THE EFFECT OF POTASSIUM IMPREGNATION ON THE BOUDOUARD REACTIVITY OF SELECTED SINGLE-SOURCE AND COMMERCIAL COKES

J. Kaczorowski[1] and T. Lindstad[1,2]

[1]Department of Materials Science and Engineering, NTNU, 7491 Trondheim, Norway
[2]SINTEF Materials and Chemistry, 7465 Trondheim, Norway
E-mail: jakub.kaczorowski@material.ntnu.no, tor.lindstad@sintef.no

ABSTRACT

The Boudouard reactivity of single cokes produced from selected bituminous coals and commercial cokes has been investigated in a thermo gravimetric furnace. The effect of potassium as a catalyst for this reaction was studied, as it is known to accumulate in ferromanganese furnaces. A gaseous impregnation technique using K_2CO_3 as the potassium source was applied to impregnate the coke samples.

The reactivity experiments were designed to simulate conditions occurring in an industrial furnace, as used for production of Mn-alloys. The coke particle size varied from 3.35 to 6.63 mm, while the temperature was maintained between 800- 1100^{o}C with a gas composition from 50 to 75% CO_2 in a CO/CO_2 mixture. To determine the catalytic effect of potassium, the concentration varied from a fraction of a percent up to 15%wt.

The results show that single chars vary significantly in reactivity, depending on the properties of the parent coal, while commercial cokes exhibit similar reaction rates. Temperature and partial pressure of CO_2 have the strongest influence on the reactivity, while the influence of gas flow rate and coke particle size were found to be negligible. The catalytic studies revealed that the relative reactivity of non-impregnated samples are preserved after the impregnation, so the least reactive coke is still the least reactive after the impregnation for the whole range of potassium loads. Also, it has been confirmed that the Boudouard reactivity reaches a plateau at potassium concentration of 4%wt.

1. INTRODUCTION

The production process of ferromanganese is carried out by reducing ores in a shaft furnace by carbonaceous materials. Higher oxides are reduced first in the upper part of the furnace where the temperature is low. The ore descends and the lower oxides are gradually reduced until the lowest oxide is left at the bottom of the furnace to be reduced by solid carbon. On the reduction path, Mn_3O_4 is reduced to MnO by carbon monoxide that flows upwards. However, when the Boudouard reaction occurs simultaneously, the reduction step will run parallel with the carbon gasification step resulting in a “direct reduction” of the ore with solid carbon according to the reactions:

$$\frac{1}{3}Mn_3O_4 + \frac{1}{3}CO_{(g)} = MnO + \frac{1}{3}CO_{2(g)} \qquad \Delta H^0_{298} = -14,8kJ \qquad (1)$$

$$\frac{1}{3}C + \frac{1}{3}CO_{2(g)} = \frac{2}{3}CO_{(g)} \qquad \Delta H^0_{298} = +57,5kJ \qquad (2)$$

$$\frac{1}{3}Mn_3O_4 + \frac{1}{3}C = MnO + \frac{1}{3}CO_{(g)} \qquad \Delta H^0_{298} = +42,7kJ \qquad (3)$$

The presence of the Boudouard reaction consumes heat, making the reduction of Mn_3O_4 highly endothermic. In addition, the Boudouard reaction consumes carbon that should be used in the lower part of the furnace. These two facts make the Boudouard reaction unwanted in the smelting process. It has been shown that the rate of the Boudouard reaction is dependent on many factors including carbon properties, gas composition and the presence of catalytic compounds of which potassium is the most important.

The presence of alkali metals and alkaline-earth metals in a furnace has a negative effect on the smelting operation [1]. Alkalis are responsible for deterioration of the coke quality, lowering the life time of lining and refractory and often causing scaffolds. Alkalis are also known for generating high furnace pressures, causing a non uniform descend of the burden and uneven distribution of gasses. Swelling of the alkali compounds causes a decrease in burden permeability due to excessive fines generation.

Alkalis enter the furnace in raw materials. The most important alkali in the smelting operation is potassium as it is present in significant amounts, is easier gasified and harder to remove by the slag of all alkalis. Over 90% of the total potassium input is associated with the ore, while 8% enters with carbon materials. Even though the amount of alkalis entering the furnace is not high, its concentration in the furnace can reach a significant value due to circulation phenomenon.

