



STUDY OF CARBOTHERMIC REDUCTION OF SELF-REDUCING BRIQUETTES OF EAF DUST AND IRON SCALE FOR USE IN ELETRIC STEELMAKING¹

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Abstract

The steel production in semi-integrated plants generates large quantities of waste, especially electric arc furnace dust (EAFD) and mill and casting scale. This paper studies the possibility of reintroduction of these wastes in the production process at the electric steel shop in the form of self-reducing composed briquettes. With the aid of the computational thermodynamic tool FactSage, an initial theoretical analysis of the genesis of iron and zinc oxides present in the EAFD was made. In sequence diagrams were made showing the amount of phases as function of the system composition at 1200 °C. The Gibbs energy change for the main carbothermic reduction reactions was determined for this temperature. This served to justify theoretically what had been proved experimentally: that the reduction of iron oxides occurs at a higher rate than the reduction of zinc oxides.

Key words: Self-reducing composite briquettes; EAFD; Mill scale.

ESTUDO DA REDUÇÃO CARBOTÉRMICA DE BRIQUETES AUTORREDUTORES DE PÓ DE FEA E CAREPA PARA USO NA ACIARIA ELÉTRICA

Resumo

A produção siderúrgica em usinas semi-integradas gera grande quantidade de resíduos, especialmente pó de aciaria elétrica (PAE) e carepas de laminação e lingotamento. Neste trabalho estuda-se a possibilidade de reintrodução destes resíduos no processo produtivo de aciaria elétrica na forma de aglomerados autorredutores compostos. Com o auxílio da ferramenta termodinâmica computacional FactSage foi feita uma análise teórica inicial sobre a gênese dos óxidos de ferro e zinco presentes na PAE. Na seqüência, foram confeccionados diagramas mostrando a quantia de fases em função da composição do sistema, a 1.200°C. A variação da energia de Gibbs para as principais reações de redução carbotérmica foi determinada para essa temperatura. Isso serviu para fundamentar teoricamente aquilo que vinha sendo comprovado experimentalmente: que a redução dos óxidos de ferro acontece a uma velocidade mais elevada que a redução dos óxidos de zinco.

Palavras-chave: Composto autorredutor briquetado; PAE; Carepas.

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1 INTRODUCTION

The steel production in semi-integrated plants involves the generation of waste such as slag, mill scale and dust from electric arc furnace (EAFD). Among the waste generated with higher metal content are the EAFD and mill scale. The EAFD is largely generated in the operation of electric arc furnace (EAF) by vaporization of ferrous and nonferrous metals and ejection and dragging of steel and slag particles. The main constituents of EAFD are oxides of iron and zinc. Yet, this waste is considered dangerous in view of its content of heavy metals like lead and cadmium.⁽¹⁾ The mill scale is generated mostly in casting and rolling operations and is primarily constituted of iron oxides.

The combined amount generated of these two waste can reach between 3 to 4 [wt.%] of the plant's production. Because of the volume, there is great pressure to recycle the iron contained in them in the production process.

In recent years, several alternatives have been proposed for the allocation of both the mill scale^(2,3) and EAFD.^(4,5) The difficulties for project implementation are related sometimes to technical / operational aspects or to aspects of economical order, which often require a minimum scale of production for its completion – alone or combined.

The presence of cement works in a near by area opens the possibility for co-processing these wastes in the cement clinker production. The mill scale has good disposition in this co-processing, however, the EAFD has encountered difficulties, due to its high chlorine content.

On the other hand, self-reducing briquettes have been studied with the purpose of a rational use of low cost raw materials in iron and steelmaking processes.⁽⁶⁾ They present themselves as an interesting alternative to the use of iron oxides from waste, agglomerated (e.g. as briquettes) with reducing agents. However, to consider the use of briquettes as a feedstock in the EAF, they must present physical properties and ability to reduce its metal oxides within the dynamics of operation of this equipment. This is particularly true since, currently, with shorter tap-to-tap times and optimization of energy consumption, the EAF reaches up to be a challenge as a reactor for waste oxide recycling.

Consequently, self-reducing agglomerates made of waste oxide may well find applicability in the electric arc furnace, but only if a reasonable fraction of the content of oxides is to be reduced within a short time period.

Also the mechanical strength is important and should be high enough to allow for maintenance of briquette's integrity during handling.

The results of a preliminary experimental study⁽⁷⁾ on the reduction of self-reducing briquettes composed of mill scales and EAFD (as well as a reducing agent) – called, for this, 'compound briquettes' –, were encouraging.

