

Evaluation of the Energy Generated In the Burning Of Residue Containing Iron and Manganese.

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Abstract: The world has been feeling the decline in coal production over the years. According to Araújo et.al, the world supply of coal in 2013 did not show significant growth compared to the year 2012. Five countries had increased production: Australia (10.9%), Indonesia (9.1%), Brazil (11.7%), Canada (4.0%) and China, which remain at the same level of 2012, with an increase of only 0.8%. For the other countries, there was a decrease in the supply of mineral coal, in particular in the case of the United States with (-3.2%), which has been reducing its production to the detriment of the substitution for the production of shale gas. Approximately 80% of the world's coal reserves are in the Northern Hemisphere. The largest producers are China, the United States, Russia and Australia, being the largest exporter of coal to the world and also to Brazil. Brazil is not among the major coal producers, since its annual production is about 0.1% of world production. However, the lack of coking coals makes it possible to use a mixture of several types of coals in most of the world, including Brazil, whose sum of their properties makes it possible to produce a suitable coke. Another important outlet for the shortage of coal production is the use of coal-fired steel waste. Such wastes are metallurgical by-products, which contain a considerable amount of iron and carbon. Recycling this waste as well as improving the efficiency of coal burning can also help reduce the environmental pollution from iron making in steelmaking processes. Thus, in this work it was possible to evaluate the reuse of iron and magnesium containing iron and steel residue. For this purpose, the statistical method of the central rotational composite design was used to evaluate the influence of the parameters of percentage of mass of residue and particle size of the particles on the burning efficiency of the coal and residue mixture. X-ray diffraction (XRD) and scanning electron microscopy (SEM) were performed for the characterization of the manganese iron residue samples. Through TGA/DSC analysis was possible to demonstrate higher values of burning of the coal and residue mixture as compared to the burning of "in natura" coal.

Keywords: Charcoal; Waste steel; Environment; Iron ore.

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I. Introduction

In the process of iron and steel, a large amount of dust and sludge are generated. They are by-products, which contain a considerable amount of iron and carbon. Recycling of these by-products containing iron can help reduce the environmental pollution of iron and steelmaking processes. In the past, the quantities of dust emitted could already be significantly reduced by better separation or by improving the technologies related to this process, thus leading to an increase in the amount of dust collected [1]. However, the powder containing iron is considered difficult to recycle due to the fine characteristics of the particles. However, a common way of using these by-products is to reuse them in the sintering plant as sinter feed materials. However, these by-products have negative effects on the quality of the sinter, the strength of the sinter and the productivity, thus,

the recycling of waste by this process must be in limited quantity [2]. Another method, for pre-treatment of iron-containing powder is the pelletizing process, however, its reduced particle size causes difficulties during pelletizing or briquetting process [3]. In addition, metallurgical by-products generally contain heavy metals and alkalis in their composition [4]. Thus, a viable recycling option for iron-containing powder consists of mixing the residue during the injection of pulverized coal (ICP), which is an effective technology to reduce the consumption of coke in the blast furnace and, consequently, to reduce the problems related to the medium environment.

Some researches have been developed with this purpose and can be cited Xuzhong et. al [5] the effects of CeO₂ and Fe₂O₃ on the combustion reactivity of various fuels, including coal, graphite and anthracite characters, were investigated using thermo-gravimetric analyzer. The results indicated that the combustion reactivity of all samples except lignite was improved with the addition of Fe₂O₃ or CeO₂. It was interesting to note that the anthracite ignition temperatures were reduced by 500C and 530C, respectively, with addition of CeO₂ and Fe₂O₃, also showing an increase in the combustion rates to 15.4% / min and 12.2% / min. Lignite ignition temperatures with CeO₂ and Fe₂O₃ were 2500C and 2260C, and the combustion rates were 12.8% and 19.3% / min, respectively.

Li-Ming Zhang et. al [6] determined the effect of an additive on the burning of the coal in the calorific values of a coal of high calorific value and one with low calorific value. The results showed that there was no significant effect on the calorific values of the two coals. The catalysts and accelerators of the additives in the two coals were studied with the help of thermogravimetric analysis. The effect of the additive occurred due to catalysis in the burning of the carbons of the carbons. Fuels with higher amounts of carbon, such as coal, make the acceleration and effect of burning more effective.M.A.

