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# Waste Corn Straw as a Green Reductant for Hematite Reduction Roasting: Phase Transformation, Microstructure Evolution and Process Mechanism

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**Abstract:** Mineral phase transformation (MPT) of hematite to magnetite by reduction roasting is a viable means of developing refractory iron ore resources. However, conventional coal-based reductants are prone to high carbon emissions and environmental pollution. Biomass, as a renewable green reductant, can make the MPT process more environmentally friendly while reducing the environmental impact associated with processing agricultural waste. This study systematically explored the feasibility of waste corn straw as a green reductant for hematite. Under the conditions of 8 min, 700 °C, a mass ratio of corn straw to hematite of 1:4, and a $N_2$ flow rate of 300 mL/min, the best beneficiation indexes were achieved, with an iron grade of 69.82% and an iron recovery of 93.95%. During the MPT process, hematite was reduced under the action of corn straw, and the new magnetite particles were loose and porous, showing an acicular crystal structure. Meanwhile, the corn straw was converted into porous biochar.

**Keywords:** corn straw; green reductant; mineral phase transformation; iron recovery; microstructure evolution

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## 1. Introduction

Iron ore, as the basic raw material in steelmaking, plays an indispensable role in the iron and steel industry [1]. With the explosive growth of steel demand worldwide, especially the rapidly expanding steel production in China, the supply of high-quality iron ore has been severely limited while the exploration and exploitation of refractory iron ore has become urgent [2]. However, traditional beneficiation techniques based on density, magnetic properties, and surface hydrophobicity pose difficulties to processing low-grade iron ores with more complex mineralogical assemblage, especially iron ores in China [3]. An effective solution is to realize the mineral phase transformation (MPT) of weakly magnetic iron minerals into strongly magnetic minerals via reduction roasting [3,4]. The roasted products can then be effectively beneficiated and upgraded through low-intensity magnetic separation.

For many years, considerable research has investigated the reduction roasting of iron ore, including: thermodynamic possibilities [2,4], kinetic mechanisms which control the process [5–7], and optimization of process parameters [6,8,9], etc. Practice shows that reduction roasting-magnetic separation can achieve excellent process indexes and obtain high-quality ironmaking raw materials from refractory iron ore resources [10,11]. Traditionally, the process has used carbon-based reductant such as coke, anthracite, and carbon monoxide. However, today this intensifies the pressure on the steel industry to further reduce carbon emissions and minimize environmental impact. To meet these challenges researchers began to explore the use of green, low-carbon, and environment-friendly reducing agents such as siderite, hydrogen, and biomass in reduction roasting [1,12,13]. These alternatives aim to make the reduction roasting process more sustainable while maintaining or improving process efficiency.

Biomass, as a renewable organic reductant, presents a promising opportunity to reduce the carbon footprint of reduction roasting and add value to agricultural waste. Ongoing research continues to optimize biomass types and roasting conditions to realize the full environmental and economic benefits. Cao et al. [13] successfully realized the magnetization roasting of hematite with straw-type biomass, achieving an iron recovery of more than 90% by magnetic separation. Wang et al. [14] discovered that biomass can be used, not only as a reductant in the magnetization roasting of red mud, but also to improve cementitious activity in tailings. Deng et al. [15] explored the effects of different types of biomass on magnetization roasting of iron tailings, and found that woody biomass is better than straw biomass, and when fir sawdust was the reductant, iron concentrate with an iron grade of 62.04% and an iron recovery of 95.29% could be obtained. Qiu et al. [16] further verified the feasibility of using fir sawdust as a reductant in iron tailings, and pointed out that the biomass oil generated during magnetization roasting would affect the operation of industrial equipment. In addition, sawdust [17,18], banana tree bark [19], leaf litters [19,20], etc., have been verified as green reductants that can be used for the reduction roasting of iron minerals, and can obtain good beneficiation indicators. Furthermore, these studies show that the reducing gases produced by pyrolysis during biomass roasting are mainly CO and $H_2$, which are the main reductants for the reduction of iron minerals [13,15,16].

The utilization of biomass as a green reductant in reduction roasting is an appealing approach. Kinetic data plays a pivotal role in the advancement and optimization of chemical reactions for industrial applications [21,22]. However, current kinetic studies in this area lack remarkable depth. Yuan et al. [23] comparatively examined the isothermal reduction kinetics of iron ore pellets using coke, charcoal, and biomass and indicated that biomass displayed greater reactivity. Liu et al. [24] explored the kinetics of sawdust reduction of pure $Fe_2O_3$, and determined that the kinetic mechanism was a first-order reaction model, but they only used a simple model fitting method. Wang et al. [14] centered on the pyrolysis kinetics of different biomasses, but failed to consider the mixed system involving iron minerals. Overall, the current kinetic data appears to focus more on the pyrolysis kinetics of biomass in the roasting process, or fails to pay attention to the roasting process of powder materials, with relatively basic kinetic research methods being employed.

