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Cyanide recombination in electric arc furnace plasma

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Abstract

Cyanide, among with NOx, CO2, and CO, is one of the adverse compounds that form in the ironmaking and steelmaking industry. High-temperature processes are suitable environments for cyanide formation, and cyanide can form as a result of recombination in electric arc plasma. Even though the cyanides might not survive e.g. the post-combustion process, understanding the formation mechanisms of hazardous materials in the steelmaking industry is important. In this work, the recombination of cyanide in a pilot-scale AC electric arc furnace is studied with optical emissions from the CN molecule. The results show how the optical emissions from the cyanide change in different process steps. Electric input, plasma temperature, and interaction of the arc with solid charge material were observed to have an impact on the CN signal. Additionally, equilibrium composition computation highlights how different sources of carbon change the recombination rate and that the highest recombination occurs at 6821 K.

1. Introduction

Cyanide (CN) and cyanide compounds, such as HCN, KCN, and NaCN, are a subject of major concern in the iron and steel industry due to their toxicity [1–3]. Cyanide forms in flames and high-temperature environments via several possible reaction routes involving e.g. C2, N2, C, and CH [4]. The cyanides are typically located in the effluents [2, 3, 5, 6] and leachates [7] in industrial processes. The sources of carbon and nitrogen in these reactions can be the atmosphere inside the furnace, charge material, additive materials, and gas or coke injections. The permissible amount of cyanide in disposed water is strictly defined [1], and this limit in the effluents is usually around 0.20mg/litre or less [1, 5–7]. Industrial electric arc furnaces (EAFs) and ladle furnaces (LFS) do not usually have water scrubbers, which means that cyanide is not a problem in the effluents. Submerged arc furnaces, on the other hand, may have a problem with the cyanide content [8]. Since a high-temperature environment is favorable for cyanide formation, it most probably is present in the industrial EAF and LF atmosphere to some extent even though it might not survive through the post-combustion process. Furthermore, post-combustion might not be implemented in semi-industrial EAFs with a capacity of only several hundred kilograms. Eventually, monitoring the formation of any hazardous gases would be beneficial e.g. in case a malfunction occurs in any process that aims to decompose harmful compounds.

A method that is viable for in situ analysis in industrial EAF and LF conditions is optical emission spectroscopy (OES). Generally, observations of molecular optical emission bands from the CN have been widely reported in various fields of applied spectroscopy, such as laser-induced breakdown spectroscopy (LIBS) studies of solid materials containing C and N [9], benzene and carbon disulfide [10], together with optical spectroscopy of plasmas including polyvinyl chloride [11], graphite [12], carbon nanotubes [13], and electrical discharge assisted combustion [14]. In LIBS studies, molecular emission bands of CN have also been used to derive the carbon content of steel [15], fly ash [16], and coal [17]. Related to steelmaking, cyanide has been suggested to have an effect on the reduction of Fe2O3 in a spectroscopic analysis including microwave treatment [18].
Due to easily identifiable optical emission band structure, the optical emission spectroscopic studies of CN offer a practical analysis method that is viable also for online applications in EAFs and LFs. In this work, the optical emissions from the CN molecule were studied during pilot-scale AC EAF process steps. The purpose of the study was to investigate the changes in the CN signal during different process steps, which may contain information on the formation and concentration of cyanide inside the furnace. The OES data has been analyzed and compared to the electrical data of the furnace, off-gas analysis of CO₂, O₂, and H₂, and camera footage.

