the reduction rate of MnO in the temperature range of 1400 to 1500°C. The experimental work was carried out in a thermogravimetric furnace in the presence of CO at atmospheric pressure. X-ray fluorescence (XRF) and electron probe microanalyzer (EPMA) techniques were performed on slags to measure the extent of MnO reduction as a function of temperature and charge composition. It was found that the rate of MnO reduction was strongly affected by temperature, while the charge composition seemed to have merely a slight effect on the reduction rate. The calculated activation energies were estimated to be in the range of 115 to 473 kJ/mol for Assmang charges, and from 457 to 517 kJ/mol for Comilog.

Keywords: FeMn, basicity, charge, reduction

INTRODUCTION

Production of high-carbon ferromanganese (HC FeMn) using the carbothermal reduction process is highly dependent on the ore properties, which are characterised by its chemical composition, mineralogical composition, CO reactivity, porosity, *etc.*, as they may influence the process itself as well as the final product quality (Sorensen *et al.*, 2010). Over the past years there have been publications from various investigations (Ostrovski *et al.*, 2002; Tranell *et al.*, 2007; among others) of the kinetics of MnO reduction during the ferromanganese production process.

At ferromanganese smelting temperatures, manganese oxide is reduced to metallic form by solid carbon, as shown by the following reaction:



Temperature and charge composition are important factors influencing the reduction rate of MnO. The MnO reduction rate will also determine the final MnO content of the slag phase.

Tangstad (1996) investigated the impact of charge composition on the reduction rate of MnO from a Broken Hill Propriety Company Ltd (BHP) manganese ore using different fluxing agents in the temperature range of 1400 to 1500°C. She showed that the reduction rate of MnO was highest for BHP ore combined with limestone, followed by BHP ore and magnesite, BHP ore and dolomite, whereas that of BHP ore with no flux had the lowest rate. It seems that the most acid charge gave the lowest reduction rate.

De Oliveira *et al.* (2011) studied MnO reduction from a manganese ore located in Erzincan (Turkey). It was found that the reduction rate of MnO by solid carbon increased with an increase of the basicity of the slag, whereas, according to Olsen *et al.* (2007), the reduction rate of a basic slag was slower in the two-phase area (MnO + liquid) compared to an acid slag, but higher in the one-liquid region.

The present study investigates the reduction rate of MnO during the FeMn production process from two different manganese ores, namely Assmang and Comilog ores. The respective basicities of unfluxed Assmang and Comilog ores are 1.3 and 0.04, which do not fall in the range of industrial ferromanganese slags (0.2 – 1.2). Therefore, lime and quartz were added to the charge to adjust the basicity to 0.4, 0.5, 0.7, 0.8, and 1.0. It is important to note that, in the present study, the charge basicity B was defined as the ratio of the total weight of CaO and MgO to the total weight of SiO₂ and Al₂O₃ in the charge, $[B=(CaO + MgO)/(SiO_2 + Al_2O_3)]$.

EXPERIMENTAL

To study the reduction rate of MnO by solid carbon, experiments were done in a thermogravimetric tube furnace which has the possibility to continuously record the mass loss during the reduction process and whose maximum temperature is 1700°C. The furnace setup is shown in Figure 1.

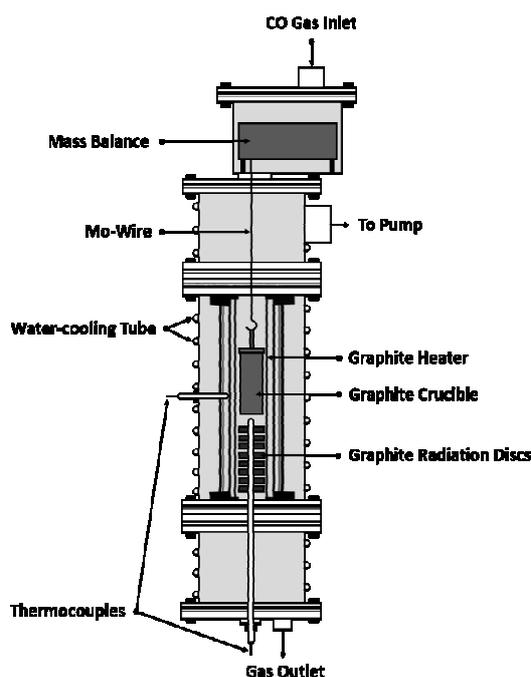


Figure 1: Schematic setup of the thermogravimetric furnace

Experimental procedure

In the experiments, predetermined amounts of manganese ore, lime, quartz, and Polish coke were carefully weighted, manually mixed, and charged into a crucible, then heated up to the target temperature. The crucible dimensions are as follows: external and internal diameter were 36 and 30 mm respectively; external and internal height were 70 and 61 mm respectively. The reduction took place as the manganese ore reacted with coke, as well as with (or without, in some cases) a certain amount of fluxing agent that it was mixed with according to the targeted basicity, in the presence of a CO gas atmosphere. The raw materials were mixed according to the following

procedure, shown in Table I, where it can be seen that the same amount of ore was used for every experiment.

