Coke Gasification in Blast Furnace Shaft Conditions with H₂ and H₂O Containing Atmospheres

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Keywords: coke gasification, water vapor, hydrogen, solution-loss reaction, blast furnace

Abstract

Most of world steel in 2018 has been produced from virgin iron ore via the blast furnace – basic oxygen furnace route. Therewith the blast furnace is one of the most important unit processes in worldwide steel making. Among others, energy efficiency of the blast furnace is dependent on coke reactivity. Coke gasification occurs via solution-loss reactions either with CO₂ or H₂O. In this work the gasification of coke is studied in CO-CO₂-H₂-H₂O-N₂ atmosphere simulating the blast furnace conditions. The simulated atmospheres are based on the measurements from an actual blast furnace. This research studies the effect of the composition of the BF atmosphere representing conditions near the wall as well as at the center and influence of the H₂O content on coke gasification under simulated blast furnace conditions.

It was found out that the location plays a role in coke gasification: wall atmosphere yields higher coke gasification compared to center. Dynamic tests showed that introduction of H₂ and H₂O in gas atmosphere fastens coke gasification by +119% at temperature range between 800 and 1200°C in wall conditions. When

H₂O was present in gas atmosphere the mass loss of coke was also greater in both wall and center conditions.

Introduction

Two third of the worlds steel production in 2018 have been produced from virgin iron ore by the blast furnace process [1]. In the blast furnace coke has many different roles including but not limited to being a source of reductant gas and energy, being material bed's structural support and carburization of hot metal.

Coke gasification occurs with CO₂ and H₂O with so called solution-loss reactions presented here

It has been shown that partial replacement of CO/CO₂ with H₂/H₂O increased the chemical reactivity of coke despite the gasifying components (CO₂ + H₂O) remaining constant. There are also indicators that the gasification behavior changes when H₂ and H₂O is added to the atmosphere thus increasing importance of existence of both in test atmospheres. [2-5] The reaction rate when mixing CO₂ and H₂O is not the sum of pure-gas reaction rates, but rather a complex mixture of the two. [3]

When blast furnaces get larger and injection rate of PCI is increased the mechanical strength of coke becomes more important. At the same time hydrogen content is also increasing in blast furnaces due to the oxygen-enriched coal injection. [5, 6]. Other potential sources of H₂ are injection of steam, oil or natural gas. When fuel injection is increased, the mechanical strength of coke becomes more important as well due to coke longer residence time. [7, 8]

There has been research about post-reaction strength of post-catalyst-added highly reactive coke. It was found out that a decrease in reaction temperature yields opposite effects depending if the catalyst is added before or after coke oven. [9]

Energy efficiency of blast furnace is dependent, among others, on coke reactivity. Furthermore, coke needs to act as a mechanical support network inside the blast furnace and enable gas movement through the

voids between the coke particles [10]. This requirement supports the need for coke to be strong enough in the lower parts of the blast furnace.

Sun *et al.* [11] compared three different coke behavior in blast furnace temperature-gas compositions achieved from literature in order to see which kinetic model should be used when describing gasification kinetics of coke in blast furnace. These temperature-atmosphere programs consisted of CO, CO₂, H₂, H₂O and N₂. However, there is not available detailed information of effect of increased water content on coke gasification reactions at the different locations at blast furnace shaft area in published literature.

This study focuses on the gasification of coke in simulated blast furnace atmosphere consisting of CO, CO₂, H₂, H₂O and N₂. The atmosphere information is accumulated from an actual blast furnace using a Multi-Point Vertical Probe (MPVP). This research examines the effect of H₂O as well as the location (wall vs center) on coke gasification under simulated blast furnace shaft conditions.

Experimental

Experimental material used was industrial metallurgical coke with chemical composition presented in **Table 1**.

Table 1. Chemical composition (wt-%) for used coke.

