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Experiment on micron-sized particle production of iron ore by rapid unloading of liquid CO_2

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The average iron content of iron ore is <30%; therefore, crushing, grinding, milling and other processing techniques must be executed before smelting. Currently, it is expensive to break iron ores using mechanical grinding. Experiments have been carried out to develop a novel approach of producing iron ore powder. First, the iron ore is placed in a high-pressure chamber, and then liquid CO_2 is injected into the chamber. Second, considering energy recycling, after the iron ore pores are filled with liquid CO_2, dissociative liquid CO_2 is substituted and collected for cyclic utilization. Third, an initial high pressure is applied inside the chamber using a water pump in order to increase the energy input. Fourth, the pressure is rapidly unloaded. After penetration and gasification expansion, the iron ore will immediately be converted into micron-sized particles. Laser grain size analysis indicated that the grain size of the iron ore particles ranges between 30 and 50 μm, which will satisfy the requirements of gravity separation, magnetic separation and the flotation process. This is a highly efficient and low-cost method that has excellent industrial promotion value.

1. Introduction

Iron powder is in great demand, especially for the auto industry, manufacturing, and the production of permanent magnets [1]. The production of special steel and alloys from iron powder is also of great interest. In this context, research is underway to expand the range and improve the quality of iron powder and also to find additional sources of raw materials for powder production. Conventional techniques used in the production of iron ore particles are based on mechanical methods, including crushing, grinding and milling processes. Mechanical crushing [2] is represented as periodic processes that accumulate energy in zones of residual stress and discharge them during fracturing. The cost of crushing accounts for a large proportion of the iron powder production costs.

Besides being a greenhouse gas and a major component of the carbon cycle in nature, carbon dioxide (CO_2) is a substance of major industrial importance [3]. Numerous industrial processes use CO_2, but the total amount of CO_2 used in industry is equivalent to about 2% of the CO_2 emitted globally by power plants into the atmosphere; it is non-toxic, non-corrosive and non-abrasive. Furthermore CO_2 is non-conducting and chemically inert. It is used for cryogenic cooling; separation technologies; chemical extractions; polymerization; powder processing; spray painting and coating; and various chemical reactions, among other processes. Liquid CO_2 blasting is a physical blasting method developed in the early 1960s in the United States and other developed countries, and the first cardox blaster was successfully developed by the US Aiduokesi Company [4–6]. The technology is based on the rapid and large volume expansion characteristics when liquid CO_2 is converted into a gas, and rock breaking could occur because of the stress wave generated by the high-pressure gas. Other countries such as England, France, Russia, Poland and Norway have also developed this method. The blasting of liquid CO_2 is non-explosive, and it accounted for 20% of total coal mining production in the United States in the early 1960s.

It is worth mentioning that CO_2 is the most widely used supercritical fluid because it has a relatively low critical value and a moderate critical pressure ($Tc = 31.0\ ^\circ\text{C}$, $Pc = 7.38$ MPa). It has unique features such as liquid-like density and gas-like diffusivity, which are important for the production of micron-sized particles of iron ore. Several methods have been broadly reviewed and implemented [7–10], including the rapid expansion of supercritical solutions (RESS), solution enhanced dispersion by supercritical fluid (SEDS), supercritical solvent impregnation (SSI), supercritical fluid extraction of emulsions (SFEE), gas antisolvent crystallization (GAS), supercritical antisolvent precipitation (SAS), precipitation by compressed antisolvent (PCA), an aerosol solvent extraction system (ASES) and particle from gas-saturated solutions (PGSS).

Liquid CO_2 blasting is considered physical blasting. The core concept underlying this technology is the blasting system, which consists of a high-pressure chamber containing an inlet control valve, seal cover and sealing ring, heating apparatus, rupture disk, and release holes following the order from top to bottom. A high-pressure chamber filled with liquid CO_2 is placed in the borehole, and the heater apparatus heats

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the liquid CO_2. The pressure in the high-pressure chamber increases rapidly, and as soon as the pressure of liquid CO_2 in the high-pressure chamber exceeds the shearing strength of the rupture disk, the rupture disk is damaged and high pressure liquid CO_2 will be released at high speed and will crush the nearby rock. This process brings about the phase change of liquid CO_2 and then causes a dramatic expansion in the volume of liquid CO_2. No sparks appear during the blasting process or the transport process, and both the storage and use of the materials are convenient. By utilizing the phase change energy of CO_2 to break the rock mass, Instantaneous high temperature is necessary. Compared with general blasting, the blasting of liquid CO_2 has the characteristics of slow expansion and steady shearing, and it is suitable for brittle rock. The blasting pressure of liquid CO_2 is lower than that of explosives, about one-third that of black powder and one-sixth that of ammonium nitrate explosives [11]. Black powder consists of KNO_3, S, and C.