2. Methods and materials

The measurements were conducted at RWTH Aachen University, Germany. The pilot-scale furnace has two graphite electrodes with 10 cm diameter, and 200 kg liquid steel capacity. The voltage, current, and power of one electrode were recorded once every two seconds. The active power is 600 kW with an arc current maximum of 2000 A. The apparent power of the transformer is 850 kVA. Six heats were performed during the measurement campaign. Before each heat, additive materials were charged into the furnace. The slag surface, which forms on top of the molten bath from the impurities of the charge material and additive materials, consists of CaO, MgO, MnO, Cr₂O₃, Fe₂O₃, SiO₂, and Al₂O₃. These components are common constituents of industrial slags. The additive materials consisted of varying amounts of CaO, MgO, SiO₂, Al₂O₃, and slag from the industrial furnace. The off-gas components CO₂, O₂, and H₂ were continuously detected with the rate of 1 Hz. The furnace operates at ambient pressure, except for the negative pressure of the dedusting system around −4 mbar.

A Baumer single-lens reflex camera was used to record the movements of the electric arc and melting of the solid material. The frame rate of the camera was 10 images per second. The camera was filtered with three green light band-pass filters that are used in welding. The spectrometers and camera were positioned on top of the furnace and looked into the furnace through an opening in the furnace roof. This represents the probable case in an industrial electric arc furnace, where the measurement heads could be attached to the roof [19]. Figure 1 displays the time-evolution of the melting of the charge material during one heat. The images have been converted from red-green-blue (RGB) to grayscale. The standard RGB pixel brightness values range from 0 to 255, 0 being the dimmest and 255 the brightest.

Czerny-Turner Avaspec-ULS2048 spectrometers were used in this study. The spectrometers covered 247.337–419.199 nm and 496.655–1000.000 nm. These spectrometers are referred to as UV and NIR, respectively. For each spectrometer, 16 spectra were recorded in a second. In order to correlate the spectrum data with the off-gas and electrical data, the brightest spectra measured during each second were used in the analysis. Example spectra are presented in figure 2. The spectra are dominated by optical emission lines from iron, calcium, chromium, aluminum, silicon, magnesium, and manganese, together with potassium, sodium, and lithium [19–23]. The emission lines from the slag components did not significantly overlap with the CN, carbon, nitrogen, oxygen, or hydrogen optical emission lines that were used in this study.

At the beginning of the measurements, a batch of charge material was melted for an hour to form the molten bath. This initial melting period has been used to observe how the optical emissions from CN behave when the furnace conditions are close to the early stages of the EAF process, namely ignition, boring, and formation of
melt. After this initial melting period, the view into the furnace was clear, and also the camera was turned on. This measurement period, on the other hand, represents the main heating period, charging of additional material, and flat bath period of the EAF process. The EAF was turned off momentarily when additive materials were charged into the furnace. Sometimes a protective shutter was placed in the view cone of the spectrometers and camera when the EAF was off.

3. Theoretical

In comparison with atomic emission lines, molecular emission spectra are more complicated due to vibrational and rotational energy states of the molecule [24]. The effect of rotational energy states was not observed in this study due to their relatively small effect on the spectra, whereas vibrational transitions are more notable and were clearly observed in the CN molecule. A pure electronic transition that does not include vibrational energy states is denoted \((0–0)\), where the first zero refers to the excited energy state from which the electron decays to the energy state denoted by the second zero. Transitions including vibrational state or states are denoted \((1–0)\), \((0–1)\), \((1–1)\), etc. The observed CN, O I, H I, N I, and C I emission lines are listed in table 1. The Roman numeral I refers to a neutral atom. The CN violet system is in the UV region between 350 nm and 450 nm, whereas the most intensive O I, H I, N I, and C I emission lines were observed to reside in the visible or near-infrared wavelength ranges. In order to remove the effect of varying arc visibility due to erratic movement of the arc on slag and solid charge partially blocking the view cone, the CN intensities have been divided with the average value of the spectrum intensity. The average intensity of the spectrum is automatically determined during spectrum acquisition.

