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The Effect of Binary and Ternary Mineral Combinations on the Reactivity of a Coke Analogue

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Keywords
effect, binary, mineral, combinations, ternary, reactivity, analogue, coke

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Abstract

The effect of the mineral matter on the reactivity of a coke analogue was examined through the addition of binary and ternary combinations of magnetite (Fe\(_3\)O\(_4\)), troilite (FeS) and quartz (SiO\(_2\)). The use of coke at a laboratory scale has issues arising from it heterogeneity and complex nature. The coke analogue was developed to help alleviate these issues, being a laboratory tool that has a simplified carbon structure and in which the mineral content, particle size and distribution is largely controlled. The reactivity of the coke analogue with CO\(_2\) was measured using a TGA system at 1100°C under conditions similar to that of the CRI test. For binary combinations of magnetite-quartz and troilite-quartz, increasing quartz content (and subsequently decreasing the iron content) was found to decrease the reactivity of the coke analogue. The change in the reactivity of the troilite-magnetite binary system was found not to be simply additive, with combinations of troilite and magnetite giving higher reactivities than would be expected from a simple proportional approach. The ternary magnetite-troilite-quartz system was found to largely follow the trends found in the bounding binary systems.

Keywords

Coke reactivity; minerals; coke analogue;

1. Introduction

Coke is a key reagent in blast furnace ironmaking, with its properties having a direct effect on the quality and productivity of the ironmaking process [1]. Coke fulfils several roles in the blast furnace, being the fuel for the furnace, the carbon source for the gaseous CO reductant for iron oxide and providing the structural support for the blast furnace burden. The performance of coke in the blast furnace is critically related to its hot strength and reactivity [2,3].

Prediction of coke reactivity from key characteristics, such as its ash content, mineralogy and rank, is desirable for both coal producers and coke users. This is not always possible and is in part limited by as yet poorly understood or unquantified effects of ash minerals on coke reactivity. Coke is a complex material, being comprised of different forms of carbonaceous materials (originating from different coal macerals) and minerals, with a pore structure dependent on the volatile mater in the source coal (and coking conditions) [2,4,5]. It also has significant heterogeneity in most metric(s) used to characterise its maceral grouping.
mineralogy, phase dispersion, morphology and porosity. When exposed to high temperatures and reactive atmospheres, the inherent compositional and structural features of a given coke render isolating the specific ash/mineral effects on its behaviour difficult.

The mineral content of the coke is an important factor affecting coke reactivity [2,6-9]. Several factors play a role in the effect that combinations of minerals can have on the reactivity of the coke analogue. These include the proportions of each mineral, the size distribution of the minerals, the phase stability of a mineral at a given temperature and the proximity of other minerals resulting in mineral-mineral reaction and possible formation of other products. These complex interactions between the coke and minerals, as well as the carbon types and porosity, in addition to the inherent heterogeneity in cokes are a focus of much research into coke reactivity.

A coke analogue has been developed for use as a tool in laboratory scale research into the mineral effects on the reactivity of coke [4-6]. The analogue is made from a number of carbonaceous materials and can be doped with minerals to simulate the mineralogy of an industrial coke. It has been used to study the effect of specific minerals and mineral combinations on coke reactivity with CO$_2$ at high temperatures [4-6], to assess the reaction rate controlling mechanisms of the analogue gasification [10] and to mimic the behaviour of industrial coke on dissolution in liquid iron [5,11-13].

Quartz is one of the most commonly found minerals in coke [6,15-16]. At temperatures around 1100°C, those used for the coke reactivity index (CRI) test, it is also known to reduce the reactivity of coke [4,9]. On the other hand, iron-bearing minerals are known to strongly increase the reactivity of coke [4,9,16]. However, the effect of combinations of quartz and iron-bearing minerals is relatively unknown, and as yet has not been quantified.