Conventional techniques used for the production of micron-sized particles of iron ore are mostly based on mechanical methods such as crushing, grinding, milling and other processes. Powder characteristics are mainly determined by the average grain size, size distribution and particle shape [12–14], and new types of iron powders with special and improved properties have been developed and have conquered the market. At present, there are generally two production methods that together account for >90% of the global production of iron powders, viz. the Hoganas sponge iron process and the water-atomizing process. The industrial production of iron powder started in 1937 based on the incentive of the General Motors Corporation in the USA [15,16].

Aluminum was not "discovered" until 1808 (about 200 years ago), although early civilizations used aluminum-bearing clays to make pottery, and aluminum salts were used to make dyes and medicines [17]. Almost all commercial aluminum and aluminum alloy powders or granulates that are used in powder metallurgy (PM) production are made through the dispersion of the molten phase by atomization [18], centrifuging or granulation [19]. Other ultrafine powders are produced by high-pressure gas atomization or ultrasonic gas atomization. For example, the Hartman Shock Wave Tube [20] that generates shock waves with ultrasonic frequencies and supersonic gas velocity to produce the finest powder with a median grain size in the range 10–20 μm is well established.

Interest in synthesizing copper powder has increased considerably during the past few years because of its high electrical conductivity, relatively higher melting point, low electrochemical migration behavior and low material cost [21,22]. Granular copper powder can be produced by a number of methods, e.g., atomization, oxide reduction, electrolysis and hydrometallurgy [23]. Of the above methods, atomization and oxide reduction are presently applied on a large global scale. Each method yields a powder with certain inherent characteristics.

Cement is a fine powder that is the principal strength-giving and property-controlling component of concrete [24]. Cement is made by crushing and grinding calcium carbonate (limestone), silica (sand), alumina and iron ore in appropriate proportions and heating the resulting mixture in a kiln to approximately 1500 °C. Common materials used to manufacture cement include limestone, shells, and chalk or marl combined with shale, clay, slate, blast furnace slag, silica sand, and iron ore. These ingredients, when heated at high temperatures, form a rock-like substance that is ground into the fine powder that we commonly think of as cement.

The production of iron powders, aluminum alloy powders, copper powders and cement utilizes mechanical and chemical methods such as crushing, grinding, milling and heating. The first observation related to the possibility of obtaining ultrafine powders through supercritical fluid (SF) processing was made in 1876 [25]. The production of particles by rapid expansion of liquid CO_2 was first noted over thirty years ago [26]. The application of liquid CO_2 as an alternative to these conventional crushing processes has been an active field of research and innovation. The main motivation for this is the possibility of exploiting the peculiar properties of liquid CO_2, such as its gas-like diffusivity, liquid-like density and low-viscosity [10], which are especially significant for micron-sized particle production processes. Moreover, these properties can easily be obtained with changes in pressure and temperature [27].

In this paper, we developed a new alternative method for the production of micron-sized particles of iron ore by rapid unloading of liquid CO_2; the grain size of the iron ore particle is between 30 and 50 μm, and 90% of the grains are smaller than 98.5 μm.

2. Introduction of rapid unloading of liquid CO_2 used in the production of micron-sized iron ore particles

The rapid unloading of liquid CO_2 is as follows: the whole process is carried out in a high-pressure chamber, which consists of a rupture disk, seal cover and sealing ring, inlet control valve and needle valve. The rupture disk is located at the end of the high-pressure chamber. The seal cover and sealing ring not only avoid liquid CO_2 leakage but also ensure the installation of the rupture disk.

