

UNIVERSIDADE FEDERAL DE MINAS GERAIS

Escola de Engenharia

Programa de Pós-Graduação em Engenharia Metalúrgica, Materiais e de Minas

Tese de Doutorado

Avaliação de *Pellet Feed* de diferentes superfícies específicas como alternativa de matéria-prima para a sinterização de minério de ferro

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Dezembro/2019

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Tese apresentada ao Programa de Pós-Graduação em Engenharia Metalúrgica, Materiais e de Minas da Escola de Engenharia da Universidade Federal de Minas Gerais, como requisito parcial para obtenção do Grau de Doutor em Engenharia Metalúrgica, Materiais e de Minas

Área de Concentração: Metalurgia Extrativa
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Belo Horizonte
Universidade Federal de Minas Gerais
Escola de Engenharia

2019

O48a

Oliveira, Vinícius de Morais.

Avaliação de *pellet feed* de diferentes superfícies específicas como alternativa de matéria-prima para sinterização de minério de ferro [recurso eletrônico] Vinícius de Morais Oliveira. - 2019.

1 recurso online (xiii, 56 f. : il., color.) : pdf.

Orientador: Maurício Covcevich Bagatini.

Coorientadora: Valdirene Gonzaga de Resende.

Tese (doutorado) - Universidade Federal de Minas Gerais, Escola de Engenharia.

Inclui bibliografia.

Exigências do sistema: Adobe Acrobat Reader.

1. Engenharia metalúrgica - Teses. 2. Metalurgia extrativa - Teses. 3. Minérios de ferro - Teses. 4. Sinterização - Teses. 5. Granulação - Teses. I. Bagatini, Maurício Covcevich. II. Resende, Valdirene Gonzaga de. III. Universidade Federal de Minas Gerais. Escola de Engenharia. IV. Título.

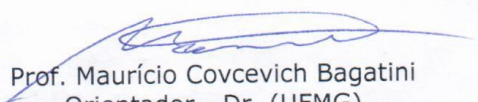
CDU: 669(043)

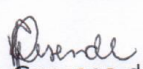


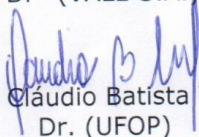
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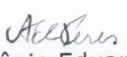



Tese intitulada "**Avaliação de Pellet Feed de Diferentes Superfícies Específicas como Alternativa de Matéria-prima para a Sinterização de Minério de Ferro**", área de concentração: Metalurgia Extrativa, apresentada pelo candidato **Vinícius de Morais Oliveira**, para obtenção do grau de Doutor em Engenharia Metalúrgica, Materiais e de Minas, aprovada pela comissão examinadora constituída pelos seguintes membros:

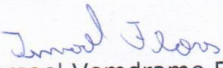

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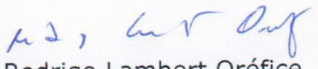

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Belo Horizonte, 06 de dezembro de 2019

Dedico este trabalho à toda minha família e em especial à minha esposa, Simone, e aos meus filhos, Gabriel e Guilherme.

AGRADECIMENTOS

Primeiramente a DEUS.

A toda minha família pelo apoio e compreensão.

A minha Mãe e a meu Pai (*in memorium*) pela educação, valores e incentivo. A senhora Mariquita pela coragem, dedicação, trabalho, força e suor na criação dos filhos. Aos meus irmãos Nádia e Norval.

Em especial a minha esposa Simone, aos meus filhos Gabriel e Guilherme, pelo incentivo, carinho, companheirismo, por estarem ali sempre quando precisei e por compreenderem a necessidade de inúmeros momentos de ausência dedicados a este trabalho.

Ao professor Maurício Bagatini pelo apoio e orientação.

A Valdirene Resende pela ajuda, apoio, orientação na consolidação e discussão dos resultados.

Ao Alei Domingues pela ajuda na execução dos planos de trabalho.

A Delciane Porfiro, aos colegas da planta piloto de sinterização, Maximiliano e Fagner. Aos colegas do laboratório de caracterização de materiais, Etevani e Ana Paula.

A Vale S.A. pelo apoio e pela oportunidade de crescimento profissional, representada pelo Rogério Carneiro. Aos demais colegas do Centro de Tecnologia de Ferrosos, laboratório de preparação de amostras, laboratório químico, laboratório metalúrgico, planta piloto de sinterização e laboratório de caracterização de materiais.

A todos aqueles que de alguma forma contribuíram para a concretização deste trabalho.

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RESUMO

Nos últimos anos, com a crescente demanda por minério de ferro e o decréscimo das jazidas de alta qualidade, as principais mineradoras ao redor do mundo vêm realizando investimentos com o objetivo de um maior aproveitamento de rochas com baixos teores de ferro, conhecidos como itabiritos. No Brasil, essas rochas passam por um processo de beneficiamento utilizando as operações unitárias de classificação, cominuição e posterior concentração. O produto obtido é o *pellet feed*, concentrado de elevado teor de ferro, baixo teor de contaminantes, porém muito fino, tipicamente com 95% das partículas menores do que 0,15 mm. Originalmente esse material é utilizado como matéria-prima para o processo de pelotização, porém, devido a sua qualidade química, este também poderia ser utilizado nos processos de sinterização. Entretanto, a sua menor granulometria afeta negativamente a permeabilidade do leito de sinterização, restringindo o seu uso a pequenas quantidades. Dessa forma, diferentes alternativas tecnológicas têm sido desenvolvidas para permitir o uso destes materiais na sinterização, tais como, uso de misturadores intensivos, pré-aglomeração, uso de aditivos especiais, tratamento mecânico através de prensa de rolos, dentre outras. O tratamento mecânico através de prensa de rolos tem o intuito de aumentar a superfície específica do *pellet feed* e conseqüentemente favorecer a etapa de granulação no processo de preparação da mistura de minérios. Neste trabalho estudou-se a utilização de um *pellet feed* hematítico natural (como ele é obtido após o processo de concentração na mina) e tratado mecanicamente em prensa de rolos, com diferentes níveis de superfície específica, como uma alternativa de matéria-prima para sinterização. Na primeira etapa do trabalho realizou-se a substituição de 25% de *sinter feed* tradicionais de mercado por *pellet feed* natural e com tratamento mecânico. As misturas foram submetidas a ensaios de granulação para avaliação das quase-partículas formadas (índice de granulação, microscopia óptica e teste de queda) e sinterização piloto para determinar o efeito dos diferentes *pellet feed* na produtividade e propriedades físicas (Shatter e tamboramento) e metalúrgicas do sínter (RI, RDI e mineralogia). Os resultados obtidos mostraram que o aumento da superfície específica do *pellet feed* melhora, de uma forma geral, o comportamento de aglomeração (melhora do índice de granulação, melhora da formação das quase-partículas e uma maior resistência das mesmas), levando a um aumento da permeabilidade do processo e permitindo a recuperação da produtividade na sinterização piloto sem comprometer a qualidade física e metalúrgica do sínter produzido. A superfície específica mínima requerida para o *pellet feed* testado ficou entre 1.400-1.500 cm²/g. A segunda parte do trabalho foi dedicada à estudos de

granulação visando um melhor entendimento dos mecanismos e fenomenologia envolvidos durante essa etapa do processo. Neste caso, foi estudado o *pellet feed* de maior e menor superfície específica, as suas frações menores do que 0,045 mm e a combinação destas frações com estes mesmos *pellet feed*. Observou-se aumento do Índice de Granulação (GI) com a média da superfície específica dos *pellet feed* (sem prensar, prensado e das misturas destes *pellet feed* com suas frações menores que 0,045 mm em diferentes proporções) com estabilização em um nível de superfície específica entre 1.400-1.500 cm²/g. Comportamento similar foi observado para a resistência das quase-partículas, medida através da quantidade de finos menor que 0,15 mm que permaneceu aglomerado às quase-partículas. Por outro lado, através das análises de microscopia óptica, observou-se um aumento contínuo do tamanho médio das quase-partículas e da espessura da camada aderente com a média da superfície específica do *pellet feed*. As quase-partículas produzidas a partir do *pellet feed* sem tratamento mecânico, apresentaram camada aderente heterogênea e com vazios, um menor GI e uma baixa resistência das quase-partículas. Já as quase-partículas produzidas pelo *pellet feed* de maior superfície específica apresentaram uma melhor formação (camada aderente homogênea e ausência/menor quantidade de vazios), o que contribuiu para um valor razoável de GI com uma resistência razoável das quase-partículas. De modo geral, o tratamento mecânico do *pellet feed* promove um melhor comportamento de granulação da mistura de minérios o que afeta positivamente o processo de sinterização, sem comprometer a qualidade física e metalúrgica do sinter produzido. Por fim, um maior tamanho médio das quase-partículas e uma maior espessura da camada aderente não significa que haverá um melhor comportamento de aglomeração da mistura.

ABSTRACT

Recently, with the increase of the demand for iron ore and the depletion of high grade ore deposits, the main mining companies around the world have been investing to increase the use of low grade iron rocks, known as Itabirites. In Brazil, these rocks passed through a treatment process by classification, comminution and subsequent concentration. The product obtained is the pellet feed, a concentrate of high iron content, low level of contaminants, but very fine, typically 95% of the particles are smaller than 0.15 mm. Normally this material is used as raw material for the pelletizing process. However, due to its chemical quality it could also be used in sintering process. On the other hand, its small particle size negatively affects the permeability of this process, restricting its use to low quantities. In this way, different technological alternatives have been developed to allow the use of these materials in sintering, such as the use of intensive mixers, pre-agglomeration, use of special additives, mechanical treatment through roller press, among others. The mechanical treatment by roller press has been widely used to increase the specific surface of pellet feed used in the pelletizing process. Thus, this work aims to study the use of a natural pellet feed (as obtained after the concentration process in the mine) and mechanically treated in roller press with different specific surface levels as an alternative of raw material for sintering. On the first step of the work, 25% of traditional market sinter feed were replaced by natural and mechanically treated pellet feed. The iron ore mixtures were subjected to granulation tests for quasi-particles evaluation and pilot sintering to determine the effect of different pellet feed on productivity and metallurgical properties of the sinter. The results obtained showed that the increase of the specific surface of the pellet feed generally improves the agglomeration behavior (improvement of granulation index, improvement of quasi-particle formation and greater resistance), leading to better permeability allowing the recovery of productivity in pilot sintering without compromising the physical and metallurgical quality of the sinter produced. The minimum specific surface required for the pellet feed tested was between 1,400-1,500 cm²/g. The second part of the work was dedicated to granulation studies aiming to better understanding the mechanisms and phenomenology involved during this process step. In this case, the highest and the smallest specific surface pellet feed, their fractions smaller than 0.045 mm and the combination of these fractions with the same pellet feed were studied. Granulation Index (GI) results increased with the mean specific surface of the pellet feed and showed a stabilization at around 1,400-1,500 cm²/g. Similar trend was observed for quasi-particle strength, measured by the amount of fines of less

than 0.15 mm that remained agglomerated to the quasi-particles. On the other hand, the optical microscopy analysis showed a continuous increase in the mean size of the quasi-particles and the thickness of the adherent layer with the average specific surface of pellet feed. The quasi-particles produced from the untreated pellet feed had a heterogeneous and poor consolidated adherent layer (some voids on it), leading to a lower GI and a low quasi-particle strength. On the the contrary, the quasi-particles produced by the highest specific surface pellet feed presented a better formation (homogeneous adherent layer and absence/smaller amount of voids), which contributed to a reasonable GI value with a good quasi-particle strength. In general, the mechanical treatment of the pellet feed promotes better granulation behavior of the ore mixture which positively affects the sintering process without compromising the physical and metallurgical quality of the produced sinter. Finally, a larger average quasi-particle size and a thicker adherent layer does not mean that there will be better agglomeration behavior of the mixture.

Capítulo 1. Considerações iniciais

1.1. Introdução

O minério de ferro, na forma de *sinter feed*, é a principal matéria-prima utilizada na sinterização. Trata-se de um processo de aglomeração a elevadas temperaturas onde o *sinter feed* juntamente com os fundentes, combustível sólido, outros materiais ferrosos e aditivos são misturados e carregados na máquina de sinterização. O sinter produto é utilizado como matéria-prima para produção do ferro-gusa nos altos-fornos, que posteriormente será transformado em aço, material versátil e que se reinventa a cada dia para estar cada vez mais inserido no cotidiano do homem moderno. Dados atuais da *World Steel Assossiation* (WSA) indicam que essa rota tecnológica é responsável por mais de 70 % da produção global de aço.

No início deste século houve um aumento considerável da produção de aço no mundo, alavancado pela China. De acordo com a WSA, a produção de aço no mundo saltou de 850 Mt (em 2000) para 1.808 Mt (em 2018). Destaca-se neste período momentos de taxas de crescimento da ordem de 7 a 9 % ao ano. Recentemente, esse crescimento limitou-se a valores inferiores a 3% ao ano, com uma estabilização nos últimos 3 anos.

Neste contexto, o mercado transoceânico de minério de ferro foi fortemente afetado e reformulado. As transações a vista, mercado *spot*, ganharam força, sendo que a cotação desta matéria-prima passou a ser diária utilizando como referência portos chineses. A forte demanda chinesa levou ao aumento considerável no preço do minério de ferro. A combinação destes fatores culminou com um grande aumento na produção de minério de ferro no mundo, especialmente na Austrália, levando este país ao posto de maior exportador dessa *commodity*, superando o Brasil. Como consequência, viabilizou-se a produção de minérios australianos de pior qualidade, com maiores valores de LOI (*Loss On Ignition*), menores teores de ferro e aumento dos contaminantes SiO₂, Al₂O₃ e P, devido à exaustão dos minérios de maior teor. Dessa forma, houve um aumento do volume de escória na produção de ferro-gusa levando ao aumento do consumo de combustível no alto-forno e a maiores emissões de CO₂.

Assim como na Austrália, no Brasil também foram realizados investimentos ao longo dos últimos anos em rotas de beneficiamento e concentração com o objetivo de aproveitar os itabiritos disponíveis, rocha que se caracteriza pelo seu baixo teor de ferro. Como produto obtém-se um concentrado de elevadíssimo teor de ferro, baixíssimo LOI e nível de contaminantes, SiO₂, Al₂O₃ e P, porém muito fino conhecido como *pellet feed*. Devido as suas

características químicas, este material é extremamente atrativo como corretivo químico nas sinterizações, porém, o seu uso é limitado devido a sua menor granulometria, o que piora a permeabilidade da mistura de minérios nos processos convencionais de sinterização. Contudo, a disponibilidade para utilização deste tipo de material como corretivo químico para a sinterização será cada vez maior, seja ele incorporado ao *sinter feed* ou usado diretamente neste processo.

Sendo assim, houve a necessidade da adaptação dos processos convencionais de preparação das misturas para sinterização motivadas pela nova realidade de qualidade granulométrica do minério de ferro. Alguns exemplos das recentes tecnologias aplicadas citados são a do aumento do comprimento dos tambores de granulação e mistura, utilização de tambor individual dedicado a granulação, granulação seletiva (*coating* de calcário e *coke breeze*), otimização do sistema de carregamento para uma melhor permeabilidade e segregação (*Segregation Slit Wires – SSW*, *Intensified Sifting Feeder – ISF*, etc.), MEBIOS (*Mosaic Embedding Iron Ore Sintering*) e HPS (*Hybrid Pelletizing Sintering*) para utilização de materiais mais finos na mistura, misturadores intensivos combinados com aditivos especiais, dentre outras. Além dessas alternativas, o tratamento mecânico de *pellet feed* e/ou concentrados através de prensa de rolos (utilizado no processo de pelotização para aumento da superfície específica devido ao seu baixo custo operacional e de investimento, baixo consumo energético e simplicidade de processo) também vem sendo estudado para aplicação na sinterização, porém sem considerar a possibilidade da substituição a *sinter feed* que são bem mais grossos.

Neste contexto, o presente trabalho avaliou o uso de *pellet feed* com diferentes superfícies específicas (sem tratamento e tratado mecanicamente em prensa de rolos) quanto ao seu comportamento de aglomeração em rotas tradicionais de sinterização em substituição a *sinter feed* convencionais, que são mais grossos, regularmente encontrados no mercado. A partir do conhecimento detalhado a respeito do comportamento desses diferentes *pellet feed*, será possível estruturar alternativas de processo e rotas tecnológicas de preparação de misturas para possibilitar a utilização de participações maiores desta matéria-prima na sinterização que poderá trazer benefícios visto o cenário atual de deterioração da qualidade e disponibilidade de *sinter feed* e de baixo investimento deste tipo de equipamento.