Table I: Amounts of raw materials for different charges

Ore	Basicity	Mass ore	SiO ₂	CaO	C
Asm	1.3	30g	-		7.5g
Asm	1.0	30g	0.45g		7.5g
Asm	0.8	30g	0.90g		7.5g
Asm	0.5	30g	3.40g		7.5g
Com	0.04	30g		-	7.5g
Com	1.0	30g		7.20g	7.5g
Com	0.7	30g		5.15g	7.5g
Com	0.4	30g		2.43g	7.5g

*Asm - Assmang ore; **Com - Comilog ore.

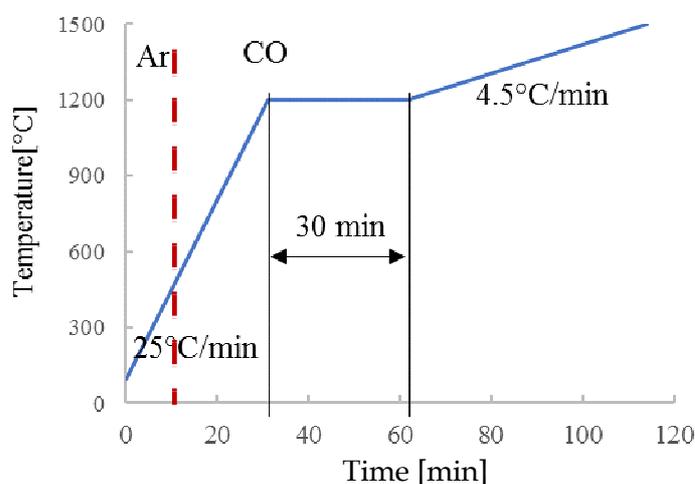


Figure 2: Temperature profile

As shown in Figure 2 above, from room temperature up to 500°C, the atmosphere inside the furnace was controlled by argon gas with a flowrate of 0.5 L/min. From 500°C to the target temperature (1400, 1450, and 1500°C), the gas atmosphere was switched from Ar to CO gas with the same flowrate. From room temperature to 1200°C, the charge was heated with a heating rate of 25°C/min. After reaching 1200°C, the charge was held at this temperature for 30 minutes, to ensure that all the pre-reduction reactions had taken place completely. Then, from 1200°C, the charge was heated up to the target temperature with a heating rate of 4.5°C; this temperature schedule is intended to simulate the industrial operation. The heating programme was then stopped after the target temperature was reached, the charge was cooled down inside the furnace using the same temperature profile but the other way around, and taken out at room temperature.

Material characterization

The materials used in this investigation were the two different manganese ores, Assmang and Comilog ores, the fluxing agents, quartz and lime, as well as the Polish coke as reducing agent. Tables 2 to 4 give details on their chemical compositions. The materials were separately crushed and screened to the size of +0.6 to -1.6 mm, suitable for use in the Thermogravimetric Graphite Tube Furnace. The raw materials were split

using the established cone and quartering method to obtain as representative a sample of the bulk as possible. Thus, finely ground samples were taken from the bulk and sent to SINTEF Molab AS for chemical characterisation using XRF, whose results are presented in the following tables.

Table II: Chemical composition of ores measured by XRF where MnO₂ is measured by titrimetric method (dry analyses, in wt%)

	MnO ₂	MnO	Fe ₂ O ₃	SiO ₂	P	Al ₂ O ₃	MgO	CaO	BaO	K ₂ O	S	CO ₂	xH ₂ O
Asm	36.7	31.4	15.8	5.14	0.03	0.90	0.77	7.10	0.57	0.03	0.21	3.70	0.80
Com	74.0	5.23	3.27	5.50	0.11	7.50	0.20	0.27	0.17	1.00	0.01	0.16	4.60

Table III: Chemical composition of lime, quartz, and coke (dry analyses, in wt%)