The aim of this study was to examine the effect of combinations of quartz and different iron-bearing minerals on coke analogue reactivity, to help build upon the knowledge gained from the study of single minerals [4]. The effect of magnetite-troilite (Fe$_3$O$_4$-FeS), troilite-quartz (FeS-SiO$_2$) and pyrite-quartz (FeS$_2$-SiO$_2$) binary combinations, and magnetite-troilite-quartz (Fe$_3$O$_4$-FeS-SiO$_2$) ternary combinations of minerals on reactivity of coke analogue was investigated in a thermogravimetric analysis (TGA) under conditions similar to that of a CRI test.

2. Experimental

Full details of the production method for the coke analogue have been reported elsewhere [4,9,10]. Minerals for examination were added to the coke analogue to give a cation concentration of 0.1 mol per 100 g. Note that unless stated otherwise the minerals quoted for the analogue represent the mineral addition to the green unfired analogue. During firing, certain minerals may transform or react, as noted for Fe$_3$O$_4$ being reduced to Fe in the single mineral study [4].

Three binary mineral combinations were examined: troilite (FeS)-quartz (SiO$_2$), pyrite (FeS$_2$)-quartz (SiO$_2$) and magnetite (Fe$_3$O$_4$)-troilite (FeS). The total mineral content, mineral particle size and porosity were all controlled to minimise their effects on reactivity. Their respective values were 0.10 mol of cations/100g, +45-75µm and 29.8 ±1.9%. Full details of the composition and porosities are given in Table 1. After preparation the mineral matter within the samples characterised using X-ray diffraction (XRD) and SEM-EDS.

Ternary mineral combinations coke analogue were produced containing two total iron cation concentrations and a range of silicon cation concentrations. The details of the mineralogy and porosities are given in Table 2. The SiO$_2$:(FeS + Fe$_3$O$_4$) ratios for each ternary mineral combination are given also in Table 2.
Table 1 – Mineral contents (in moles of cations per 100 g of carbonaceous material) in coke analogues containing binary mineral combinations and the measured porosity for each coke analogue. The porosity was measured as described below.

<table>
<thead>
<tr>
<th>Mineral combination</th>
<th>Iron mineral content (mol/100g)</th>
<th>Quartz content (mol/100g)</th>
<th>Si:Fe ratio</th>
<th>Porosity (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Pyrite-Quartz (FeS₂-SiO₂)</td>
<td>0.10</td>
<td>0.00</td>
<td>0</td>
<td>29.2</td>
</tr>
<tr>
<td></td>
<td>0.0676</td>
<td>0.0333</td>
<td>0.5</td>
<td>31.7</td>
</tr>
<tr>
<td></td>
<td>0.050</td>
<td>0.050</td>
<td>1.0</td>
<td>30.5</td>
</tr>
<tr>
<td></td>
<td>0.0333</td>
<td>0.0667</td>
<td>2.0</td>
<td>31.2</td>
</tr>
<tr>
<td>Troilite-Quartz (FeS-SiO₂)</td>
<td>0.10</td>
<td>0.00</td>
<td>0</td>
<td>28.4</td>
</tr>
<tr>
<td></td>
<td>0.067</td>
<td>0.033</td>
<td>0.5</td>
<td>29.1</td>
</tr>
<tr>
<td></td>
<td>0.050</td>
<td>0.050</td>
<td>1.0</td>
<td>30.1</td>
</tr>
<tr>
<td></td>
<td>0.033</td>
<td>0.067</td>
<td>2.0</td>
<td>30.1</td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>Mineral combination</th>
<th>Magnetite content (mol/100g)</th>
<th>Troilite content (mol/100g)</th>
<th>Porosity (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Magnetite-Troilite (Fe₃O₄-FeS)</td>
<td>0.10</td>
<td>0.00</td>
<td>31.0</td>
</tr>
<tr>
<td></td>
<td>0.0667</td>
<td>0.033</td>
<td>28.5</td>
</tr>
<tr>
<td></td>
<td>0.050</td>
<td>0.050</td>
<td>27.9</td>
</tr>
<tr>
<td></td>
<td>0.0333</td>
<td>0.0667</td>
<td>31.0</td>
</tr>
</tbody>
</table>

Table 2 – Mineral contents (in moles of cations per 100 g of carbonaceous material) in coke analogues containing ternary mineral combinations and the measured porosity for each coke analogue.