C	Fe	CaO	SiO ₂	Al ₂ O ₃	MgO	Mn	P ₂ O ₅	S	Na ₂ O	K ₂ O	ZnO	TiO ₂
88.82	0.49	0.20	5.24	3.05	0.07	0.007	0.06	0.73	0.06	0.13	0.002	0.16

Coke samples were shaped into a cylinder form with a diameter of 18.8 mm and a height of 12.0 mm. The initial weight of samples varied between 3.0 and 4.0 g. The TGA-device used in the experiments is presented in a previous work by Haapakangas et al. [2] and it was used to gasify coke particles in 1 atm pressure. The furnace allows using different atmospheres having gas components such as N₂, CO, CO₂, H₂ and H₂O. The water is provided using a peristaltic pump and then vaporized directly to the gas line. The furnace tube has diameter of 32 mm and is made from Al₂O₃ allowing temperature to be as high as 1500°C. The gas flow rate was 2l/min. The samples were placed in a crate which was made from drag wire. The furnace allows for the atmosphere and temperature to change with time. Coke samples were held in 110°C

overnight in order to remove moisture before tests. The samples were weighted before and after each experiment with a calibrated scale (Mettler Toledo model MS204TS/00, Mettler-Toledo GmbH, Greifensee, Switzerland) in order to verify the mass loss that was measured with the scale of the custom-made TGA. One coke cylinder at a time was placed in a TGA device with changing temperature and gas composition. The used temperature-gas pairs containing H_2 and H_2O were acquired from actual measurements taken place in an industrial blast furnace. Herewith, a Multi-Point Vertical Probe (MPVP) equipped with six temperature measuring and gas sampling points was used. The MPVP probe was introduced into the blast furnace, deposited above the burden surface and then descended with burden downwards. During the descent of the probe, the radial temperature mapping and gas analyses were determined, including contents of CO , CO_2 and H_2 . In addition to those gas components, H_2O and N_2 are always present in the BF shaft gas, but the contents of those were not measured. A detailed description of the multi-point vertical probing technology can be found elsewhere. [12, 13] For our experimental work, we used the results of a MPVP trial performed at No 4 blast furnace of ROGESA (Roheisengesellschaft) in Dillingen, Germany. Since H_2O measurements were not available, the H_2O content was calculated using thermodynamic software, HSC Chemistry version 9.6 [14] so that the partial equilibrium pressure of oxygen for the carbon dioxide and water vapor formation reactions were equal in CO - CO_2 - N_2 gas (atmosphere A) and in CO - CO_2 - H_2 - H_2O - N_2 gas (atmosphere B). The reduction potential of CO and H_2 were equal during the reduction process since CO/CO_2 and H_2/H_2O were thermodynamically balanced to achieve this. The remainder of the gas was assumed to be N_2 .

Time information for dynamic tests as well gas profiles are presented in **Table 2** and a presentation for dynamic test conditions for wall and center profiles for atmosphere B can be seen in **Figure 1**, note that gas composition used in isothermal tests at $1200^\circ C$ are bolded. Holding time in isothermal tests was 360 minutes. In dynamics tests the sample was put in a pre-heated furnace ($800^\circ C$) under nitrogen atmosphere and test program started after temperature of the sample had stabilized. Cooling period was also made in nitrogen atmosphere. Each test was repeated 3–5 times.

Table 2. Gas compositions, temperatures and cumulative time scale used in dynamic tests. Isothermal test gas composition at $1200^\circ C$ is bolded.