1.2.Objetivos

O objetivo geral do presente trabalho foi avaliar qualitativamente o comportamento de granulação (aglomeração a frio) de *pellet feed* hematítico de diferentes superfícies específicas, com e sem tratamento em prensa de rolos, considerando-se a rota convencional de preparação de misturas das matérias-primas para sinterização. Para atingir o objetivo geral proposto, o presente estudo foi desenvolvido de acordo com os seguintes objetivos específicos:

- Determinação do impacto da adição de *pellet feed* de diferentes superfícies específicas, em substituição parcial de *sinter feed* tradicionais disponíveis no mercado, nos diferentes parâmetros de processo da sinterização piloto e sobre a qualidade do sinter produzido.
- Avaliar o comportamento de granulação das misturas a serem sinterizadas com *pellet feed* de diferentes superfícies específicas.
- Compreender comportamento de granulação das misturas de *pellet feed* de diferentes superfícies em diferentes proporções com as suas frações menores do que 0,045 mm;
- Estudar como as características microestruturais, dos *pellet feed* de diferentes superfícies específicas e de suas frações menores que 0,045 mm, se relacionam com o comportamento de granulação destes materiais.
- Por fim, obter uma maior compreensão dos mecanismos e fenômenos envolvidos na etapa de aglomeração a frio quando se utiliza *pellet feed* de diferentes superfícies específicas.

1.3. Estrutura da Tese e Descrição dos Artigos

A redação da presente tese fundamentou-se em dois artigos internacionais, um já publicado (Artigo A - Capítulo 2) e outro sob revisão (Artigo B – Capítulo 3). Além do presente capítulo que introduz o leitor ao tema e aos objetivos do estudo, a tese também apresenta o capítulo de considerações finais (Capítulo 4) onde se fez a conexão entre os dois artigos supracitados. O capítulo seguinte (Capítulo 5) é dedicado às principais contribuições científicas do trabalho e encerrado com um capítulo dedicado às sugestões para possíveis investigações futuras envolvendo o tema são descritos de maneira sucinta, Capítulo 6. A seguir são citados os artigos que são corpo principal do presente trabalho:

- **Artigo A - Alternative to deal with high level of fine materials in iron ore sintering process:** Nessa primeira etapa do trabalho procurou-se estabelecer a condição ideal de superfície específica de um *pellet feed*, através do tratamento de prensagem, para viabilizar a substituição de *sinter feed* convencionais de mercado mantendo-se a produtividade da sinterização e as características físicas e metalúrgicas do sinter produto;
- **Artigo B - Study of the granulation behavior of an iron ore sintering mixture containing high grade pellet feed with different specific surface:** Neste trabalho, procurou-se entender o comportamento de granulação dos *pellet feed* com e sem o tratamento em prensa de rolos, de sua frações menores do que 0,045 mm, bem como da misturas destas frações com estes *pellet feed*.

Capítulo 2. Artigo A - Alternative to deal with high level of fine materials in iron ore sintering process

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Paper submetido e publicado pela revista Journal of Materials Research and Technology (JMRT) <https://doi.org/10.1016/j.jmrt.2019.07.032>

ABSTRACT

Nowadays, as the demand for iron ore increases together with the depletion of high grade ore deposits, mining companies have been investing to produce iron ore concentrates, such as pellet feeds (95% lower than 0.15 mm) with low level of contaminants. It is well-known that its lower particle size negatively affects the permeability of the sintering process, restricting its use to small quantities. In this way, this work was focused on the use of this fine material in sintering process by replacing regular sinter feed. The pellet feed was prepared in roller press aiming different levels of specific surface. The iron ore mixtures were evaluated in a regular preparation route composed by two drums, one for mixing and another for granulating. To carry out this study, 25% of pellet feed was added to the mix replacing sinter feed. The mixtures were tested in pilot sintering pot test under process conditions close to the industrial practice. The results obtained in pot test showed that the previous mechanical treatment of pellet feed is suitable to enable the use of this fine material in sintering process. It was possible to obtain an optimum performance in the granulation step, promoting good process permeability conditions without causing any significant metallurgical or strength demerit in sinter product. The productivity increased from 25.8 t/day/m² to 29.4 t/day/m² by adding raw pellet feed and treated

by roller press, respectively. Additionally, solid fuel decreased from 69.3 Kg/t to 65.9 Kg/t, respectively.

Keywords: iron ore, pellet feed, roller press, specific surface, sintering pot test.

2.1. Introduction

The iron ore sintering process consists in high temperature agglomeration method where mainly sinter feeds together with fluxes, solid fuel, other ferrous materials and additives are mixed and charged into the sintering machine. The sinter product is the main raw material for hot metal production, which will later be transformed into steel. According World Steel Association (WSA), this technological route accounts for more than 70% of global steel production and its world production increased from 850 Mt (in 2000) to 1,628Mt (in 2016). In this scenario, the massive use of iron ore leads to the depletion of high grade sinter feed and make viable the exploitation of lower grade iron-containing rocks, leading to the production of finer, lower contaminants and high grade iron ore concentrates, known as pellet feed. This material is extremely attractive to be used as a chemical corrective in sintering due to its good chemical quality, however, its use is limited due to its small particle size.

To use pellet feed in sintering process it is necessary to adjust the conventional raw materials preparation route, normally mixing and granulating steps, which requires different process conditions, and also adjustments in additives consumption. Some examples of recent applied technologies were cited in literature, such as [1,2]: (i) selective granulation, allowing to use lower grade sinter feed; (ii) MEBIOS (Mosaic Embedding Iron Ore Sintering) and HPS (Hybrid Pelletizing Sintering), for use of finer materials in the mix; and (iii) intensive mixers combined with special additives. Additionally, the mechanical treatment of pellet feed or concentrates by roller press to increase the specific surface area has been studied to prepare pellet feed for agglomeration processes [3-5]. Other alternatives raised were: (i) the production of an artificial sinter feed with similar characteristics of natural ones, by using additives which allow to produce high strength agglomerates [6]; and (ii) the implementation of a previous briquetting process to enlarge the size of iron ore fines particles, before its use in sintering [7]. Both alternatives show good results due to the decrease in the amount of fine materials, below 0.15 mm, in the iron ore mixture. Recently, the combination of intensive mixer with different level of binder were investigated by different authors as an alternative to use pellet feed in sintering process. One of these authors achieved similar level of productivity of reference case without any pellet feed [8]. Another one reported an improvement in productivity in cases when the concentrates were grinded (mean size of 0.01 mm), mixed with intensive mixer and when a dedicated granulation process were applied [9].

In this context, the present work aims to evaluate the pretreatment of pellet feed in roller press as an alternative to replace regular sinter feeds in iron sintering process. The amount of pellet feed tested was fixed with different levels of specific surface, achieved by passing it several times in roller press. The focus was on the evaluation of this alternative in the regular preparation route, which consists of one drum for mixing and another drum, in sequence, for granulation. Sintering pot test was carried out with different mixtures to verify the effect of these replacement on the sintering process and in sinter product chemistry, physical and metallurgical quality. Additionally, to better understanding of the mechanism involved during the use of pellet feed, granulation test and quasi-particles evaluation by drop test and optical microscopy were carried out.

2.2. Methodology

2.2.1. Pellet feed

A Brazilian pellet feed was previously prepared in a laboratory roller press (LABWAL model, manufactured by Polysius AG). The pellet feed passed through roller press once and 5 times. The pressing parameters were the same for both situations, i.e. 80 bar of pressure and 8% of moisture.

The specific surface of natural and pressed pellet feed was determined using different laboratories techniques, one based on air permeability test (Blaine index) [10] and another one based on nitrogen adsorption method (B.E.T.) [11-13], using Quantachrome equipment (NOVA 1000e model). Through determination of hysteresis of adsorption-desorption isotherms, information regarding the distribution of the pores was obtained.

Qualitative image analyses of the samples, aiming to investigate the shape and roughness of the particles, were performed by means of Scanning Electron Microscopy (SEM), using a Carl Zeiss® microscope, EVO MA15 model. The mineralogical composition of the pellet feed was determined by optical microscopy, using a Carl Zeiss microscope.

2.2.2. Sinter feeds and sintering mixtures

Sinter feeds from Australia and Brazil were used in this work. Table 2.1 shows the iron ore mixtures tested. The reference case was formed by a mixture of sinter feeds from Australia and Brazil. The pellet feed of Cases I, II and III was introduced at a fixed ratio of 25%, with different specific surface and different size distribution. It mainly replaces the Australian regular sinter

feed, Ore A, which has a high level of coarse particles. The mineralogical composition of the sinter feeds was determined by optical microscopy.

Table 2.1 - Iron ore mixtures tested.

Parameter	Reference Case	Case I (25% PF A)	Case II (25% PF B)	Case III (25% PF C)
Australian sinter feeds (ores A and B), %	69	40	40	40
Brazilian sinter feeds (ores C, D and E), %	31	35	35	35
Pellet Feed A (PF A), %	0	25	0	0
Pellet Feed B (PF B), %	0	0	25	0
Pellet Feed C (PF C), %	0	0	0	25

2.2.3. Sinter pot test

To evaluate the sintering behavior of the materials, sintering pot tests were conducted. In these tests, the cold agglomeration route was composed by two drums in series, one for mixing and another one for granulating.

Sintering pot test evaluation was based on the French simulation technique, which consists in balancing return fines condition, where coke breeze was added until the amount of return fines achieves the aimed value [14-16]. After the definition and establishment of moisture and fuel for each condition, the valid sintering pot tests were performed for a minimum of three times. Process parameters, as solid fuel consumption and productivity, were determined. Table 2.2 shows the experimental conditions established for sinter pot test evaluation and details of the conditions employed for the preparation route tested.

To collect samples, the sinter cake was disintegrated using an ASTM drum device with 50 revolutions. After that, the sinter was screened in different sizes from 5 mm to 80 mm and size distribution of the sinter was determined. This procedure was repeated for each valid burn and each size fractions is separated and later used to samples preparation for characterization. To

obtain representative samples for all characterizations, the required weight is obtained through quartering the material in an automatic equipment.

Table 2.2 - Sintering pot test parameters and granulation conditions.

Parameters	Experimental conditions
Bed height, mm	550
Return fines, %	30
Suction pressure, mmH ₂ O	1,500
Burnt lime level, %	3
Mixing step (dry) time, s	120
Balling step (wet) time, s	240
Drum rotation, rpm	18

Beyond chemical analysis, the quality of sinter obtained from each mixture was evaluated considering physical properties, size distribution and mechanical strength (tumbler ISO 3271 and shatter index JIS M 8711), and metallurgical performance (Reduction Degradation Index, RDI – ISO 4696-2 and Reduction Degree, RI – ISO 7215). Additionally, the mineralogical composition and porosity were determined by optical microscopy and mercury intrusion porosimetry, respectively.

2.2.4. *Quasi-particles evaluation and granulation test*

For a better understanding of the phenomena involved during the granulation step, quasi-particles evaluation and granulation test were carried out. A quasi-particle is formed by a nucleus containing a coarse particle surrounded by fine particles [17].

The sample of quasi-particles were collected just before the loading of the sinter pot test and evaluated by optical microscopy, using a Carl Zeiss® microscope, Axio Imager Z2m model.

The granulation and quasi-particles drop tests were carried out using the same mixtures, applying a small drum. The setup conditions for mixing and granulating are reported in Table 2. After mixture preparation, samples were collected, dried out at 120°C and split for the two tests.

Concerning the granulation test, the sample passed through a dry sieving process and the amount of fines below 0.25 mm was determined. Then, the sample passed through wet sieving process and quasi-particles, which grew up and formed micropellets, were disaggregated, and

finally the amount of fines below 0.25 mm was measured again. The granulation index was calculated and represents the amount of fines that remains forming the quasi-particles and micropellets. The higher this parameter, the better the granulation and permeability in sintering process.

The quasi-particles drop test was carried out with a procedure based on the standard JIS M 8711 used for Shatter Index determination of sinter product. In this test, the amount of fines below 0.15 mm was measured before and after 2 drops. These results were compared with the amount of fines below 0.15 mm of the iron ore mixture before drum mixing. Finally, an estimative of the amount of fines that is still joined to the nucleus particles was determined.

2.3. Results and Discussion

2.3.1. Raw materials characterization

Table 2.3 shows the chemical analysis of the iron ores, whereas Table 2.4 shows their mineralogical composition. The sinter feeds A and B, from Australia, have the highest Loss On Ignition (LOI) due to their mineralogical composition, mostly formed by goethite. On the other hand, sinter feeds C, D and E, from Brazil, have lower LOI and are mainly composed by hematite. In terms of level of contaminants, i.e. Al_2O_3 and P, the Brazilian sinter feeds have the lowest value, while SiO_2 content, except for sinter feed C, have the highest values. The pellet feeds, PF A, B and C, have the lowest amount of contaminants and LOI, as they are mostly composed by hematite.

Concerning hematite crystals morphology (Table 2.5) ore B presents a mixture of martite and lobular hematite. Ore C were mostly formed by microcrystalline hematite. Ores D and E present a mixture of granular and specular hematite and finally, pellet feeds were mostly formed by specular hematite.

Table 2.3 - Chemical characterization of the iron ores (%).

Iron Ores	Fe	SiO₂	Al₂O₃	P	LOI
Ore A	57.4	5.41	1.64	0.032	10.5
Ore B	61.0	4.24	2.61	0.089	5.5
Ore C	64.8	2.61	1.59	0.027	1.8
Ore D	62.5	6.81	0.93	0.057	2.3
Ore E	60.0	10.85	0.98	0.036	1.5
PF A, B and C	68.0	1.93	0.33	0.016	0.6

LOI: Loss On Ignition

Table 2.4 - Mineralogical composition of iron ores (wt. %).

Iron ore	Hematite	Goethite	Magnetite	Quartz	Other minerals
Ore A	3	86	0	7	4
Ore B	46	45	1	6	3
Ore C	85	9	1	4	0
Ore D	76	13	3	9	0
Ore E	83	6	1	10	0
PF A, B and C	91	5	2	2	0

Table 2.5 - Hematite morphology of iron ores (Vol. %).

Iron ore	Microcrystalline	Martite	Lobular	Granular	Specular
Ore A	0	1	2	1	0
Ore B	0	31	20	0	0
Ore C	47	5	20	13	5
Ore D	0	17	0	27	39
Ore E	0	7	0	33	51
PF A, B and C	0	7	1	15	69

The fluxes and solid fuel, used in sinter pot tests, have similar characteristics of that one used in industrial scale sintering plant in steel mills. Table 2.6 and Table 2.7, show the chemical characterization and immediate chemical analysis of the fluxes and solid fuel, respectively.

Table 2.6 - Chemical characterization of the fluxes (wt. %).

Materials	Fe	SiO ₂	Al ₂ O ₃	CaO	MgO	LOI	others
Flux 1	0.43	3.78	0.73	51.85	0.66	41.42	1.13
Flux 2	5.56	50.80	2.00	2.60	31.10	5.30	2.64
Flux 3	0.65	2.95	0.33	92.14	0.38	3.93	0.00

Table 2.7 - Immediate chemical analysis of solid fuel (wt. %).

Material	Fixed Carbon	Volatile Matter	Ashes	S
Coke breeze	84.98	3.06	11.96	0.58

Table 2.8 shows the results of specific surface obtained for each pellet feed, both techniques, Blaine and B.E.T., presented the same trends although with different absolute values. The increase on the number of times passing in roller press leads to a higher specific surface area. The differences in absolute values is that the B.E.T. measures the total specific surface area, i.e. including the surface area relative to pores.

Table 2.8 - Specific surface of pellet feeds.

Pellet Feed	Number of times in roller press	Blaine Index (cm ² /g)	B.E.T. (m ² /g)
PF A	0	433	0.80
PF B	1	854	1.00
PF C	5	1,468	1.70

Isotherms of adsorption-desorption were presented in Figure 2.1 (a). The characteristics of this curves show similar results of the ones reported in literature for iron ores [13,18]. The amount of nitrogen adsorbed increases with the increase of specific surface of pellet feed. The

shape of the isotherms indicates a small distribution of size of pores for all considered pellet feed (Figure 2.1, b).