Currently, the overall process includes CO_2 liquefaction, CO_2 filling, CO_2 substitution, high pressure initialization, unloading and CO_2 gasification.

First, iron ores are placed in the high-pressure chamber, and after sealing, liquid CO_2 is injected. The injection temperature and pressure are controlled at −10 °C and 3 MPa, respectively, in order to ensure that the injected CO_2 is liquid and energy consumption is minimal. As indicated in a previous study of CO_2 liquefaction characteristics [28], the total power of the liquefaction device, which is combined with the compression and refrigeration components, is smallest at a 3-MPa compression pressure, and the transition temperature of CO_2 at that pressure is approximately −10 °C. The most economical process of CO_2 liquefaction is to first compress the CO_2 gas until a 3-MPa pressure is reached and then to cool the gas to cause the CO_2 to condense. If the liquid CO_2 is produced at the same site as particle production, the most economical option is to keep the liquid CO_2 at the T and P under which it was produced. In this way, the economically optimal CO_2 injection process occurs at −10 °C and 3 MPa.

After approximately 20–30 s, the saturated penetration time of the liquid CO_2 under 3 MPa is reached. Second, dissociative liquid CO_2 between the iron ore will be substituted by water and collected for cyclic utilization. It is emphasized that the water pressure is slightly higher than 3 MPa. Third, higher pressure is applied to the water inside the chamber through the water pump in order to increase the energy input. Fourth, the liquid CO_2 is rapidly unloaded.

The liquefaction process of CO_2 includes controlling the pressure and temperature; based on the physical characteristics of CO_2, when temperature and pressure are controlled at −10 °C and 3 MPa, respectively, economic benefits are maximized. The density of CO_2 under different pressures and temperatures is illustrated in Table 1.

For the purpose of recycling the liquid CO_2, several procedures relating to CO_2 substitution should be carried out. Dissociative liquid CO_2 can be replaced by water, and liquid CO_2 inside the iron ore does not spread out during the CO_2 substitution process. Based on the porosity of the iron ore, about 95% of the dissociative liquid CO_2 can be substituted. The CO_2 substitution apparatus is illustrated in Fig. 1.

First, the outlet of the high-pressure chamber is equipped with a needle valve to control the amount of CO_2 substitution and the sealing process.

Second, the initial filling pressure of the liquid CO_2 in the high-pressure chamber is approximately 3 MPa; the saturated penetration time of the iron ore is approximately 20 s, illustrated in Fig. 2. We performed a penetration test of the iron ore. After the iron ore is placed in the sealed container, the high pressure liquid CO_2 is injected at one end of the sealed container, illustrated in Fig. 3. The discharge of liquid CO_2 is observed at the other end of the sealed container, illustrated in Fig. 4. When the discharge of liquid CO_2 becomes stable, it is believed that the iron ore reaches infiltration saturation. According to the

Table 1
Density of CO_2 under different pressure and temperature.

T(℃) \ P (Mpa)	0.1	1	2	3	4	5	6	7	8	9	10
-10	🟧 2.03	🟧 21.9	🟧 48.8	🟦 985	🟦 991	🟦 996	🟦 1002	🟦 1007	🟦 1012	🟦 1016	🟦 1021
0	🟧 1.95	🟧 20.8	🟧 45.6	🟧 77.3	🟦 932	🟦 941	🟦 948	🟦 955	🟦 962	🟦 968	🟦 974
10	🟧 1.88	🟧 19.9	🟧 43	🟧 71	🟧 108	🟦 869	🟦 882	🟦 893	🟦 903	🟦 912	🟦 920
20	🟧 1.82	🟧 19.1	🟧 40.8	🟧 66.2	🟧 97.5	🟧 141	🟦 783	🟦 809	🟦 828	🟦 843	🟦 856
30	🟧 1.75	🟧 18.4	🟧 38.8	🟧 62.2	🟧 89.8	🟧 124	🟧 171	🟧 267	🟦 702	🟦 744	🟦 772

🟧 G 🟦 L

equation $K = 2000\frac{P_{atm}\mu Q_a L}{A(P_1^2 - P_2^2)}$($P_{atm}$—atmospheric pressure; μ—viscosity; Q_a—velocity; L—length of iron ore; A—cross-sectional area; P_1—import pressure; P_1—outlet pressure), the permeability of the iron ore at 15 MPa is $1 * 10^{-6}$ μm^2.