Temperature			800°C	850°C	900°C	950°C	1000°C	1050°C	1100°C	1200°C (also isothermal)
CO (vol-%)	Wall	Atm. B	27.8	28.9	30.0	31.0	34.1	37.9	40.2	39.3
		Atm. A	29.7	31.0	32.6	33.9	37.3	41.1	43.5	42.4
	Center	Atm. B	33.7	35.4	37.1	40.7	43.6	45.0	45.4	41.4
		Atm. A	36.4	38.4	40.2	44.0	47.3	48.9	49.4	45.3
CO ₂ (vol-%)	Wall	Atm. B	19.3	18.1	15.7	13.1	10.4	6.4	4.0	2.4
		Atm. A	20.7	19.5	17.0	14.3	11.4	6.9	4.3	2.6
	Center	Atm. B	12.1	9.5	7.3	3.2	1.3	1.0	1.0	1.0
		Atm. A	13.1	10.3	7.9	3.5	1.4	1.1	1.1	1.1
H ₂ (vol-%)	Wall	Atm. B	3.8	4.0	4.6	5.2	5.6	6.0	6.2	6.3
		Atm. A	-	-	-	-	-	-	-	-
	Center	Atm. B	5.5	5.9	6.1	6.6	7.4	7.6	7.7	8.0
		Atm. A	-	-	-	-	-	-	-	-
H ₂ O (vol-%)	Wall	Atm. B	2.5	2.8	3.2	3.3	2.9	1.9	1.3	1.0
		Atm. A	-	-	-	-	-	-	-	-
	Center	Atm. B	1.9	1.8	1.6	0.8	0.4	0.3	0.4	0.5
		Atm. A	-	-	-	-	-	-	-	-
N ₂ (vol-%)	Wall	Atm. B	46.5	46.2	46.5	47.4	47.0	47.9	48.2	51.0
		Atm. A	49.7	49.5	50.4	51.8	51.4	52.0	52.2	55.0
	Center	Atm. B	46.8	47.4	48.0	48.6	47.3	46.1	45.5	49.1
		Atm. A	50.6	51.4	52.0	52.5	51.4	50.1	49.5	53.7
P_{O_2} (atm)	Wall		1.85 $\cdot 10^{-19}$	2.52 $\cdot 10^{-18}$	2.29 $\cdot 10^{-17}$	1.59 $\cdot 10^{-16}$	7.31 $\cdot 10^{-16}$	1.64 $\cdot 10^{-15}$	3.75 $\cdot 10^{-15}$	4.03 $\cdot 10^{-14}$
	Center		4.95 $\cdot 10^{-19}$	4.61 $\cdot 10^{-19}$	3.24 $\cdot 10^{-18}$	5.61 $\cdot 10^{-18}$	6.96 $\cdot 10^{-18}$	2.89 $\cdot 10^{-17}$	1.82 $\cdot 10^{-16}$	6.17 $\cdot 10^{-15}$
Time (min)	Wall		0	6.7	18.7	26.8	65.7	75.3	82.0	90.2
	Center		0	5.9	11.8	17.7	22.0	25.4	28.9	36.6

Figure 1. Experimental conditions for wall and center profiles for atmosphere B.

Center gas composition is nearer to the equilibrium of gasification reaction of coke with CO_2 (Boudouard reaction), whereas wall conditions are more on the side of the product. The experimental programs and Boudouard reaction are presented **Figure 2** as a function of temperature and gas composition.

Figure 2. Experimental conditions and chemical equilibrium for coke gasification reactions as a function of temperature and gas composition at 1 atm.

When there is carbon dioxide and water vapor present in the atmosphere, water-gas-shift (WGS) reaction $\text{CO(g)} + \text{H}_2\text{O(g)} \leftrightarrow \text{CO}_2\text{(g)} + \text{H}_2\text{(g)}$ is also present. This also affects the balance between the gasifying components.

Results and discussion

Isothermal tests

In order to see the effect H_2 and H_2O have on the coke gasification, two different gas compositions (A+B) were used. The gas compositions differed on the water vapor and hydrogen contents (see Table 2). **Figure 3** presents sample mass loss at studied gas atmosphere in 1200°C for all repetition experiments. Average mass losses in water vapor and hydrogen atmospheres are 27.5% and 12.5% in wall and center conditions respectively. Similarly, the mass losses in $\text{CO-CO}_2\text{-N}_2$ atmospheres are 19.3% and 6.5% in wall and center conditions. It can be seen from the figures that water vapor has an effect on the coke reactivity in both gas compositions (wall and center). This effect could happen because water vapor is thought to be more penetrating into the porous structure when compared with CO_2 . This could be because the H_2O molecule is smaller in size and thus internal diffusion resistance is smaller when compared with CO_2 and CO_2 has

smaller effective diffusion coefficients (f. ex. CO_2 : $3.53 \cdot 10^{-7}$ (900°C), $8.39 \cdot 10^{-5}$ (1050°C) and H_2O : $1.03 \cdot 10^{-5}$ (900°C), $1.16 \cdot 10^{-2}$ (1050°C))[5].