<table>
<thead>
<tr>
<th>Magnetite (Fe₃O₄) content (mol/100g)</th>
<th>Troilite (FeS) content (mol/100g)</th>
<th>Quartz (SiO₂) content (mol/100g)</th>
<th>Si:(total Fe) ratio</th>
<th>Porosity (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>0.0125</td>
<td>0.0375</td>
<td>0.05</td>
<td>1.0</td>
<td>29.1</td>
</tr>
<tr>
<td>0.025</td>
<td>0.025</td>
<td>0.05</td>
<td>1.0</td>
<td>28.6</td>
</tr>
<tr>
<td>0.0375</td>
<td>0.0125</td>
<td>0.05</td>
<td>1.0</td>
<td>30.0</td>
</tr>
<tr>
<td>0.0333</td>
<td>0.0333</td>
<td>0.0333</td>
<td>0.5</td>
<td>27.9</td>
</tr>
<tr>
<td>0.0167</td>
<td>0.0167</td>
<td>0.0667</td>
<td>2.0</td>
<td>28.9</td>
</tr>
</tbody>
</table>

Coke analogue samples were reacted in a pseudo-CRI test, using a TGA [4,6,10]. In each test, a sample of coke analogue, approximately 18 mm diameter and 30 mm height weighing approximately 8 grams, was placed in a perforated alumina crucible and suspended in a TGA. The samples were heated under a flowing argon atmosphere (1L/min) to 1100°C at 10°C/min and stabilised at temperature for 5 min. The gas flow was then switched to CO₂ at a flow rate of 2 L/min for two hours and the weight change logged. At the end of 2 hours the gas flow was returned to argon and the sample cooled to room temperature at 10°C/min. The pseudo-CRI test is designed to reflect conditions in a standard CRI test with the primary exception that a much smaller mass of materials is examined (approximately 8 g in the pseudo-CRI test versus 200 g in the standard test). Reactivity results from the pseudo-CRI test are expressed as fractional weight change (FWC), defined in equation 1,

\[ FWC = \frac{W_t - W_i}{W_i} \] (1)

where \( W_t \) is the weight (g) at time \( t \), and \( W_i \) is the initial weight. The more negative the FWC value, the more reactive the coke analogue in CO₂.
Fired coke analogues were sectioned and mounted in cold setting resin. The mounted coke analogue samples were polished to a 0.5 μm finish. Coke analogue porosities were measured in the pore size range 10–500 μm using optical microscopy and subsequent image analysis using ImageJ. Details of the optical microscopy porosity measurement technique are given elsewhere [17]. This technique is unable to resolve porosities < 10μm in diameter. Selected samples were subsequently carbon coated for examination in scanning electron microscopy (SEM) and energy dispersive spectroscopy (EDS).

3. Results and Discussion

The initial porosities of the samples with binary and ternary combinations of minerals are given in Tables 1 and 2, respectively. As intended, the porosity the coke analogue samples was largely constant and independent of the minerals added. The minerals were found to be well distributed throughout the coke analogues.

3.1 Coke analogue containing binary combinations

In Fig. 1(a) the FWC for troilite-quartz and pyrite-quartz at different Si:Fe mole ratios is shown. The FWC was plotted against Si:Fe as a convenient way to present the composition for the binary mineral combinations. The reactivity of analogues containing pyrite-quartz combinations was lower than for those containing troilite-quartz. The reactivity of coke analogues containing troilite-quartz and pyrite-quartz combinations followed similar trends. The reactivity decreased (higher FWC) with increasing Si:Fe ratio until a plateau or local minimum in reactivity at Si:Fe = 1.0. The overall decrease in reactivity with increasing quartz (Si:Fe ratio) content may have been expected, as it was shown in the investigation of the effect of addition of single minerals in the coke analogue reactivity that troilite increases reactivity (more negative FWC) while quartz decreases reactivity (less negative FWC) [4]. The presence of a plateau or local minimum may indicate interaction/reaction between the quartz and the troilite.