Due to the high energy of the arc, plasma diagnostics can be used to analyze the OES data. The temperature of the plasma was determined with the Boltzmann equation [27]

\[
\ln \left( \frac{\varepsilon^z \lambda_{mn}}{g_{mn} A_{mn}} \right) = -\frac{1}{kT} E_m^z + \ln \left( \frac{\hbar c N^z}{4\pi U'(T)} \right)
\]

where \(z\) is ionization state of the particle, \(\varepsilon^z\) is wavelength-integrated emissivity \(n\) is lower atomic energy state, \(m\) is upper atomic energy state, \(\lambda_{mn}\) is wavelength of the emission line, \(g_{mn}\) is degeneracy of the upper atomic energy state, \(A_{mn}\) is transition probability, \(k\) is the Boltzmann constant, and \(T\) is the plasma temperature, \(E_m^z\) is energy of the upper atomic energy state, \(N^z\) is number density, and \(U'(T)\) is partition function. At a fixed temperature, the term containing the partition function is constant and can be neglected when the left-hand-side of equation (1) is plotted with respect to \(E_m^z\). The plasma temperature can thus be solved from the slope of the plot.
EAF is on, the CO2 is saturated at 20% which is the observation limit of the off-gas analysis system. When

### 4.1. Early stages of EAF process: ignition, boring, and formation of melt

Compared to the data of the later stages of the EAF process, the highest CO2 content is observed during this initial

Table 1. List of observed CN, N, C, O, and H emission lines.

| Line | \( \lambda \) (nm) | Line | \( \lambda \) (nm) |
|------|----------------|------|----------------|
| CN (0–0) \([9, 10, 12, 25]\) | 388.34 | N I \([26]\) | 744.23 |
| CN (1–1) \([9, 10, 12, 25]\) | 387.14 | N I \([26]\) | 746.83 |
| CN (2–2) \([9, 10, 12, 25]\) | 386.19 | N I \([26]\) | 818.49 |
| CN (3–3) \([9, 10, 12, 25]\) | 385.47 | N I \([26]\) | 881.80 |
| CN (4–4) \([9, 10, 12, 25]\) | 385.09 | N I \([26]\) | 820.04 |
| CN (5–5) \([23]\) | \(\approx\)384.90 | N I \([26]\) | 821.07 |
| CN (1–0) \([9, 10, 12]\) | 359.00 | N I \([26]\) | 821.65 |
| CN (2–1) \([9, 10, 12]\) | 358.58 | N I \([26]\) | 822.31 |
| CN (3–2) \([9, 10, 12]\) | \(\approx\)358.40 | N I \([26]\) | 824.24 |
| CN (4–3) \([12]\) | \(\approx\)358.40 | N I \([26]\) | 856.77 |
| CN (2–3) \([10, 12]\) | \(\approx\)418.00 | N I \([26]\) | 859.40 |
| CN (3–4) \([10, 12]\) | \(\approx\)416.70 | N I \([26]\) | 862.92 |
| CN (4–5) \([10, 12]\) | \(\approx\)415.80 | N I \([26]\) | 868.03 |
| CN (5–6) \([10]\) | \(\approx\)415.10 | N I \([26]\) | 868.34 |
| CI \([26]\) | 906.14 | N I \([26]\) | 868.62 |
| CI \([26]\) | 906.25 | N I \([26]\) | 870.33 |
| CI \([26]\) | 907.83 | N I \([26]\) | 871.17 |
| CI \([26]\) | 908.85 | N I \([26]\) | 871.88 |
| CI \([26]\) | 909.38 | N I \([26]\) | 872.89 |
| CI \([26]\) | 911.18 | &nbsp; | &nbsp; |
| O I \([26]\) | 777.42 | &nbsp; | &nbsp; |
| O I \([26]\) | 844.68 | &nbsp; | &nbsp; |
| O I \([26]\) | 926.09 | &nbsp; | &nbsp; |
| O I \([26]\) | 926.60 | &nbsp; | &nbsp; |
| H I (H\(_\alpha\)) \([26]\) | 656.28 | &nbsp; | &nbsp; |

Table 2. List of O I lines used in plasma temperature analysis.