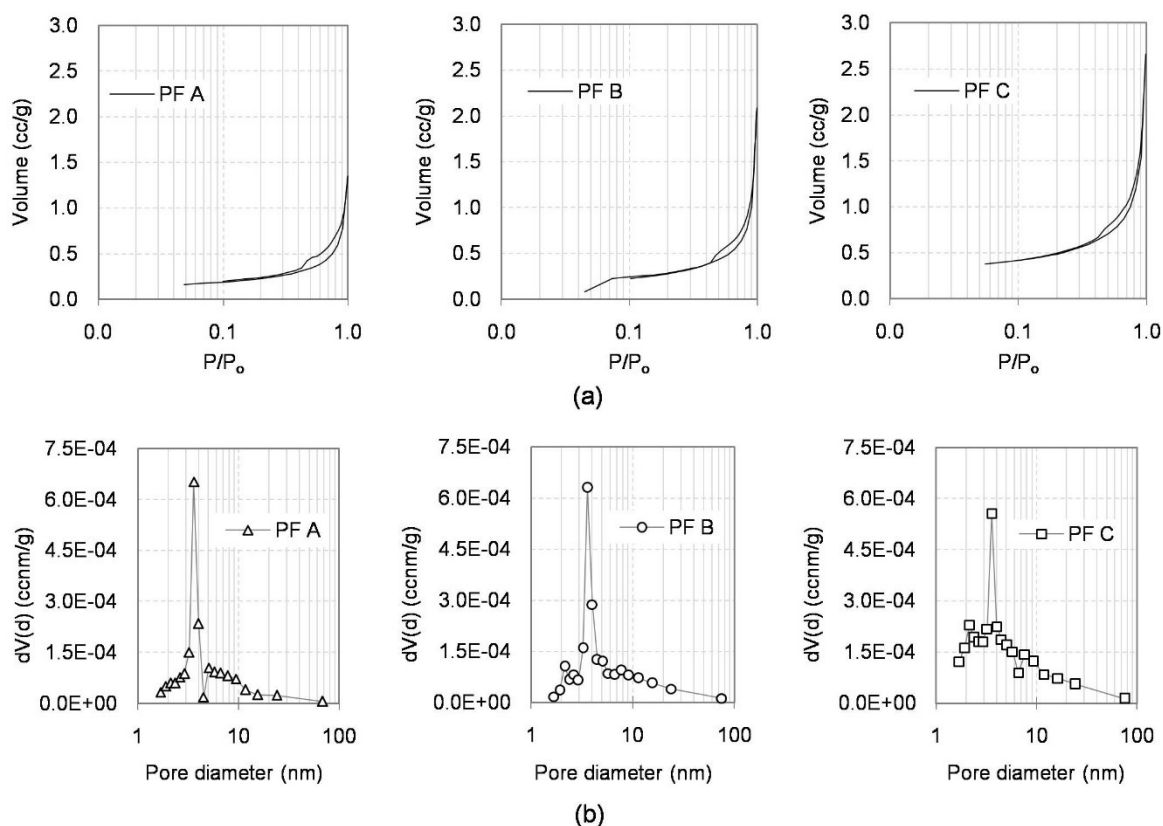


Figure 2.1 - (a) Isotherms of adsorption-desorption with N₂ at 77K of the pellet feeds A, B and C; (b) pore size distribution of the pellet feeds.

Figure 2.2 shows the size distribution of sinter feeds, natural pellet feed (PF A) and pressed pellet feeds (PF B and C). As expected, the pellet feeds, in comparison to sinter feeds, are much finer. Comparing the pellet feeds, the increase in the number of times passing in roller press leads to a production of a much finer material. SEM images were collected and shown in Figure 2.3. Note that the higher the specific surface is, the higher the amount of ultra-fines particles produced.

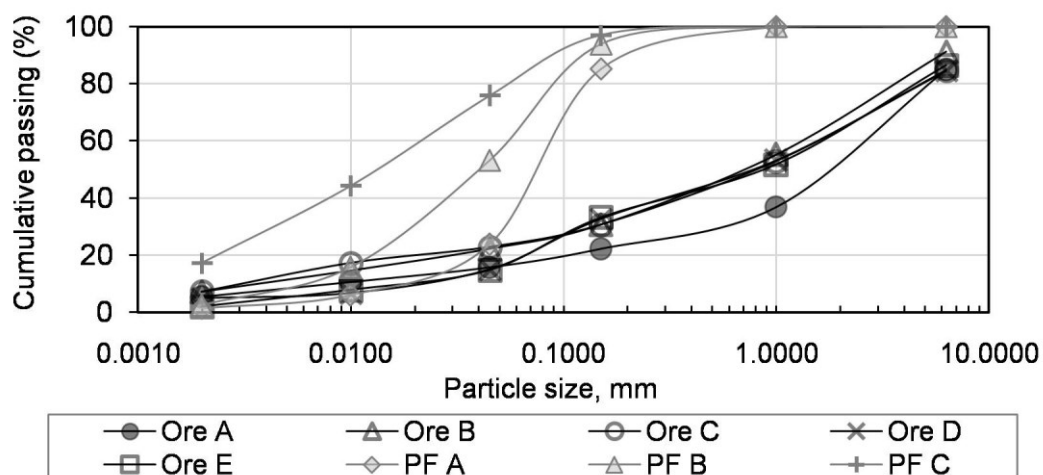


Figure 2.2 - Size distribution of the iron ores used in the present work (sinter feeds from Australia: Ores A and B; sinter feeds from Brasil: Ores C to E; natural pellet feed: PF A; pellet feed A treated once in roller press: PF B; pellet feed A treated five times in roller press: PF C).

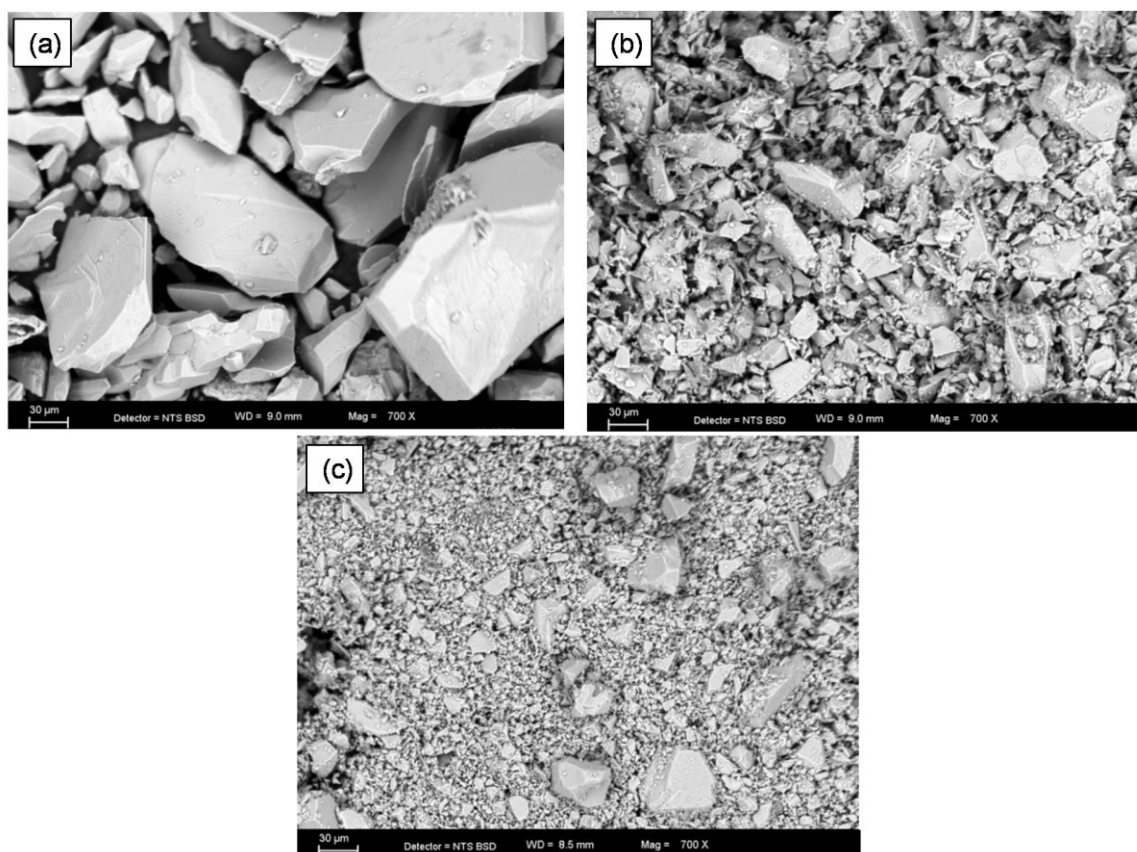


Figure 2.3 - SEM images:(a) PF A, natural pellet feed; (b) PF B, pressed pellet feed once in roller press; and (c) PF C, pellet feed pressed five times in roller press.

2.3.2. Sinter pot test results

Table 2.9 shows more details of the iron ore mixture tested. The use of pellet feed allowed an improvement in the quality of the mixture and as consequence in sinter product, the Fe content increased with the decrease of SiO₂, Al₂O₃, P and LOI. On the other hand, the iron ore mixture became much finer.

Table 2.9 - Details of iron ore mixtures tested.

Parameter	Reference	Case I (PF A)	Case II (PF B)	Case III (PF C)
Iron ore mixture				
Sinter feeds	100	75	75	75
Pellet feed	0	25	25	25
LOI, %	7.4	4.9	4.9	4.9
+1.000 mm, %	53.1	39.3	39.3	39.3
-0.150 mm, %	24.9	40.7	42.9	43.7
-0.045 mm, %	17.0	18.9	26.3	31.9
Sinter product				
Fe, %	56.8	57.5	57.5	57.5
SiO ₂ , %	5.97	5.73	5.73	5.73
Al ₂ O ₃ , %	1.72	1.41	1.41	1.41
P, ppm	50	44	44	44
CaO/SiO ₂	1.6	1.6	1.6	1.6

The results of productivity and fuel consumption obtained in sinter pot tests for each case are reported in Figure 2.4. These results showed that when natural pellet feed is introduced in the iron ore mixture, a decrease in productivity was observed. This result could be explained by the increase of the fine particles in iron ore mixture affecting the permeability of the process and was in line with the one reported in literature [7,8,19]. Partial recovery of productivity was achieved when pressed pellet feed with intermediate specific surface was used. Fully recovery of productivity was achieved only with the highest level of specific surface of the pellet feed. So, in these cases, even with the increase of the amount of fines (pellet feed replacing coarse sinter feed) better productivity was achieved contradicting the literature reported. Similar behavior was reported by Jian *et al.* [5] only when high specific surface pellet feed replaces iron ore concentrates.

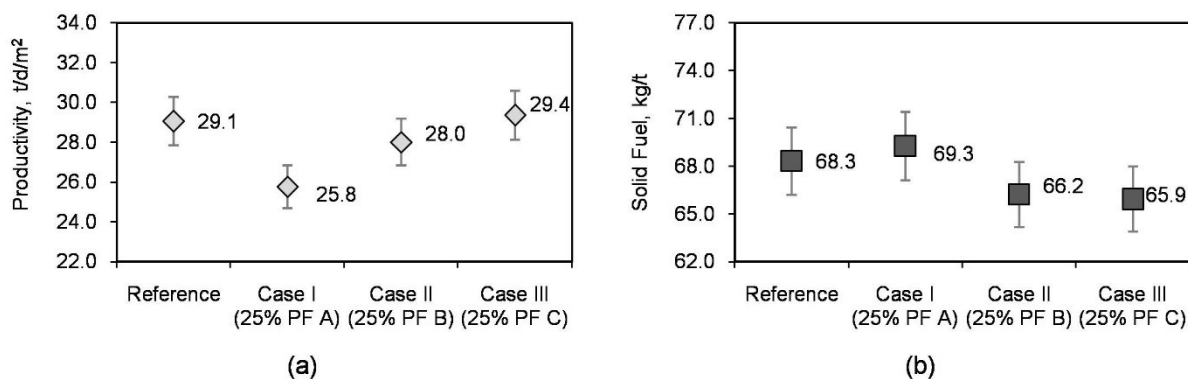


Figure 2.4. Effect of the use of pellet feed replacing regular sinter feeds in sintering productivity (a) and solid fuel consumption (b), relative to base case for regular granulation process.

Table 2.10 shows the main parameters which affect productivity in sintering process, i.e. flame front speed, sintering yield and charge density. Additionally, it also shows the moisture of the mixture and a permeability index, JPU (Japanese Permeability Unit). The flame front speed is an indicative of the permeability of the process and should be as high as possible without compromising other sinter properties, for instance strength and mineralogy. If the flame front speed is too fast part of the iron ore mixture could be unburned or if it is too slow excessive melting formation may happen.

Table 2.10. Parameters which affects sintering productivity.

Parameter	Reference	Case I (PF A)	Case II (PF B)	Case III (PF C)
Flame front speed, mm/min	22.5	18.1	19.4	20.4
Charge density dry, t/m ³	1.62	1.72	1.75	1.72
Sintering yield, %	55.3	57.5	57.3	57.6
Moisture, %	7.4	7.3	7.4	7.5
JPU*	15	7	13	14

* Japanese Permeability Unit

Sintering yield is the ratio of the amount of sinter in the size range able to be used in blast furnace to the amount of iron ore mixture (including return fines, fuel and fluxes). The higher the sintering yield is, the better the productivity is and less fines are generated by the produced sinter. The charge density is also another parameter that directly affects productivity: the higher the charge density, the better the productivity is.

So, when the reference case is compared with the other cases (Table 2.10), where pellet feed was introduced in the iron ore mixture, the parameter that was negatively affected is the flame front speed which had decreased. The permeability index, JPU, also decreased. As previously mentioned, this result was expected, as the size distribution of the iron ore mixture became finer. On the other hand, sintering yield and charge density increased in all cases with the addition of pellet feed, independently of the mechanical treatment. These results, sintering yield and charge density, were in line to those reported in literature [20-24] when high LOI ores (goethite ores) were replaced by hematite ores.

Coming back to the flame front speed results, it increased with the increase of specific surface of pellet feed and also with the amount of particles below 0.045 mm of the iron ore mixture, see Figure 2.5. For Case III, with pressed pellet feed with 1,460 cm²/g of specific surface (Blaine Index), flame front speed reaches a value enough to allow the full recovery of the productivity. Additionally, it is also observed an improvement in the permeability index. So, the improvement in granulation step could be the main hypothesis that better explain this result.

About solid fuel consumption, Figure 2.4 (b), no expressive changes were observed for the Case I with natural pellet feed. Concerning Cases II and III, a decrease on this parameter was achieved. This result was in accordance with the one reported in literature [20-24] when high LOI ores were replaced by hematite ores. The hypothesis raised to explain the behavior of Case I was related to the lower permeability of the process which leads to a higher fuel requirement to achieve suitable sinter strength and was in accordance with the findings of Yang *et. al.*[25].

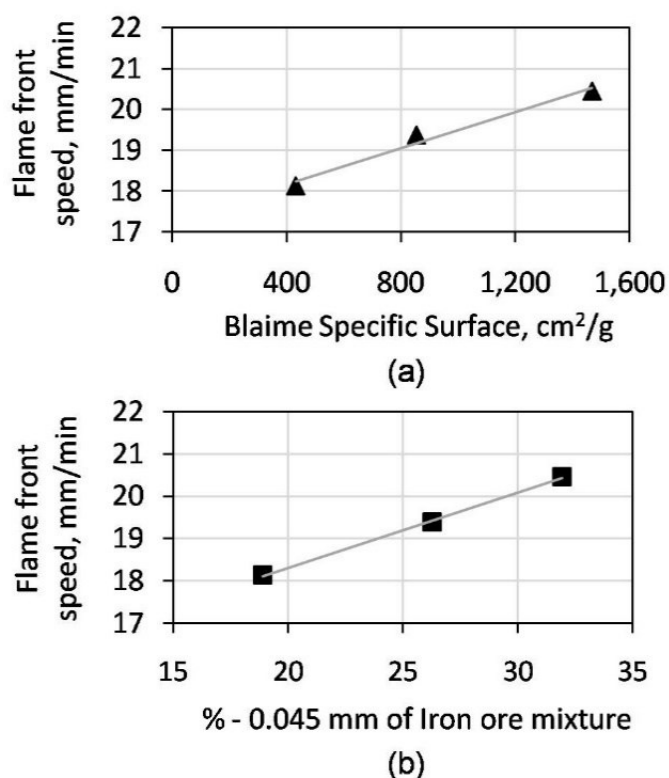


Figure 2.5. Flame front speed for the cases with pellet feed, in iron ore mixture replacing Australian sinter feed's, Cases I, II and III. (a) Relation between pellet feed Blaine Index (specific surface) and flame front speed; and (b) relation between the % of fines below 0.045 mm and flame front speed.