To further deepen the mechanism of the hematite biomass reduction reaction, the potential application of corn straw as a green reductant for MPT of hematite was systematically investigated. The effect of fluidization roasting conditions on the beneficiation indexes and saturation magnetization is discussed. The phase transformation during the MPT process was analyzed by X-ray diffraction (XRD) and in-situ XRD. The microstructure evolution of corn straw and hematite was investigated using scanning electron microscopy and energy dispersive spectroscopy (SEM-EDS). Detailed kinetic studies will be carried out in a separate study on biomass pyrolysis during roasting, using advanced methods recommended by ICTAC.

## 2. Materials and Methods

### *2.1. Materials*

The corn straw used in the study was collected from Chaoyang City, Liaoning Province, China, and was crushed to less than 1 mm. After sieving, the corn straw was dried at 60 °C for 12 h. Table 1 presents the results of the preliminary, elemental, and constituent analyses of corn straw. The volatile content was 77.52%, while its fixed carbon content, moisture content, and ash content were 15.54%, 5.57%, and 1.37%, respectively. The most prevalent elements found in the corn were C, H, and O, accounting for 41.82%, 5.60%, and 48.6%, respectively. The main components of corn straw are cellulose (40.91%), hemicellulose (28.57%), and lignin (5.48%).

**Table 1.** Preliminary, elemental, and constituent analyses of the corn straw (%).

| Preliminary Analysis | | Elemental Analysis | | Biomass Constituent | |
|---|---|---|---|---|---|
| Moisture $^{ad}$ | 5.57 | C | 41.82 | Cellulose | 40.91 |
| Ash $^{ad}$ | 1.37 | H | 5.60 | Hemicellulose | 28.57 |
| Volatile $^{ad}$ | 77.52 | O | 48.60 | Lignin | 5.48 |
| Fixed carbon $^{ad}$ | 15.54 | N | 1.25 | Others | 24.92 |
| – | – | P | 0.034 | – | – |
| – | – | S | 0.07 | – | – |

$^{ad}$: based on air dried basis.

The hematite was sourced from Anshan City, Liaoning Province, China, and 70% of the hematite had a particle size of less than 0.074 mm. The composition of different iron minerals in the hematite samples was analyzed by chemical iron phase, showing that hematite or limonite accounted for 96.13% and magnetite accounted for 2.67%. The XRD pattern in Figure 1 indicates that the major mineral is hematite, with minor amounts of quartz and magnetite. Table 2 presents the results of the chemical composition analysis of the hematite sample. The sample was found to have a total iron (TFe) content of 67.47%, with an FeO content of 0.88%. The major impurities were determined to be $SiO_2$ and $Al_2O_3$, which were present at levels of 0.80% and 0.38%, respectively.

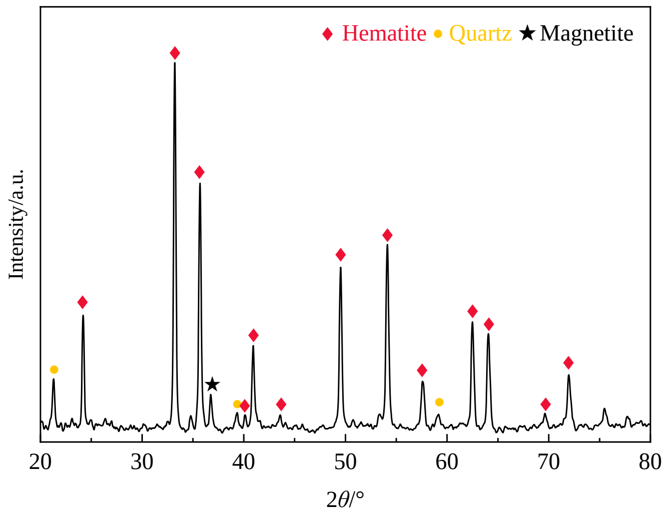


**Figure 1.** XRD analysis of the hematite sample.

**Table 2.** Chemical composition analysis of hematite sample.

| Component | TFe | FeO | $SiO_2$ | $Al_2O_3$ | CaO | MgO | Mn | $TiO_2$ | S | P |
|---|---|---|---|---|---|---|---|---|---|---|
| Content/% | 67.34 | 0.88 | 0.80 | 0.38 | 0.05 | 0.24 | 0.13 | 0.047 | <0.01 | 0.04 |

## *2.2. Methods and Equipment*

### 2.2.1. Experimental Apparatus and Procedure

The experiments in this study utilized suspension furnace and magnetic separation, as depicted in Figure 2. The experimental procedure started with heating the suspension furnace (OTF-1200X-S-VT, Hefei Kejing Materials Technology Co., Ltd., Hefei, China) and adjusting the temperature. Additionally, the experimental system was purified using $N_2$. Next, the raw sample consisting of different proportions of hematite and corn straw was placed on a perforated quartz plate located in the center of the furnace tube. During the MPT process, $N_2$ was used to suspend the sample particles. When the predetermined time was reached, the MPT was stopped and the roasted sample was cooled to room temperature in $N_2$. The cooled sample was subjected to magnetic separation using a Davis magnetic tube (XCQS73-Φ50, JILIN EXPLORATION MACHINERY PLANT, Jilin, China) with a magnetic field of 198 mT, and a water flow of 500 mL/min. All experimental conditions were repeated three times and averaged to obtain errors.