| Line | \( \lambda_{\text{obs}} \) (nm) | \( \lambda_{\text{calc}} \) (s\(^{-1}\)) | \( E^* \) (eV) | \( g \) |
|------|----------------|----------------|----------------|------|
| O I \([26]\) | 777.42 | 3.69e+07 | 10.74 | 5 |
| O I \([26]\) | 844.68 | 3.22e+07 | 10.99 | 3 |
| O I \([26]\) | 926.09 | 1.56e+07 | 12.08 | 5 |
| O I \([26]\) | 926.60 | 4.45e+07 | 12.08 | 9 |

The errors of the plasma temperatures can be obtained with

\[
\frac{\delta T}{T} = \sqrt{\frac{kT}{\sum_{i=1}^{q} E_i^2 - \frac{1}{q} (\sum_{i=1}^{q} E_i)^2}} \left( \frac{\delta e}{e} + \frac{\delta A}{A} \right),
\]  

(2)

where \(q\) is the number of emission lines, \(\delta e/e\) is the general relative error of line intensity, and \(\delta A/A\) is the general relative error of transition probability \([28]\). The plasma temperature was determined with four O I lines, which are listed in table 2.

In an earlier study involving the same pilot-scale furnace under similar conditions, the plasma temperatures were determined to be within a range from 4500 to 9000 K, electron densities between \(10^{18}\) and \(10^{20}\) cm\(^{-3}\), and local thermodynamic equilibrium criterion densities around \(2 \times 10^{15}\) cm\(^{-3}\) \([21]\). The electrical properties of the arc, such as voltage gradient and anode-cathode voltage drop, together with conductivity and effect of slag composition have been discussed in another study \([29]\).

### 4. Results and discussion

#### 4.1. Early stages of EAF process: ignition, boring, and formation of melt

The time-evolution of the CN (0–0) intensity, the average intensity of the spectra, plasma temperature, electrical data of the furnace, and off-gas components have been presented in figure 3 for initial melting. The different CN transitions that are listed in table 1 have nearly exactly the same trends, so only CN (0–0) is presented. When the EAF is on, the CO2 is saturated at 20% which is the observation limit of the off-gas analysis system. When compared to the data of the later stages of the EAF process, the highest CO2 content is observed during this initial melting of the solid charge material.
The EAF power with respect to the CN (0–0) intensity has been displayed in figure 4 with O₂, CO₂, H₂, and current in the colorbars. The power values near 300 kW are from the very beginning of melting, which can mean that the low amount of CN is also related to solid charge blocking the view cone to the electric arc. The increase of CN intensities starting at 1100 s coincides with the increase in the O₂, which indicates that the CN intensity starts to increase when the solid charge material has melted for the most part. At 1100 s also the power and current are lowered and the plasma temperatures start to decrease.
Plasma temperature shows high fluctuation in the figure 3, ranging from 5000 to 15 000 K. The majority of R² larger than 0.85, however, are between 6000 and 8000 K. Figure 5 shows crude trends between the plasma temperature, power, and CN (0–0) intensity. The recombination of CN in the plasma tends to be highest when plasma temperature is between 6000 and 8000 K. This is also the range with the highest R² values. Parigger et al [30] state in their LIBS study that the optical emissions from CN depend on the plasma temperature. In their work, the highest CN recombination occurs between 6000 and 9000 K with a maximum of around 7000 K. On average, higher plasma temperature results to lower the CN intensity in figure 5, which is in line with the results of Parigger et al for lower CN signal above 9000 K. Furthermore, figures 4 and 5 show that higher power and current result to lower CN intensity, which could be explained with increasing plasma temperature when the electric input is increased.