	MnO	Fe ₂ O ₃	SiO ₂	Al ₂ O ₃	MgO	CaO	S	C-fix	CO ₂
Lime	-	-	1.00	0.27	1.00	54.0	0.01	-	42.3
Quartz	0.14	-	93.9	1.19	0.05	0.09	-	-	-
Coke	0.04	0.86	5.60	2.79	0.22	0.42	0.44	87.7	-

RESULTS AND DISCUSSION

Weight loss behaviour

The weight loss was measured (in the TG Furnace) during heating, in order to investigate the MnO reduction rate. At this stage, the charge consists of MnO oxide or MnO-MgO solid solution and a liquid slag where dissolved MnO is reduced from. Attention was paid only to the reduction of MnO, while SiO₂ reduction was neglected, as this oxide is more stable than MnO, and will not be reduced at these temperatures and these basicities. During the reduction of MnO, the formation and removal of CO gas causes the weight loss, which is then recorded. For each ore, four different charges were heated up to 1400 and 1500°C, and the obtained weight loss curves are shown in Figures 3 to 6.

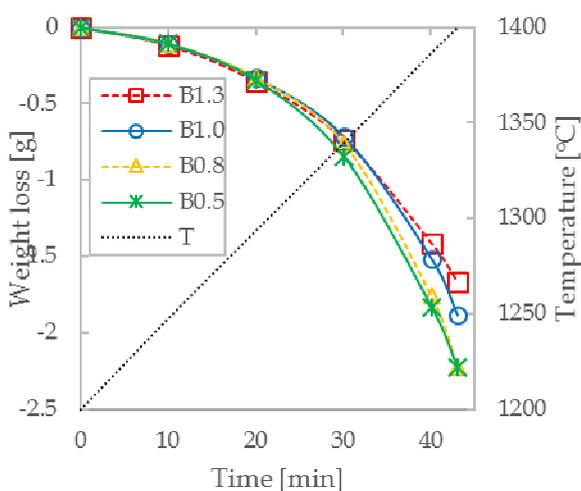


Figure 3: Reduction curves of Assmang charges with different basicities heated to 1400°C

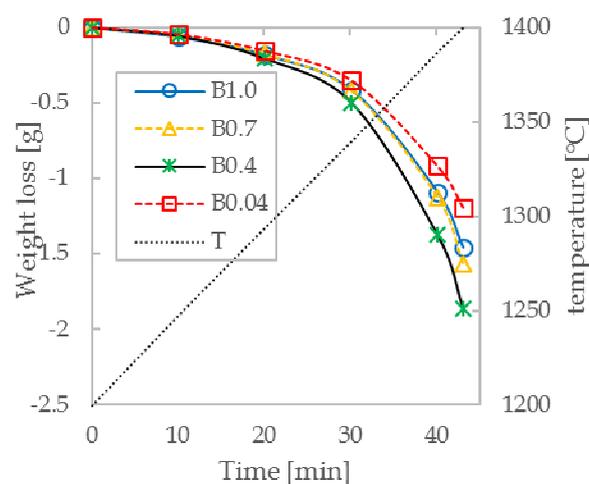


Figure 4: Reduction curves of Comilog charges with different basicities heated to 1400°C

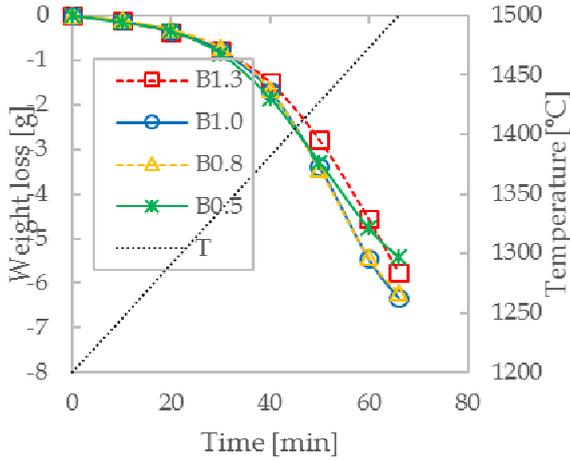


Figure 5: Reduction curves of Assmang charges with different basicities heated to 1500°C