Fig. 1 – The effect of binary combinations of minerals on the fractional weight change of coke analogue after reaction with CO₂ at 1100°C for 2 hours. (a) Troilite-quartz and pyrite-quartz combinations; and (b) magnetite-troilite combinations.
In Fig. 1(b) the effects of the combinations of troilite and magnetite at a constant iron cation content of 0.1 mol of per 100 grams of fired carbonaceous material are shown. Unlike the troilite-quartz and pyrite-quartz combinations, there is a nonlinear additive effect occurring between the magnetite and troilite resulting in a larger increase in reactivity when the minerals are combined together than would be expected from a simple law of mixing approach. This effect of magnetite-troilite combinations on the reactivity indicates interaction between the minerals in the coke analogue.

The reactivities of the pyrite-quartz combinations were lower than those for the troilite-quartz combinations. This is in line with what was observed for coke analogues with pyrite and troilite added as individual minerals [4] where pyrite had a lower reactivity compared to troilite. In a previous study, combinations of magnetite and quartz were also investigated [4]. While the effect of magnetite-quartz combinations on the reactivity of the coke analogue was more linear, it was similar to those for both the troilite-quartz and pyrite-quartz combinations in being largely additive.

While the effects of troilite-quartz and pyrite-quartz combinations on the reactivity of coke analogue were similar, troilite-magnetite combinations had a different effect on reactivity. The increase in the reactivity (more negative FWC) of coke analogues over that which would be predicted from a simple proportional mixing approach is quite different to the largely additive effects of combinations of the iron sulfides and quartz.

The reactivities of the mineral combinations indicated possible interactions/reactions between the iron sulfides and quartz, as well as between troilite and magnetite. Thermodynamic analysis was conducted on each of the binary mineral combinations investigated to assist in predicting and understanding what interactions/reactions were possible between the minerals in the coke analogue. The analysis was carried out using FactSage [18], with the mineral combinations investigated at 1100-1200°C with an excess of carbon to simulate coking conditions. A summary of the phases predicted to be stable under these simulated coking conditions is given in Table 3.

Table 3 – Summary of condensed phases predicted by FactSage [18] for the investigated mineral combinations under coking conditions, and those identified by XRD in the fired coke analogues.

<table>
<thead>
<tr>
<th>Mineral combination</th>
<th>Phases predicted by FactSage</th>
<th>Phases identified by XRD</th>
</tr>
</thead>
<tbody>
<tr>
<td>Magnetite-quartz (Fe$_3$O$_4$)-(SiO$_2$)</td>
<td>Metallic iron (liquid at 1200°C), cristobalite (SiO$_2$)</td>
<td>Iron ($\alpha$-Fe), quartz (SiO$_2$)</td>
</tr>
<tr>
<td>Troilite-quartz (FeS)-(SiO$_2$)</td>
<td>Troilite (FeS), cristobalite (SiO$_2$)</td>
<td>Troilite, quartz</td>
</tr>
<tr>
<td>Pyrite-quartz (FeS$_2$)-(SiO$_2$)</td>
<td>Troilite (FeS), cristobalite (SiO$_2$)</td>
<td>Troilite, quartz</td>
</tr>
<tr>
<td>Magnetite-troilite (Fe$_3$O$_4$)-(FeS)</td>
<td>Metallic iron (liquid at 1200°C), troilite (FeS)</td>
<td>Iron ($\alpha$-Fe), troilite</td>
</tr>
</tbody>
</table>

Little interaction between the minerals was predicted for both the troilite-quartz and pyrite-quartz combinations, consistent with the reactivity results given in Fig. 1(a). Pyrite was predicted to decompose to troilite below ~1200°C, while troilite remained stable. From this it might be expected that troilite and pyrite may be similar in the coke analogue and have similar effects on the reactivity. This was not the case. For the magnetite-troilite combinations it was predicted that the magnetite would be reduced to metallic iron, while the troilite was again unchanged. There was little indication of any interaction between the two minerals.
The thermodynamic analysis indicated that little interaction is possible between any of the minerals in the combinations investigated. To further investigate any possible interactions between the minerals in the coke analogues, the mineral phases present in the fired coke analogues were characterised by XRD (Table 3) and SEM-EDS (Fig. 2). The phases identified by XRD were generally consistent with those predicted by FactSage. One discrepancy was that the quartz added appeared to still be present as quartz after firing, while FactSage predicted the presence of cristobalite. There are two possible explanations of cristobalite being predicted by FactSage but quartz being found in the XRD.