4.2. Later stages of EAF process: additions, main heating, and flat bath

The time-evolution of the CN (0–0), average spectrum intensity, image brightness, O I plasma temperature, electrical data of the furnace, and off-gas data for O₂, CO₂, and H₂ is presented in figure 6. The plasma temperatures are within the range from 4000 to 8000 K with a mean R² value of 0.93 for the Boltzmann plots and an error between 8.6 and 17.4%. Between 0 and 1800 s, it can be seen that the CN intensities fluctuate significantly and that the highest CN intensities are observed in this time period. Charge material and carbon were added into the furnace prior to the measurement of figure 6, which could further increase the formation of CN. Because of this, the data from 0 to 1800 s has been presented apart from the 1800–5800 s data. It should be noted that the camera was turned on around 900 s in order to capture the arc movement and the slag surface when the initial charge had melted.

The relation between the CN (0–0) intensity and power, current, and off-gas components have been presented in figure 7, where (a) to (d) describe the melting of the charge material (0–1800 s in figure 6) and (e) to (h) the additions, main heating, and flat bath (1800–5800 s in figure 6). The relations of figures 7(a) to (d) are very irregular when compared to the (e) to (h). Higher CN intensities are observed when CO₂ content of the off-gas is
between 10 and 12%, i.e. the highest CO$_2$ content in the range from 0 to 1800 s. Figures 7(e) to (h) have more systematic trends between the power and CN formation where the CN intensities are, generally, higher with a higher power. Similar to (a) to (d), CO$_2$ content is higher with higher CN intensities. It should be pointed out that between 1800 and 5800 s the instances of highest power and CN formation are observed between 4500 and 5000 s, i.e. towards the end of the measurement period.

The trends of figure 7 are very different from figure 4. The difference in the measurement conditions between the two melting periods is that in the case of figure 4 the furnace initially has only solid charge material which is then melted, whereas in figure 7 the charge material is added on top of the molten bath. Another clear difference is in the plasma temperatures of these two cases. The majority of plasma temperatures in figure 6 fall between 5000 and 8000 K, which is within the temperature range that Parigger et al [30] reported for CN signals. One explanation for the clearer trends in figures 7(e) to (h) could be the lower plasma temperatures with a narrower range in comparison to figure 3 where the plasma temperatures vary from 5000 to 15 000 K. In the conditions of figures 7(e) to (f), higher power and current leads to an increase in CN intensity. The irregularity of figures 7(a) to (d), on the other hand, can be attributed to erratic arc behavior during melting of solid charge material, where the arc moves between the solid charge material and the slag surface. This can also be seen in the voltage of figure 6 that fluctuates significantly between 1000 and 1800 s as the arc wanders between the molten surface and the solid charge. This time period corresponds to CO$_2$ off-gas contents between 10 and 12% in
figure 7(b) and to the highest CN intensities. Hence, the highest CN intensities are observed when the arc interacts with the solid charge material.

4.3. Equilibrium composition computation

It was evident from the spectra that intensive N₂, O₂, and C₂ optical emission bands were not observed. C₂ Swan band has numerous optical emissions in the UV and visible range \[11, 12, 14, 18\], whereas intense N₂ optical emissions can be found in the UV \[13, 14, 18\]. The natural amount of N₂ and O₂ in the air suggests that these molecules are dissociated into atoms. However, the majority of C₂ can be expected to originate from the molten bath or the electrode. At least from the location of the spectrometers that were used in this study, the optical emissions from the C₂ could not be observed. However, C I lines were observed above 800nm, which could mean that the C₂ molecules tend to dissociate into atomic C.

Due to the lack of N₂, O₂, and C₂ optical emissions, the dissociation of air was analyzed using HSC Chemistry software made by Outotec Oyj. In order to take into account some of the impurities in the furnace atmosphere, the composition of the air entering the plasma was defined as 72.40% N₂, 24.05% O₂, 0.50% H₂, 0.50% CO₂, 0.50% CO, 0.05% C, 0.50% Ar, 0.50% H₂O, 0.50% K, and 0.50% Na. Intensive K I and Na I optical emissions can be observed whenever the EAF is turned on, which means that they could contribute to the formation of either KCN or NaCN. H₂O and H₂ were added to the mixture in order to see if HCN forms in the plasma.