Third, after saturated penetration, the water pump starts to run. When the water pressure is slightly higher than 3 MPa, we need to open the inlet control valve and the needle valve of the high pressure chamber simultaneously to substitute liquid CO_2; when water flows slowly from the needle valve, the inlet control valve and needle valve should be closed. The whole process of liquid CO_2 substitution is accomplished. The results confirm that the grain size of the iron ore particles after liquid CO_2 substitution is slightly larger than that resulting from direct powder production but is similar to that resulting from the conventional crushing methods.

The total energy input is related to the grain size of the iron ore powder. The higher the energy input, the greater the pressure of the liquid CO_2 stored in the iron ore. A higher pressure gradient is generated when gasification occurs, and a finer iron ore powder is obtained. In order to increase the input energy, water pressure is applied through the inlet control valve by the water pump after CO_2 substitution; the high water pressure apparatus is illustrated in Fig. 5. Because the compression coefficient of water is small, a high initial water pressure (50 MPa, 80 MPa) is easily reached.

When the pressure increases, the rupture disks will burst. The time of rupture disk bursting is approximately 5–8 ms, and the high pressure is reduced to zero during this time. During the unloading process of the liquid CO_2, the gasification boundary of liquid CO_2 is in a certain position within the iron ore where the gasification pressure is maintained at 3 MPa, and the iron ore body between the outer surface and gasification boundary is then converted into micron-sized iron ore particles. Subsequently, the gasification boundary gradually moves inwards, and finally, the iron ore body is turned into powder layer by layer. Under the high pressure gradient, micron-sized iron ore particles are obtained.

3. Implementation

3.1. Model test for micron-sized particles of iron ore

In addition to the above procedures (CO_2 liquefaction, CO_2 filling, CO_2 substitution, high pressure initialization, unloading and CO_2 gasification), the model test includes the collection of iron ore powders. Different types of model tests have been carried out, including direct powder production, powder production after CO_2 substitution, and powder production after CO_2 substitution and high pressure initialization.

The iron ore is weighed before being placed in the high-pressure chamber. The equivalent diameter of the iron ore is <60 mm because the inner diameter of the high pressure chamber is 63 mm. The inner volume of the high pressure chamber is approximately 0.01 m^3, and according to the volume ratio of densely packed particles of equal diameter, the porosity is approximately 35–40%; therefore, the mass of the iron ore is about 7 kg. After sealing, CO_2 filling, liquid CO_2 substitution and high water pressure initialization, the high pressure chamber is placed in the collection container to effectively collect the iron ore powder; the apparatus is illustrated in Fig. 6. Eventually, the rupture disks burst, and the iron ore powder sprays into the collection container.

3.2. Grain size and grain size distribution measurements

Grain size is a key factor that determines the performance of different organic and inorganic materials. Data analysis indicates that both the average grain size and the grain size distribution (GSD) are related to the initial water pressure and the liquid CO_2 substitution. The direct powder production result is illustrated in Fig. 7. The grain size of the iron ore particles ranges from 30 μm to 50 μm, and grain sizes <98.5 μm account for 90% of the grains, as illustrated in Fig. 8. The comparison between the iron ore powder under the condition of direct powder production and powder production after CO_2 substitution is illustrated in Table 2. The grain size of the iron ore after liquid CO_2 substitution includes more coarse powders of iron ore and a few fine powders. From the experiment of direct powder production, the percent grain size <43 μm accounts for 40%, which makes it too fine for the gravity separation, magnetic separation and flotation processes. Based on the experiment of powder production after CO_2 substitution, the percent grain size <147 μm accounts for 60%, which is suitable for gravity separation, magnetic separation and flotation processes. In summary, the micron-sized particles of iron ore produced by powder

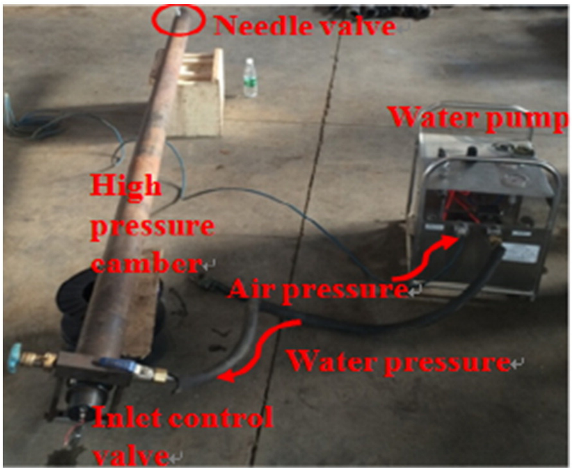

Fig. 1. CO_2 substituting apparatus.