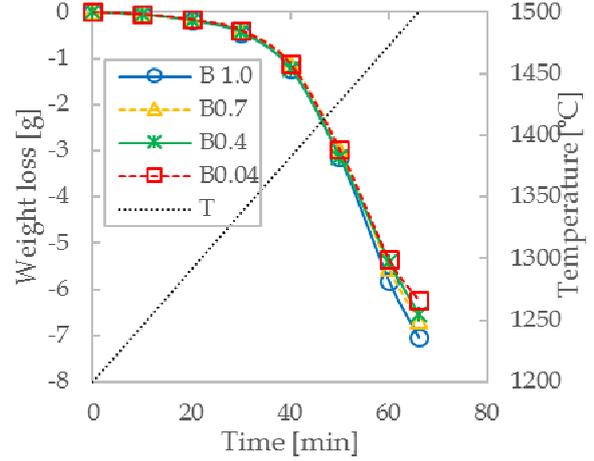


Figure 6: Reduction curves of Comilog charges with different basicities heated to 1500°C

Table IV: Measured weight loss values at each temperature from different charges, from Fig.5

	B0.5	B0.8	B1.0	B1.3
Asm T1400 [g]	-2.23	-2.22	-1.97	-1.67
Asm T1450 [g]	-4.06	-4.40	-4.43	-3.61
Asm T1500 [g]	-5.43	-6.21	-6.33	-5.75

Table V: Measured weight loss values at each temperature from different charges, from Fig.6

	B0.04	B0.4	B0.7	B1.0
Com T1400 [g]	-1.19	-1.87	-1.56	-1.52
Com T1450 [g]	-4.17	-4.29	-4.21	-4.42
Com T1500 [g]	-6.23	-6.54	-6.67	-7.03

Figures 3 to 6 show the reduction curves of Assmang and Comilog charges respectively, while Tables IV and V show the recorded weight loss for different experiments in the predetermined conditions. From these results, one can easily see how the temperature affects the MnO reduction for both ores, as the weight loss increases with temperature. On the other hand, the effect of adding quartz and lime to Assmang and Comilog charges respectively can also be perceived when looking at the values listed in Tables IV and V, the trend being that the weight loss increases when the basicity increases. However, at $T = 1400^{\circ}\text{C}$, the weight loss seemed to decrease when increasing the basicity for both ores; this fact is difficult to explain and not in agreement with results from previous studies (Olsen & Tangstad, 1995; De Oliveira *et al.*, 2011; Cengizler & Eric, 2016), but seems to be in accordance with Olsen *et al.* (2007) who found that, at the MnO + liq area, the acid charges reduce faster, whereas at high temperatures 1450 and 1500°C the degree of reduction increased with temperature. In view of these results, one can notice that there are relatively small differences between the weight loss and hence the extent of reduction with different basicities. On the other hand, in the industrial basicity area 0.4 - 1, it can be seen that at low temperatures and extents of reduction, that is in the MnO + liquid phase area, the reduction rate increases with lower basicity, as stated earlier on (Olsen *et al.*, 2007). At higher temperatures, and higher extents of reduction, that is in the liquid area, the reduction rate is faster with increasing basicities. It must, however, be noticed that the extreme basicities of 1.3 and 0.04 show some deviation to this. The reduction rate is given by Equation [2].

$$r_{\text{MnO}} = \frac{g \text{ MnO}}{\text{min}} = k * A * (a_{\text{MnO}} - a_{\text{MnO,eq}}) = k * A * \left(a_{\text{MnO}} - \frac{a_{\text{Mn,eq}}}{K_T} \right) \quad [2]$$

where r_{MnO} is the reduction rate (g/min), A is the interfacial area (cm^2), a_{MnO} is the activity of MnO in the slag phase, a_{Mn} is the activity of Mn in the metal and K_T is the

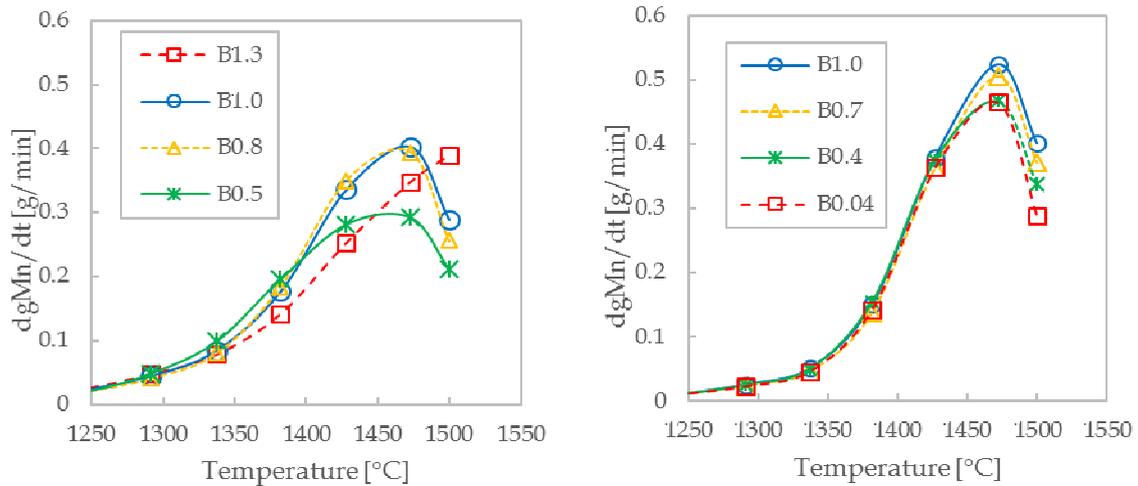
equilibrium constant at temperature T , k is the rate constant ($\text{g}/\text{min cm}^2$) described by Equation [3] below, where k_0 is the frequency factor, E_a is the activation energy of MnO (kJ/mol), R is the ideal gas constant ($\text{J}/\text{K} \cdot \text{mol}$), and T is the temperature (K).