Diez et. al [7] evaluated two oils for lubrication, and plastic waste composed mainly of polyolefins (95%) and their mixtures, for the purpose of possible use as minor components in coal blends for the production of metallurgical coke. The addition of plastic waste at 2% by weight causes a decrease in the maximum flowability of the coal during the heat-up between 400 and 5000 ° C. However, it has been observed that the two oils are good additives for the coal / plastic mixture in order to partially restore the agglomeration capacity of the co-carbonization systems. Co-carbonization of the coke mixture with the different types of residues were carried out on a oven with a capacity of 15 kg. Although the bulk density remained unchanged, the addition of the plastic waste produced an increase in coke pressure to high values. At the same time, the mechanical strength of the partially carbonated coke was improved as presented by the RSE index. The oils, however, had the effect of reducing the density and coke pressure generated during the process. When mixed with coal and plastic materials, the oils appeared to act as good polyolefin solvents and also proved to be effective in reducing coke pressure without adversely affecting the quality of the coke.

Chong Zou et. al [8] evaluated the effects of powder containing iron on the behavior of coal combustion. Thermogravimetry (TG) and differential calorimetric scanning (DSC) were used. The results indicated that the presence of hematite, BOF dust and sludge reduced the ignition temperature of BOF, thus accelerating the combustion rate, improving the burning behavior and increasing the rate of heat release during the coal combustion process . However, BF dust generally showed a negative effect on the combustion behavior of coal. Based on the results of the kinetic study, XRD and SEM analysis, a possible combustion mechanism intensified by these powders was proposed. The activation energy of the combustion reaction decreased with the addition of powder. In addition, the surface morphology seems to be more important than the chemical composition in the catalytic effect of the iron-containing powder in the coal combustion.

II. Materials And Methods

2.1 Characterization of the residue used.

X-ray diffraction (XRD) and scanning electron microscopy (SEM) was performed to characterize the samples of waste added to pulverized coal. The residue samples originate from the region of Minas Gerais, Brazil. All the samples were comminuted in a ball mill at the facilities of the School of Industrial and Metallurgical Engineering of the Fluminense Federal University, generating granulometric fractions of 0.0036; 0.053; 0.061; 0.107 and 0.234mm. The obtained fractions were submitted in powder form to analyzes and presented the chemical compositions presented in Table 1,

Table 1. Chemical composition of the evaluated waste sample

Powder	Mn	Fe	SiO ₂	Al ₂ O ₃	P	CaO	MgO	BaO	K ₂ O	TiO ₂
Conc (%)	21,00	1,39	19,42	2,83	0,023	1,62	0,275	0,76	13,6	0,001

The crack and microcrack structures were visualized by scanning electron microscopy (SEM). The X-ray diffraction technique allows the generation of a spectrum which, through the Ritveld method, can simulate an entire diffractive profile based on structural parameters of the possible component phases, allowing more information to be extracted from the diffractograms. The Ritveld method takes into account the overlapping of the peaks of all phases present with the background noise (background) contributions with a standard spectrum for the analysis of the ore in question.

For the analysis of the variables of the waste - coal mixing process, the central composite planning with central and axial points was used. The central composite design is a symmetric and second order design, consisting of two parts: the factorial 2k, with one or more central points, and the axial part.

Among the standard designs, the Central Composite Design (DCC) is considered a satisfactory delineation. According to Atkinson and Donev (1992) [9] the composite designs belong to a family of efficient designs, which require few trials for their realization. Regarding efficiency measures, Lucas (1976) and Donev and Atkinson (1988) show that the DCC performs well according to the optimization criteria. [9]

In addition, it has interesting characteristics for the search of the point that gives the optimal answer, which are: a smaller number of treatments in relation to the complete factorials and can be performed sequentially, in order to move towards the optimization of the system, that is, by performing a part of the experiment (applying factorial experiments 2k or fraction-first-order experiment) through the "steepest ascent" technique. Samples from different iron ore masses were reacted according to the factorial planning methodology with central and axial points including levels from -1.41 to +1.41 following a factorial 22 including 4 trials at the axial conditions and 3 replicates at the center, totaling 2 trials. Thus, the following variables were analyzed: percentage of residue in the coal and mean particle size of the particles being presented in Table 2.