Reactions happen also through cracks and pores with CO_2 and H_2O . It has been discussed that water vapor has more difficulties than CO_2 to diffuse to the center of coke. This is true especially at higher temperature. [10, 15] Due to this in higher temperature range coke degradation by CO_2 is stronger than that of H_2O . In lower temperature range H_2O has stronger destructive effect on coke. [10, 15]

Furthermore, it is suggested that due to the weaker bonds of hydrogen and oxygen in H_2O compared to the double bonds of oxygen and carbon in CO_2 molecule, the coal char reactivity is enhanced towards H_2O . [16] The presence of CO_2 seems to inhibit the faster $\text{C-H}_2\text{O}$ reaction [3] supporting also the theory of shared active sites. CO inhibits the CO_2 gasification [17, 18] just like hydrogen inhibits the steam gasification reaction [19, 20]. One reason for this inhibitive behavior it suggested to be the result from CO and H_2 blocking active sites [17-20].

Figure 3. Effect of H_2 and H_2O on coke gasification in wall and center conditions for all experiments.

The location where coke is situated inside the blast furnace (i. e. wall or center) has also an effect on the coke gasification. When looking at the sample mass loss in water vapor containing atmosphere (**Figure 3**), it can be seen that even when considering the variation between samples, the mass loss is greater in wall conditions. This is also to be expected since the main difference in the gas compositions (see Table 2 and Figure 1) between wall and center was that the wall composition had greater amount of CO₂ and H₂O compared to that of the center composition. This increase happens even with inhibition behaviors of CO and H₂.

It has been reported in literature that coke reactions with CO₂ are more affected by temperature than reactions with H₂O. This effect can be seen especially at temperatures above 1100°C. The weight loss rate of coke is higher with H₂O than with CO₂, average reaction rate with H₂O being 1.3–6.5 times that with CO₂ in temperature range 950–1250°C. The difference between these reaction rates decreases when temperature increases. [10]

Dynamic tests

In order to see more realistic effect of water vapor has on coke gasification, dynamic tests were carried out from 800°C to 1200°C under simulated BF conditions. The results for all repeated experiments can be seen in **Figure 4**. In wall conditions, high acceleration rate of coke can be found after 1100°C. Similar findings have been noted in previous study by Babich *et al.* [21], where it was found out that the acceleration of mass loss at 1100°C is not dependable on the coke reactivity. In other studies reaction rate has also been seen to change substantially at temperatures above ~1000°C (1300K [11], 1373–1674 K [22]). This happens because the resistance to chemical reaction is weakened in these high temperature conditions. Below this temperature chemical reaction was controlling step. In temperatures over ~1000°C (1300K) the chemical reaction and gas diffusion (porous diffusion [23]) are both controlling factors. [11]

The analogous effects compared to isothermal tests can also be seen in dynamic tests. The water vapor containing gas yields higher coke gasification. Of course, the difference is not as great as in isothermal tests

because the experiment time is not as long. This is still important to notice when making conclusions about industrial blast furnaces function.

Figure 4. Effect of H_2 and H_2O on the coke gasification in wall and center conditions in dynamic tests for all experiments.

The wall vs center conditions have also very similar effect on the coke sample mass loss as in the isothermal test. The wall conditions with water vapor yield again always greater mass loss than any tests made in the center conditions.

Threshold temperatures are calculated in order to see if the hydrogen and water vapor addition has an impact on the temperature when coke gasification starts. The threshold temperatures based on apparent reaction rate (ARR) method [24] in wall conditions are presented in **Table 3** and with an example graph for one experiment in **Figure 5**. Threshold is marked when the 10-value medium reaction rate is greater than

8% of maximum ARR indicating the onset of coke gasification. It can be seen that the apparent reaction rate of coke is slightly lower when water vapor and hydrogen are present in atmosphere.

Table 3. Threshold temperatures in wall conditions for all experiments.