The initially trials showed that EAFD – a fine-grained material – has great difficulty in forming agglomerates of satisfactory mechanical strength. However, the compound briquettes produced by mixing mill scale with EAFD resulted in tough and compact agglomerates, allowing their handling without major damage.

In tests of carbothermic reduction of briquettes made in the temperature of 1200°C, differences were observed in the evolution of the degree of metallization of iron and reduction / volatilization of zinc in function of time. These tests revealed that the kinetics of reduction of iron compounds were faster than the kinetics of reduction and volatilization of zinc compound - as will be seen below.

Aim of this study is to perform a theoretical analysis, capable to base the study of the reduction reactions of compounds containing iron and zinc found in self-reducing briquettes under heating at a temperature of 1200°C. This hopefully will lead to a better understanding (thermodynamics and kinetics) of the occurring phenomena.

Also there will be a short introduction to the topic EAFD generation based on some thermodynamic considerations.

2 METHODOLOGY

The equilibrium state for every system under study in the present work was determined by the Gibbs energy minimization method using the *Equilib* module of the *FactSage* software (version 6.1) – software is described elsewhere by Bale et al.⁽⁸⁾

The following databases were employed:

- FSstel - FactSage steel intermetallic compounds and alloy solutions [2009];
- FToxid - FACT oxide compounds and solutions [2009];
- FACT53 - FactSage compound database [2009].

For the sake of simplicity, most of the analysis was conducted at the temperature of 1200°C.

3 THEORETICAL CONSIDERATIONS

3.1 EAFD genesis

Fe, Zn and oxygen are known, the three most abundant elements on EAFD. A brief thermodynamic analysis, considering only those elements, will be conducted next.

The goal is to identify the phases that are established at steady state, simulating the genesis of EAFD. The chosen conditions are temperatures close to those existing in the EAF and ancillary equipment and oxygen partial pressure of 0.21atm.

Results show that above 1430°C – the most interesting temperature range – Fe_3O_4 is the single iron oxide in the equilibrium state for $\text{Zn}/(\text{Fe}+\text{Zn})$ smaller than 0.37, replacing hematite, Fe_2O_3 , Figure 1. For greater values of that ratio, however, there are no more ‘pure’ iron oxides (Fe-O system). franklinite, ZnFe_2O_4 , on the other hand, is always present in the equilibrium state, either with magnetite, Fe_3O_4 , or zincite, ZnO .

These results are in good agreement with those from the work of Sofilic et al.⁽⁹⁾ These authors found that magnetite makes up the matrix of all examined samples (mass share in excess of 50wt.%). Moreover, the three major mineral phases identified by XRD analysis in the EAFD from bag filters were: spinels (magnetite and franklinite) and zincite.⁽¹⁰⁾

Based on thermodynamic results and taking into consideration the experimental analyses from literature, it can be concluded that oxidation products of material emanating from the EAF can not be the outcome of a single (overall) state of equilibrium.