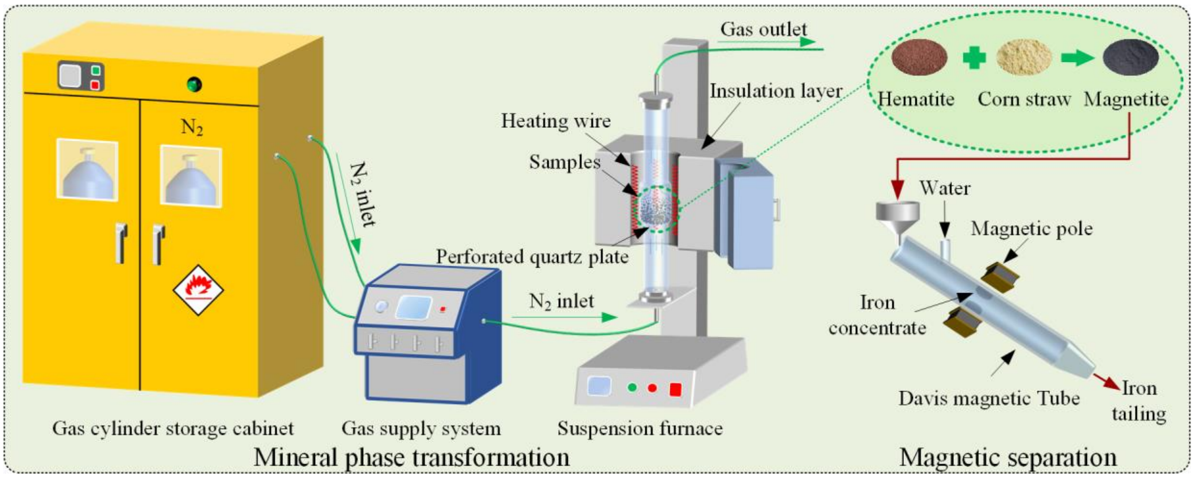


**Figure 2.** Schematic of hematite mineral phase transformation test with corn straw as green reductant.

2.2.2. Characterization Methods

The content of the chemical component in the sample was determined using titration and X-ray wavelength dispersive fluorescence spectrometry. The phase composition of iron was determined by iron chemical phase analysis [25]. The saturation magnetization of the samples was analyzed using a vibrating-sample magnetometry (VSM, JDAW-2000D, LAKESHORE, Columbus, OH, USA). XRD analysis was performed using an X'Pert pro MRD diffractometer (PANalytical B.V., Almelo, The Netherlands) at a scan rate of 12°/min. And the in-situ XRD analysis was performed using a Rigaku Smartlab diffractometer (Rigaku Corporation, Tokyo, Japan) at a scan rate of 15°/min with a heating rate of 20 °C/min. Microstructural evolution was studied by SEM-EDS analysis (Thermo Scientific Apreo 2C, Thermo Fisher Scientific Inc., Waltham, MA, USA; Oxford Ultim Max 40, Oxford instrument, Abingdon, Oxfordshire, UK). The thermogravimetric analysis was performed using an STA 449F3 thermal analyzer (NETZSCH Group, SELB, Bavaria, Germany) under a nitrogen atmosphere.

## 3. Results and Discussion

### *3.1. Effect of Roasting Conditions*

The effects of the main parameters during the roasting process on iron recovery, and the saturation magnetization of the roasted samples were investigated, as shown in Figure 3.

Optimizing the roasting time can lead to improved separation performance while keeping energy consumption under control. The roasting time was optimized at a temperature of 700 °C, a mass ratio of corn straw to hematite of 1:3 and a $N_2$ flow rate of 500 mL/min. As depicted in Figure 1a, a significant increase in both iron grade and recovery was observed from 2 to 6 min, with an increase of 2.30% and 28.42%, respectively. After 6 min, the iron grade continued to improve slightly while the iron recovery gradually decreased. Meanwhile, the saturation magnetization first increased significantly and then decreased slightly. This was thought to be caused by the transformation process of hematite and limonite to magnetite to wustite [13]. The saturation magnetization reached a maximum value of 75.37 A·m$^2$/kg at 8 min, corresponding to the best beneficiation indexes. Therefore, the roasting time was set at 6 min, resulting in an iron grade and recovery of 69.75% and 93.65%, respectively.