Potassium enters the furnace mostly in the form of potassium silicates. Most of this silicate travels unreacted downwards to the coke zone, where some part of it goes to the slag and the rest is reduced with carbon. This reduced potassium is in gaseous state and flows upwards together with other gasses. Some part of this potassium gas is reversed via the reaction opposite to the reduction reaction (recirculation cycle 1), and another part flows higher to the colder region. At 700- 800°C, gaseous potassium reacts with CO and CO_2 to form potassium carbonate, both in the liquid and the solid state. The solid part partially leaves the furnace with flue gasses, and partially descends with the charge. The liquid potassium carbonate drips and impregnates the charge particles. As the potassium carbonate descends to the hotter region, it is reduced to metallic potassium that flows up again and the formation of the potassium carbonate is repeated. At this point, the second recirculation cycle is closed. This makes the two potassium recirculation cycles complete. The first cycle, lower recirculation, takes place by the reversed reduction reaction of the silicate in the temperature region between 1100 and 1400°C. The upper recirculation via the carbonate takes place between 1000-1050°C and 700-800°C.

The recirculation phenomenon besides providing catalyst for the Boudouard reaction causes the shift of the heat to the upper part of the furnace and involves carbon consumption. Assuming that 10 kg of K_2O is circulating per ton of metal, Sterneland [2] estimated that the lower circulation causes the transfer of 18 kWh/ton from the lower part of the furnace (1400°C) to the higher part at about 1100°C. Under the same assumption, the upper circulation will cause the shift of 27 kWh/ton from the 1000°C-region to higher region at 700°C. The carbon consumption was estimated to 0.64 kg of carbon per ton of metal.

Swamy [3] used a model to calculate the amount of carbon that is lost due to the Boudouard reaction for catalyzed and uncatalyzed coke samples. Assuming that the reaction is dependent on the Mn_3O_4 reduction characteristics (hence production of CO_2 by the reduction) in the temperature region of 800- 1200°C, the amount of carbon consumed was calculated to 1.67 kgC/t alloy for uncatalyzed and 14.8 kgC/t alloy for catalyzed reaction. From the above calculation it is clear that the alkali circulation will cause a strong increase in carbon loss due to the catalytic effect of potassium on the Boudouard reaction.

2. EXPERIMENTAL

2.1 Material

For the reactivity studies 6 different cokes have been selected, 3 commercial metallurgical cokes and 3 single source cokes. The commercial cokes were produced in Poland (PBC), Russia (RBC) and England (BBC) from unknown blends of coal. The cokes were received in the form of lumps of average size 10-30 mm.

The single cokes originate from a single seam coals produced in Poland (St), Australia (PD) and the USA (BG). The Australian coal comes from Peak Downs mine located in Queensland, and operating in the Bowen Basin of the Permian age. The sample was delivered in the form of lumps, sizing from coal dust up to 40 mm. The Polish coal had been produced in Staszic mine in the Upper Silesian basin. The coal was deposited during carboniferous age and represents lower rank coking coal. The lumps were of the size 30- 70 mm. The American coal was received from the Blue Gem mine, located in south-east Kentucky being a part of Appalachian basin where the coal was deposited during the Pennsylvanian age.

After the coals had been received they were air-dried for three days and sent to an external accredited laboratory for basic analyses. In addition to the analysis, the coal samples were carbonized to cokes of which properties were also determined. The Blue Gem coal was received as the run of mine coal and therefore prior to coking it was washed from ash using density separation method with $ZnCl_2$ solution as the washing medium.

2.2 Sample Preparation

The coal samples were carbonized in a laboratory scale furnace. The coking process took place in a 4 kg-capacity crucible placed in a retort furnace. The final temperature was 950°C and the heating rate 1.5- 2°C/min. After the final temperature was reached, the charge was left for an additional time (soaking time) of about 3 hours.

The Boudouard reactivity of alkali catalyzed cokes required impregnation of samples by potassium. The impregnation method was specially developed at NTNU for testing the resistance of carbon electrodes in aluminum industry [4]. A schematic representation of the impregnation is given in Figure 1. A graphite crucible with coke lying on the top of potassium carbonate mixed with activated carbon was inserted into an electric furnace where the temperature of about 1000°C was maintained for a number of hours depending on the desired potassium content. The heating rate of 300°C/h and free cooling after the impregnation has been used. To prevent the sample from oxidation, ambient gas (argon) is allowed to circulate inside the retort. The potassium content in the samples was measured by Inductively Coupled Plasma Mass Spectrometry (ICP/MS).