Table 2. Variables and levels used in experimental planning

Variables	-1,41	-1	0	1	+1,41
Mean Particle Size (mm)	0,0036	0,053	0,061	0,107	0,234
Percentage of residue in the coal sample	1	5	10	15	20

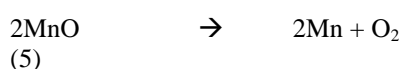
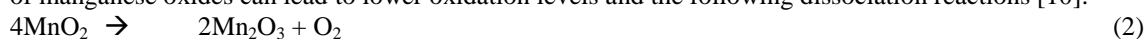
In this study, the enthalpy change was used to identify the energy generated during the burning of the coal mixture and the added residue in the tga. For the evaluation of the best conditions of generation of energy in the form of heat flow, the experimental factorial planning method with axial points and central points was used where it was possible to obtain useful information about the behavior of the acquired responses in relation to the levels and factors initially taxes can also evidence the quality of the process's repeatability. The mixture of iron and manganese-containing residue was added on a thermogravimetric scale located inside the equipment and then the mixture was burned at a rate of 150C / min in order to reach a temperature of 100 ° C having an isotherm of 1 min in a 50 ml air flow.

After obtaining the graphs the calculation of the amount of energy released follows:

$$\int_a^b f(x)dx = \int_a^{a+\Delta x} f1(x)dx + \int_a^{a+2\Delta x} f2(x)dx + \dots \quad (1)$$

Where a and b represent the initial and final energy of a given fraction within the heating curve thus obtaining different energies as a function of time and corresponding function. In this way it is possible to obtain the area of the heating chart as well as the energy released during the burning of the coal-residue mixture.

Manganese may have oxidation states between Mn⁺³ and Mn⁺⁷, with the most known oxidation states being Mn⁺⁴ and Mn⁺². The oxidation state of the main ores is Mn⁺⁴ and the oxidation state of the major chemical compounds of manganese is Mn⁺². Heating under controlled conditions (decomposition of these oxides to air) of manganese oxides can lead to lower oxidation levels and the following dissociation reactions [10]:



Where the following free energy variations are obtained:

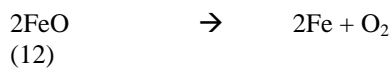
$$\Delta G^\circ = 40185 - 51,28 T \text{ cal / mol O}_2 \quad (6)$$

$$\Delta G^\circ = 44204 - 36,89 T \text{ cal / mol O}_2 \quad (7)$$

$$\Delta G^\circ = 104844 - 55,46 T \text{ cal / mol O}_2 \quad (8)$$

$$\Delta G^\circ = 186742 - 36,65 T \text{ cal / mol O}_2 \quad (9)$$

In the case of iron, it presents the following stages of decomposition [10]:



The free energy variations for these dissociation reactions are [10]:

$$\Delta G^\circ = 114287 - 65,93 T \text{ cal / mol CO}_2 \quad (13)$$

$$\Delta G^\circ = 158969 - 54,89 T \text{ cal / mol CO}_2 \quad (14)$$

$$\Delta G^\circ = 126645 - 30,90 T \text{ cal / mol CO}_2 \quad (15)$$

Then, considering the reactions with their thermodynamic data, it was possible to develop the Gibbs energy diagrams for the manganese and iron observed in Figures 1 and 2.