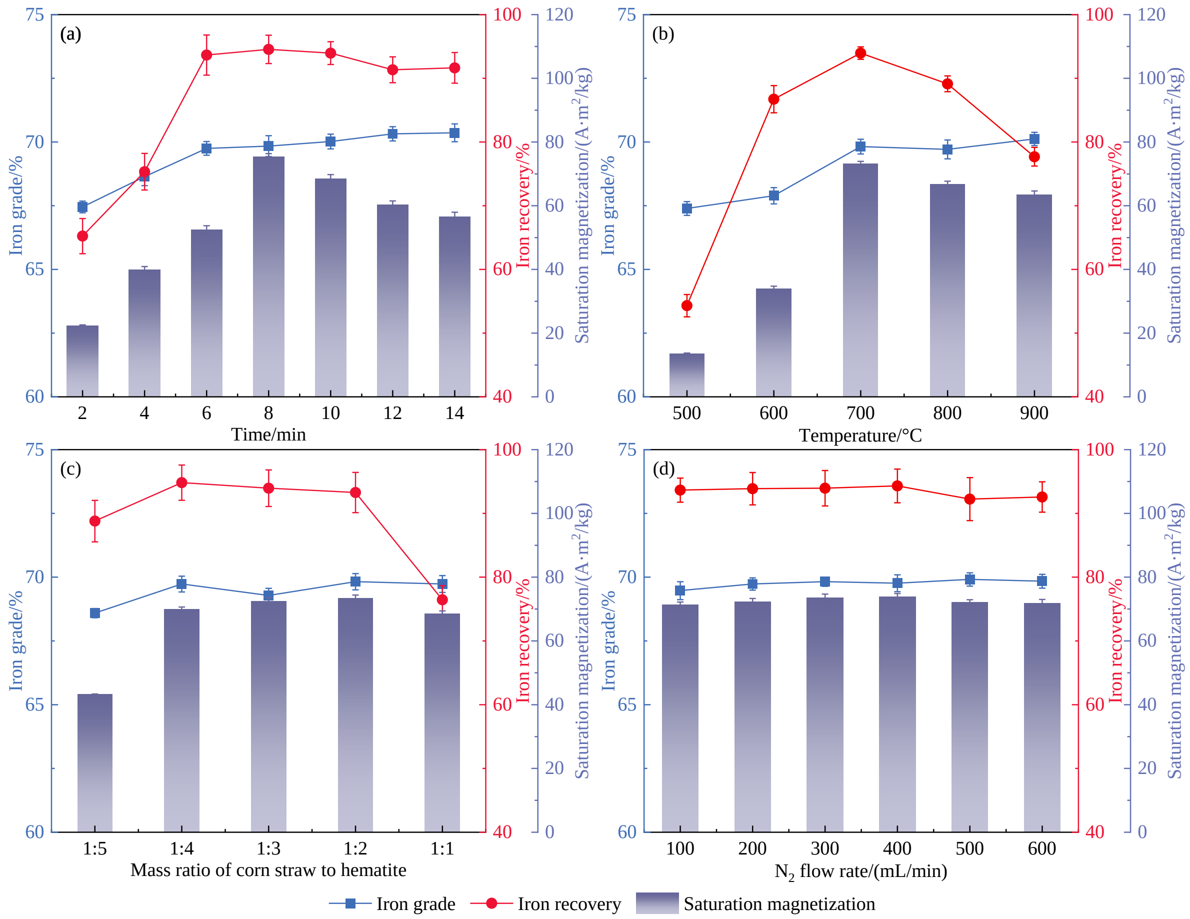


**Figure 3.** Effect of roasting conditions on magnetic separation performance and saturation magnetization, (**a**) roasting time, (**b**) roasting temperature, (**c**) mass ratio of corn straw to hematite, (**d**) $N_2$ flow rate.

Temperature is an important factor affecting corn straw pyrolysis and hematite reduction [13]. After setting the roasting time to 6 min, the effect of roasting temperature was explored, as shown in Figure 1b. The iron grade increased from 67.39% at 500 °C to 69.82% at 700 °C, but then reached a plateau. Iron recovery, on the other hand, showed a single peak of 93.95% at 700 °C. This indicates that increasing the temperature promoted the reduction of hematite. However, at higher temperatures, the pyrolysis of corn straw was more intense, resulting in the reduction of hematite to antiferromagnetic wustite, which affected the separation performance [13]. This also brought about a trend, that the saturation magnetization increased first and then decreased, and the peak value of 75 was obtained at 700 °C. Thus, the optimum roasting temperature was determined to be 700 °C, with an iron grade and recovery of 69.82% and 93.95%, respectively.

In the MPT process, corn straw acts as a reductant. However, when the dosage of corn straw is low, complete reduction of hematite to magnetite will not occur. Conversely, at higher dosages, excess corn straw is wasted and can lead to increased $CO_2$ emissions. Figure 1c shows that for mass ratio between 1:5 and 1:2, the iron grade fluctuated between 69.28% and 69.82%, and the iron recovery was between 93.27% and 94.82%. However, increasing the corn straw dosage to 50% (mass ratio of 1:1) resulted in a significant decrease in both the iron grade and recovery. This suggests that the reductants produced by the pyrolysis of corn straw exceeded the amount required for the reduction of $Fe_2O_3$ to $Fe_3O_4$. The saturation magnetization increased significantly only when the mass ratio was increased from 1:5 to 1:4, and the saturation magnetization did not change significantly when the dosage of corn straw continued to be increased. Therefore, the appropriate mass ratio of corn straw to hematite was determined to be 1:4, the iron grade obtained to be 69.82%, and the iron recovery to be 93.95%.