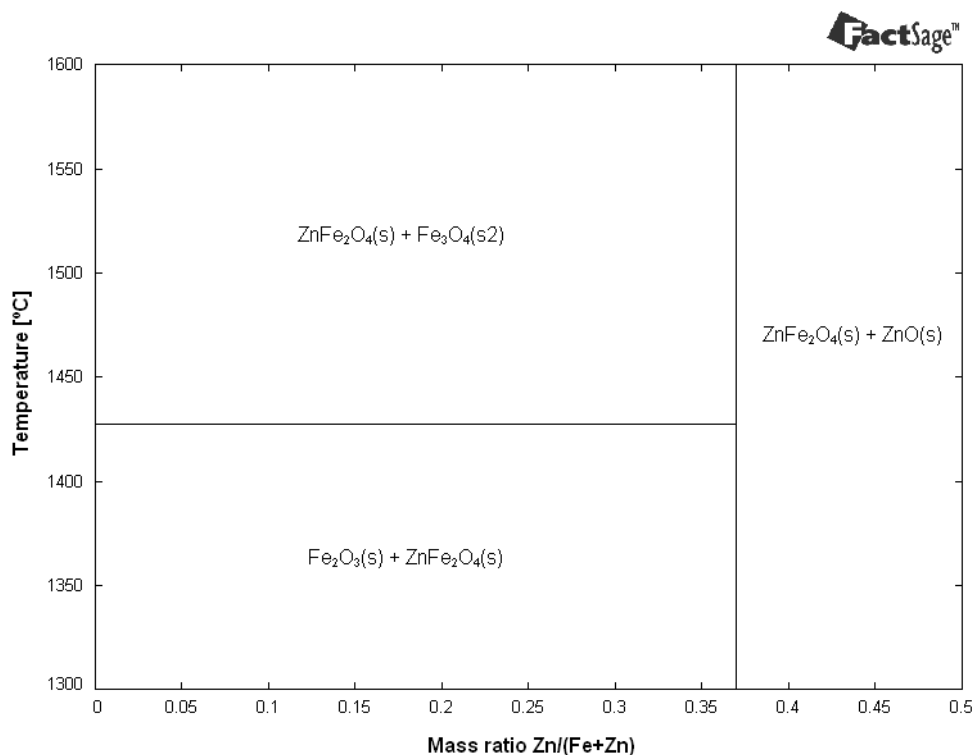


Figure 1. Phase diagram, system Fe-Zn-O, $p_{O_2} = 0.21\text{atm}$.

One might think that, somehow, they represent rather the result of an oxidation reaction on two different *types* of materials: the first, most easily found, is rich in iron – this is the reason why magnetite is so common – and the second, conceivably more seldom, has Zn/(Fe+Zn) value higher than 37% by weight. Amounts of each of these oxides in EAFD will be, of course, variable, dependent on the availability of iron-rich and Zn-rich raw materials in furnace.

3.2 Phases in equilibrium

The phases related to this thermodynamic analysis were treated as if they were stoichiometric compounds. That does not invalidate the analysis itself, because in thermodynamics, enlightening simplifications are regularly made. However, if the question is not addressed more fully, the complex nature of condensed matter will become less visible.

In order to determine the phases present in equilibrium state, the same temperature at which the experiments were done, of 1200°C, was chosen. The pseudo ternary system Fe₂O₃-FeO-ZnO was chosen to represent the relationships among the major oxides present in the raw material. It lies within the composition tetrahedron of the quaternary system Fe-Zn-O-C – a system that is able to encompass all the questions relating to the reduction of oxides of Fe and Zn by carbon.

As can be seen from the results, Figure 2, many of the phases previously reported as intermetallic (stoichiometric compounds) have a variable composition. The most notable case is that of spinel, which changes its composition continuously, from ‘pure’ Fe₃O₄ to ‘pure’ ZnFe₂O₄. As if this were not enough, one may also check, disregarding of the Fe or Zn content, the spinel phase can dissolve a variable amount of oxygen. Within this logic, it can also be observed that zincite dissolves Fe as well as wustite dissolves Zn.

Paradoxically, in the region of pseudo ternary diagram FeO-Fe₂O₃-ZnO where

the three phases are in equilibrium, at 1200°C, the phases FeO, and ZnO Spinel - all solid *solutions* – display a *fixed* composition.

It is important to note, however, that the cited diagram is strongly dependent on oxygen pressure. High pressures, for example 0.21 atm, follow tie lines between Fe₂O₃ and ZnO. This is in accordance with Figure 1. As the oxygen pressure lowers (reduction), the lines approach the ‘corners’ where the reduced metals are located.

Since the element carbon is at one of the vertices of the composition tetrahedron, the possibility of formation of carbides and the possible dissolution of carbon in phases where metallic iron is predominant should also be considered. Zinc, however, does not dissolve this element.