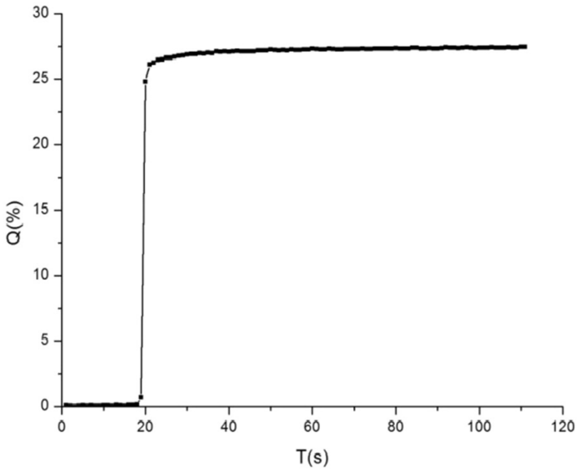

Fig. 2. Saturated penetration time of iron ore.

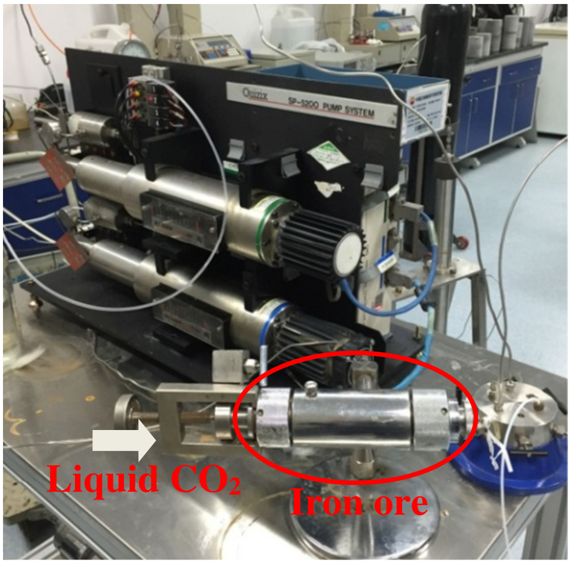

Fig. 3. Penetration test of the iron ore.

production after CO_2 substitution are ideal because of the optimized grain size distribution and low cost.

The grain size distribution of the iron ore powder under different initial water pressure is illustrated in Fig. 9. The initial water pressures are 50 MPa (explosive 300 g), 50 MPa (explosive 400 g), 80 MPa (explosive 200 g), and 80 MPa (explosive 440 g). Fewer particles larger than 297 μm and more particles smaller than 147 μm will be obtained under high initial water pressure. It is well accepted that a grain size smaller than 147 μm accounts for the main component when the initial water pressure reaches 80 MPa. The initial water pressure provides high energy to the liquid CO_2 stored in the iron ore; therefore, when the rupture disk is broken, there is a large volume expansion, and more small-sized iron ore powders will be obtained. Based on Fig. 9, the percent grain size <147 μm accounts for 80% when the initial water pressure is 80 MPa, compared with 50% when the initial water pressure is 50 MPa. The effect of the initial water pressure is greater than the amount of the explosive in the powder production of the iron ore, and based on the above conclusion, the explosive can gradually be replaced by a high initial water pressure.