1. The analogue is fired at a temperature of 1200°C but characterised at room temperature. It is therefore possible that cristobalite was present after firing and that it transformed to quartz on cooling.
2. SiO₂ was added as quartz to the analogue and may not have transformed to cristobalite during the firing process. FactSage calculations predict equilibrium phases at the temperatures specified and do not consider any kinetic impact on phase evolution.

The iron and sulfur maps in Fig. 2 show differences between the iron sulfide particles remaining in the pyrite and troilite containing analogues. Troilite particles show iron-rich/sulfur depleted centres (Fig.2(a)). This contrasts with pyrite, where the composition was consistent across the iron sulfide particles (Fig. 2(b)). This indicates that the pyrite remained as an iron sulfide, while the troilite was at least in partially reduced. No metallic Fe was identified in fired coke analogue samples containing troilite by XRD. There are a few possibilities for metallic iron not to be identified by XRD. The micrographs showed that only the centre of the troilite particles were metallic iron, so that there was only a low concentration of iron. It is also possible that the X-rays might not penetrate to the centre of the particles. The XRD patterns of the fired analogues necessarily include a large carbon peak, which might also help to mask any minerals with only low concentrations.

The higher reactivities of the troilite-quartz containing analogues compared with pyrite-quartz may be in part caused by this partial reduction of the troilite. However, there was little indication of any interaction between the iron sulfide particles and the quartz particles within the coke analogues. The plateau and peak in the reactivity with changing Si:Fe ratio for troilite and pyrite respectively, as shown in Fig. 1, are likely not caused by an interaction between the two minerals. This point is not yet fully understood, and requires further investigation.

The magnetite-troilite containing samples were different again. Magnetite was reduced to metallic iron, while troilite particles showed signs of reduction to metallic iron in the centre, surrounded by edges with compositions close to that expected for troilite (Fig 2(c)). Fig. 2(c) also shows sulfur present at the outer edges of the metallic iron (ex-magnetite) particles. This apparent transfer of sulfur from the troilite to the metallic iron (ex-magnetite) indicates that there is some interaction between the minerals. This likely interaction between the two minerals may cause the non-linear behaviour seen in the reactivity in the coke analogues containing combinations of magnetite and troilite (Fig. 1 (b)).

3.2 Coke analogue containing ternary combinations of troilite, magnetite and quartz

The mineral contents of the coke analogues with troilite-magnetite-quartz combinations were shown in Table 2. The characterisation of the minerals was similar to that seen for each of the minerals in the binary combinations. Troilite showed signs of reduction with the sulfur-
depleted centres, magnetite was reduced to metallic iron but there were indications of sulphur present at the particle surface, and the quartz was unchanged.

![Typical iron and sulfur maps](image)

Fig. 2 – Typical iron and sulfur maps of the fired analogue containing (a) troilite-quartz; (b) pyrite-quartz and (c) magnetite-troilite binary combinations. The scale bar is 200μm.

The FWC for coke analogues containing troilite-magnetite-quartz ternary combinations are given in Fig. 3. FWC was plotted against the FeS:Fe₃O₄ molar ratio is given in Fig. 3(a) and against the Si:(FeS + Fe₃O₄) molar ratio in Fig. 3(b) to help visualise any trends in reactivity.