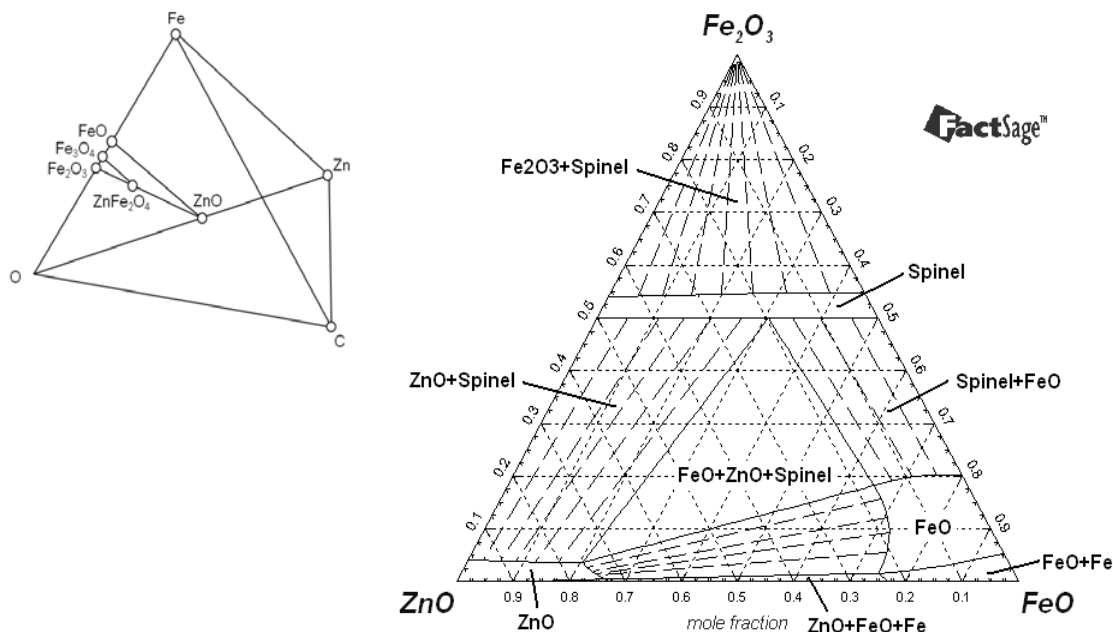


Figure 2. Composition tetrahedron (mole base), system Fe-C-O-Zn and phase diagram, pseudo ternary system Fe₂O₃-FeO-ZnO, temperature: 1200°C.

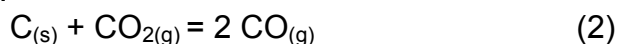
3.3 Carbothermic reduction of self-reducing briquettes

The *self-reduction* concept applies to the metallurgical processing of metal oxides where a strong reducing agent – usually a substance with reasonable levels of fixed carbon – is intimately mixed with metal oxides to be reduced, forming a mechanically coherent aggregate.

It has been demonstrated, since the preliminary work of Rao,⁽¹¹⁾ that the reaction between balanced mixtures of iron oxides with carbon, in the suitable environment and temperature, *e.g.*, in the so-called *direct reduction*, occurs preferentially *via* gaseous intermediates (CO and CO₂). In general, the carbothermic reduction of iron oxides can be viewed with the help of the following reaction between wustite and gaseous CO:



The source of CO can be varied. In reactors where the carbothermic reduction is achieved by the use of solid carbon, the use of ‘Boudouard reaction’ (actually, it is the *sum* of other basic reactions between carbon and oxygen gas) explains the origin of CO:



One can demonstrate that the composition of the gas-phase of each of these reactions (or systems) at steady state – both at a total pressure of 1 atm – will be identical only to the temperature of $\sim 710^{\circ}\text{C}$. Thus, at the temperature of interest, 1200°C , the equilibrium atmosphere of one of them will certainly be different from that of another reaction. Consequently, the relative amount of reagents will be crucial in defining the balance of the ‘overall’ reaction. This reasoning expresses the concept of ‘excess reagent’.

There are places inside the reactor where there are chunks of carbon – realm of the ‘Boudouard reaction’ (2) – and places where iron oxide can be found – now the equilibrium will be given by the stoichiometric reaction (1). That is to say, the two stoichiometric reactions ‘compete’ with each other. Globally, there is an ‘excess of carbon’ in the system – which is a basic condition in the carbothermic reduction. Because of this, the atmosphere of the ‘overall’ equilibrium state will be ideally given by reaction (2) and, accordingly, among the phases FeO and Fe, the only *stable* phase will be the *iron* – this way, the reactor’s goal, which is the production of metallic iron, will be fully achieved.