Figure 1d demonstrates that the iron grade remained stable as the $N_2$ flow rate increased, ranging between 69.47% and 69.84%. The iron recovery showed minimal change until the $N_2$ flow rate reached 400 mL/min, maintaining between 93.65% and 94.61%. However, when the $N_2$ flow rate was increased to 500 mL/min, the iron recovery slightly decreased. This was likely due to the high $N_2$ flow rate during fluidization process, which removed the less dense corn straw and the reducing gas produced by pyrolysis, resulting in poorer reduction of hematite. And the change of saturation magnetization explained the slight effect of $N_2$ flow rate change on the beneficiation indexes. Thus, the appropriate roasting gas flow rate is determined to be 300 mL/min, at which the iron concentrate grade was 69.82% and the iron recovery was 93.95%.

### *3.2. Phase Transformation*

The phase transformation during the MPT process was investigated using XRD and iron chemical phase analysis, and the results are shown in Figure 4.

Figure 4a illustrates the appearance of characteristic diffraction peaks of magnetite at 2 min. As the roasting time was increased to 8 min, the characteristic diffraction peaks of hematite disappeared, while those of magnetite gradually increased and became dominant. Further extension of the time to 10 min resulted in the appearance of characteristic diffraction peaks of wustite, indicating excessive reduction of hematite by corn straw [13,15]. Figure 4c reveals that the trending change in hematite content was consistent with that of the characteristic hematite diffraction peaks. Meanwhile, when the roasting time was 6 min, although a small amount of hematite was still present, the generated magnetite was sufficient to ensure the recovery of the roasted particles by magnetic separation [22,25]. These results were also consistent with the magnetic separation performance obtained.

Figure 4b,e illustrates the effect of temperature on the phase transformation. As the roasting temperature increased, the transformation process of hematite was like that of prolonging the roasting time. The magnetite content reached a maximum value of 88.23% at 700 °C. At high temperatures of 800 and 900 °C, the formation of wustite became more apparent. Based on this observation, the previously determined roasting temperature of 700 °C was considered reasonable.

During the MPT process, the dosage of corn straw was a critical factor in determining the concentration of reducing gas [16]. Compared with the time and temperature factors, the same phase transformation trend was observed (Figure 4c,f). Specifically, a 1:4 mass ratio of corn straw to hematite resulted in a magnetite content of 82.69%. This level of magnetite content was enough to achieve a satisfactory magnetic separation index. The results showed that the 1:4 mass ratio produced enough combined reduction gas to reduce most of the hematite to magnetite.