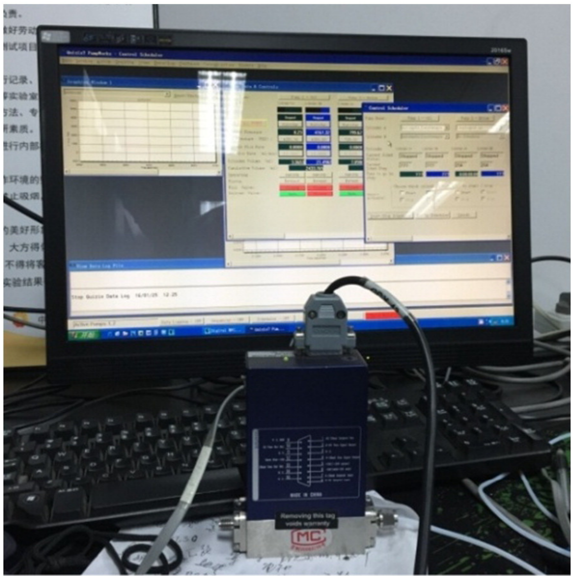

Fig. 4. Discharge of liquid CO_2 monitoring.

Fig. 5. High pressure initialization apparatus.

4. Reliability and reusability

In order to verify the reliability and the reusability of the powdering method, a series of experiments on iron ore powdering were carried out, including hematite, refractory iron ore, low-grade iron ore, and magnetite powdering, as illustrated in Fig. 10. In addition to iron ore powdering by unloading of liquid CO_2, bauxite and limestone powdering experiments were also carried out, and the particles obtained are illustrated in Fig. 11. The grain size of the bauxite and limestone particles ranged from 4 μm to 6 μm and from 10 μm to 20 μm, respectively, as illustrated in Fig. 12. The results confirmed that obtaining

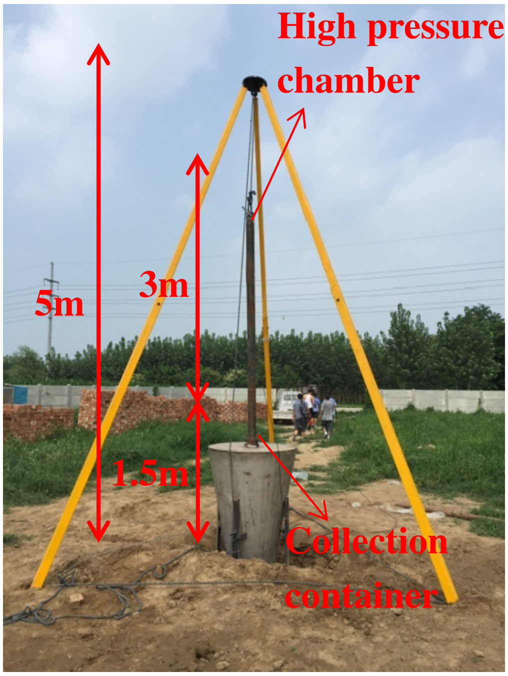

Fig. 6. Platform for iron ore powder production.

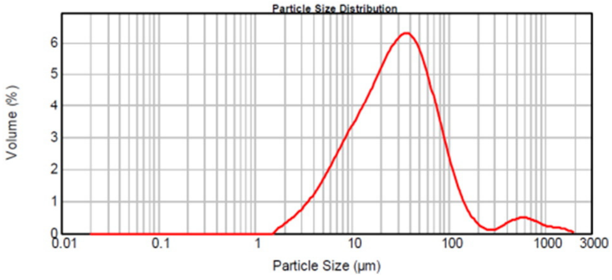

Fig. 7. Grain size distribution of iron ore particles.

Table 2
Grain composition of the iron ore particles.

Diameter of the powder (μm)	Direct powder production (explosive 440 g)	Powder production after CO_2 substituting (explosive 440 g)
+50 (297)	14.99	40.57
+100 (147)	7.47	14.57
+150 (104)	9.43	11.61
+200 (74)	14.66	13.31
+260 (51)	10.24	7.02
+325 (43)	4.99	2.36
−325 (<43)	38.22	10.56
Total	100.00	100.00

micron-sized powder by rapid unloading of liquid CO_2 is feasible. In addition, the powdering method is green and energy efficient.

5. Analysis of economical benchmarking

The current cost of iron ore powder produced by ball milling in China is approximately 5.7 US dollars per ton of iron ore. The estimated cost of the rapid unloading procedure is approximately 1.0 US dollars per ton. The cost of the rapid unloading procedure has two components: power consumption and wear.