2.3.3. *Quasi-particles and granulation evaluation*

To better understand the sinter pot test performance of the mixtures with natural and pressed pellet feed and to confirm the hypothesis raised about the improvement in granulation step, an investigation of the quasi-particles formed during balling step before the pot test was performed using optical microscopy. Figure 2.6 shows the main differences between Case I and Case III with natural pellet feed and with pressed pellet feed, respectively. The images show a better quasi-particles formation in the case where pressed pellet feed was used (five times in roller press, Blaine index of 1,468 cm²/g), which explains the improvement in permeability observed in this case, leading to a higher flame front speed and better productivity.

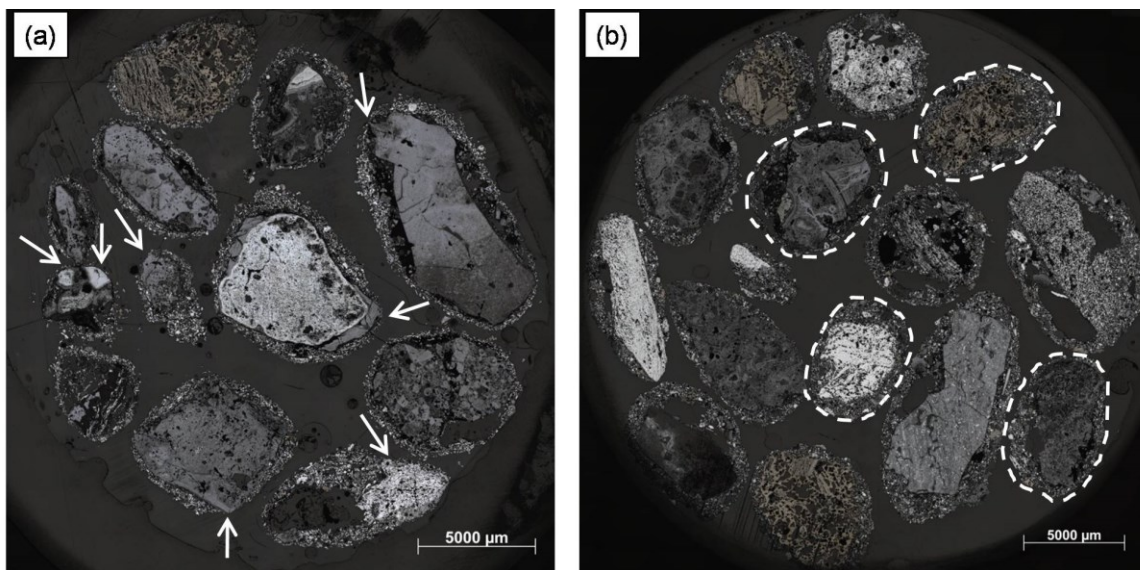


Figure 2.6. Quasi-particles collected after granulating step before sinter pot test for regular granulation process (two drums in sequence, one for mixing and another for balling): (a) PF A (Blaine index: $433 \text{ cm}^2/\text{g}$) with not well formed quasi-particles (white arrows) and (b) PF C (Blaine index: $1,468 \text{ cm}^2/\text{g}$) with well-formed quasi-particles (rounded shape aspect).

Additionally, the drop test carried out with samples collected for these two cases (Figure 2.7) showed that more fines below 0.15 mm remain agglomerated to quasi-particles or micropelletized for Case III as compared to Case I. The quasi-particles formed with the pressed pellet feed (Case III) was stronger than the quasi-particles formed with natural pellet feed (Case I).

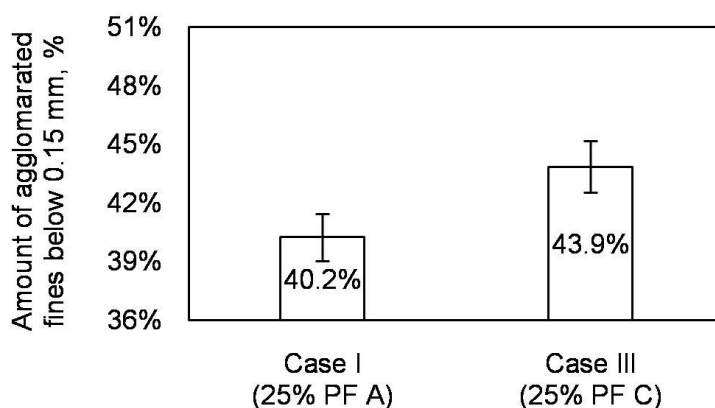


Figure 2.7. Amount of fines below 0.15 mm that remain agglomerated after drop test for the Case I (25% PF A) and Case III (25% PF C), moisture of 7.5% of iron ore mixture.

Finally, the granulation test results were also in line with the observations previously reported confirming the hypothesis about the improvement of the granulation of the Case III in comparison with Case I (Figure 2.8).

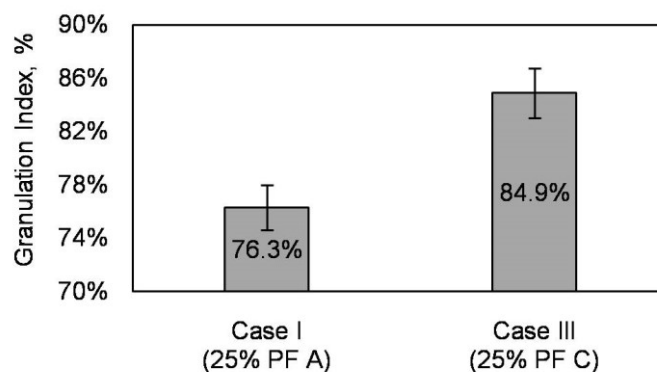


Figure 2.8. Granulation index results for Case I (25% PF A) and Case III (25% PF C), moisture of 7.5 % of iron ore mixture.

2.3.4. Sinter characterization

Figure 2.9 shows the results of sinter strength, i.e. shatter and tumble index. In general, an increase in sinter strength with the introduction of pellet feed (hematite ore) replacing coarse Australian sinter feed (goethite ores) was achieved. The increase of fines particles in iron ore mixtures does not compromise sinter strength. Some reports in literature [23,24] about the replacement of goethite by hematite sinter feeds presents similar results.

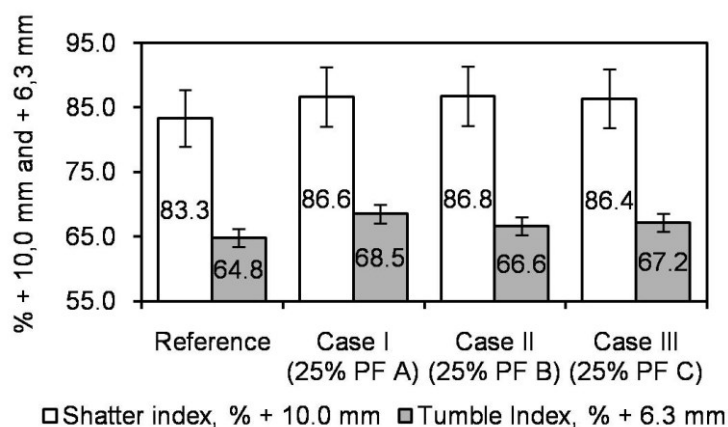
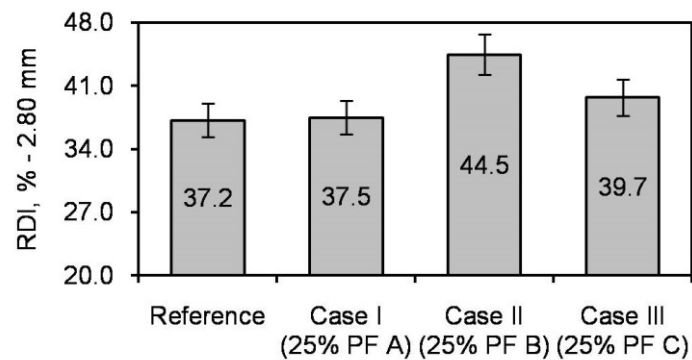
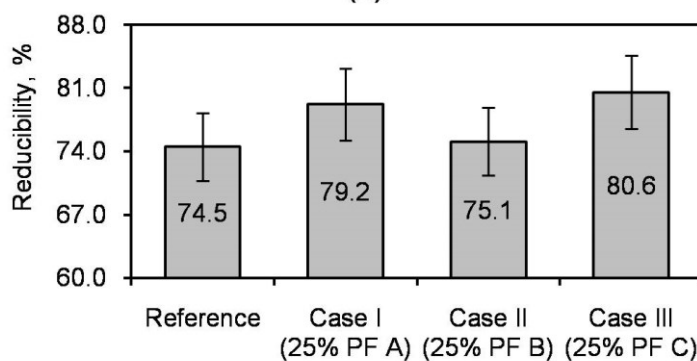


Figure 2.9. Shatter and tumble index of the sinters produced with pellet feed replacing regular sinter feeds.

About metallurgical performance, Figure 2.10 shows the results of RDI and reducibility. Regarding RDI, Cases I and III presents similar values of the reference case. These results were in line with microstructural characterization of these sinters, Table 11. Case II presents the highest level of RDI and could be explained by the higher level of magnetite, lower level of acicular ferrites and higher porosity.



(a)



(b)

Figure 2.10. Metallurgical quality of the sinters produced with pellet feed replacing regular sinter feeds: (a) RDI, % - 2.8 mm, and (b) reducibility, %.

Table 2.11. Mineralogical composition and mercury intrusion porosimetry of the sinters produced in pot test.

Parameter	Reference	Case I (PF A)	Case II (PF B)	Case III (PF C)
Mineralogical composition (wt. %)				
Granular Ferrites	11±1	8±1	2±1	6±1
Acicular Ferrites	22±1	20±1	17±1	20±1
Primary Hematite	4±1	2±1	3±1	5±1
Secondary Hematite	34±1	37±1	37±1	35±1
Magnetite	20±1	21±1	34±1	24±1
Silicate	8±1	10±1	7±1	10±1
Quartz	0±1	2±1	1±1	1±1
Mercury intrusion porosimetry (%)				
Hg Porosity	21.3±0.5	20.9±0.5	23.6±0.5	20.6±0.5

The results of reducibility were slightly improved when the hematite pellet feed replaces goethite sinter feed. This result could be explained by the decrease in alumina content of the iron ore mixture which were concentrated in adherent fines, introduction of pellet feed with very low alumina content, which promote similar segregation obtaining when selective granulation method is applied in sintering process leading to an overall better sinter quality as reported in literature [26-28]. The results were also in line with microstructural characterization, except for Reference Case, which should have higher reducibility.

In general, the use of hematite pellet feed with different levels of specific surface replacing goethite sinter feeds from the market does not compromise the overall quality of the produced sinter.

2.4. Conclusions

The use of pellet feed in sintering process replacing regular sinter feeds of the market was studied through sinter pot tests (25% of iron ore mixture). It was shown that the alternative of pre-treatment of the pellet feed in roller press is interesting to promote the use of it in this process. Based on the present work, the main conclusions were:

- The use of hematite pellet feed in sintering process is a good alternative to improve the chemical composition due to the high iron content and a lower level of contaminants, i.e. SiO_2 , Al_2O_3 and P. Additionally, depending on the ore replaced, better solid fuel consumption could be achieved due its lower LOI.
- The mechanical treatment of pellet feed in roller press leads to an increase in the ultrafine fraction of the regular pellet feed, i.e. amount less than 0.045 mm and less than 0.010 mm, which is helpful for the granulation process. On the other hand, without pretreatment the present pellet feed does not have a positive effect on the granulation step due to its lower ultrafine fraction.
- The use of pellet feed with higher specific surface leads to an increase on the productivity of the sinter pot test. This is due to the better granulation behavior of pressed pellet feed when it is compared to the non-pressed pellet feed, leading to a better permeability (higher flame front speed). Comparing with the reference case, which has no addition of pellet feed, charge density as well as sintering yield (higher sinter strength) also contributed to a better productivity.
- In terms of sinter quality, the sinter strength results showed an increase of tumble and shatter index as consequence of the introduction of pellet feed (hematite ore) replacing Australian sinter feed (goethite ores). Considering metallurgical performance, interesting results were achieved and RDI results were in line with the microstructural characterization of the sinters. Case III, with the highest specific surface (pressed pellet feed five times in roller press), showed better results than Case I, with lowest specific surface (natural pellet feed, not pressed).

Conflicts of interest

The authors declare no conflicts of interest.

Acknowledgments

The authors would like to thank CAPES-PROEX, FAPEMIG and CNPQ, as well as all collaborators involved in the work at Vale S.A. and Federal University of Minas Gerais (UFMG).

References

- [1] Lu L and Ishiyama O. Iron ore sintering. In: Lu L., editor. Iron ore: Mineralogy, Processing and Environmental Sustainability. 1st ed. Elsevier Ltd; 2015, p. 395-433.
- [2] Naito M, Takeda K, and Matsui Y. Ironmaking technology for the last 100 years: Deployment to advanced technologies from introduction of technological know-how, and evolution to next-generation process. *Isij International* 2015; 55(1):7-35.
- [3] Abazapoor A, Halali M, Hejazi R and Saghaein M. HPGR effect on the particle size and shape of iron ore pellet feed using response surface methodology. *Mineral Processing and Extractive Metallurgy* 2018; 127: 40-48.
- [4] Zhu D, Pan J, Lu L and Holmes R J. Iron ore pelletization. In: Lu L., editor. Iron ore: Mineralogy, Processing and Environmental Sustainability. 1st ed. Elsevier Ltd; 2015, p. 435-473.
- [5] Jian P, De-Qing Z, Pimenta H, Xian-Lin Z and Lin W. Improving granulating and sintering performance by pretreating specularite concentrates Through Mechanical Activation. *Isij International* 2013; 53(12):2013-2017.
- [6] Dutra F C, Vieira M B and Pimenta H P. Development of minipellets for use in sintering. In: Associação Brasileira de Metalurgia, Materiais e Mineração – ABM, organizer. Proceedings of the 6th International Congress on the Science and Technology of Ironmaking-ICSTI; 2012; Rio de Janeiro, Brasil. 12p.
- [7] Pan J, Shi B, Zhu D, and Mo Y. Improving sintering performance of specularite concentrates by pre-briquetting process. *Isij International*, 2016; 56(5):777-785.
- [8] Pereira H C. Produção de sinter com pellet screenings e pellet feed com emprego de granulação intensiva [Doctoral thesis]. Belo Horizonte: Federal University of Minas Gerais; 2015.
- [9] L. Hsieh. Effect of Iron Ore Concentrate on Sintering Properties. *Isij International*, 2017; 57(11):1937-1946.
- [10] Arvaniti E C, Jurenger M C G, Bernal S A, Duchesne J, Courad L, Leroy S, et al. Determination of particle size, surface area, and shape of supplementary cementitious materials by different techniques. *Materials and Structures/ Materiaux et Constructions*, 2014; 48(11): 3678-3701.
- [11] Lowell S and Shields J E. Powder Surface Area and Porosity. 2nd ed. Chapman and Hall; 1984.