$$k = k_0 * e^{-E_a/RT} \quad [3]$$

When the slag is a complete liquid, MnO activity is determined by applying the following expression, obtained by fitting and modelling of MnO activity data from FactSage 6.4, as suggested by Olsen (2016), in the temperature range of 1500-1600°C and %MnO range of ≤ 60 wt%.

$$\begin{aligned} a_{MnO} = & 0.0007576T - 123.7C_{MnO} + 30.14C_{SiO_2} + 47.84C_{MgO} + 49.54C_{CaO} - 47.96C_{Al_2O_3} \\ & + 122.8C_{MnO}^2 - 67.78C_{SiO_2}^2 - 46.32C_{MgO}^2 - 47.68C_{CaO}^2 + 22.51C_{Al_2O_3}^2 + 78.35C_{MnO}C_{CaO} \\ & + 77.56C_{MnO}C_{MgO} + 176.6C_{MnO}C_{Al_2O_3} + 101.2C_{MnO}C_{SiO_2} - 71.52C_{SiO_2}C_{CaO} \\ & - 70.58C_{SiO_2}C_{MgO} + 27.35C_{SiO_2}C_{Al_2O_3} + 46C_{SiO_2}^3 - 92.97C_{CaO}C_{MgO} + 2.44C \end{aligned} \quad [4]$$

where, C_{MnO} , C_{SiO_2} , C_{CaO} , C_{MgO} , $C_{Al_2O_3}$ designate the weight % of MnO, SiO₂, CaO, MgO, and Al₂O₃ respectively in the slag phase. When the slag contains a solid MnO phase in coexistence with a liquid phase, the activity of MnO will be equal to the molar fraction of MnO in the solid phase, which is found by EPMA analysis.



(a) Assmang series

(b) Comilog series

Figure 7: Reduction rate of MnO ($d\text{gMn}/dt$) versus temperature of Assmang (a) and Comilog (b) charges for different basicities, based on Figures 5 and 6

Figure 7 shows the calculated reduction rate as a function of temperature; the trend is that the rate increases with an increase in temperature. However, it can be observed that at relatively low temperatures, about 1300 to 1400°C, for both ores, the lower the basicity is, the higher the reduction rate is, as shown by their respective curves being on top, while, at temperatures higher than 1430°C, the reduction rate abruptly increases as the charge basicity increases until it reaches a peak at about 1475°C, then it decreases with about the same speed until the temperature of 1500°C was reached. This fact was observed in a similar study conducted by Li (Li & Tangstad, 2017). This decrease of the reduction rate observed at high temperature was due to the fact that the MnO content of the slag which dropped significantly at high temperatures decreased thus leading to a decrease of the driving force as reported in Table VII. One can also notice that charges with the same basicity ($B = 1$) had the highest rate for both ores at

about 1475°C, as their respective curves appeared on top, but Comilog charge showed a rate much higher than Assmang, thus indicating how much more reactive Comilog ore is at high temperatures than it is at low temperatures.

Slag composition

The XRF analysis method was performed on the obtained slags to determine their chemical composition.