Power consumption is a linear function of liquid CO_2 usage. The power consumed during liquid CO_2 production is approximately 175 KWh per ton. The amount of liquid CO_2 used to produce iron ore powder varies based on the scale of the production. If the container scale has a radius of 1 m and a height of 10 m and 95% of the dissociative liquid CO_2 can be recycled, then the average usage of liquid CO_2 is 15 kg per ton of iron ore. Another source of power consumption is the pumping system. The average pumping cost for one ton of iron ore is approximately 0.7 KWh. The total power consumption for the processing of one ton of iron ore is $175\ \text{KWh/ton} * 0.015\ \text{ton} + 0.7\ \text{kWh} = 3.3\ \text{KWh}$. The power price in China is 0.15 US dollars per KWh for industry usage. The power consumption is estimated as 0.5 US dollars per ton.

The wear cost is also affected by the scale of the production. Because the rapid unloading procedure does not have a liner cost in the current production procedure, the main wear cost is the rupture of disks. By using a variable-diameter outlet design, the size and thickness of the rupture disks could be reduced. The estimated wear cost is 0.52 US dollars per ton of iron ore.

6. Discussion

Rapid unloading of liquid CO_2, as demonstrated, can produce micron-sized particles of iron ore. This process is different from conventional methods, and the grain size distribution can be regulated by the controllable methods of the initial water pressure and CO_2 substitution. From the two briefly illustrated cases, the production of micron-sized particles of iron ore can evidently be employed advantageously. It should be pointed out that the method is an effective way to improve efficiency and save energy.

Based on the comparison of direct powder production and powder production after CO_2 substitution, there is now a general consensus that the grain size of iron ore powder after CO_2 substitution is larger than that resulting from direct powder production. In this case, further studies are needed to determine the extent of CO_2 substitution, which is the basis for the optimization of iron ore powder grading. However, what has not been delineated is the phase change of CO_2 at the time the rupture disk opens. It is well accepted that a pressure of 3 MPa is the gas-liquid conversion limit point, but whether the gas-liquid conversion interface of iron ore is the dominant factor needs to be determined, in other words, how does the gas-liquid conversion interface affect iron ore powdering? Among the questions that remain to be resolved is the process of iron ore powdering. Because the whole powdering process occurs inside a high-pressure chamber, only a pressure-time history curve can be obtained. To further determine the mechanism of iron ore powdering, X-rays or a transparent chamber deployed with a high-speed camera need to be adopted. The current iron ore powdering production experiments are under the combined effect of explosives and liquid CO_2. The experiments have indicated that the effect of explosives on powdering is not significant. Therefore, we can gradually increase the initial water pressure and reduce the use of explosives until no explosives are used. Optimization of the powdering process is likely to reduce the cost of iron ore powder production.

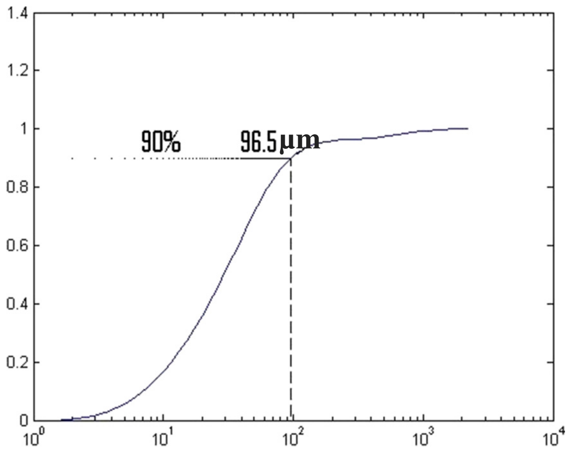

Fig. 8. Cumulative distribution of iron ore particles.

7. Conclusions

The results of the laboratory-scale experiments indicate that it is possible to produce micron-sized particles of iron ore using controllable methods of CO_2 substitution and initial water pressure.