- [12] Pena E Q, Vieira C B, Silva C A and Silva F G. Aplicação do método de adsorção de nitrogênio para caracterização dos parâmetros de porosidade de pellet feed de minérios de ferro. In: Associação Brasileira de Metalurgia, Materiais e Mineração – ABM, organizer. Proceedings of the VII Simpósio Brasileiro de Minério de Ferro; 2006; São Paulo, Brasil. p. 473 – 482.
- [13] Mao H, Zhang R, LV X, Bai C and Huang X. Effect of surface properties of iron ores on their granulation behaviour. *Isij International*, 2013; 53(9):1491-1496.
- [14] Clary A, Dutra E R, Dutra F C and Junior F L C C. Desenvolvimento integrado de carga metálica para cliente asiático baseado na análise da sua cadeia de valor. In: Associação Brasileira de Metalurgia, Materiais e Mineração – ABM, organizer. Proceedings of the 39º Seminário de Redução de Minério de Ferro e Matérias-primas; 2009; Ouro Preto, Brasil. 2009, 11p.
- [15] Ribeiro M R and Pimenta H P. Desenvolvimento de misturas de sinterização de elevado desempenho com ênfase na escolha do calcário e do combustível sólido. In: Associação Brasileira de Metalurgia, Materiais e Mineração – ABM, organizer. Proceedings of the 40º Seminário de Redução de Minério de Ferro e Matérias-primas; 2010; Belo Horizonte, Brasil. p. 763-773.
- [16] Napoleão A, Pinheiro P, Capporali L, Vieira M B, Harumi L and Oliveira D. CVRD methodology to evaluating iron ore for sintering process. In: Associação Brasileira de Metalurgia, Materiais e Mineração – ABM, organizer. Proceedings of the Ironmaking Conference; 2001; Belo Horizonte; Brasil. p. 913-918.
- [17] Ishikawa Y, Sugawara K and Umezu Y. Latest development of sintering technology. In: K.V.S. Sastry (ed.), *Agglomeration 77*, AIME, New York, 1977, p. 503-525.
- [18] Pena E Q, Vieira C B, Silva C A, Seshadri V and Araujo F G S. Caracterização dos parâmetros de porosidade de concentrados de minérios de ferro pelo método de adsorção de Nitrogênio. *Tecnologia em Metalurgia e Materiais*, 2008; 4(4): 53-57.
- [19] Silva T P and Honorato E P. Avaliação do efeito da adição de pellet feed usando misturamento intensivo em sinterização piloto. In: Associação Brasileira de Metalurgia, Materiais e Mineração – ABM, organizer. Proceedings of the 43º Seminário de Redução de Minério de Ferro e Matérias-primas; Belo Horizonte; Brasil. p. 89-99.
- [20] Oliveira V M, Kumar P and Oliveira D. Influence of high grade iron ore usage on the performance of SEA Blast Furnaces. In: SEASI, organizer. Proceedings of the SEASI Conference & Exhibition; 2017; Singapore. 15p.

- [21] Di Giorgio N, Brace D, Bennett A and Wijekulasuriya K. Reengineering Bluescope steel's port kembla plant for high productivity and reduced steelworks greenhouse emissions. In: Engineers Australia, organizer. Proceedings of the CHEMECA 2011 - Engineering a Better World; 2011; Sidney; Australia. p. 1038-1052.
- [22] Loo C E. A perspective of goethitic ore sintering fundamentals. *Isij International*, 2005, 45(4):436-448.
- [23] Sakamoto N, Noda H, Ichikawa K, Sato H and Kawata H. A commercial production test of iron ore sinter using high amounts of pisolite ores and quality evaluation of the sinter products. *Isij International*, 1997; 37(11):1066-1071.
- [24] Caporali L, Pinheiro P, Ottoni R and Vieira M. B. H. Iron ore microstructure-properties-performance relationship in sintering process. In: ISS, organizer. Proceedings of the 2nd International Congress on the Science and Technology of Ironmaking – 2nd ICSTI; 1998; Canada. p. 123-135.
- [25] Yang C, Zhu D, Shi B, Pan J, Lu L, Li X and Mao Y. Comparison of sintering performance of typical specular hematite ores with distinct size distributions, *J. Iron Steel Res. Int.*, 24 (2017) 1007-1015.
- [26] Lu L, Holmes J R and Manuel J R. Effects of alumina on sintering performance of hematite iron ores. *Isij International*, 2007; 47(3):349-358.
- [27] Jeon J, Kim S, Suh I and Jung S. Assimilation behaviour of quasi-particles comprising alumina pisolitic ore. *Isij International*, 2014; 54(12):2713-2720.
- [28] Haga T, Ohshio A, Nakamura K, Kozono T and Uekawa K. Control technique of the melting reaction in sintering process by the fine part selective granulation of clayish iron ores. *Tetsu-to-Hagané*, 1997; 83(2):103-108.

Capítulo 3. Artigo B - Study of the granulation behavior of an iron ore sintering mixture containing high grade pellet feed with different specific surface

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Paper submetido a Revista *Mineral Processing and Extractive Metallurgy Review* Código de submissão: GMPR-2019-0275

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ABSTRACT

High-grade iron ores became more attractive due to the searching for lower slag rate operation in blast furnaces aiming to reduce CO₂ emissions as the environmental regulation became even more restricted. The granulation behavior of high-grade ores individually and together with other iron ores played an important role for sintering process. In this context, this work aims to evaluate the granulation behavior of a pellet feed with different specific surfaces. To carry out this study, 25% of pellet feed was added to an iron ore mix in a bench scale drum. The Granulation Index (GI) was determined and samples were collected after granulation step for quasi-particles investigation. The results showed that a previous mechanical treatment of the pellet feed by roller press is suitable in order to enable a good granulation behavior of this fine material, which was essential to guarantee its use as raw material in sintering process. The fraction below 0.045 mm of the pressed pellet feed helped to improve the granulation of the natural pellet feed. The thickness of the adherent layer and means size of quasi-particles increased with the specific surface. The GI results increase with the pellet feed specific surface, up to 1,400-1,500 cm²/g stabilizing around 86-90 %. The fines below 0.15 mm that remained agglomerated after drop test, had similar behavior of GI. Finally, it was possible to obtain a

minimum specific surface level (1,400-1,500 cm²/g) to achieve an optimum performance in the granulation step which may promote a good sintering process permeability conditions.

Keywords: iron ore, granulation, pellet feed, roller press, specific surface.

3.1. Introduction

One important step of the iron ore sintering process is the raw materials preparation, which means that these materials need to be mixed and subsequently granulated. Recently, due to the increase of fine portion of the sinter feeds and also the availability of concentrates, such as pellet feeds, this step played an important role to guarantee good permeability conditions for sintering process [1,2].

Granulation is the term used to name the process of increasing particle size to a certain optimal size with spherical shape by using fine and very fine particles. According to Walker [3] the granulation happens when a bed of solid particles moves with simultaneous intensive mixing in the presence of a liquid phase. This movement provides particle collisions and individual particles coalesce. Further granule growth takes place by layering on to this nuclei. The granulation is considered a physical phenomenon, involving physical interactions mainly due to capillary forces.

The iron ore sintering can be divided in two important steps which are according to Lu *et al.* [4] the granulation and the thermal densification, which determine the final quality of the sinter as well the sintering process performance. The characteristics of iron ore affect the sintering process, because it impact on these two steps of the process. Vieira *et al.* [5] mentioned that many studies in the literature have discussed the importance and the relationship between the microstructural characteristics of the ore fines and the efficiency of the cold agglomeration stage in the sintering process and the final quality of the sinter. Kohsa and Manuel [6] highlighted that the granulation is needed to improve the bed permeability in sintering process.

Ishikawa *et al.* [7] and Satoh [8] described an investigation about the granulation process, disclosing the studies about the concept of pseudo-particles or quasi-particles formed by nucleus and adherent fines. They introduced a method to quantify the iron ore mixture behavior, which is the Granulation Index (GI). The authors proposed that particles larger than 1.00 mm act as nuclei, particles less than 0.20 mm act as adhesive powder and particles between 0.20 mm and 1.00 mm work as intermediate particles during the granulation process. Other authors [4, 9, 10] also mentioned similar classification of iron ore particles and its importance to better understanding of the granulation behavior of iron ores. Lu *et al.* [4] showed that for the same type of ore, the increase of the quantity of adhesive powder improves the morphology of the granules and their size distribution becomes narrower, but with a smaller average size. However, this improvement is not translated into an increase in permeability the bed as a whole.

Umadevi *et al.* [11] reported that finer fraction i.e. -0.15 mm increases with the decrease in iron ore mean particle size and a large quantity of finer particle in the sinter mix decreases the granulation efficiency and reduces the Flame Front Speed (FFS). It is a consensus in the literature that the permeability of the ore mixture to be sintered decreases as the amount of fines increases.

Other physical characteristics such as porosity, shape and particle surface characteristics also affect granulation performance. The forces that bind the ultrafines to larger particles are due to the capillarity effect by the presence of water, so the interaction between water and ore particles is very important. Zhu *et al.* [12] described the concentrates and pellet feeds as raw materials for pelletizing process and mentioned the need of the roller press treatment to improve its use. Abzavapor *et al.* [13] mentioned the benefits of using roller press in terms of the shape of the particles to produce pellets. Other authors [14-16] investigated the use of concentrates and pellet feed, which were previously pre-treated in roller press aiming to increase its specific surface, in sintering process showing that the pre-treatment allows to use these materials without losing process permeability. Those findings were recently reinforced by Yang *et al.* [2] who investigated different size distribution of specular hematite ore types and Oliveira *et al.* [17] whom reported that it was also possible to replace coarse sinter feeds without losing productivity.

Finally, the present work aims to evaluate the granulation behavior of pellet feed together with other iron ores. The pellet feed was used as it was produced in mining (from here on called natural), treated in a roller press and prepared from the fraction below 0.045 mm. The iron ore mixtures were evaluated in a small drum which was designed to reproduce the granulation behavior of the mix, with one step for mixing and another for granulation at fixed process conditions (rotation, drum speed, time and loading conditions). For this evaluation, 25% of pellet feed was added to the mix. The GI was determined and samples were collected after granulating step for quasi-particles evaluation by optical microscopy. Additionally, drop test were carried out to determine the strength of the quasi-particles formed.

3.2.Methodology

3.2.1. The pellet feed and iron ore mixtures

A Brazilian pellet feed natural and after mechanical treatment by roller press (LABWAL model, manufactured by Polysius AG) was used in this work. The pellet feed passed through roller

press to achieve a high specific surface. The pressing parameters were 80 bar of pressure and 8 % of moisture. The natural and pressed pellet feed were submitted to a screening process and samples of both materials with size below 0.045 mm were collected. The samples were codenamed as PF A for the natural pellet feed, PF A.1 for the fraction below 0.045 mm of the natural sample, PF B for the pellet feed after roller press, and finally PF B.1 for the fraction for the fraction under 0.045 mm of sample PF B.

The specific surface of the samples was determined using a ZEB PC Blaine equipment (Zunderwerke model) and by nitrogen adsorption method (B.E.T. method), using Quantachrome equipment (NOVA 1000e model). Additionally, the ballability index (K) of the pellet feeds was determined using an apparatus specially manufactured for such analysis and based on the method mainly used and developed in China [15] which is based on water retention capacity and the maximum capillarity forces of the iron ore sample.

Scanning Electron Microscopy (SEM) images of the pellet feed particles were collected using a Carl Zeiss® microscope, EVO MA15 model.

Sinter feeds from Australia and Brazil were used in this work. Table 3.1 shows the iron ore mixture tested. The pellet feed level was fixed in 25% of the total mixture. The natural and pressed pellet feeds (PFA and PFB respectively) and its fraction below 0.045 mm (PFA.1 and PFB.1) were tested individually.

Additionally, the fractions below 0.045 mm were mixed with the regular pellet feeds (natural and prepared in roller press) in different proportions ratio 9%/16% and 16%/9% maintaining the total ratio of pellet feed in the iron ore mixture of 25%. This investigation aims to evaluate if the incorporation of the fractions below 0.045 mm could be effective to improve the granulation behavior of the iron ore mix containing more ultrafine particles and also to evaluate the effect of the average specific surface of the pellet feed on the granulation behavior of the iron ore mixture.

Table 3.1 - Iron ore mixtures tested.

Iron ores	Participation
Australian sinter feeds (ores A and B), %	40
Brazilian sinter feeds (ores C, D and E), %	35
Pellet Feeds, %	25

For a complete chemical and mineralogical characterization of the pellet feed, sinter feeds, as well as of the raw materials used in the present work, the reader is referred to the work of Oliveira *et. al.* [17].

3.2.2. Granulation tests

The iron ore mixtures were granulated in a bench scale drum, according to the conditions listed in Table 3.2. The procedure established for this test was based on the methodology reported in literature [7,8] and recently studied by different authors [18-21].

After mixing and granulating, according to the conditions listed in Table 2, the agglomerated particles were transferred to a carousel divider and splitted in four aliquots of approximately 400 g each. The samples were divided as following: two for granulation test, one for microscopy analysis and another for moisture determination.

The portion for the granulation test was screened in sieve sizes of 4.76 mm, 2.00 mm, 1.00 mm, 0.50 mm and 0.25 mm. Subsequently, it passes through disaggregation process to determine the amount of particles smaller than 0.25 mm which were adhering to agglomerates larger than 0.25 mm. To infer the granulation performance, the GI was calculated from the equation below:

$$GI (\%) = \frac{(a - b)}{a} \times 100$$

where:

a: the amount of particles smaller than 0.25 mm agglomerated plus the no agglomerated particles, reported in grams.

b: the amount of particles with less than 0.25 mm no agglomerated, reported in grams.

Table 3.2 - Experimental procedure for mixing and granulating.

Drum characteristics	Step	Parameters
Volume: 0.125 m ³ Length: 0.250 m Diameter: 0.240 m Typical samples weight: 2,400 grams	Mixing	Time: 120 s Rotation speed: 18 RPM
	Granulation	Time: 360 s Rotation speed: 18 RPM Water addition: 100%

The test was performed twice and the final value of GI represents the average of these two tests. If the difference between the results were higher than 3%, the test was repeated. Additionally, the particle size distribution of the agglomerated particles (mean size) was also determined.

3.2.3. Quasi-particles evaluation

For a better understanding of the phenomena involved during the granulation step the quasi-particles (agglomerated particles) were analyzed by optical microscopy and drop test.

The samples of agglomerated particles were classified in the following size ranges: > 4.76 mm, > 2.83 mm and < 4.76 mm, and > 1.00 mm and < 2.83 mm, Figure 3.1(a). Figure 3.1(b) shows the agglomerated particles in a polished section. Figure 3.1 (c) shows detailed images of the types of agglomerated particles obtained by optical microscopy and their classification into quasi-particles (nucleus particles surrounded by adherent fines), micropellets (composed by adherent fine particles agglomerated) and non-agglomerated particles. The quasi-particles were described initially by Ishikawa *et al.* and Satoh [5,6] and later studied by several authors. The micropellet particles became more important recently due to the increase of fine particles below 0.15 mm as a result of the incorporation of pellet feed or concentrates into the iron ore mixtures as reported different authors [14-17, 20,21].