The reactivity of the coke analogues with the ternary mineral mixtures were similar to those for the magnetite-troilite binary combination, with a non-linear relationship between
reactivity and the FeS:Fe$_3$O$_4$ ratio. The reactivity initially increased quite sharply when the FeS:Fe$_3$O$_4$ ratio increases from 0.5 to 1.0, before decreasing slightly as FeS:Fe$_3$O$_4$ is further increased to 3.0. The comparison between the binary magnetite-troilite combination and the ternary combination (at constant SiO$_2$) is shown in Fig. 3(a). The magnetite-troilite binary combination represents one of the axes of the ternary system. The non-linear trend for the ternary mineral combination agrees well with the binary combination. The FWC from the ternary combination were lower, as might be expected with SiO$_2$ being present in the analogue. The trend in the axis binary combination holds true for the ternary mineral combination.

Similar to the troilite-magnetite binary combination, transfer of sulfur between the troilite and the magnetite particles may possibly be the cause of the non-linearity found in troilite and magnetite mixtures on the reactivity.

![Graph](image)

Fig. 3 – (a) Effect of troilite and magnetite on the reactivity of coke analogue, as represented by the FeS:Fe$_3$O$_4$ molar ratio; (b) effect of quartz on the reactivity of coke analogue for the magnetite-quartz binary system [4], the troilite-quartz binary system and the troilite-magnetite-quartz ternary system. The quartz level is represented by the Si:(FeS + Fe$_3$O$_4$) molar ratio.

The effect of SiO$_2$ content on the reactivity of the magnetite-troilite-quartz ternary with the magnetite-quartz [4] and the troilite-quartz binary combinations is compared in Fig 3(b). These two binary systems each represent an axis of the ternary system. The reactivity decreased as the Si:(FeS + Fe$_3$O$_4$) ratio increased for each mineral combination. The trends in the axis binary combinations hold true for the ternary mineral combination.

For a more complete representation of the reactivities of coke analogues within the ternary troilite-magnetite-quartz combinations, the FWC results for ternary mixtures and each of the axis binary combinations have been plotted on a ternary diagram in Fig. 4. Also included on the diagram are results for the magnetite-quartz binary from [4]. As was illustrated in Fig. 3, the trends for the axes can be extended across the whole diagram. Increasing the SiO$_2$ content decreases the reactivity across the ternary diagram. The non-linear relation for troilite and magnetite on the reactivity also holds true across the ternary diagram.

The reactivities of coke analogues containing minerals in the troilite-magnetite-quartz ternary combination follow similar trends as those for the bounding binary combinations. This allows interpolation for prediction of the reactivity of coke analogues for further compositions within the troilite-magnetite-quartz combination, which in turn, will aid understanding of the effect of these minerals on the reactivity of industrial cokes.
The reactivity of coke analogues reacted with CO$_2$ at 1100ºC, as represented by FWC at 120 minutes, plotted against composition on a ternary diagram.

**4. Conclusions**

A pseudo-CRI reactivity test was used to study the effect of binary and ternary combinations of quartz and iron-bearing minerals on the reactivity of coke analogues. Binary mineral combinations tested were troilite-quartz, pyrite-quartz, and magnetite-troilite, while the ternary troilite-magnetite-quartz combination was also examined. The key outcomes from the testing of combinations of quartz and iron-bearing mineral additions to the coke analogue were:

- Troilite-quartz and pyrite-quartz combinations showed a non-linear relationship between reactivity and the Si:Fe molar ratio, with a plateau or local minimum in the reactivity at a Si:Fe ratio of 1.0. There was no evidence of interaction between the minerals by either thermodynamic analysis or characterisation of the minerals in the analogue sample.
- Magnetite-troilite combinations showed a non-linear effect on the reactivity in that the reactivity of coke analogues containing mixes of these two minerals show a higher reactivity than would be expected from a simple proportional composition/law of mixing approach.
- The reactivity of the coke analogue in the ternary troilite-magnetite-quartz combination show trends from the axis binary combinations were largely followed across all compositions. This should allow interpolation of the reactivity of coke analogues for further compositions, which in turn, will allow better understanding of the effect of these minerals on the reactivity of industrial cokes.
References