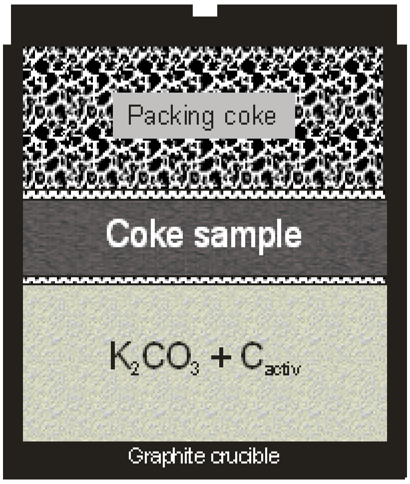

Figure 1: Schematic representation of the impregnation crucible

For the image analysis and petrography, polished pellets were produced. Particles of size 3.35 to 4.75mm for pore size distribution and 0.5- 1.0 mm for optical textures were embedded in a fluorescence epoxy. For better repeatability two pellets of larger particles were produced.

For the reactivity experiments a sample of 40 g was prepared from each coke by crushing the coke in a jaw crusher and sieving to the size range 3.35- 4.75 mm.

The X-ray technique requires use of fine powder, so for that purpose the cokes were crushed in a ball mill until they passed 106 µm test sieve. To be able to correct the position of the carbon peak due to shifting, a fine pure silicon standard was added in the amount of 10wt%.

2.3 Procedure

The proximate and ultimate analyses were done by en external laboratory following adequate ISO and PN standards.

For the petrographic studies a Leitz DM RXE microscope with attached MPV-SP intensity counter was used. In addition, the microscope was coupled with a digital camera, KAPPA CF 20 DXC. Three oil immersion lenses of magnification 20, 32 and 50 times were selected. The reflectance of about 300 vitrinite points was measured based on two reflectance standards and presented in the form of histogram. For the maceral constitution, a point-count method was employed. Maceral identification is based on size, structure, reflectance, degree of relief and association with other macerals. A minimum of 500 points was counted. The automatic point counter generated a uniformly spaced grid of points that under the magnification of 300 times were recognized and counted. The grid spacing was such that the majority of entities encountered during analysis coincide with only one grid point.

The physical properties were determined using standard methods and image analysis. The surface area was measured in FlowSorb II 2300 instrument using BET method. For the image analyses, a metallographic microscope, Leica Reichert MeF3A with an analog camera and dry lenses were used. Images of the surface were collected from computer-chosen locations and processed by special software to give crystallinity as the percentage of anisotropic areas.

The Boudouard reactivity was assessed using a thermobalance apparatus. The experimental station contained; an electric furnace, a steel crucible, a scale, CO/CO_2 analyzer, temperature controller, inlet gas regulator and a PC recording; time, temperature, mass and outlet gas composition. The sample was placed at the bottom of the crucible on a ceramic perforated plate. The crucible was suspended from the scale and inserted into the furnace so the hottest area surrounded the sample. The inlet gas was fed to the top of the crucible wherefrom it traveled to the sample between double-wall construction of the crucible. A detailed description of the reactivity apparatus can be found elsewhere [5].

To compare the reactivities of various cokes, standard experimental conditions have been chosen. The reactive gas contained 50% CO_2 in a CO_2/CO mixture was fed with the flow rate 4 Nl/min. The gas was introduced after the sample was heated in argon to the temperature of 1000°C.

For the influence of temperature, particle size, gas composition and flow rate, a factorial design of experiments at two levels has been applied. Setup conditions are presented in Table 1.

Table 1: Factorial design for evaluation of the importance of experimental conditions on the Boudouard reaction

	Temperature [°C]	Gas composition [% CO_2 in CO/CO_2 mixture]	Particle size [mm]	Flow rate [Nl/min]
Level 1	959±6	50	3.36 – 4.75	2
Level 2	991±4	75	4.75 – 6.68	4

3. RESULTS AND DISCUSSION

3.1 Carbon Properties

Since the single cokes have been produced from single source coals, it was important to identify the basic properties of the coals so the coke gasification characteristics could be related to the properties of the parent coals. Basic properties of the coals are presented in Table 2.