The results of the HSC equilibrium composition analysis have been presented in figure 8. CO₂ mostly dissociates already below 4000 K, whereas CO is persistent up to 6000 K. Above 6000 K, minor C₂ recombination occurs. Contrary to CO₂, N₂ and O₂ are persistent up to 4000 K. NO forms between 4000 and 6000 K, where also minor NO₂ formation is observed. The amount of atomic oxygen in the plasma increases already above 4000 K, whereas atomic carbon and nitrogen start to increase around 6000 K. Atomic optical emissions of N I, O I, and C I were observed in all spectra. Interestingly, no N₂ optical emissions were observed even though the molecule is, according to the HSC calculations, present in the plasma up to 7000 K.

The HSC calculation for recombination of the cyanide species HCN, NaCN, KCN, and CN is presented in figure 8(d). The formation of HCN is the lowest, followed by NaCN and KCN. The CN formation is several magnitudes higher than those of HCN, NaCN, and KCN, but still only fractions of a percent. The amount of Na and K in the process depends on the charge material, and a charge material with a high alkali metal concentration might result in a higher yield of NaCN and KCN. The peak of the CN is at 6821 K. The chemical equilibrium computation reported by Parigger et al \[30\] resulted in a similar peak close to 7000 K. In another study, they studied graphene with LIBS where the CN spectrum had a temperature of 6840 K \[31\]. In our study, the majority of the O I plasma temperatures with high R² are between 4000 and 8000 K. As can be seen from the figure 8, the amount of atomic O starts to increase near 4000 K. Recombination of CN is negligible at this low temperature.
which could mean that the CN recombination takes place closer to the center of the arc, where the plasma temperature is higher and reaches temperatures near and above 6000 K.

With additional carbon input, the cyanide recombination will consequently increase. Figure 9 demonstrates how additions of CO₂, CO, and C₂ affect the CN recombination at 6821 K, which corresponds to the plasma temperature at which CN recombination is at its peak according to figure 8. C₂ additions have the most prominent effect. The electrodes are a potential source of C₂ due to the evaporation of electrode material. An increase in CO, on the other hand, can be observed during slag foaming practice. In this work, the CO₂ content of the off-gas saturated at 20%, which also corresponds to the highest intensities of CN optical emissions in the
later stages of the melting, i.e. 1800–5800 s in figure 6. It can be seen from figure 7(f) that the CO2 is saturated at 20% when the CN intensities are the highest.

5. Conclusions

The recombination of cyanide has been studied in a pilot-scale AC EAF with optical emission spectroscopy combined with camera footage, off-gas data, and electric data of the furnace. The molecular optical emissions of the CN violet system were clearly observed in the ultra-violet wavelength range and the empirical results show how the CN intensity changes in different EAF process steps. Several atomic carbon, nitrogen, oxygen, and hydrogen emission lines were also identified from the spectra. Plasma temperature was derived with four O I emission lines, and plasma temperatures with the highest R2 values were observed to be between 4000 and 8000 K. Plasma temperature, electrical input, and interactions between the arc and solid charge material had an apparent effect on the CN intensities. The industrial applicability of OES measurements has been established in previous studies [19, 22, 23, 32], and the optical emissions from the CN molecule have been observed e.g. in spectra from industrial LF [23].

Analysis of CN within the wavelength range of this study was found to be viable, whereas molecular optical emissions from other molecules, such as C2, N2, and O2, were not detected. Equilibrium composition analyses showed that CN recombines around 6821 K in the plasma. Similar calculations resulted in increasing CN recombination rate as either C2, CO2, or CO content of the gas was increased. Molecular optical absorption and emission bands from other molecules than CN may reside also outside the wavelength range of this study, mainly in the infrared in the case of molecules. Ultraviolet, visible, and near-infrared range, on the other hand, provide beneficial information about the CN molecule and abundance of atomic optical emissions.

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Conflict of interest

The authors declare that they have no conflict of interest.

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