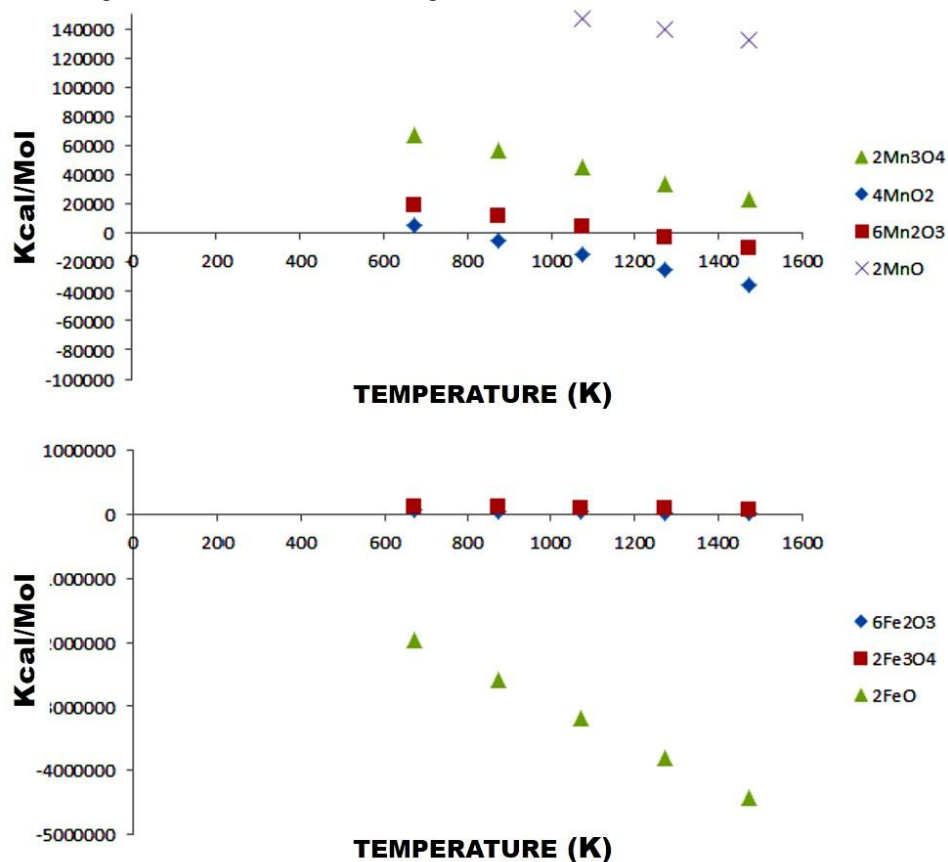


Figure 1 - Gibbs Energy Variations for equilibrium with pure oxides of manganese and iron.

Figure 1 shows the variation of the Gibbs energy for the equilibrium with pure oxides of manganese and of iron in total pressure of 1 atm. Analyzing the temperature at 1250 ° C and 1523 K, the spontaneity of the decomposition of the oxides 4MnO_2 and $6\text{Mn}_2\text{O}_3$ in equilibrium with the decomposition of 2FeO was observed. It was also observed the absence of spontaneity for the decomposition of the MnO species, thus preventing the obtaining of pure metallic Mn. According to Benique [10] As the partial pressure of O_2 at equilibrium in the $\text{CCO} - \text{CO}_2$ system at this temperature is of the order of 10 - 16 atm, it was possible to conclude that iron reduction is possible to obtain pure metallic Mn activities of C, Fe, Mn and their oxides.

III. Results And Discussions

3.1 Analysis of the sample of ferro - manganese residue used in the tests.

In figure 2 are presented images by means of scanning electron microscopy technique of particles of residues with a mean particle size of 0.061 and 0.234mm.

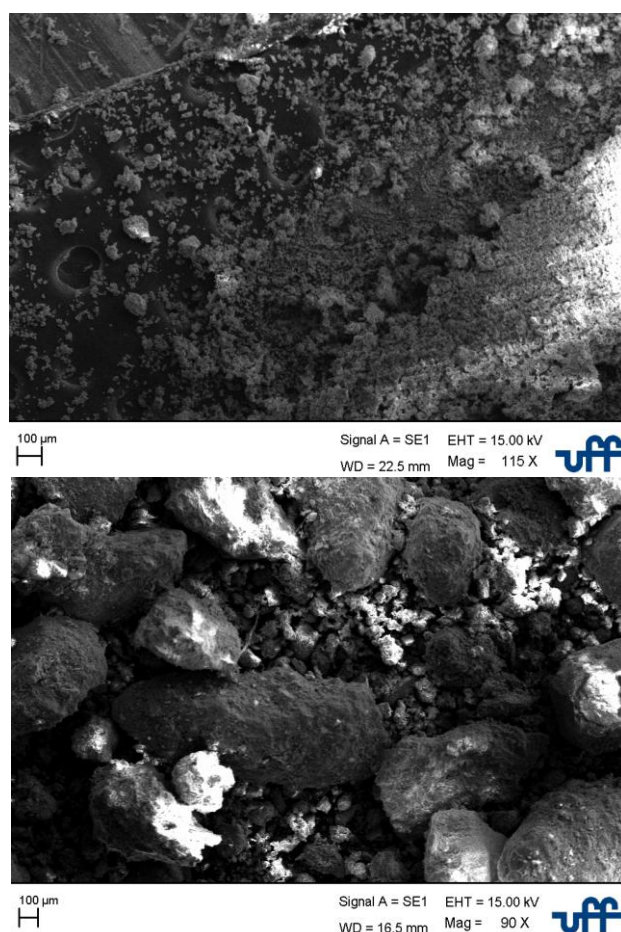


Figure 2. Images made by scanning electron microscopy technique in waste granulometric mean particle size of 0.061 mm and 0,234mm.