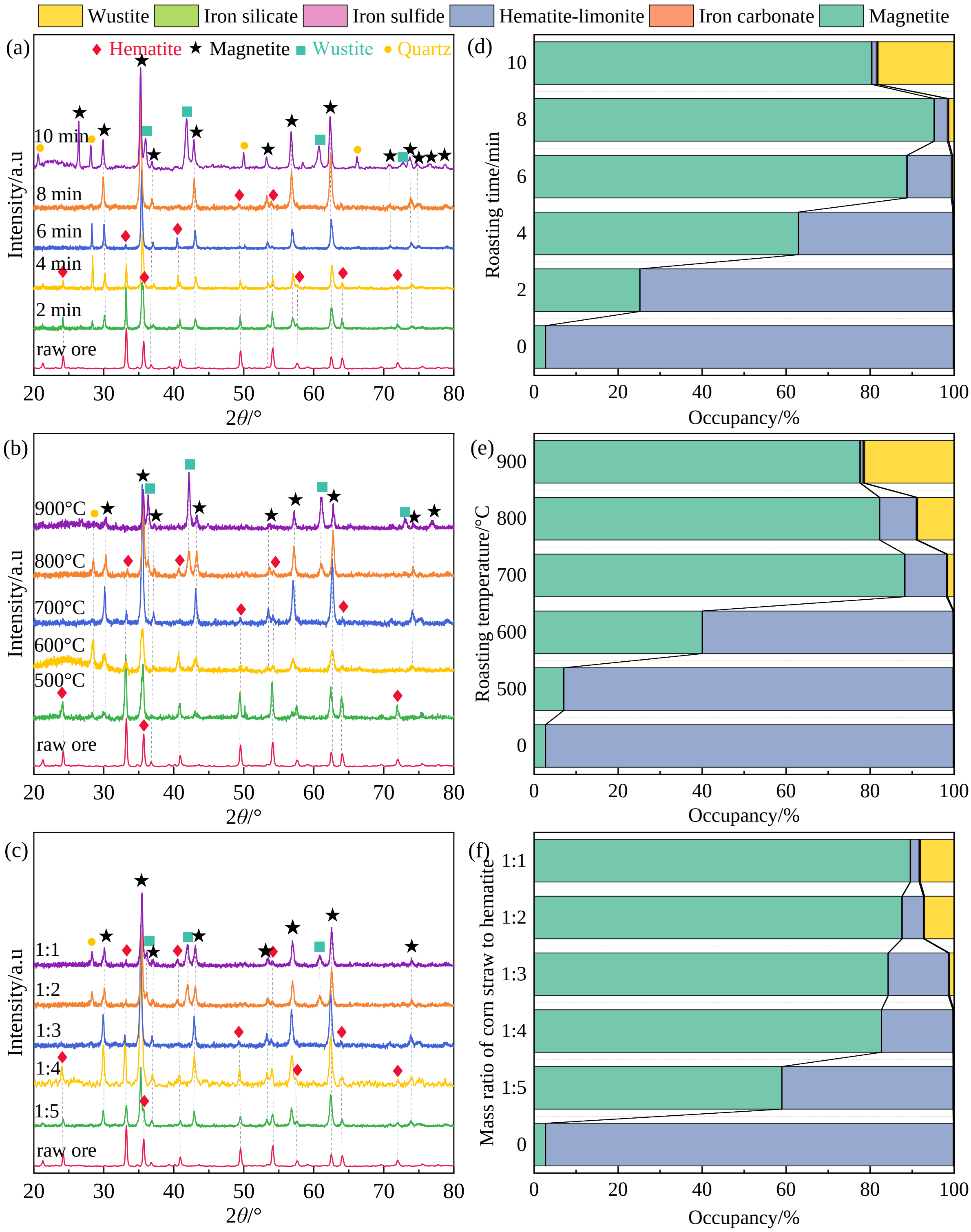


**Figure 4.** XRD pattern and iron phase composition of roasted samples: (**a**,**d**) effects of roasting time; (**b**,**e**) effects of roasting temperature; (**c**,**f**) effects of corn straw dosage.

To investigate the phase transformation of hematite during the MPT process, an in situ heating XRD analysis was carried out, and the results are presented in Figure 5. As the temperature increased, the characteristic diffraction peaks of hematite decreased while those of magnetite increased. The characteristic diffraction peaks of hematite were significantly weakened at 500 °C, indicating that the reduction of hematite occurred mainly before 500 °C. At 700 °C, magnetite became the dominant phase, indicating that most of the hematite had been reduced to magnetite. At 800 °C, over-reduction occurred, resulting in the formation of wustite [19]. The characteristic diffraction peaks of carbon were also observed, indicating that carbon with great crystallization was deposited on the particle surface [16]. These observations suggest that during the MPT process of corn straw and hematite, hematite was first reduced to magnetite, followed by the formation of wustite at higher temperatures.

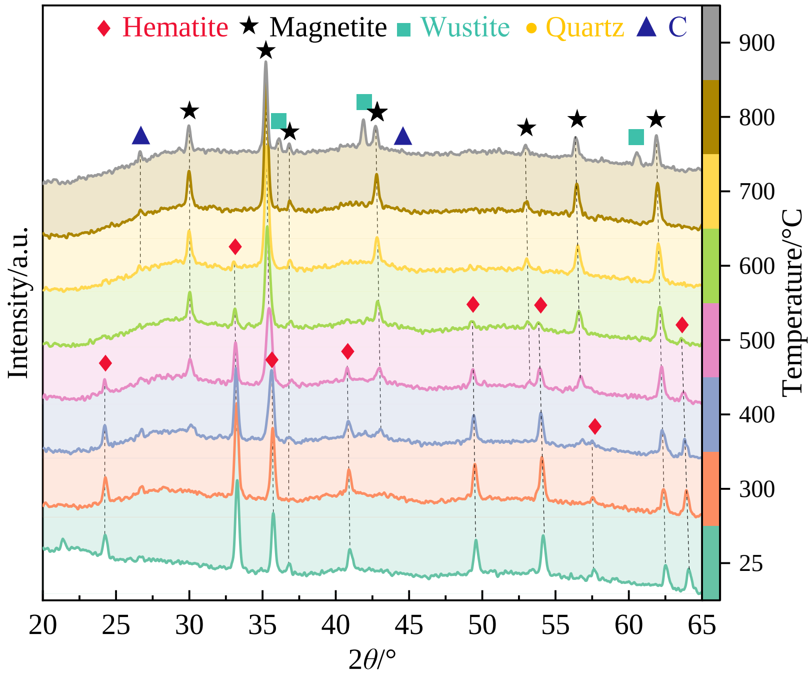


**Figure 5.** In situ heating XRD pattern of corn straw and hematite in $N_2$ atmosphere (heating rate: 20 °C/min).

*3.3. Microstructure Evolution*

The evolution of microscopic morphology before and after MPT of corn straw and hematite was explored by SEM-EDS, as shown in Figure 6.

Figure 6a illustrates the complex and diverse morphology of corn straw, showing fiber rods and irregular crumbs and flakes. Additionally, there are many regular cylindrical holes and crack-shaped holes within the corn straw. This natural porosity is conducive to the release of volatiles. As shown in Figure 6b,c, hematite was mainly irregularly granular, with a relatively flat and dense surface, and no holes or cracks inside.