The production of iron ore particles by rapid unloading of liquid CO_2 is a truly original approach, which is based on the physical characteristics of liquid CO_2, such as low viscosity, high permeability, and a high dilation coefficient. This approach is different from the traditional mechanical methods that use crushing, grinding and milling. Iron ore powder production after CO_2 substitution is an effective method, which not only optimizes the grain size distribution but also saves 95% of the liquid CO_2. The initial water pressure provides high energy to the liquid CO_2 stored inside the iron ore. The higher the initial water pressure, the higher the number of fine particles obtained. The effect of the initial water pressure is greater than that of the explosives in the powder production of the iron ore, and breaking through the rupture disk is the main effect of the explosives. Based on the above conclusion, the explosives can gradually be replaced by the initial water pressure.

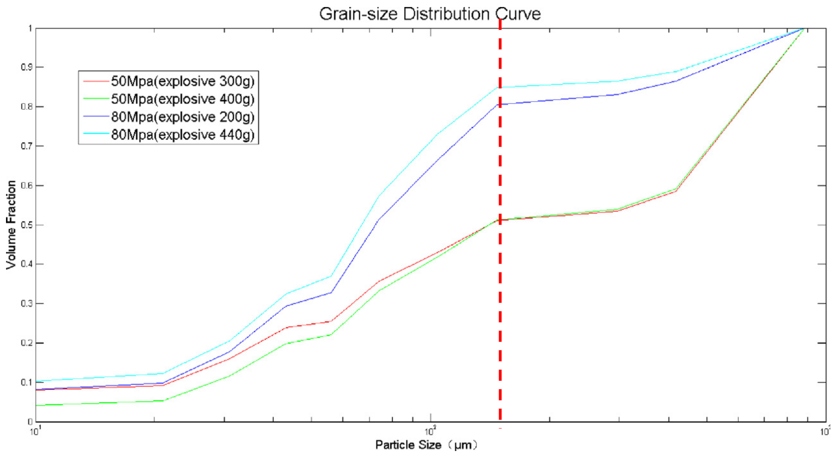

Fig. 9. grain size distribution curve of iron ore particles.

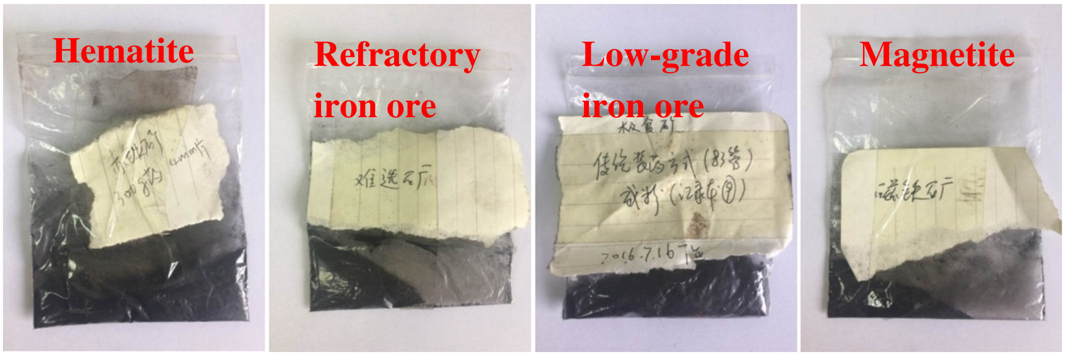

Fig. 10. Different types of iron ore powder.

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Fig. 11. Bauxite and limestone particles.

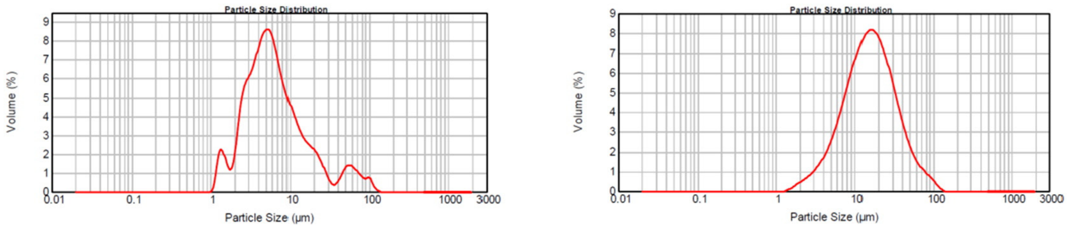

Fig. 12. Grain size distribution of bauxite and limestone particles.

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