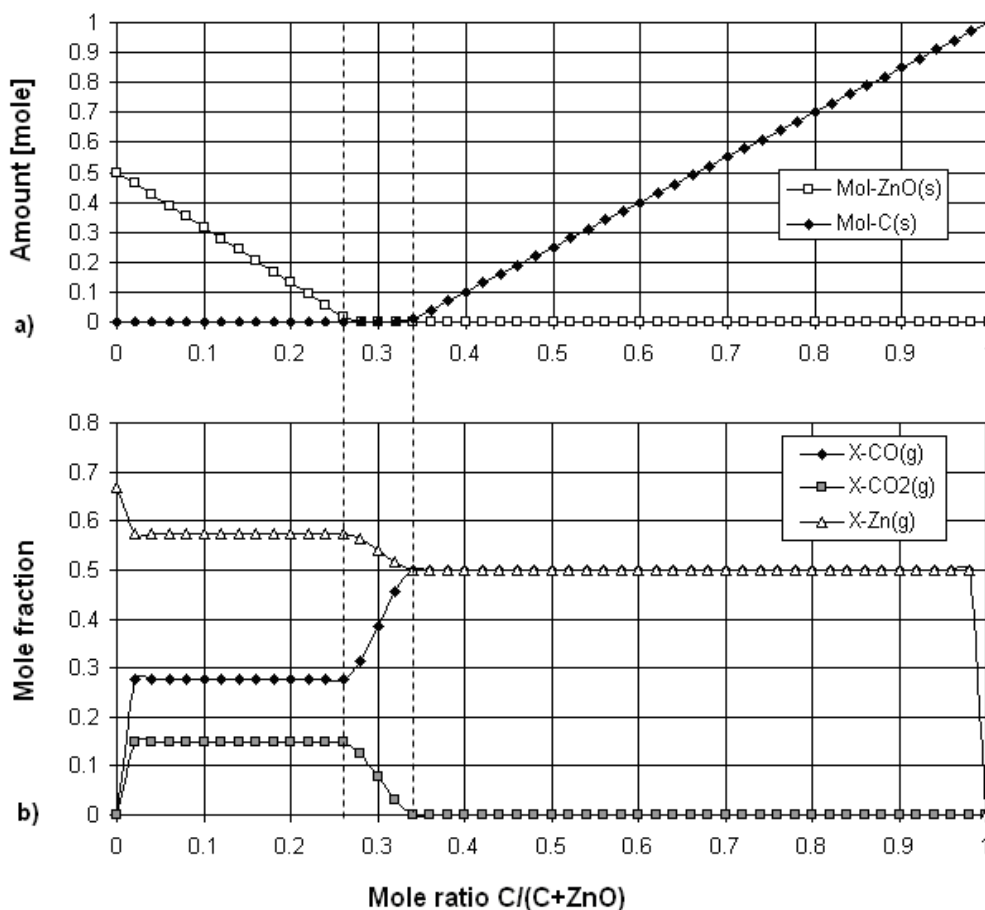


Figura 3. Amount of condensed phases (a) and gas phase composition (b), one mole system, system ZnO-C, temperature: 1200°C .

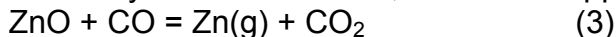
The concept ‘excess reagent’ can also be applied to the carbothermic reduction of zincite (ZnO). The result of applying this concept (system containing *one* mole of matter) can be seen in Figure 3. The composition axis used here corresponds to the imaginary line ZnO-C in the composition tetrahedron (Figure 2).

For a system containing a total of *one* mole of matter, for $C/(ZnO+C) = 0.26$ and up, due to ‘excess solid carbon’ – that is, because of the highly reducing

conditions – there is no more free zinc oxide in the equilibrium state, as can be seen in Figure 3 (a).

Adding a little more carbon, graphite becomes a stable phase, remaining at equilibrium state from now on (between these two points there is a small intermediate region where there is no solid phase present at equilibrium).

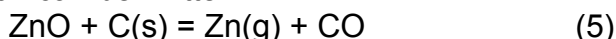
In this case, unlike the reduction of wustite, next to CO and CO₂ there is Zn(g) in the atmosphere, since the temperature is higher than that of the boiling point of metallic zinc. By the same reason, it does not appear as a phase (Figure 3 b):



Once the temperature is low, the predominance of CO and CO₂ in the atmosphere establishes again:



In case of having solid carbon in the system ('excess reagent') the following reaction can be written:



In this case, the ratio CO₂/CO at equilibrium as a function of inverse temperature – as pointed out by Rosenqvist⁽¹²⁾ – will parallel to that of Boudouard reaction for a total pressure of 1 atm (Figure 4).