It was presented in Figure 2 images obtained by the scanning electron microscopy technique of residuals of residues with a mean particle size of 0.061 and 0.234mm, with the particle agglomeration phenomenon being observed in the particles of mean particle size of 0.061mm. Phenomenon not observed in particles with a mean particle size of 0.234mm. According to Vamuka et. al [11] the use of larger particles improved the degree of combustion relative oxygen / coal inside the blast furnace. Going against Kucukbayrak et. al [12] stated that for large particles ($> 1\text{mm}$), the reaction rate increases with the specific external surface area, which can be explained by the increase of the contact surface between the particle and the oxygen during the combustion process.

3.2 Analysis of the burning of coal with different levels of residues.

Table 3 presents the table with the tests indicated by the rotational central delineation method followed by the average enthalpy of the combustion reactions. In Figure 3 all overlapping tests indicated by the rotational central design method are presented.

Table 3. Assays indicated by the rotational central delineation method.

Test	% Of Waste	Granulometry : Average (mm)	Enthalpy : Average (mW)
1	5	0,107	10481
2	15	0,107	10481
3	5	0,053	10567
4	15	0,053	10696
5	1	0,061	10021
6	20	0,061	10715
7	10	0,0036	9826
8	10	0,234	9525
9	10	0,061	9889
10	10	0,061	9277
11	10	0,061	9921

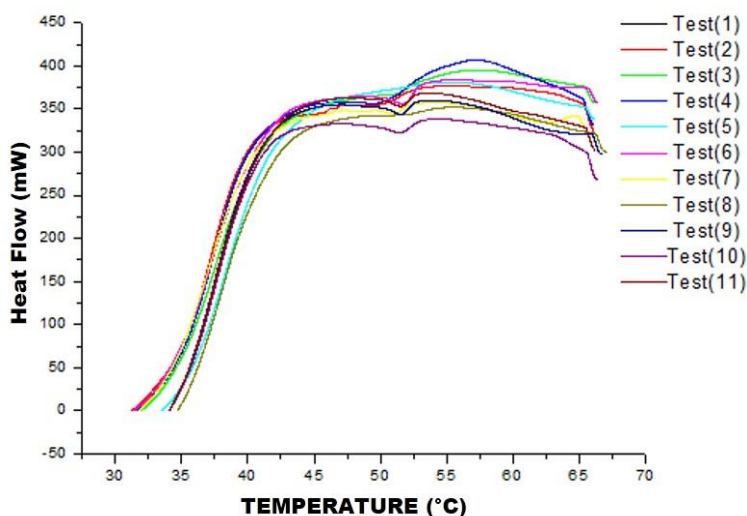


Figure 3. Overlapping heating curves corresponding to the 11 tests.

Figure 3 shows the heating curves corresponding to the 11 tests carried out as a function of the design of the rotational central composite design. It was observed the occurrence of better results in the tests 4 and 6 indicating that the increase of the heat flow occurs due to the increase of percentage of residue in the sample and the increase in the sieve value of the same. Figure 4 shows the percentages of heat flux increase as a function of the granulometric fraction and the percentage of iron manganese residue during the combustion process of the coke-waste mixture.

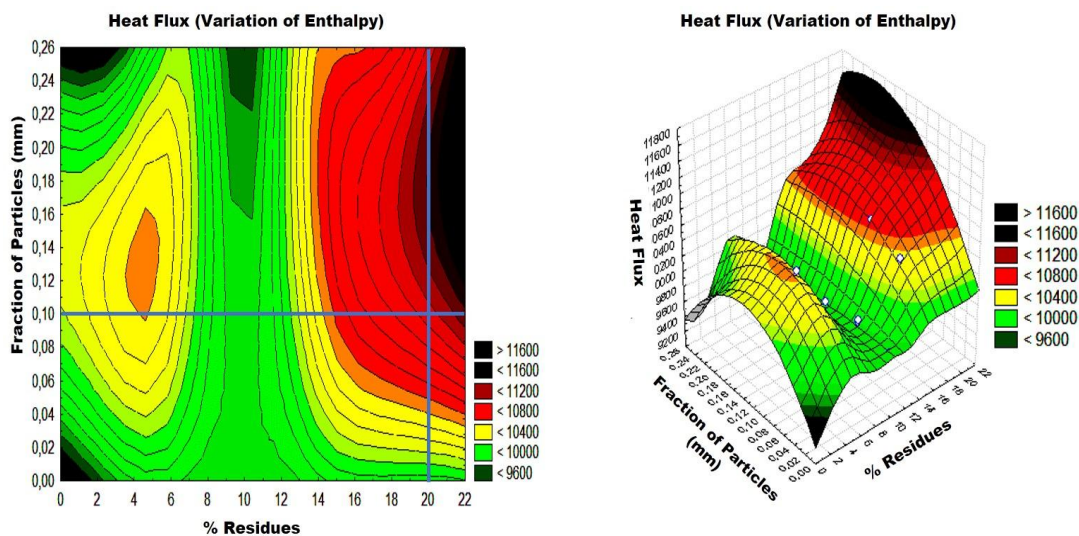


Figure 4. Heat flux values as a function of the percentage of residues and the mean particle size fraction of the particles..