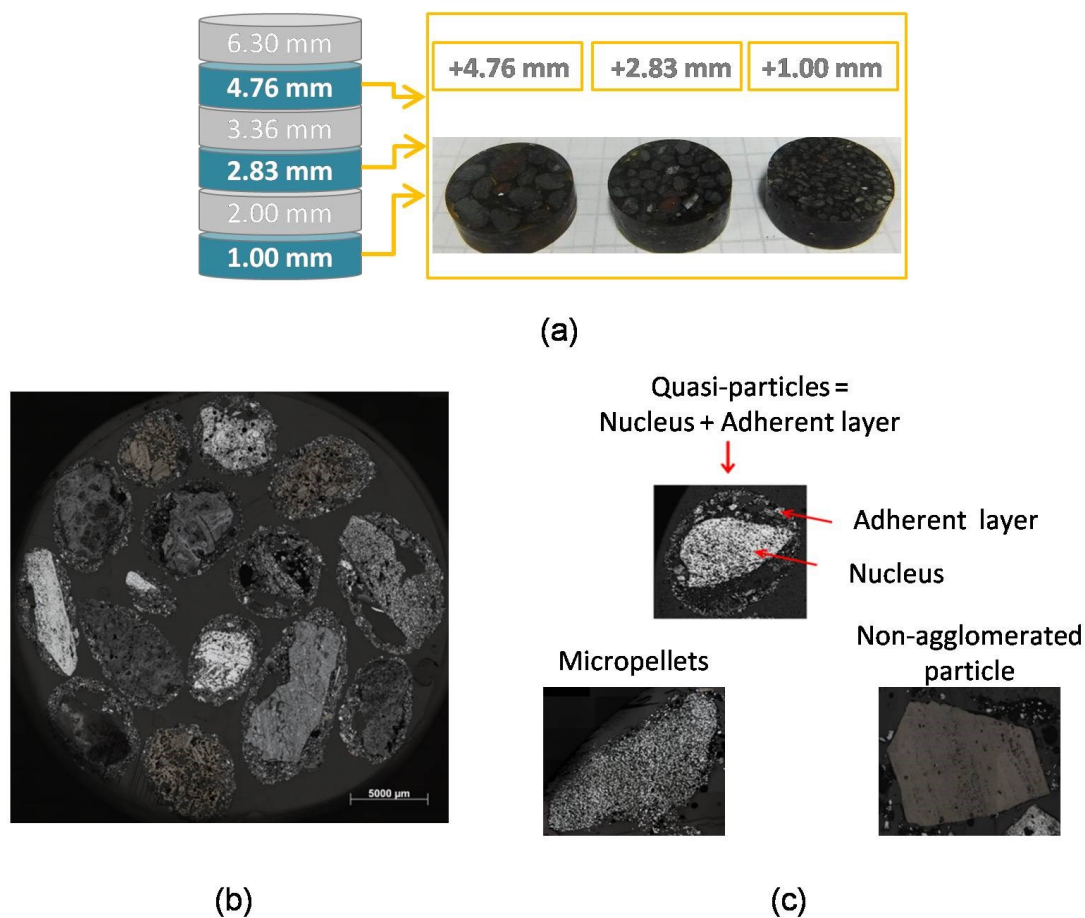


Figure 3.1. (a) Sample preparation at different granulometric sizes; (b) agglomerated particles in a polished section; and (c) details of particles classification.

The samples were analyzed by optical microscopy, using a Carl Zeiss® microscope, AxioImager Z2m model. An image analyzer software was used and the agglomerated particles (quasi-particles, micropellets, non-agglomerated particles) were quantified in different types by dot counting. The nucleus type particles and the thickness of the adherent layer were also quantified.

The quasi-particles drop test was carried out following a procedure based on the standard JIS M 8711 used for Shatter Index determination of sinter product. In this test, the amount of fines below 0.15 mm was measured before and after 2 drops. These results were compared with the amount of fines below 0.15 mm of the iron ore mixture before drum mixing. Finally, an estimative of the amount of fines that were still joined to the nucleus particles was determined.

3.3.Results and Discussion

3.3.1. Pellet feed characterization

Figure 3.2 shows the size distribution of the pellet feed samples used in this work. As expected, the fractions below 0.045 mm of the pellets, PF A.1 and PF B.1, are finer than the pellet feeds PF A and PF B.

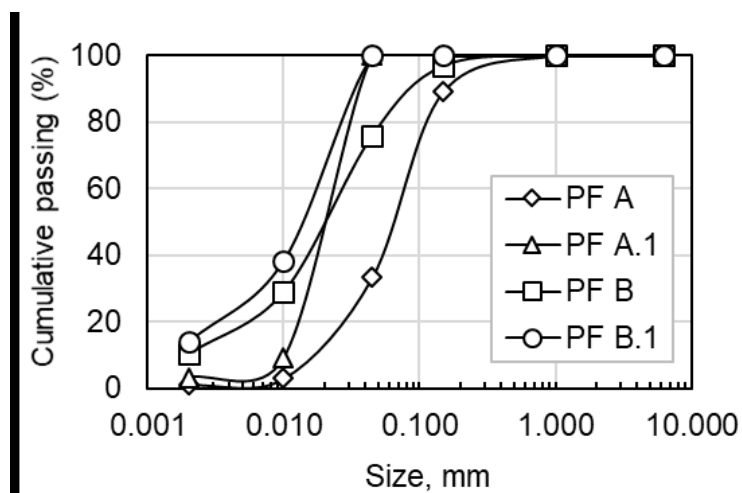


Figure 3.2 - Size distribution of the pellet feed samples.

Figure 3.3 (a) and (b) shows SEM images of the fractions below 0.045 mm of the natural pellet feed and after pressing (samples A.1 and B.1, respectively). The PF B.1 sample contains much more ultrafine particles as compared to PF A.1 and the large particles were surrounded by those ultrafine. Additionally, the large particles became rare being replaced by smaller particles and some cracks (Figure 3 a and b). Both effects were due to the action of the roller press as reported in literature [12,13].

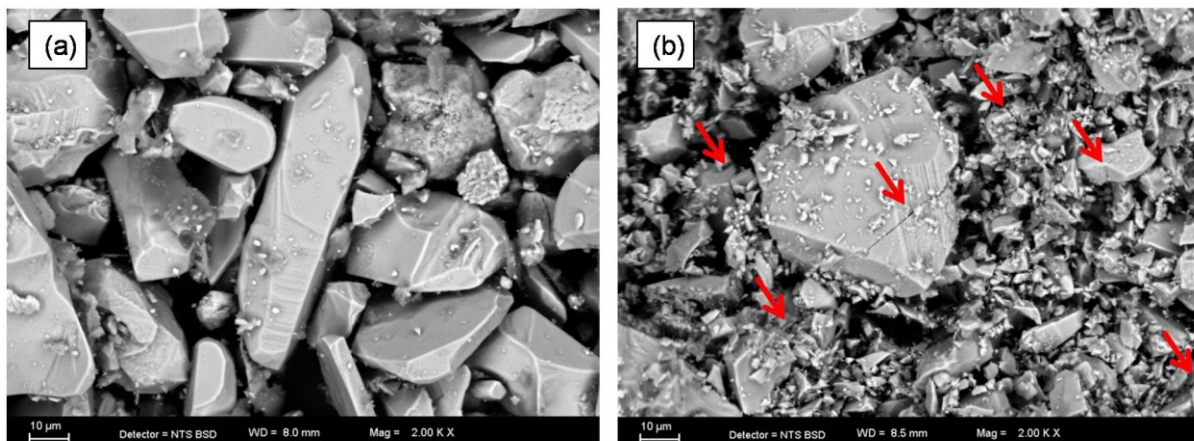


Figure 3.3 - SEM images:(a) PF A.1, fraction below 0.045 mm of the natural pellet feed (PFA); and PF B.1, fraction below 0.045 mm of the pressed pellet feed (PF B). The arrows indicate cracks on the surface of the particles.

Table 3.3 shows the results of specific surface of the samples using different techniques, one based on air permeability test (Blaine Index) and another one based on nitrogen adsorption method (B.E.T.). It can be observed that the fraction below 0.045 mm of the pressed pellet feed presents the highest specific surface area through both techniques, similar results were reported in literature [17, 22]. Concerning the ballability index (K), it increases as the specific surface of the pellet feed increases, reaching values over 0.6, which is considered a good level of K factor for a good agglomeration behavior according to Jian et al. [15].

Table 3.3 - Specific surface and ballability index of pellet feeds.

Pellet Feed	Blaine Index (cm ² /g)	B.E.T. (m ² /g)	Ballability (K) (m ² /g)
PF A	433	0.80	0.17
PF B	1,468	1.70	0.58
PF A.1	750	1.44	0.20
PF B.1	2,100	2.13	0.80

3.3.2. Granulation test results

The granulation test was performed at fixed moisture level of 7.5 %. Tests were performed with moisture level higher than 7.5 %, but it was not practical due to the impact on the

homogenization of the iron ore mixture causing problems in the reproducibility of the test. Table 3.4 shows more details of the iron ore mixture tested in the present work. The level of particles lower than 0.010 mm of the iron ore mixture increases with the increase of the specific surface of the pellet feeds.

The aimed chemical quality of the sinter (Fe: 57.5%, SiO₂: 5.70%; Al₂O₃: 1.40%; P: 0.044 %; and ratio %CaO/%SiO₂: 1.60) is based on a typical Asian blast furnace operation (high level of sinter product in ferrous burden) to guarantee a optimum permeability, good metallurgical performance and low slag rate.

Table 3.4 - Details of iron ore mixtures tested.

Parameter	Case PF A	Case PF A.1	Case PF B	Case PF B.1
Iron ore mixture				
Sinter feeds	75	75	75	75
Pellet feed, PFA	25	0	0	0
Pellet feed, PFA.1	0	25	0	0
Pellet feed, PFB	0	0	25	0
Pellet feed, PFB.1	0	0	0	25
Pellet feed Specific Surface*, cm ² /g	430	750	1,468	2,100
Iron ore mixture size				
+1.000 mm, %	39.3	39.3	39.3	39.3
-0.150 mm, %	41.7	44.4	43.6	44.4
-0.045 mm, %	21.3	38.0	32.0	38.0
-0.010 mm, %	9.7	11.3	16.2	18.5
Fluxes and additives				
Coke Breeze, %	4.0			
Burnt lime, %	3.0			
Fluxes total , Kg/t	126			

* Blaine Index

Table 3.5 shows the results of GI and agglomerated particles mean size for the iron ore mixtures containing pellet feed and its fraction below 0.045 mm. The pressed pellet feed, PF B,

and its fraction below 0.045 mm, PF B.1, presented better better GI and mean size of agglomerated particles results than natural pellet feed, PF A, and its fraction below 0.045 mm, PF A.1. Comparing the pellet feeds with its fraction below 0.045 mm similar results were achieved. The mean size of agglomerated particles increased with the increase of specific surface of the pellet feeds and with the fraction below 0.010 mm of the average iron ore mixture.

Table 3.5 - Results of GI and agglomerated particles mean size for the iron ore mixtures

Case	GI, %	Mean size of agglomerated particles, mm
Case PF A	76.3	2.25
Case PF A.1	78.2	2.82
Case PF B	89.5	2.91
Case PF B.1	88.9	3.35

Figure 3.4 shows the results of GI when the fractions below 0.045 mm of the pellet feeds were mixed with natural pellet feed in different proportions as described previously. The fraction below 0.045 mm of the pressed pellet feed (PF B.1) improved the granulation behavior of the natural pellet feed. No significant difference on the granulation behavior was observed when the fraction below 0.045 mm of natural pellet feed (PF A.1) was mixed with natural pellet feed. These results showed that the fraction below 0.045 mm of this material is different, indicating that the roller press treatment contribute to guarantee a good cold agglomeration granulation performance of this material.

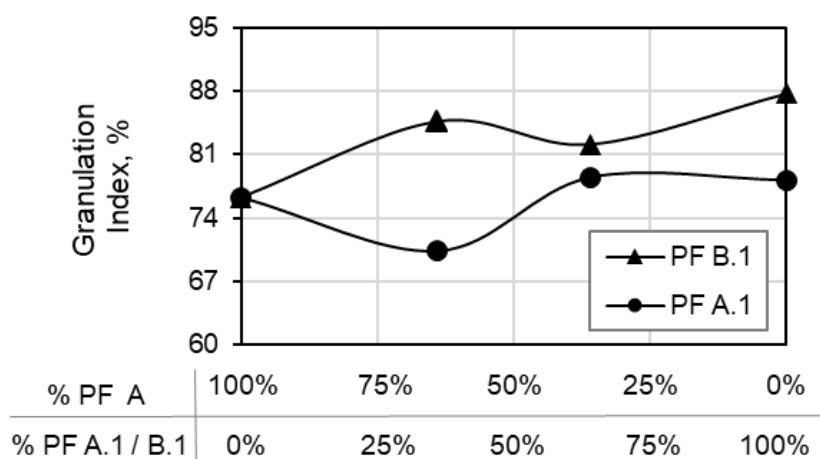


Figure 3.4 - GI results obtained when the fraction below 0.045 mm (samples PF A.1 and PF B.1) was mixed with natural pellet feed (PF A).

The Table 3.6 shows the average specific surface of the cases where the fractions below 0.045 mm were mixed with the regular pellet feeds (natural and prepared in roller press) in different proportions maintaining the total pellet feed ratio in the iron ore mixture in 25%.

Table 3.6 - Average specific surface of the mixtures of pellet feeds

PF A (%)	PF B (%)	PF A.1 (%)	PF B.1 (%)	Pellet Feed Total (%)	Average Specific Surface (cm ² /g)
25	--	--	--	25	430
--	--	25	--	25	750
--	25	--	--	25	1,470
--	--	--	25	25	2,100
9	--	16	--	25	545
16	--	9	--	25	635
16	--	--	9	25	1,000
16	--	--	9	25	1,031
12	--	--	13	25	1,300
9	--	--	16	25	1,499
--	9	--	16	25	1,697
--	16	--	9	25	1,873

Figure 3.5 (a) and (b) show GI results and mean size of agglomerated particles after granulation test in function of the average pellet feed specific surface (Blaine Index). It is possible to note an increase of GI with the specific surface up to 1,400-1,500 cm^2/g . For specific surface higher than this value a stabilization of GI around 86-90 % was observed. Concerning the mean size of the agglomerated particles formed after the granulation step, it is observed a trend to increase with the increase of specific surface, Figure 5 (b). These results were not in line with GI results as mentioned previously. The hypothesis that possible explain this behavior is that even the higher mean size, the agglomerated particles did not have enough strength, thus resulting in a higher GI. To better understand these results, the investigation of the quasi-particles formed during the granulation behavior is of paramount importance.

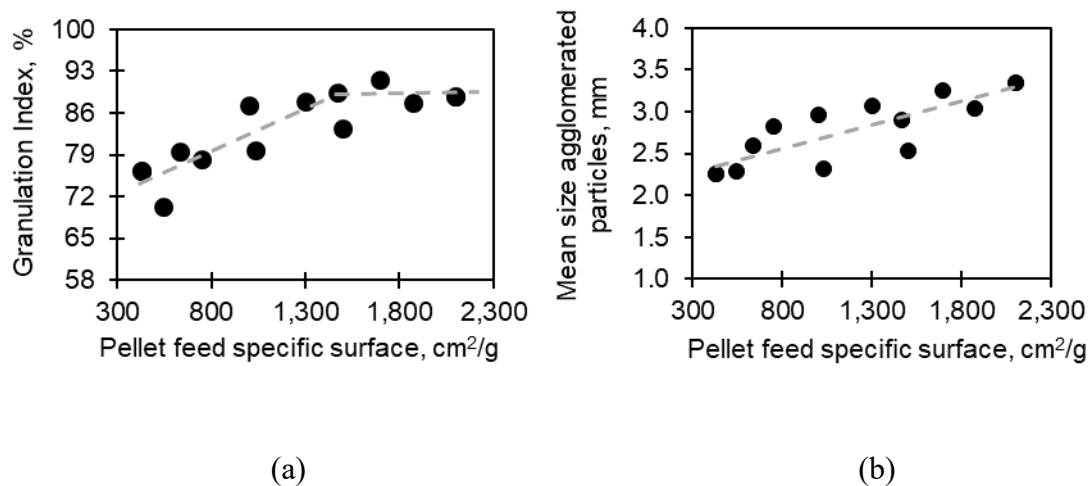


Figure 3.5 - (a) GI results and specific surface of the pellet feeds; and (b) mean size of agglomerated particles after granulation test and specific surface of the pellet feeds.

3.3.3. Microscopy analysis and drop test results

The quasi-particle evaluation by optical microscopy was done aiming to better understand the cold agglomeration phenomena involved during granulation step and also investigate the hypothesis previously raised.

Table 3.7 shows a summary of the main results obtained by optical microscopy analysis. About thickness of quasi-particles, an increasing trend with specific surface was observed. The growing of the adherent layer due to the deposition of ultrafines seems to be the mechanism of growing phenomena (layering), as other authors recently reported in literature [20]. No clear correlation was observed with circularity.