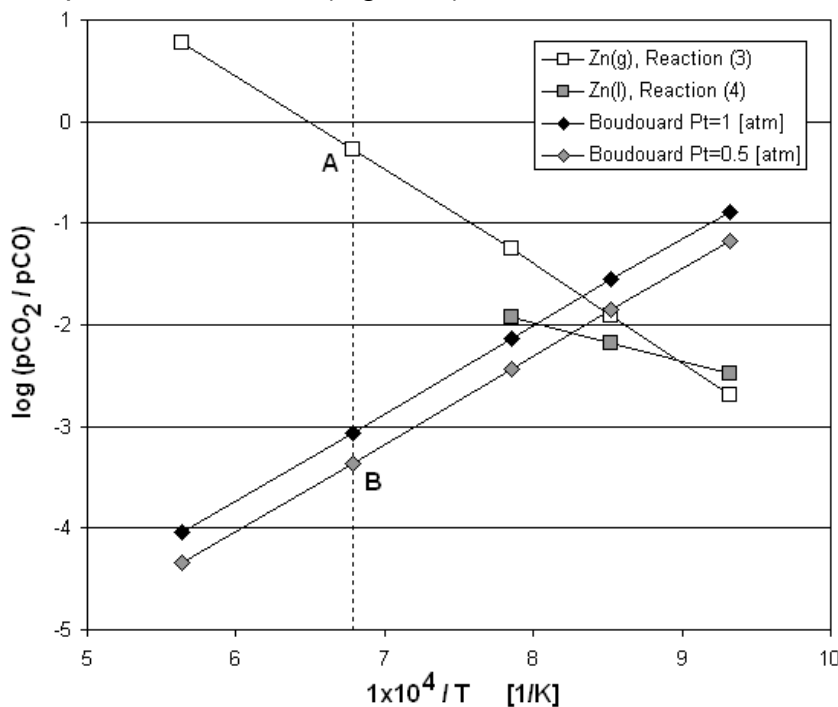


Figure 4. Logarithm of equilibrium CO₂/CO gas ratio for reduction of ZnO(s) to Zn(g) as well as to Zn(l) and for the Boudouard reaction for total pressures of 1 and 0.5 [atm] as a function of inverse of the absolute temperature; A – B distance gives the *driving force* for the carbothermic reduction of ZnO(s) at 1200°C.

The point 'A' of this figure shows the value of logarithm of that ratio for a place with an excess of ZnO ($p_{\text{Zn}} \approx 0.5$ atm) and point 'B' does the same for a site where there is an excess of solid carbon ($p_{\text{CO}} \approx 0.5$ atm). The 'difference' between these two states is proportional to the driving force (see more on this subject later) for the ZnO reduction by solid carbon, for a total pressure of 1 atm and temperature of 1200°C.

Reducing franklinite is somewhat more complex, as one mole of iron and 0.5 mole of Zn is obtained simultaneously from 1/2 mol franklinite.

The phases present in the equilibrium state (system containing *one* mole of matter) as a function of $C/(ZnFe_2O_4+C)$, during the franklinite reduction, can be seen in Figure 5. The composition axis used there – similarly to the previous case – corresponds to the imaginary line $ZnFe_2O_4-C$ in the composition tetrahedron (Figure 2).

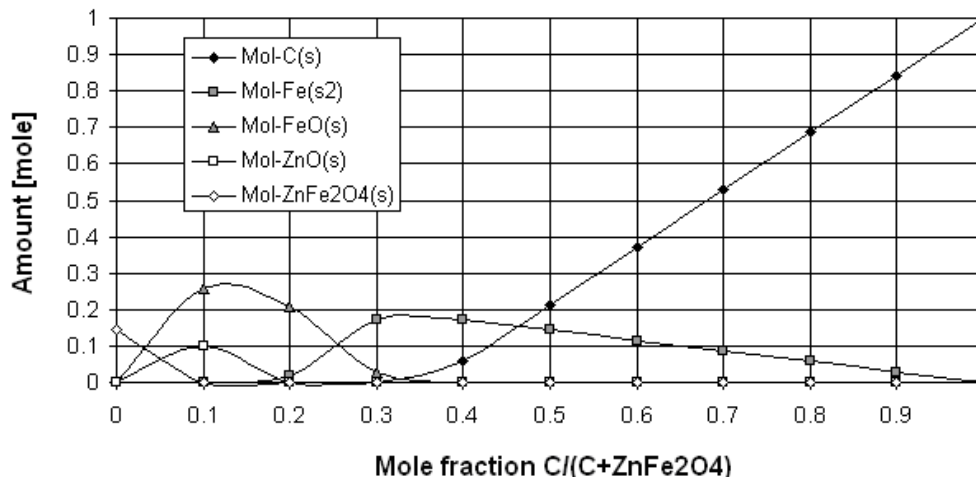


Figure 5. Amount of condensed phases, one mole system, system $ZnFe_2O_4-C$ at 1200°C.

As seen in Figure 5, franklinite ‘vanishes quickly’, giving rise initially to FeO and ZnO-phases, and then, depending on the degree of ‘excess carbon’, graphite and Fe appear. Zinc metal – unlike Fe – by taking part in the gas phase, is also not represented here.