Table 2: Basic properties of the parent coals

		St	PD	BG
Proximate analysis [%wt]				
Moisture	W^a	2.5	0.9	2.4
Ash	A^a	5.9	11.6	1.5
Volatile matter	V^a	31.76	19.8	36.14
	V^{daf}	34.67	22.63	37.61
Fix C	$C_{Fix}{}^a$	56.93	64.87	58.49
Maceral constitution [vol %]				
Vitrinite		57	73	84
Liptinite		11	0	10
Inertinite		30	21	5
Mineral matter		3	6	1
Vitrinite Reflectance	R_0	0.74	1.32	0.77

^{a}- as received, ^{daf}- dry ash free, 0- mean random

From the table above, it can be seen that the three coals represent different coals in terms of their maceral structure, rank and mineral matter. The amount of the volatile matter and the reflectance show that the Peak Downs coal represents medium volatile bituminous coals, while Staszic and Blue Gem represent high volatile bituminous coals of similar rank. All those three coals are coking coal with Peak Downs as primary coking coal. The characteristic feature of the Blue Gem coal is the very low amount of mineral matter.

The Staszic and Blue Gem coals are of similar rank, both within high volatile bituminous coals. Although the two coals are of similar rank, the maceral constitution differs significantly. Staszic contains more inertinites and ash in comparison to Blue Gem. Additional non-maceral analysis (not presented here) showed the Staszic is mostly a dull coal with coarse inert macerals and ash particles. Blue Gem similar to Peak Downs was described as a bright coal with fine inerts and minerals.

After the single coals had been carbonized, the properties of now six cokes were determined. The characterization included proximate analysis, ash analysis, porosity, BET and anisotropy by image analysis. The properties of the coke samples are presented in Table 3.

Table 3: Basic properties of the coke samples

		Single cokes			Commercial cokes		
		St	PD	BG	PBC	RBC	BBC
Proximate analysis	[%wt]						
Moisture	W^a	0.5	0.2	0.2	0.3	0.3	0.1
Ash	A^a	10.0	13.9	2.3	9.5	14.0	10.9
Volatile Matter	V_{daf}	1.09	0.49	0.41	1.32	1.42	1.03
Fix C	$C_{Fix}{}^a$	88.41	85.41	97.09	88.88	84.28	87.97
Alkali index*	B	7.65	2.23	1.52	2.67	3.28	0.93
Total potassium	[wt %]	0.082	0.189	0.031	0.024	0.029	0.009
Crystallinity by Image Analysis	%	8.8	49.2	2.9	41.6	21.2	46.6
BET surface area	[m^2/g]	1.41	0.78	0.22	1.02	3.0	0.94

$$^*B = \frac{Fe_2CO_3 + CaO + MgO + K_2O + Na_2O}{SiO_2 + Al_2O_3} \square ash\%$$

The image analysis used to determine the crystallinity of the cokes showed that both Staszic and Blue Gem are in general free from optical textures, with incipient anisotropy present in some areas. Such a low crystallinity is in agreement with rank of their parent coals. Such a low ordering of carbons is an indication of their higher reactivity as isotropic carbon structures are known to be highly reactive, [6] and [7]. However, low ash content [8], low surface area [9] and low inert content [10] in BG might shift the reactivity towards lower reaction rates. The Peak Downs coke, in contrary has visible anisotropic domains, in slightly elongated shape. The size and the shape corresponds well to the medium volatile bituminous rank of the parent coal with R_o= 1.32%. It has been previously establish that with increasing rank of coal, larger anisotropic domains are formed as the lamellar shape of the enlarged aromatic clusters enhances their fusion into uniformly oriented pre-graphitic crystallites during carbonization [11]. Due to its high anisotropy, PD is expected to be of low reactivity towards CO_2.

Blended cokes have in general higher total anisotropy as they are produced by blending coals of different rank, always including some amount of medium to high rank coals. Surprisingly, the Russian coke showed low crystallinity. Coke carbon forms counting (not included) revealed that the Russian coke has higher amounts of low rank coal used for the blend and in addition contains metal particles as contaminants (originating from the smelting plant wherefrom the coke was taken).