Table VI: Chemical composition of end slags

T1500°C								
Asm	MnO	SiO ₂	CaO	MgO	Al ₂ O ₃	Fe ₂ O ₃	C	Tot.
B1.3	47.1	18.8	25.5	2.8	3.3	0.2	-	97.7
B1.0	32.9	26.5	28.5	2.8	4.4	0.2	-	95.3
B0.8	33.9	30	25.2	2.7	3.7	0.3	-	95.8
B0.5	37.4	34.5	18.5	2	3	0.2	-	95.6
Com								
B1.0	18.6	17.3	39.3	0.42	22.4	0.09	-	98.0
B0.7	26.9	16.2	27.7	0.60	26.1	0.07	-	97.6
B0.4	36.3	19.5	16.4	1.05	16.2	0.08	-	95.1
B0.04	48.8	20.7	1.00	0.75	28.2	0.10	-	99.5
T1400°C								
Asm	MnO	SiO ₂	CaO	MgO	Al ₂ O ₃	Fe ₂ O ₃	C	Tot.
B1.0	58	11	9.9	0.9	1.6	8.7	4.6	95
B0.8	57	15	11	0.9	1.5	7.6	4.7	97.7
B0.5	55	22	11	1.1	1.4	4.3	2.4	97.2
Com								
B0.4	56	8.4	14	0.2	13	1.9	5.4	99.2

The presence of iron as well as some carbon in the slag (T = 1400°C) shows that the slag was contaminated, as it was assumed that almost all the iron content would end up in the metal phase, therefore these results are discarded, thus leading to the use of results from experiments at 1500°C to recalculate the slag compositions for experiments at 1400°C and 1450°C, by considering the recorded weight loss in order to determine the kinetic parameters.