3.4 Thermodynamics as a basis for kinetics

It is known that thermodynamics provides the first indication of the kinetics of any process (reaction, transformation). This indication or trend is called generically *driving force*. The net driving force for a proposed process depends upon the difference between the two quantities: energy in final state minus energy in initial state. This can be seen as the ‘degree of separation’ or ‘distance’ between the initial state (any) and the state of thermodynamic equilibrium of the system. The system whose initial state has the highest ‘degree of deviation’ from equilibrium – other factors disregarded – will be favored kinetically. Whoever, in contrast, shows a small driving force, will tend not to develop.

The driving force of a chemical reaction is measured by the *Gibbs energy variation*, ΔG , on going from the reactants to the products. Intended to be a measure of spontaneity, it can be an indication of a trouble-free kinetics – since large negative values tend to indicate that small kinetic-related *resistances* will be easily surpassed. On the other hand, small ΔG negative values signalize that initial and final states are ‘located’ at a short ‘distance’. In this case, even the smallest resistance represents an insurmountable obstacle, indicative of a slow kinetics. Nevertheless, both are spontaneous reactions.

The determination of ΔG of a chemical reaction depends on a reference value, the variation in *standard* Gibbs energy, ΔG° , plus the knowledge of the initial and final reagents conditions (activities). For ease of determining, the value of ΔG° is often used in place of the Gibbs energy change for an initial, or immediate, evaluation, of the driving force of a chemical reaction. It must be kept in mind, that a reaction which

presents a large negative value for ΔG° , with great probability will also have a large negative ΔG value.

Aligning different reactions, one can make a comparison between them to obtain, at the same time, indications of their spontaneity and the likely driving forces.

The reaction (1) represents a reduction with CO(g). The presence of solid carbon in a reaction can be obtained from the sum of reactions (1) and (2). It represents in a better way the situation of an 'excess carbon' inside the reactor. This general reduction reaction can be written for all essential compounds of this study.

Since the Gibbs energy is an extensive property, standardization is necessary so that the comparison between the reactions is valid – as is the case of the Ellingham diagrams. To establish a common basis for comparing the reactions of carbothermic reduction of oxides, we used one mole of C(s) reagent. Thus, all reactions of interest were written as a carbothermic reduction by 1 [mole] of solid carbon (Table 1). The results of the standard Gibbs energy change determination for the selected reactions can be also seen in Table 1.

Table 1. Standard Gibbs energy variation for selected carbothermic reduction reactions of some iron and zinc oxides at 1200°C

Reaction	ΔG° [J]
$1/3 \text{ Fe}_2\text{O}_3 + \text{C} = 2/3 \text{ Fe} + \text{CO}$	-92 447.4
$1/4 \text{ ZnFe}_2\text{O}_4 + \text{C} = 1/4 \text{ Zn(g)} + 1/2 \text{ Fe} + \text{CO}$	-85 279.1
$1/4 \text{ Fe}_3\text{O}_4 + \text{C} = 3/4 \text{ Fe} + \text{CO}$	-79 454.6
$\text{FeO} + \text{C} = \text{Fe} + \text{CO}$	-72 072.0
$\text{ZnO} + \text{C} = \text{Zn(g)} + \text{CO}$	-71 929.3

The results show, based on ΔG° values, that all the selected reactions would be *spontaneous*. The interpretation of results according to suggested criteria, suggests that the kinetics of hematite reduction should be the most favored. Finally comes the reduction reaction of zincite, which has the lowest driving force of all.

This is also observable using the ratio CO_2/CO as criterion: to reduce zincite, the 'distance' between the initial and final states is small (Figure 4), unlike, for example, this same situation involving hematite.

4 COMPOSED BRIQUETTES REDUCTION EXPERIMENTS

In Table 2 and Figure 5 the experimental results regarding the extent of metallization of iron and the extent of removal of (volatile) zinc in self-reacting compound briquettes made of EAFD and mill scale, at the temperature of 1200°C, can be observed.⁽⁷⁾

It is clearly seen that for the lower test times (10 and 15 min), the percentage of reduced iron is greater than the percentage of zinc reduced (volatilized). These results confirm to some extent, based on the criterion of Gibbs energy change, the framework provided previously, that the reactions which have the highest driving forces are those involving iron oxides.