As shown in Figure 6d–h, the morphology of corn straw was softened and deformed due to the release of volatiles after roasting. The surface of the corn straw became rougher, and the number of pores increased significantly with irregular characteristics. This change in morphology indicates that the deposited carbon participated in gasification reactions and was consumed, resulting in the formation of irregular porous biochar [16]. After MPT, the roasted particles exhibited visible irregular cracks and holes on their surface, while the newly formed magnetite exhibited a porous acicular structure. This is due to the thermodynamic instability of the magnetite formed after the reduction of hematite during crystal growth. Magnetite nuclei grow more easily along the magnetic axis, with lower surface energy and an acicular structure [5]. The observed results suggest that the gas produced during the pyrolysis of corn straw reacted with the hematite to form magnetite [13]. The temperature stress and phase transformation stress experienced during this reaction caused the initially dense structure of hematite to loosen, thus facilitating the entry of reducing gas molecules and leading to a further increase in the degree of reduction.

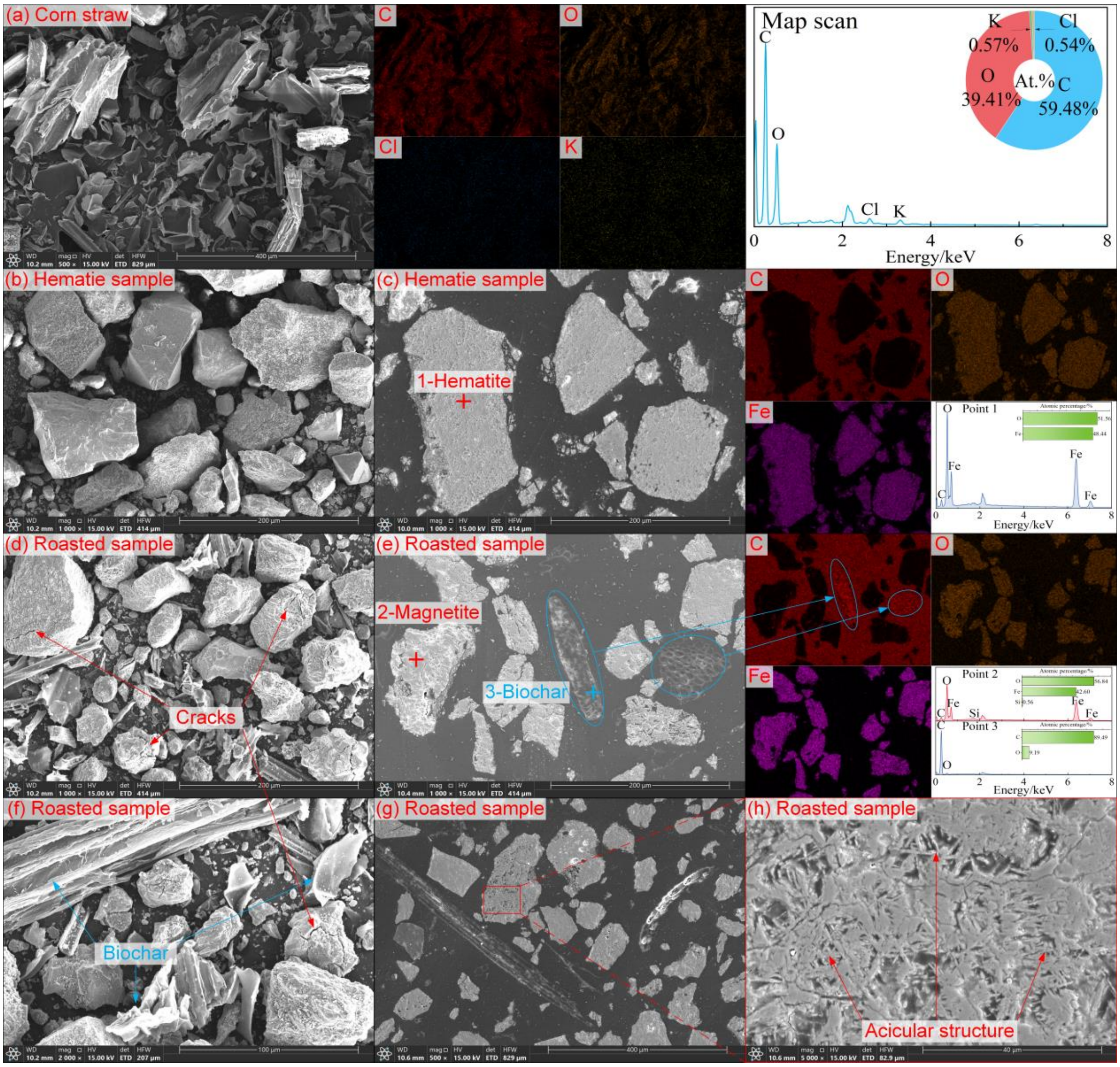


**Figure 6.** Microstructure analysis: (**a**) SEM image and map scan of corn straw, (**b**) SEM image of hematite sample particles, (**c**) SEM image and map scan of cross section of hematite sample, (**d**,**f**) SEM images of roasted sample particles, (**e**) SEM image and map scan of cross section of roasted sample, (**g**,**h**) SEM image of roasted sample.