3.2 The Boudouard reactivity of non-impregnated samples

The reactivity towards CO_2 mixture can be expressed by the conversion of carbon in a unit time. The conversion is the ratio between the carbon mass at a given time and the carbon mass before reaction according to the equation:

$$X = \frac{dM}{M_0 \times \%FixC} \tag{4}$$

The reactivity plots for non-impregnated samples are presented in Figure 2 and the reactivity values in the Table 4.

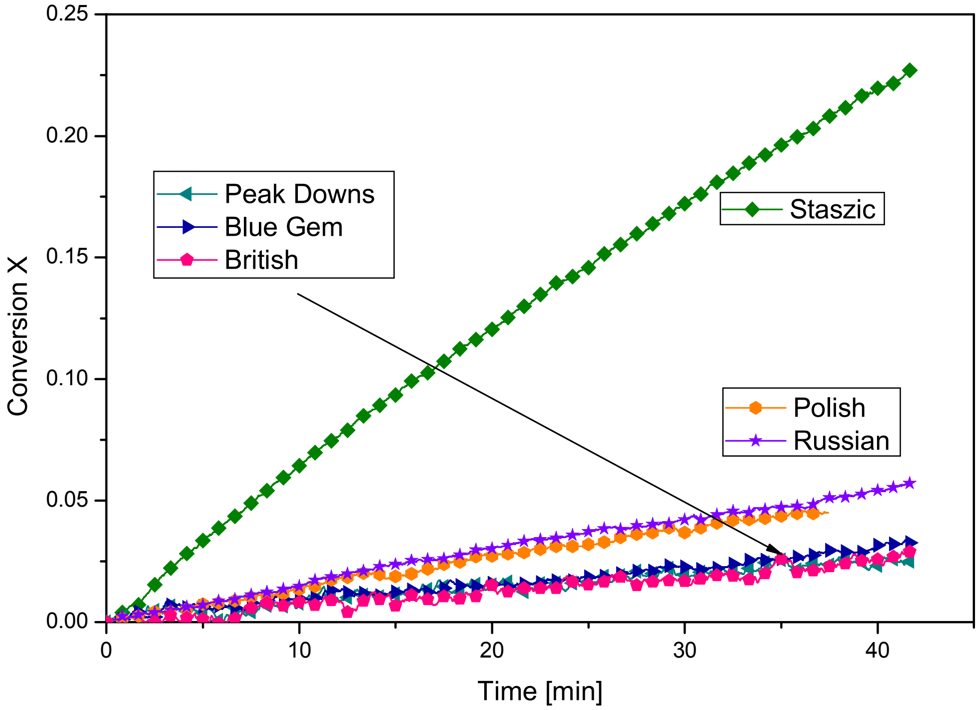

Figure 2: Reactivity as carbon conversion vs. time of non-impregnated samples

Table 4: The Boudouard reactivities of non-impregnated samples

	Single cokes			Commercial cokes		
	St	PD	BG	PBC	RBC	BBC
dX/dt [10^{-4}/min] ±5%	53.7	5.8	6.9	12.3	13.1	6.4
R^2	0.9963	0.9496	0.9785	0.9942	0.9922	0.9439

From the plots it can be noticed that the cokes divide themselves into three groups of similar reactivities. The low reactive group includes Peak Downs, British and Blue Gem cokes. The difference in reactivity between them is negligible in the view of 5% relative uncertainty. The second, medium reactivity group includes Polish and Russian industrial cokes, and the high reactive group consists of Staszic coke only.

The highest reactivity of the Staszic coke can be attributed to its properties. This coke has high BET surface area, thus providing a better utilization of micropore surface area for the reaction. In addition most of the carbon is present in isotropic areas, thus carbon atoms are easily detached from the coke structure. The chemical analysis of ash showed very high concentration of basic oxides that are reported to catalyze the reaction [10]. Also the filler phase analysis showed very high concentration of porous inert particles.

The Blue Gem coke, similar to Staszic, was produced from a high volatile bituminous coal of similar rank. The isotropic structure suggested its high reactivity; however its reactivity was much lower than this of Staszic. Low alkali index, together with low surface area and small amount of small pores must have suppressed the impact of the low anisotropy.

The third, Peak Downs coke, originating from medium rank, reacted slowly. Here, the presence of large optical domains being the result of higher rank of the parent coal, together with low alkali index and surface area helped in retarding detachment of carbon atoms from the carbon mass.