Table 2. Iron Metallization Degree (Fe^o MD) and zinc Removal Degree (Zn RD) at 1200°C of samples after some reduction time periods

Sample	Fe ^o MD [wt.%]	Zn RD [wt.%]
A (10 [min])	47.6	16.2
B (15 [min])	86.7	40.5
C (20 [min])	90.1	96.5
D (30 [min])	93.4	98.6

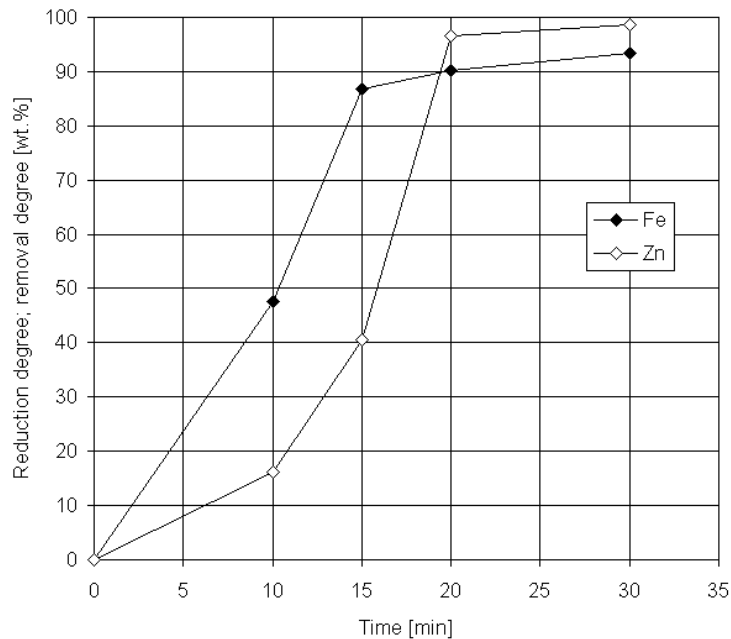


Figure 5. Iron metallization degree (Fe° MD) and zinc removal degree (Zn RD) at 1200°C of samples after some reduction time periods.

Moreover, from 15 min, there is a dramatically reduction in the degree of metallization of iron.

For an increasing metallization degree there is always a tendency for a decrease in the rate of the reduction reactions. This decrease with the passage of time is a characteristic of the self-reducing briquettes of iron oxides and is explained by the topochemical model of reduction, which shows a core of unreacted material gradually more difficult to reach.

Anyway, a number of factors can influence the kinetics of the reduction reactions of iron oxides in the self-reducing agglomerates, e.g. process temperature and rate of heat transfer. Specific properties of oxides such as: density, crystal structure and porosity, or the amount of reactive surface of iron oxides exposed to reducing gases will interfere as well. The slowest step of all (rate-determining step) will determine the overall rate of reaction – once no ‘mixed control’ exists.

With reference to the reduction and removal of zinc from the samples, it can be said that gaseous zinc is formed and that means (in addition to the difficulties of removing Zn) that a high amount of heat must be transferred to Zn gas – since its reduction is an endothermic phenomenon (it can be seen by applying the van't Hoff equation to the data of Figure 4). These facts could also influence and slow down the kinetics of the reactions of reduction of ZnO.

5 CONCLUDING REMARKS

Other factors may be considered interesting in using self-reducing agglomerates of EAFD with the EAF mill scale – beyond the recovery of iron found in these wastes. They are related with:

- the decrease in the total amount of EAFD generated and,
- new uses for the Zn-enriched residues.



The concretization of these possibilities arises from the repeated EAFD recycling and reduction in EAF. Since all contained Zn tends to go only to the EAFD, the two factors listed above have a tendency to take place.

In parallel there is also a tendency to increase the *zincite* content at the expense of franklinite during the recycling of waste.

More studies should be made to explore these possibilities.

6 CONCLUSIONS

The thermodynamic simulations on EAFD genesis – considering the appropriate temperature range – showed that primarily iron oxides (mostly magnetite) and spinels of iron and zinc are generated. However, this composition is subject to some variations, according to the proportion of components Fe and Zn in the charge, depending on the different equilibria that can establish in the system Fe-O-Zn.

It could be said, based on thermodynamics, the difference in the kinetics of reduction of iron (faster) and zinc oxides contained in self-reducing briquettes, has direct relation to the greater stability of zinc oxide in relation to iron oxides.

However, there may be other factors interfering in the process, factors related to the transport of reducing gas (CO) to the interior and product gas (CO₂) out of the briquette, coverage of metal oxides by reduced Fe, difficulties in the removal of gaseous Zn from the interior of briquettes and in providing the necessary heat for the endothermic reduction reactions. That will be dealt with in a forthcoming work.

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