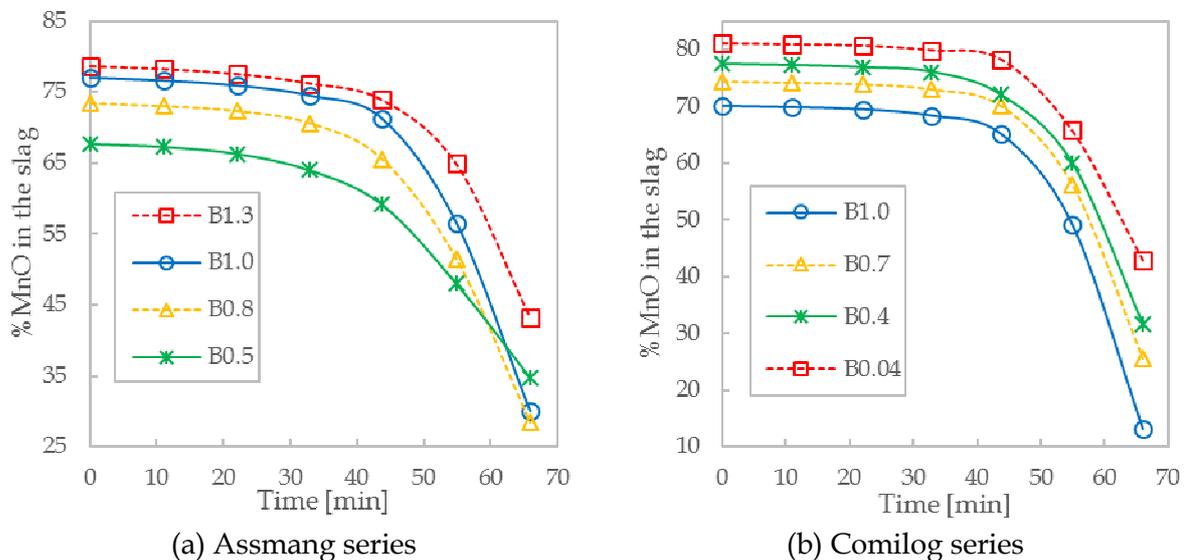


Figure 8: Effect of basicity on the residual %MnO in the slag

Table VII: Parameters for kinetic calculations

Ore	T[°C]	Reduction rate gMnO/min	a_{MnO}	Metal mole composition			a_{Mn}	K_T	Driving force
				Mn	Fe	C			
Assmang B=1.3	1400	0.17	0.952	0.466	0.477	0.057	0.218	21.8	0.942
	1450	0.30	0.411	0.637	0.302	0.062	0.328	36.9	0.402
	1500	0.38	0.396	0.719	0.215	0.065	0.375	60.5	0.390
Assmang + Quartz B=1.0	1400	0.24	0.988	0.449	0.454	0.058	0.235	21.8	0.977
	1450	0.38	0.997	0.676	0.261	0.064	0.345	36.9	0.988
	1500	0.29	0.306	0.734	0.199	0.066	0.387	60.5	0.225
Assmang + Quartz B=0.8	1400	0.25	0.930	0.533	0.409	0.058	0.260	21.8	0.896
	1450	0.39	0.844	0.675	0.262	0.063	0.358	36.9	0.834
	1500	0.26	0.378	0.732	0.202	0.066	0.260	60.5	0.372
Assmang + Quartz B=0.5	1400	0.23	0.952	0.533	0.409	0.058	0.340	21.8	0.936
	1450	0.30	0.410	0.675	0.262	0.063	0.368	36.9	0.400
	1500	0.21	0.150	0.710	0.224	0.065	0.359	60.5	0.144
Comilog + Lime B=1.0	1400	0.25	0.988	0.750	0.185	0.066	0.378	21.8	0.971
	1450	0.48	0.870	0.859	0.072	0.069	0.437	36.9	0.858
	1500	0.40	0.192	0.883	0.047	0.070	0.454	60.5	0.184
Comilog + Lime B=0.7	1400	0.25	0.922	0.756	0.180	0.064	0.400	21.8	0.904
	1450	0.46	0.900	0.856	0.076	0.068	0.448	36.9	0.887
	1500	0.38	0.191	0.880	0.049	0.070	0.448	60.5	0.184
Comilog + Lime B=0.4	1400	0.24	0.900	0.779	0.155	0.066	0.401	21.8	0.882
	1450	0.44	0.681	0.857	0.074	0.069	0.437	36.9	0.669
	1500	0.34	0.155	0.88	0.050	0.071	0.448	60.5	0.148
Comilog B=0.04	1400	0.23	0.920	0.713	0.223	0.064	0.363	21.8	0.903
	1450	0.43	0.497	0.855	0.076	0.069	0.438	36.9	0.485
	1500	0.29	0.121	0.877	0.052	0.070	0.449	60.5	0.114

Figure 8 shows the effect of basicity on the residual MnO in the slag, for (a) Assmang and (b) Comilog charges respectively, while Table VII lists the kinetic parameters. From Figure 8, it can be seen that the addition of silica and quartz to Assmang and Comilog charges respectively, dilutes the MnO concentration of the primary slag. With an increase in slag basicity from 0.4 to 1.0 for Comilog ore, the MnO content of the slag decreased from 36.3 to 18.6% MnO, which, in turn, increases the extent of MnO reduction, while with an increase of basicity from 0.5 to 1.0 for Assmang ore, the MnO content decreased from 37.4 down to 32.9% MnO in the slag. This shows how strong the effect of basicity is in the acid slags compared to basic slag.

Assuming that manganese oxide is reduced solely by coke, whose particles are spherical with a diameter of 1.1 mm, and density considered equal to unity, the coke consumption can be determined based on the weight loss listed in Tables IV and V. Thus, the rate constant can be calculated as shown in Figure 8, by the Arrhenius plots of MnO reduction for all Assmang and Comilog charges.