The commercial cokes also varied in reactivity. The Russian one reacted fastest as it had the highest surface area. In addition the alkali index was high and the structure was not as anisotropic as the other commercial cokes. The British coke had the lowest surface area and high anisotropy. In addition the alkali index was low, thus the catalytic effect of ash components was negligible. As the result the British coke exhibited the lowest reactivity.

For the verification of the importance of various experimental conditions on the rate of the Boudouard reaction, a simple factorial analysis has been used. The factorial analysis did not include potassium impregnation as it was previously shown [12] that the influence of potassium has the strongest influence on the reactivity of all factors.

The response is given by the Boudouard reactivity, $R = \frac{dM_{carbon}}{dt}$ [1/1000min]. The standard error has been calculated from the highest order interactions assuming that the last five of them are largely due to noise. The results are presented in Table 5.

Table 5: Factorial analysis of experimental conditions for Staszic coke

Main (average) effect	Symbol	Response [*10^{-3}]	Standard error s [10^{-3}]
Temperature	T	**3.88**	0.28
Concentration of CO_2	C	**2.08**	0.28
Particle size	P	0.68	0.28
Flow rate	F	**1.13**	0.28
Two-factor interactions			
Temp by Concentration	TC	0.19	0.28

Table 5: Factorial analysis of experimental conditions for Staszic coke (Continued)

Main (average) effect	Symbol	Response [*10^{-3}]	Standard error s [10^{-3}]
Temp by Particle	TP	-0.04	0.28
Temp by Flow	TF	-0.14	0.28
Con by Particle	CP	-0.13	0.28
Con by Flow	CF	-0.09	0.28
Particle by Flow	PF	0.26	0.28

From the above table it can be seen that the strongest influence on the reactivity has temperature followed by CO_2 concentration and flow rate. Particle size, within the range used does not play an important role, as its effect is lower than 2-3 times the standard error [13].

3.3 Impregnated samples

After the impregnation, coke structure was studied using SEM. The micrographs of selected cokes are presented in Figure 3. Visible white areas around pores are potassium compounds present within the pore walls. As it can be seen, the distribution of the potassium compound is non-uniform, with some walls covered by thick layer and other being potassium free. It can be also seen that the white areas are on both sides of original pore wall border line. This suggests that carbon from the coke is involved in the formation of potassium compound and that the higher volume of potassium carbonate results in its expansion outside the coke mass border. Further analysis by electron probe micro analyzer (EPMA) of the potassium layers showed the atomic ratio to be very close to that of K_2CO_3. Therefore it can be assumed that the catalysis in the present work involves potassium carbonate.

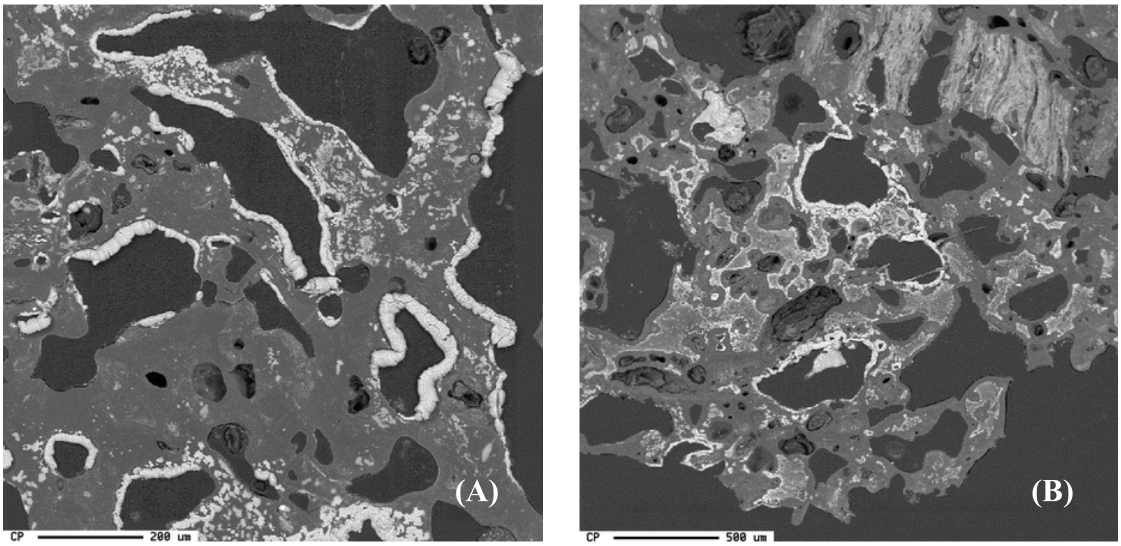

Figure 3: SEM micrographs of impregnated cokes, PBC 4.91% K

Under the light microscope, the impregnated samples showed presence of potassium compound as layers around some particles, Figure 4 (colorful/bright areas along the edge). Here, it can also be seen that the potassium compounds are present within the coke mass leaving the original shape of the coke structure.