It is observed in Figure 4 the growth of the heat flux values as a function of the increase of the percentage of residues in the coal - residue mixture to values above 20%. It was also observed that in values of mean particle size fraction above 0.1mm a significant increase of the heat flow inside the thermogravimetric analyzer occurred.

In Figure 4 the presence of particle agglomerates smaller than 0.04 mm in size was observed. The agglomerates formed may hinder the oxidation kinetics of the reduced minerals thus preventing the increase of the generated heat flow. Going against Zou et. al [8] stated that mineral species and exert a strong catalytic effect on the combustion of mineral coal samples. It was observed that the presence of mineral phases is of great importance in increasing the combustion rate of coal. Manganese oxides (MnO_x) including Mn₃O₄, Mn₂O₃, and MnO₂ are among the most active oxide catalysts for the oxidation of volatile organic compounds [9].

According to Mozer et. al [14] the Mars-van Krevelen mechanism suggests that oxidation reactions of oxides catalyzed by oxides occur in a first step between oxide and hydrocarbons [14]. The hydrocarbon is oxidized and the oxide is partially reduced. In a second step, the oxide reacts with the O₂ returning to the initial state. The oxidizing agent is O²⁻ from the oxide. The tendency of an oxide (or combination of oxides) to donate its oxygen should be of major importance in determining whether the oxide is a selective oxidizing catalyst. If the oxide reduction is easy (dissociation-free energy and presumably the enthalpy of dissociation is small), then the oxygen can be donated to the molecule from the gas phase and the catalyst must be active rather than selective. On the other hand, if it is difficult to dissociate O₂ due to the strong metal-oxygen bond, the oxide should have low catalytic activity. In an intermediate range, oxides should be moderately active and still selective. Generally this mechanism is applied in oxidation reactions catalyzed by oxides surfaces., Ramesh et. al [15] reported that the reactivity shows an order of MnO < MnO₂ < Mn₂O₃ oxidation in the oxidation of CO. MnO_x have various structures and morphology. According to Zou et. al [8] the surface morphology seems to be more important than the chemical composition in the catalytic effect of iron-containing powder in the coal combustion. The coupling of mNO_x with other metal oxides, eg Co₂O₃ [14], CuO [15], Fe₂O₃ [16], CeO₂ [17], is generally favorable for catalytic performance. CeO₂ oxide increases oxygen storage capacity, while transition metal oxides improve compound reducibility. As large surface area aids in increasing the activity of the microporous and mesoporous materials with large surface area, for example, Al₂O₃ anatase, zeolites, clays, clays and pillared, are used as carriers for excellent making well dispersed mNO_x nanoparticles. Materials with large surface area, abundant porosity, strong surface acidity, and high thermal stability are suitable substrates for species that promote oxidation.

IV. Conclusion

Based on the analysis methodology and results obtained in this work, it is considered that the feasibility of reuse of wastes containing manganese together with pulverized coal. Each of these steps was studied and its impact on pulverized coal burning was analyzed. In general, the following points can be highlighted:

- ✓ The morphology of the residue is considered important for the combustion of the same in the blast furnace. The particles of larger particle sizes are considered to be better for the combustion process.
- ✓ Given the diagrams presented and the thermodynamic data obtained it is possible to conclude that iron reduction is possible and it is impossible to obtain pure metallic Mn during combustion inside the blast furnace.
- ✓ Data obtained through the statistical tests it was possible to conclude that the heat flow occurs due to the increase of the percentage of residue in the sample and the increase in the sieve value of the same.
- ✓ Given the presented results being understood through the mechanism of Mars-Van Krevelen (MVK) it was observed the possibility of the manganese to oxidize in several oxides where it allows the catalysis of the combustion of the carbon coming from the coal being extremely favorable for the combustion process of the inside the blast furnace.

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