### *3.4. Process Mechanism, Environmental Benefits and Feasibility*

Based on the above experimental results, a process mechanism was derived as shown in Figure 7. The use of biomass reductants proved to be a more environmentally friendly and sustainable option compared to coal-based reductants, reducing the environmental impact. Additionally, the mineral phase transformation process used in this study produced satisfactory indexes (iron grade 69.82%, iron recovery 93.95%). During the MPT process, corn straw underwent pyrolysis, producing reducing gases consisting mainly of CO and $H_2$ [13,16,26]. These gases, in turn, participated in the phase transformation process, transforming hematite into magnetite. The result was the production of porous magnetite, which was easily beneficiated by the magnetic separation process.

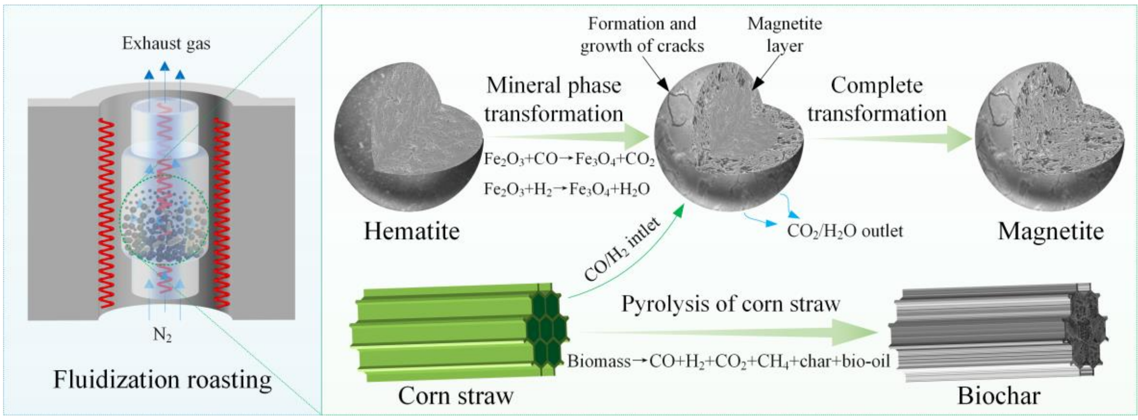


**Figure 7.** Mechanism schematic of corn straw as a green reductant to promote the phase transformation of hematite.

China produces about 900 million tons of straw annually, 30% of which is corn straw [27,28]. Unfortunately, much of this is burned directly in the fields, resulting in significant $CO_2$ emissions. The burning of straw produced approximately 38 million tons of $CO_2$ [27,28]. The issue is particularly critical in northeast China, where more than 80% of straw is burned each year, two-thirds of which is corn straw. This has led to environmental pollution and a significant waste of resources [27,28]. The use of randomly burned straw as a green reductant for hematite not only reduces airborne dust pollution and $CO_2$ emissions, but also offers considerable economic benefits.

Based on the MPT process, a semi-industrial pilot plant has been established within northeast China. Previous experiments involving various minerals such as hematite, siderite, ferromanganese ore, and flotation tailings from different sources have yielded favorable results, leading to the industrialization of this technology. To assess the feasibility of industrial implementation utilizing the abundant straw-based biomass resources within Northeast China, semi-industrial experiments on biomass roasting are planned for the near future.

## 4. Conclusions

The feasibility of using waste corn straw as a green reductant to promote the MPT of hematite to magnetite was explored. Optimal process parameters were determined to be a roasting time of 8 min, a roasting temperature of 700 °C, a mass ratio of corn straw to hematite of 1:4, and a $N_2$ flow rate of 300 mL/min. The roasted products were subjected to magnetic separation, and an iron concentrate with an iron grade of 69.82% and an iron recovery of 93.95% were obtained. CO and $H_2$ were the primary reducing gases generated during the roasting of corn straw and participated in the reduction of hematite. In the MPT process, hematite was reduced to magnetite, and further over-reduction produced wustite. Meanwhile, the corn straw was turned into porous biochar. The phase transition of hematite led to a loose and porous roasted particle structure. The newborn magnetite exhibited an acicular structure, which was beneficial for the subsequent grinding process. This study demonstrated the potential of using corn straw as a green and environmentally friendly reductant for iron recovery. Further studies on the detailed pyrolysis mechanism of corn straw and semi-industrial trials are planned to guide the design of large-scale prototype plants. This research has added value to agricultural waste and greened the hematite roasting process, paving the way for environmentally friendly and sustainable technologies.

**Author Contributions:** Q.Z.: Data curation, Formal Analysis, Validation, Writing—original draft. Y.S.: Conceptualization, Funding acquisition, Resources, Writing—review & editing. G.J.: Experiment, Data curation. Y.C.: Data curation, Resources. Y.H.: Data curation, Resources, Supervision. All authors have read and agreed to the published version of the manuscript.

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**Data Availability Statement:** The data supporting the results of this study are available from the corresponding author on reasonable request.

**Conflicts of Interest:** The authors declare that they have no conflict of interest.

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