After the coke samples had been impregnated to various potassium concentrations, the Boudouard reactivity was studied. The reactivity rates were corrected for the presence of potassium i.e. the reactivity was cal-

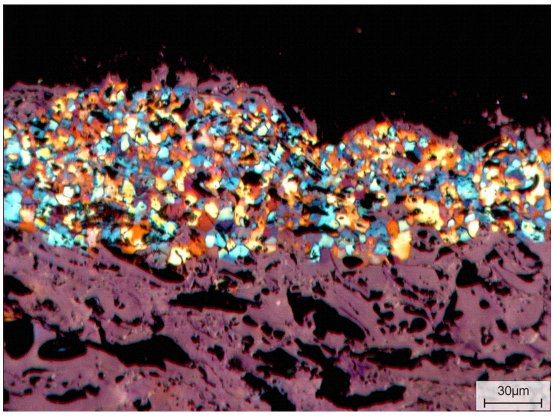

Figure 4: Potassium compounds as bright colorful fillings. RBC 4.97% K, polarized light, oil immersion, 320x

culated per mass of coke (excluding potassium, ash, volatile matter and moisture). The results for three single and one blended cokes are presented in the Figure 5.

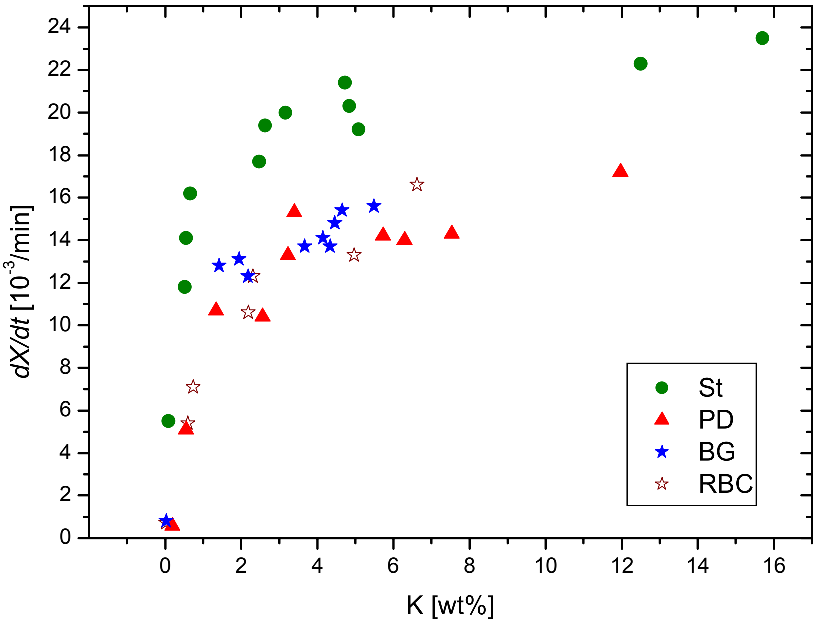

Figure 5: Boudouard reactivity vs. potassium concentration

From the above graph a few conclusions can be drawn. First of all, the reactivity increases with potassium concentration in a logarithmic fashion. At the concentrations around 4 wt% K, the reaction rate reaches a plateau and higher potassium concentrations give little increase in reactivity. The saturation level is similar for all the samples irrespectively of the inherent properties. The increase in gasification rate due to potassium was different for different cokes, ranging from 2- to 20-increse for Staszic and RBC respectively. It can also be noticed that the inherent reaction rate order between the single cokes is preserved in impregnated samples for all the potassium concentrations. This means that strong catalytic effect of potassium does not exclude the effect of the inherent properties of non-impregnated cokes. This fact shows that even though the coke is impregnated with alkalis in the smelting furnace, the inherent properties should not be neglected in evaluation of coke reactivity towards CO_2.