	Threshold temperature (°C)	
	H ₂ /H ₂ O/CO/CO ₂ /N ₂	CO/CO ₂ /N ₂
Experiment 1	904	919
Experiment 2	902	918
Experiment 3	920	930
Experiment 4	904	881
Average	907.5	912

Figure 5. Mass loss of Experiment 1 at threshold temperature determination in wall conditions.

Discussion about results in context of industrial blast furnace

In industrial blast furnaces water vapor is always present and it has an accelerating effect on the coke gasification. As expected, the longer the experiment time is the greater the effect of water vapor is, but the effect can also be seen in dynamic tests where experiment times are shorter (about 36 min in the center conditions and about 90 min in the wall conditions) as mentioned in **Table 2**. These experimental times are determined such that the 0 min mark is

placed at the temperature of 800°C when the gasifying atmosphere was introduced in experiments. This difference in experiment details between wall and center conditions is due to that in the center the temperatures are higher thus heating the material faster and on the other hand near the wall the material flow is assumed to be faster because coke is burned in the raceways near walls. Therefore, the total material retention time difference is exactly converse to the difference between the experiment durations.

The industrial blast furnace can be operating at overpressure f. ex. 2-5 atm [13], the question of effect of pressure has on the chemical equilibrium of coke gasification reactions is raised. The effect of pressure for both carbon dioxide and water vapor is presented in **Figure 6**. Overall, it can be said that increase in pressure shifts the equilibrium curves towards higher temperatures. The pressure does not have an effect on the WGS reaction since the molecular amount does not change during the aforementioned reaction. On the other hand, the equation (1) has fewer moles on the source material side regardless of whether coke is gasified by CO₂ or H₂O. When pressure increases, the emphasis shifts to the source material side thus increasing the threshold temperature.

Figure 6. Effect of pressure on chemical equilibrium for coke gasification reactions.

It is known that the pass-through time for coke in blast furnace differs between the center and near the wall. As expected, the measured gas compositions also vary between center and near the wall areas. The

center condition has less water vapor and carbon dioxide than that near the wall. Both isothermal and dynamic tests made in this work show analogous results when looking at the gasification of coke – the sample mass loss is higher in near the wall atmosphere than in the center. According to the present study, coke gasification is +119 % faster in wall conditions than center conditions up to 1200°C temperature in atmosphere where water vapor and hydrogen are present.

If hydrogen injection is increased leading to elevated amounts of water vapor in the blast furnace shaft, coke reacts faster thus resulting in stricter requirements (for example mechanical strength) for coke quality especially in the lower part of the furnace. Elevated amounts of hydrogen affect also the reduction of the iron bearing layers in the furnace [25]. Thus, further research on the topic of hydrogen and water vapor in blast furnace is important.

Conclusions

This work focuses on how the presence of hydrogen and water vapor as well as the location inside blast furnace affect the coke gasification. These temperature – gas composition profiles were acquired from an actual industrial blast furnace using a Multi-Point Vertical Probe.

Isothermal tests were carried out at 1200°C in order to determine the effect H_2 and H_2O in gas on coke gasification. As expected, a difference could be seen: coke in a gas atmosphere without H_2 and H_2O gasified slower. This tendency could be seen in both – wall and center – atmospheres. The effect of location (i. e. wall or center) on coke gasification seems natural: the center atmosphere has less H_2O and thus mass loss of coke is slower.

Dynamic tests were also performed with wall and center atmospheres. The wall atmosphere yielded higher coke gasification than the center atmosphere, for example when hydrogen and water vapor were present in atmosphere average weight losses were in wall and center conditions –27.5% and –12.5%, respectively. When H_2O was present in gas the mass loss of coke was also greater –27.5% vs –19.3% in wall conditions and –12.5% vs –6.5% in center conditions, compared to $CO-CO_2-N_2$ gas.

Acknowledgements

The work presented here has been carried out with a financial grant from the Research Fund for Coal and Steel (RFCS) of the European Community with Grant Agreement No:709816 and project title “Online Blast Furnace Stack Status Monitoring”.

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