Table 3.7 - Optical microscopy results of the quasi-particles

Parameter	PF A	PF A.1	PF B	PF B.1
Pellet Feed Specific Surface				
Blaine Index, cm ² /g	430	750	1,470	2,100
Characteristics of quasi-particles				
Thickness of quasi-particles, mm	0.115	0.149	0.137	0.185
Circularity	0.660	0.657	0.657	0.660
Particle type formed, %				
Quasi-particles	81.7	92.7	83	92.1
Micropellets	11.1	5.4	15.5	7.1
Non-agglomerated	7.2	1.9	1.5	0.8
Nucleus particles characterization, %				
Hematite	13.7	13.4	13.5	23.3
Goethite	43.6	39.9	41.3	33.1
Sinter return fines	42.0	44.7	40.2	42.8
Magnetite	0.2	0	0	0
Others	0.5	2.1	5	0.8

In terms of particle type formed, a downward trend of non-agglomerated particles was observed with the increase of specific surface. No clear correlation was observed between quasi-particles nether micropellets formed. Finally, the type of nucleus particles did not show a clear trend when the specific surface was varied.

Figures 3.6, 3.7 and 3.8 shows the correlation between the optical microscopy results of the quasi-particles with the specific surface of the pellet feed.

Figure 3.8 (a) shows that the thickness of the quasi-particles increases with the specific surface. This result was in line with the increase of the mean size of agglomerated particles obtained in granulation test. Additional tests were required to better understand this behavior. On the other hand, the circularity of agglomerated particles did not demonstrate any correlation (Figure 3.6 b), confirming the results of individual tests (Table 3.7).

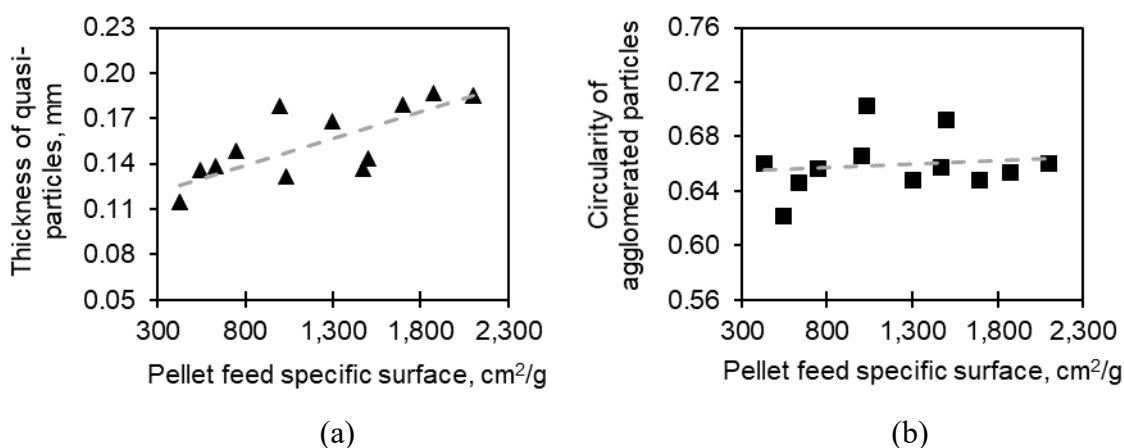


Figure 3.6 - (a) Thickness of quasi-particles and specific surface of pellet feeds; and (b) Circularity of agglomerated particles and specific surface of pellet feeds.

Figure 3.7 (a), (b) and (c) show the particle type formed during the granulation process for the different specific surfaces of pellet feed. The non-agglomerated particles decreased with the increase of specific surface of pellet feeds, showing that the increase of specific surface promote a better agglomeration behavior of the fines (Figure 3.7(a)). The micropellets formation demonstrated a maximum for a specific surface around 1,300-1,500 cm^2/g (Figure 3.7 (b)) whereas the quasi-particles formed reached a minimum, Figure 3.7 (c). However, the amount of quasi-particles formed remained the majority of the agglomerated particles. Based on these results, it was confirmed that the mechanism of formation of agglomerated particles was governed by quasi-particles growing because of the deposition of ultrafines at the adherent layer (layering). Additionally, there was also a formation of micropellets with the ultrafines (coalescence) up to around 1,300-1,500 cm^2/g , but in a lower scale. This behavior explains the better granulation performance of the mixture up to this level of specific surface. On the other hand, values of specific surface higher than 1,500 cm^2/g indicate that the most important mechanism was the quasi-particles growing due to the deposition of ultrafines on the adherent layer (layering). This explains the agglomerated mean size and thickness of quasi-particles increasing with the increase of specific surface, but it not explains why the GI stabilized. Based on these findings it may be inferred that the thickness of quasi-particles reaches a critical value for specific surfaces (around 1,500 cm^2/g) and above that it became weak.

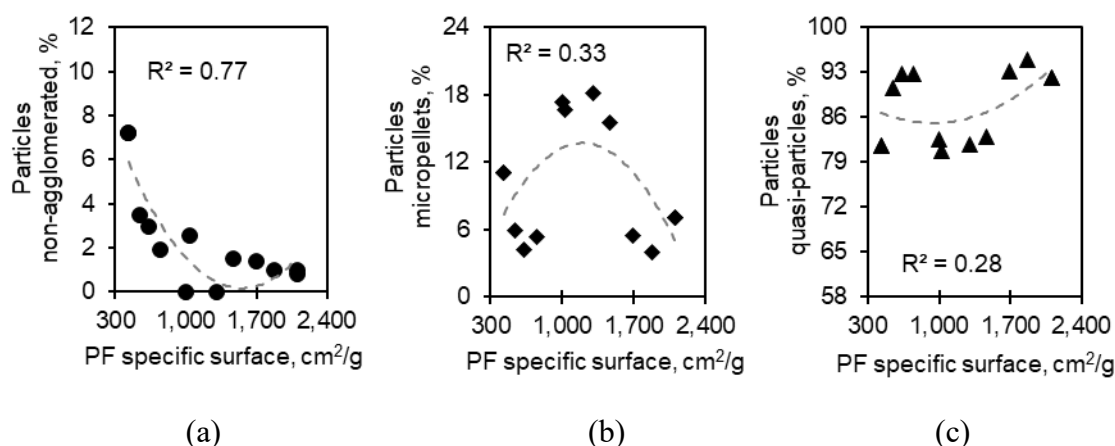


Figure 3.7 - Types of agglomerated particles with the specific surface of pellet feeds: (a) Non-agglomerated particles; (b) micropellets; and (c) quasi-particles.

Once the most agglomerated particles formed were quasi-particles (Figure 3.7) it is important to evaluate the nucleus particles characteristics, see Figure 3.8. In general, an upward increase of nucleus particles formed by hematite is observed when the specific surface increases, whereas the nucleus particles composed by goethite decrease. Normally, goethite ores are more porous, retaining more water in comparison to hematite ores, and require more water to have a good agglomeration behavior as reported in literature [4,6,23]. It means that these particles will compete with the fine particles of the pellet feed for the water available leading to decrease of nucleus particles formed by those ores. No clear trend was observed for the nucleus particles formed by the sinter return fines.

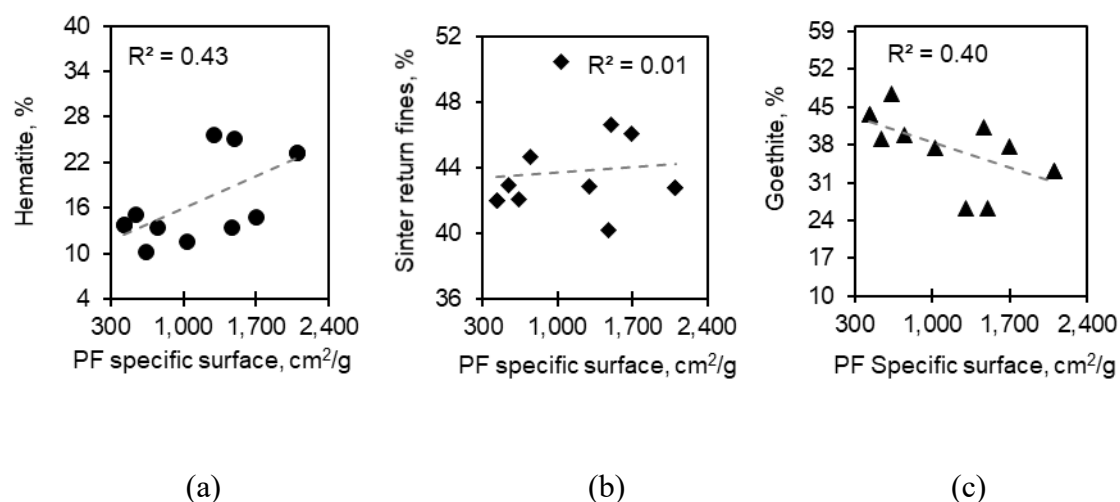


Figure 3.8 - Nucleus particles characteristics: (a) hematite; (b) sinter return fines; and (c) goethite.

Detailed optical microscopy images of the quasi-particles were collected to compare the case with the lowest (PF A) and the highest (PF B.1) specific surface of pellet feed, Figure 3.9 (a) and (b), respectively. The pellet feed with the lowest specific surface lead to a low mean size of agglomerated particles, low thickness of adherent fines layer and low GI. This behavior is explained by the incomplete adherent layer formed, some voidages in this layer and coarse particles forming it, as indicated by arrows in Figure 3.9 (a). On the contrary, the pellet feed with the highest specific surface leads to high mean size of agglomerated particles, high thickness of adherent layer and good level of GI. This behavior may be explained by a good adherent layer formed with fine particles well distributed, but with some voidages (less than in the case with PF A) in this layer which contribute to the stabilization of GI for specific surface higher than 1,400-1,500 cm^2/g , as indicated by arrows in Figure 3.9 (b). Thus, a high thickness of the adherent layer does not mean a high GI or high strength of this layer.

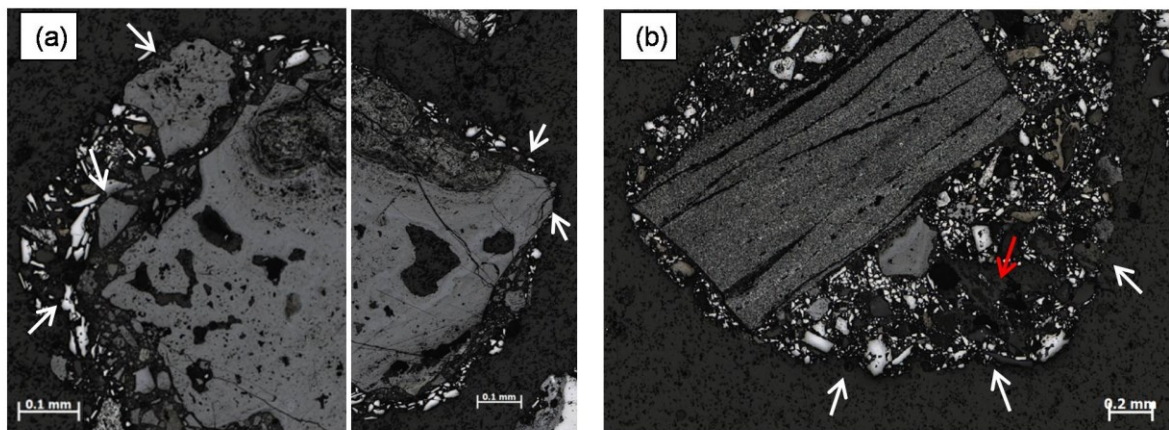


Figure 3.9 - Detailed images of the quasi-particles: (a) case with the lowest specific surface of pellet feed (PF A); and (b) case with the highest specific surface tested (PF B.1).

In order to reinforce the hypothesis raised about the strength of the adherent layer of the quasi-particles, a drop test was carried out with the cases containing the pellet feeds A, A.1, B and B.1. Figure 3.10 shows that more fines below 0.15 mm remain agglomerated to quasi-particles or micropelletized with the increase of the specific surface up to 1,400-1,500 cm^2/g and getting stable above these values.

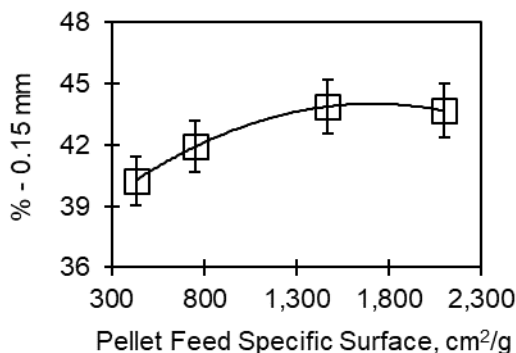


Figure 3.10 - Amount of fines below 0.15 mm that remain agglomerated after drop test as a function of the pellet feed specific surface.

The results presented in Figure 3.10 are in line with the GI results confirming the hypothesis raised that for the pellet feeds of higher specific surface the quasi-particles grew (Figure 3.9 (b)), but they did not have enough strength to maintain its size. So, values higher than 1,400-1,500 cm²/g will not impact the permeability of the sintering process as its GI and strength of quasi-particles stabilized, meaning that for the level of pellet feed tested, 25%, it was the minimum value of specific surface needed for good sintering performance as confirmed through sintering pot test results reported by Oliveira *et. al.* 2019 [17].

3.4. Conclusions

The granulation phenomena involved during the preparation of iron ore mixtures for sintering played an important role in the process impacting directly the permeability of the bed and as consequence the productivity of sintering machine. In this context, an iron ore mixture containing 25% of pellet feed with different specific surfaces, i.e. natural, roller pressed and its fractions below 0.045 mm, were evaluated and studied through bench scale granulation test. Based on the results achieved, the main conclusions are:

- The characterization of the pellet feeds used in this work showed that the fractions below 0.045 mm presents higher specific surface than the natural and pressed pellet feeds and higher ballability index, K. A complementary analysis obtained by SEM images showed the presence of small particles, cracks on the remaining larger particles and particles assuming a more rounded shape surface.

- The GI test results showed that the pressed pellet feed and its fraction below 0.045 mm presented higher values than the natural pellet feed and its fraction below 0.045 mm. The mean size of agglomerated particles increased with the increase of specific surface of pellet feeds. The addition of the fractions below 0.045 mm of the pressed pellet feed to the natural sample improved the granulation behavior of the iron ore mixture.
- The GI results increase with the increase of pellet feed specific surface up to 1,400-1,500 cm²/g. For values of specific surface higher than these values a stabilization of GI around 86-90 % was observed. On the other hand, the mean size of the agglomerated particles formed after the granulation step increases continually with the pellet feed specific surface.
- Concerning the agglomerated particles, the thickness of quasi-particles increases with the specific surface. About the particles type formed, the non-agglomerated particles decreased with the increase of the specific surface of pellet feeds, showing that the increase of specific surface promote a better agglomeration behavior of these fines. The quasi-particles formed remain most of the agglomerated particles.
- In general, an increasing trend of nucleus particles formed by hematite was observed with the increase of specific surfaces of pellet feed, whereas a decrease of nucleus particles formed by goethite was observed. No clear trend was observed for the nucleus particles formed by sinter return fines.
- The adherent layer formed with PF A (lowest specific surface) was incomplete with some voidages and coarse particles forming it. This explains the low mean size of agglomerated particles, low thickness of adherent fines layer and low GI. On the other hand, a good adherent layer formed with fine particles well distributed and less voidages was observed for the case with PF B.1 (the highest specific surface), which explains a high mean size of agglomerated particles, high thickness of adherent layer and good level of GI.
- Finally, the drop test carried out with the cases containing the pellet feeds and its fraction below 0.045 mm showed that the fines below 0.15 mm that remained agglomerated to quasi-particles or micropelletized increases with the increase of specific surface stabilizing for values higher than 1,400-1,500 cm²/g, confirming the hypothesis that the quasi-particles strength also stabilizes.

Conflicts of interest

The authors declare no conflicts of interest.

Acknowledgments

The authors would like to thank CAPES-PROEX, FAPEMIG and CNPQ, as well as all collaborators involved in the work at Vale S.A. and Federal University of Minas Gerais (UFMG).