In Figure 6, the Arrhenius plots of selected impregnated samples are plotted together with non-impregnated samples, with slope values presented in Table 6.

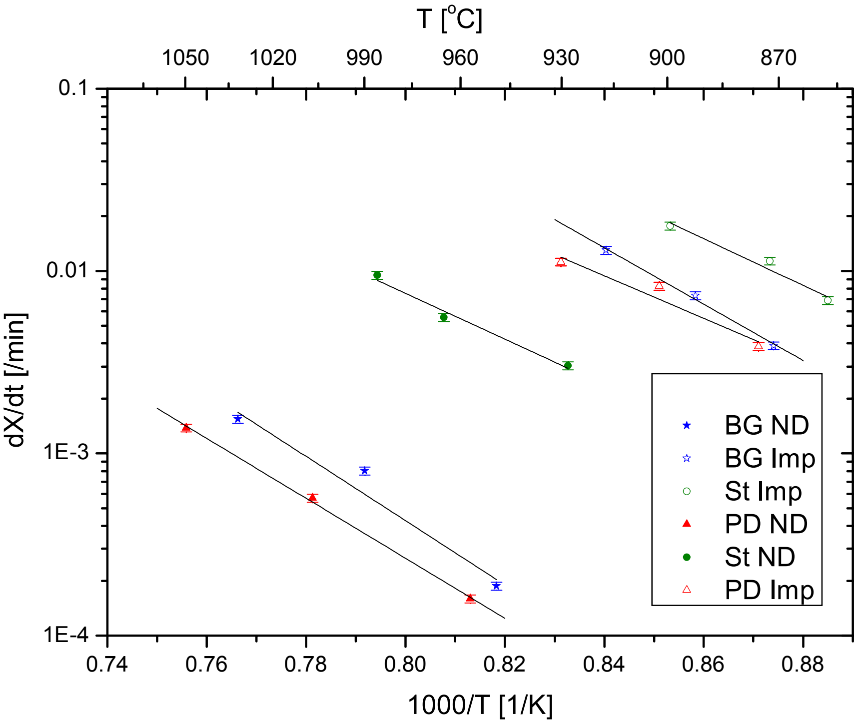

Figure 6: Arrhenius plots of non- and impregnated single coke samples

Table 6: Activation energies for impregnated and non-impregnated single cokes

	Staszic	Peak Downs	Blue Gem
Inherent Activation Energy E_A [kJ/mol]	240±16	315±10	338±10
K- impregnated [wt%]	2.47	2.56	1.94
Activation Energy E_A [kJ/mol]	241±18	223±14	296±17

From the Arrhenius plots, it can be seen that the activation energy is different for different cokes, both non-impregnated and impregnated. It can also be seen that the catalyzed higher gasification is attributed to the lowering of the activation energy. Small change in E_A for Staszic corresponds to small catalytic effect of potassium on the gasification rate for this coke. Very high inherent reactivity seems to reduce the effect of potassium.

3. CONCLUSIONS

In the present work, the Boudouard reactivity of selected commercial and laboratory produced cokes have been investigated. Cokes of high rank, low ash and low amount of inert carbon inclusions from coal tend to have low reactivity. However cokes from coals of similar rank can result in significantly different reactivity, mainly due to different ash composition and petrographic structure.

Of the experimental conditions (excluding potassium impregnation), the temperature, partial pressure of CO_2, and gas flow rate has the strongest influence on the reactivity, respectively. The reaction rate was similar for the particle size-range 3.35- 4.75 and 4.75- 6.68 mm, suggesting that the particle size in the range studied does not influence the Boudouard reactivity. The combination of the experimental conditions did not show any significant change in reactivity.

Impregnation of the coke with potassium resulted in formation of potassium carbonate on pore walls with involvement of carbon present in the coke. The distribution of carbonate was inhomogeneous through the particle.

The Boudouard reactivity of impregnated samples was significantly higher than the raw sample. The increase in reactivity with the amount of potassium was steep, up to about 4%, after which the reaction rate increased slightly. The increase in reactivity was different for different cokes; however the reactivity order of raw cokes was preserved after the impregnation.

The activation energy for the catalyzed Boudouard reaction was in general lower than for raw cokes, suggesting that the mechanism of catalysis involves lowering of the activation energy.

4. ACKNOWLEDGEMENTS

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