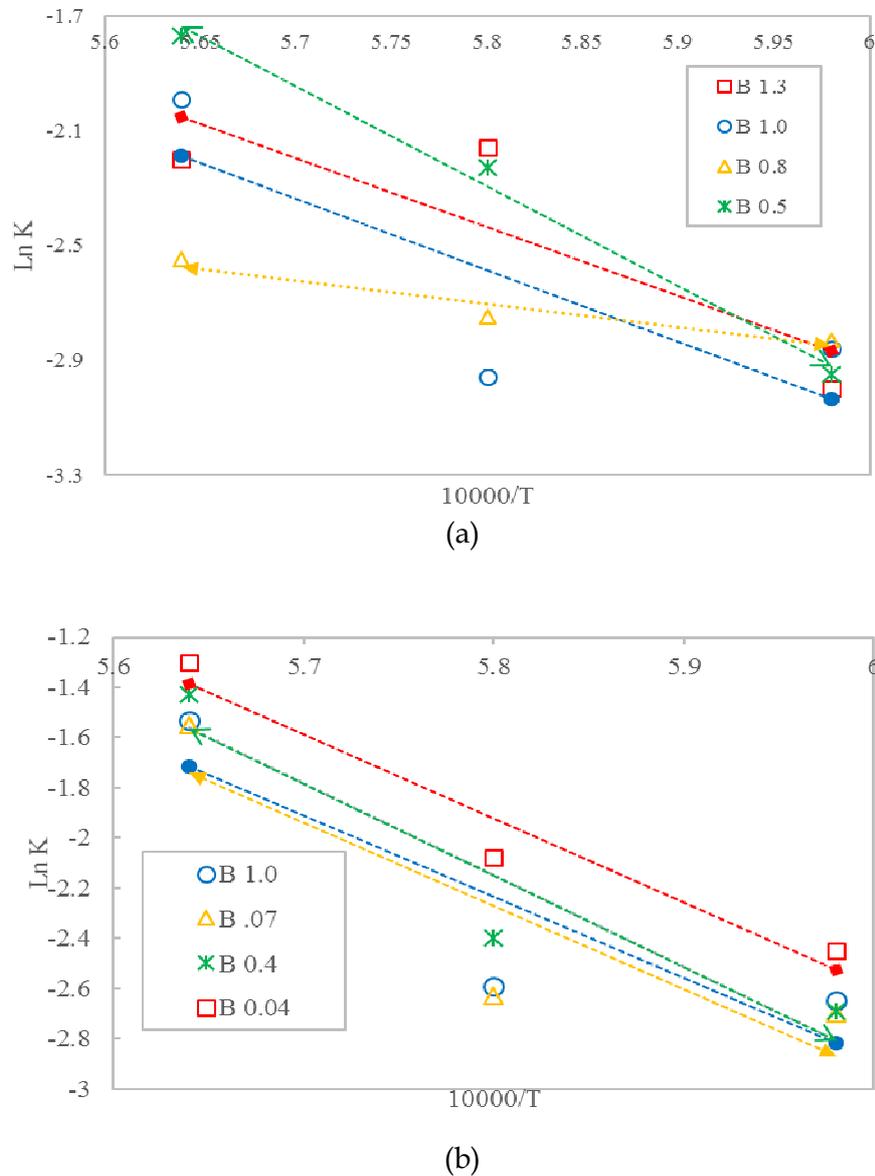


Figure 9: Arrhenius plots of MnO reduction for all Assmang (a) and Comilog (b) charges

Figure 9 shows the Arrhenius plots for MnO reduction for all charges, where it can be seen that the rate constant increases with an increase in temperature, in accordance with its expression [3].

Table VIII: Estimated activation energies for all Assmang and Comilog charges ($1400^{\circ}\text{C} < T < 1500^{\circ}\text{C}$)

Charge	E_a [kJ/mol]	Temperature [$^{\circ}\text{C}$]	k [g/min cm^2]
Asm	388	1450	$91.3 \cdot 10^{-3}$
Com	479	1450	$90.8 \cdot 10^{-3}$

From the results listed in Table VIII, it can be seen that Comilog ore has a higher activation energy than that of Assmang ore, while their rate constants are about the same.

CONCLUSIONS

The reduction rate of MnO from two differently based charges was investigated. Basicity and temperature were used as experimental parameters. Upon investigation of the effect of temperature on the reduction rate of MnO, it was found, for both ores, that the reduction rate increased significantly with an increase in temperature. The addition of quartz to Assmang charges, to vary the charge basicity from $B = 1.3$ to $B = 1.0$, and from $B = 0.8$ to $B = 0.5$, resulted in increasing the weight loss for temperatures of 1450 and 1500°C, while the opposite was observed for the temperature of 1400°C. The same observation was made when adding lime to Comilog charges for different basicities from $B = 0.04$ to $B = 0.4$, and from $B = 0.7$ to $B = 1.0$, at 1400°C, the addition of lime seemed not to promote the MnO reduction, while at 1450 and 1500°C it resulted in increasing the weight loss. Thus, it should be said that the rate of MnO reduction will at high temperatures, that is at a high degree of reduction, increase with the basicity. At lower temperatures, that is at a lower degree of reduction, the trend is opposite, that the rate of MnO reduction will decrease with increasing basicity, as shown in Figure 7. This is in accordance with Olsø's results (Olsø, Tangstad, & Olsen, 1998) on MnO reduction, where it was found that, at low degrees of reduction, the amounts of solid phase would be higher for higher basicities, and for high degrees of reduction, the extent of MnO reduction would increase with increasing basicity, as the MnO reduction follows the viscosity. The exception in this work seems to be the extreme basicities of 0.04 and 1.3 for Comilog and Assmang ores respectively, which will always have a lower rate of MnO reduction compared to this trend. It must also be said that the basicity has a very minor influence on the rate of MnO reduction. At 1500°C, the difference between the reduction degree for Comilog based charges at the minimum value ($B = 0.04$) is still almost 90% of the maximum value ($B = 1$). The activation energy of MnO reduction between 1400 and 1500°C was calculated to be an average of 388 kJ/mol for Assmang charges, with a higher average value of 479 kJ/mol for Comilog charges. This also shows how temperature dependent Comilog ore is, compared to Assmang ore.

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