References

- [1] Wu S, Que Z and Li K. Strengthening granulation behavior of specularite concentrates based on matching of characteristics of iron ores in sintering process, *Journal of Iron and Steel Research International* 2018; 25:1017-1025. DOI:10.1007/s42243-018-0153-9
- [2] Yang C, Zhu D, Shi B, Pan J, Lu L, Li X and Mo Y. Comparison of sintering performance of typical specular hematite ores with distinct size distributions. *International Journal of Iron and Steel Research*, v.24, p1007-1015, 2017.
- [3] Walker G M. Chapter 4 - Drum Granulation. In: Salman, A D, Hounslow, M J, Seville, J P K. *Handbook of Powder Technology – Granulation*. 1st Edition. Elsevier Science, 2007. Volume 11, p.219-254.
- [4] Lu L and Ishiyama O. Iron ore sintering. In: Lu L., editor. *Iron ore: Mineralogy, Processing and Environmental Sustainability*. 1st ed. Elsevier Ltd; 2015, p. 395-433.
- [5] Vieira C B, Rosière C A, Pena E Q, Seshadri V, Assis P S. Avaliação técnica de minérios de ferro para sinterização nas siderúrgicas e minerações brasileira: Uma análise crítica, *REM, Escola de Minas de Ouro Peto*, 56(2), 97-102, 2003.
- [6] Kohsa J and Manuel J. Predicting granulating behaviour of iron ores based on size distribution and composition. *Isij International* 2007; 47(7):965-972.
- [7] Ishikawa Y, Sugawara K and Umezu Y. Latest development of sintering technology. K.V.S. Sastry. *Agglomeration* 77, AIME. New York. 1977, pp.503-525.
- [8] Satoh K. Preparation of raw materials centering around the improvement of quasi-particles. *Flotation*. v.28, n.2, p99s-109s, 1981.

- [9] González D F, Bustinza I R, Mochón J, Casca C G, Verdeja L F. Iron ore sintering: raw materials and granulation, mineral processing and extractive metallurgy review. London, 38:1, 36-46, 2017. DOI: 10.1080/08827508.2016.1244059
- [10] Wu S, Zhu J, Bei J, Zhang G and Zhai X. Effects of particle characteristics on the granulation ability of iron ores during the sinter process, *International Journal of Minerals, Metallurgy and Materials*, Berlin. v. 22, n. 9, p907-916, 2015. DOI:10.1007/s12613-015-1149-0
- [11] Umadevi T, Brahmacharyulu A, Roy A K, Mahapatra P C, Prabhu M and Ranjan M. Influence of iron ore fine feed size on microstructure, productivity and quality of iron ore sinter. *Isij International* 2011; 51(6):922-929.
- [12] Zhu D, Pan J, Lu L and Holmes R J. Iron ore pelletization. In: Lu L., editor. *Iron ore: Mineralogy, Processing and Environmental Sustainability*. 1st edition. Elsevier Ltd; 2015, p.435-473.
- [13] Abazapoor A, Halali M, Hejazi R and Saghaein M. HPGR effect on the particle size and shape of iron ore pellet feed using response surface methodology. *Mineral Processing and Extractive Metallurgy* 2018; 127: 40-48.
- [14] Zhu D Q, Pan J, Chun T. J. and Chen D. Improving the granulation and sintering performance by pretreating concentrates using roller press. *Iron Ore Conference*. 2011. The Australian Institute of Mining and Metallurgy - AusIMM, Perth, WA, Australia, 11-13 July 2011, 575-578p.
- [15] Jian P, Zhu D, Pimenta H P, Xian-Lin Z and Liin W. Improving granulating and sintering performance by pretreating specularite concentrates through mechanical activation. *Isij International* 2013; 53(12):2013-2017.
- [16] Gan M, Fan H, Ji Z Y, Chen L X, Yin L, Jiang T, Yu Z Y and Huang Y S. Optimizing method for improving granulation effectiveness of iron ore sintering mixture. *Ironmaking and Steelmaking*, London. v. 42, n. 5, p351-357, 2015. DOI:10.1179/1743281214Y.0000000237
- [17] Oliveira V M, Resende V G, Domingues A L A, Bagatini M C and Castro L F A. Alternative to deal with high level of fine materials in iron ore sintering process. *Journal of Materials Research and Technology*, 2019; 8(5):4985-4994.
- [18] Avila L S C, Azevedo R N, Bagatini M C, Castro L F A. Influência das características do itabirito concentrado no desempenho do processo de sinterização. In: Associação Brasileira de Metalurgia, Materiais e Mineração – ABM, organizer. *Proceedings of the 45^o*

Seminário de Redução de Minério de Ferro e Matérias-primas; 2015; Rio de Janeiro, Brasil. 10p.

- [19] Silva T P, Lemos L R, Castro L F A. Uso de calcário na forma de polpa com Dispersante na etapa de aglomeração a frio para sinterização de minério de ferro. In: Associação Brasileira de Metalurgia, Materiais e Mineração – ABM, organizer. Proceedings of the 49º Seminário de Redução de Minério de Ferro e Matérias-primas; 2019; São Paulo, Brasil. 12p.
- [20] Oliveira A F L, Júnior R A L, Flores I V, Avila L S C, Bagatini M C. Adição de pellet feed na aglomeração a frio de minério de ferro para sinterização. In: Associação Brasileira de Metalurgia, Materiais e Mineração – ABM, organizer. Proceedings of the 49º Seminário de Redução de Minério de Ferro e Matérias-primas; 2019; São Paulo, Brasil. 13p.
- [21] Shatokha V, Korobeynikov I, Maire E, Adrien J. Application of 3D X-ray tomography to investigation of structure of sinter mixture granules. *Ironmaking & Steelmaking*. 2009; 36(6):416-420.
- [22] Mao H, Zhang R, LV X, Bai C and Huang X. Effect of surface properties of iron ores on their granulation behaviour. *Isij International*, 2013; 53(9):1491-1496.
- [23] Loo C E. A perspective of goethitic ore sintering fundamentals. *Isij International*, 2005,45(4):436-448.

Capítulo 4. Considerações Finais

No presente trabalho avaliou-se o comportamento de aglomeração de *pellet feed* com e sem tratamento mecânico em prensa de rolos de diferentes superfícies específicas, como uma alternativa de matéria-prima para sinterização.

O artigo apresentado no Capítulo 2 discorre sobre o estudo em sinterização piloto com utilização de *pellet feed* em substituição a *sinter feed* presentes do mercado (25% da mistura de minério de ferro). Nesse trabalho foi demonstrado que a alternativa de pré-tratamento do *pellet feed* na prensa de rolos é interessante para promover o seu uso neste processo. Observou-se que o *pellet feed* composto essencialmente por hematita é uma boa alternativa para melhorar a composição química do sinter devido ao alto teor de ferro e a um nível mais baixo de contaminantes (SiO_2 , Al_2O_3 e P). Além disso, dependendo do minério substituído, observa-se uma redução do combustível sólido na sinterização devido ao seu menor LOI. O tratamento mecânico do *pellet feed*, na prensa de rolos, leva a um aumento na fração ultrafina (frações menores que 0,045 mm e 0,010 mm) o que se mostrou positivo para o processo de granulação. O uso de *pellet feed* com superfície específica mais alta, em torno de 1.400-1.500 cm^2/g , levou a um aumento na produtividade da sinterização piloto. Isso ocorre devido ao melhor comportamento de granulação das misturas formadas pelos *pellet feed* que foram prensados, levando a uma melhor permeabilidade (maior velocidade de frente de chama). Nestes casos, as quase-partículas formadas apresentaram-se com a camada aderente bem formada, coesa e com poucos vazios (finos aderentes circundando praticamente quase todas as partículas nucleantes) quando comparadas a aquelas formadas com *pellet feed* sem prensagem. Adicionalmente, este caso apresentou melhor resultado do índice de granulação (GI) e maior resistência, medida através do teste de queda, das quase-partículas formadas. Em termos de qualidade do sinter, os casos onde o *pellet feed* foi utilizado em substituição ao *sinter feed* australiano goetítico, independentemente da superfície, mostraram melhores resultados da resistência mecânica sem comprometer a sua qualidade metalúrgica.

O Capítulo 3 foi dedicado ao segundo artigo do trabalho cujo objetivo principal foi investigar com uma maior profundidade, através de ensaios de granulação, o comportamento dos *pellet feed* sem e com tratamento de prensagem (o *pellet feed* natural e o de maior superfície específica), suas frações menores do que 0,045 mm e a mistura destas frações com estes mesmos *pellet feed*. Alguns indicativos puderam ser vistos no artigo anterior visto que, o *pellet feed* de maior superfície específica, que apresentou melhor performance na sinterização,

apresentou um melhor GI e maior resistência das quase-partículas. Como essa melhora de permeabilidade foi atribuída, a princípio, as frações ultrafinas dos *pellet feed* prensados optou-se por estudar as frações menores do que 0,045 mm dos *pellet feed* natural, sem tratamento na prensa de rolos, e o *pellet feed* de superfície específica que foi suficiente para recuperação da produtividade quando este substitui *sinter feed* de mercado. De maneira geral, este trabalho mostrou que as frações menores que 0,045 mm dos *pellets feed* são fisicamente diferentes, sendo que o *pellet feed* prensado apresentou trincas nestas frações numa proporção muito maior do que do *pellet feed* sem prensar, conforme evidenciado por imagens de microscopia eletrônica de varredura. Os resultados dos testes de GI mostraram que a adição das frações abaixo de 0,045 mm do *pellet feed* prensado à amostra natural melhorou o comportamento de granulação da mistura de minério de ferro. Os resultados de GI aumentam com o aumento da superfície específica dos *pellet feed* até 1.400-1.500 cm²/g, enquanto que para valores de superfície específica superiores a esses valores, foi observada uma estabilização do GI em torno de 86-90%. Por outro lado, o tamanho médio das partículas aglomeradas formadas após a etapa de granulação aumentou continuamente com a superfície específica do *pellet feed*. Ainda no que diz respeito a investigação das partículas aglomeradas, houve uma diminuição das partículas não aglomeradas com o aumento da superfície específica do *pellet feed*, mostrando que o aumento da superfície específica promove um melhor comportamento de aglomeração da mistura de minérios. As quase-partículas foram as partículas predominantemente formadas e a espessura da camada aderente aumentou com a superfície específica indicando o mecanismo da deposição de ultrafinos e crescimento na formação destas. Quanto a identificação da morfologia do núcleo das quase-partículas, foi observada uma tendência destes núcleos serem formados preferencialmente por partículas de hematita com uma diminuição das partículas de goethita com o aumento de superfícies específicas do *pellet feed*. A camada aderente formada com o *pellet feed* de superfície específica mais baixa ficou incompleta com vazios e partículas grossas na sua estrutura. Isso explica o baixo tamanho médio das partículas aglomeradas, a pequena espessura da camada de partículas aderentes e o baixo GI. Por outro lado, uma camada aderente formada com partículas finas bem distribuídas e menos vazios foi observada no caso do *pellet feed* de superfície específica mais alta, o que explica o maior tamanho médio de partículas aglomeradas, uma maior espessura da camada aderente e bom nível de GI. Finalmente, o teste de queda realizado mostrou tendência similar ao GI com uma estabilização da resistência dos aglomerados a partir da superfície específica dos *pellet feed* entre 1.400-1.500 cm²/g. Sendo assim, pode-se concluir que o tratamento mecânico do *pellet feed* através de prensa de rolos

para aumento da sua superfície específica do *pellet feed* é fundamental para garantir o bom desempenho para a sua utilização na sinterização.

Por fim, neste trabalho foi possível demonstrar que o tratamento mecânico do *pellet feed* é uma alternativa para permitir a sua utilização, ao nível de 25% de participação da mistura de minério, na sinterização de minério de ferro sem comprometer a produtividade do processo e sem causar qualquer inconveniente nas qualidades, física e metalúrgica, do sínter produzido. Quando comparado ao *pellet feed* sem tratamento mecânico observa-se uma melhora na permeabilidade do processo de sinterização que pode ser explicada por uma melhora do comportamento durante a etapa de granulação, conforme já mencionado. Isso se deveu a presença dos ultrafinos no *pellet feed* prensado e da presença de trincas nestas partículas o que favoreceu o crescimento e a formação de uma camada aderente mais resistente. Essa camada aderente permaneceu coesa e bem consolidada até o nível de superfície específica do *pellet feed* entre 1.400 e 1.500 cm²/g a partir do qual há uma estabilização dos resultados de GI e da resistência das quase-partículas.

Capítulo 5. Contribuições originais ao conhecimento

As principais contribuições do presente trabalho para o conhecimento científico e tecnológico encontram-se listadas a seguir:

- O tratamento mecânico do *pellet feed* em prensa de rolos mostrou-se uma alternativa interessante para viabilizar a utilização deste material mais fino na sinterização inclusive sendo capaz de potencializar o uso destes materiais em substituição de *sinter feed* disponíveis no mercado. Trata-se de uma quebra de paradigmas para o processo de sinterização. Neste caso quanto mais fino o material, no caso o *pellet feed*, melhor será seu desempenho no processo desde que sejam dadas condições para que granulação (aglomeração a frio) ocorra de maneira satisfatória. Dessa forma, deverá ser dada especial atenção ao tipo de processamento a que estes materiais deverão ser submetidos para uso neste processo, características de superfície específica e granulometria (conceito similar ao aplicado ao processo de pelletização). A utilização deste material na sinterização piloto permitiu, além de recuperar a produtividade, produzir um sinter com qualidade física e metalúrgica satisfatória.
- A fração menor do que 0.045 mm do *pellet feed* tratado na prensa de rolos e sem tratamento são diferentes. Além disso, pode-se utilizar essa fração ou mesmo o *pellet feed* tratado na prensa de rolos para corrigir o *pellet feed* sem tratamento (natural) em termos de comportamento de granulação e espera-se com isso obter um bom desempenho na sinterização.
- Através da análise das quase-partículas foi possível verificar uma melhora da granulação das misturas com o aumento da superfície específica do *pellet feed*. Foi possível quantificar a formação preferencial de quase-partículas, com algumas micropelotas formadas e praticamente nenhuma partícula sem aglomerar. Análise mais detalhada das quase-partículas mostrou um aumento da espessura da camada aderente com o aumento da superfície específica do *pellet feed* devido a deposição destes ultrafinos (partículas mais finas e com a presença de trincas devido ao tratamento mecânico na prensa de rolos) nesta camada, sendo este o mecanismo predominante.
- Ainda sobre a camada aderente formada, para o caso com o *pellet feed* de superfície específica mais baixa observou-se que esta ficou incompleta com vazios e partículas grossas na sua estrutura. Por outro lado, uma camada aderente formada com partículas finas bem distribuídas e menos vazios foi observada no caso do *pellet feed* de superfície específica

mais alta. Dessa forma, o caso com maior superfície específica apresentou um melhor índice de granulação e uma melhor resistência das quase-partículas quando comparado ao caso de menor superfície específica.

- Para o nível de *pellet feed* testado foi possível estabelecer um nível mínimo de superfície específica para se obter uma boa performance na sinterização piloto e na etapa de granulação, 1.400-1.500 cm²/g, além de ser possível estabelecer uma faixa ótima de trabalho para o GI entre 86-90%. Para valores de superfície específica maiores observa-se a estabilização do GI, mesmo com o aumento da espessura da camada aderente das quase-partículas.

Capítulo 6. Perspectivas de trabalhos futuros

Dentre os principais pontos recomendados para execução de trabalhos futuros destacam-se:

- Avaliar maiores participações de *pellet feed* prensado (> 25%) na mistura de minérios para verificar se essa alternativa tecnológica seria viável.
- Estudar a combinação de maiores participações do *pellet feed* prensado (> 25%) na mistura de minérios com aditivos, tais como cal ou outros aglomerantes disponíveis no mercado, e / ou a combinação com diferentes rotas tecnológicas, tais como misturadores intensivos, que promovam uma melhor etapa de mistura.
- Avaliar o uso de aglomerantes e aditivos para estabilizar as quase-partículas formadas em misturas contendo *pellet feed* de elevada superfície específica e com alta participação de ultrafinos (fração menor que 0,045 mm).
- Estudar a utilização de dispersantes para garantir distribuição homogênea dos ultrafinos da camada aderente e assim evitar a formação de partículas aglomeradas de baixa resistência.
- Avaliar utilização de diferente método de tratamento mecânico (exemplo moagem) para produção do *pellet feed* de maior superfície específica e maior quantidade de ultrafinos na mistura de minérios (25% de participação na mistura).
- Estudar diferentes tipos (origem e mineralogia) de *pellet feed* prensado (25%) na mistura de minérios para verificar se essa alternativa tecnológica seria viável.