

## UTILIZATION OF MSW-DERIVED BIOCHAR FOR PRODUCTION OF IRON THROUGH SOLID-SOLID REACTION

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**ABSTRACT:** Every year, the increasing human population has contributed to the abundance of Municipal Solid Waste (MSW) at designated landfills, especially in Malaysia. On the other hand, in the iron and steel industry, a high demand for steel production has caused an increase in CO<sub>2</sub> production, which would, unfortunately, contribute to global warming. Therefore, sustainable solutions are needed to avoid this environmental crisis from worsening. This study employed an effective approach, utilizing low-grade iron ore from Pahang to produce iron by using MSW-derived biochar as an alternative to coal as a reductant and energy source. This project aims to study the mechanism of the solid-solid reduction reaction of iron ore with carbon. MSW was collected and prepared before the slow pyrolysis to obtain the MSW-derived biochar. Iron ore was characterized by using X-Ray Fluorescence (XRF) for elemental composition determination and X-Ray Diffraction (XRD) to observe the presence of mineral phases. In this research, the solid-solid reaction was conducted for the reduction reaction at 900°C with residence times of 1 hour, 3 hours, and 5 hours. From the research that has been conducted, temperature and residence time were found to influence the elemental composition, reduction degree, and mineral phase transformation of iron.

**ABSTRAK:** Setiap tahun, peningkatan populasi manusia telah menyumbang kepada kelimpahan Sisa Pepejal Perbandaran (MSW) di tapak pelupusan yang ditetapkan, terutamanya di Malaysia. Manakala dalam industri besi, permintaan yang tinggi terhadap pengeluaran keluli telah menyebabkan peningkatan pengeluaran karbon dioksida, yang malangnya menyumbang kepada pemanasan global. Oleh itu, penyelesaian yang mampan diperlukan untuk mengelakkan krisis alam sekitar ini menjadi lebih buruk. Dalam kajian ini, pendekatan yang berkesan digunakan dengan memanfaatkan bijih besi gred rendah untuk pembuatan besi dengan menggunakan arang yang dihasilkan daripada MSW sebagai alternatif kepada arang batu sebagai bahan agen penurunan serta sumber tenaga. Tujuan projek ini adalah untuk mengkaji mekanisme tindak balas dalam pembuatan besi. MSW telah dikumpulkan dan disediakan sebelum menjalani proses pirolisis untuk mendapatkan arang yang dihasilkan daripada MSW. Bijih besi dianalisa menggunakan Fluoresens X-Ray (XRF) untuk penentuan komposisi unsur dan Penghabluran X-Ray (XRD) untuk memperoleh komposisi fasa mineral. Dalam penyelidikan ini, eksperimen dijalankan untuk tindak balas penurunan pada suhu 900°C dengan masa selama 1 jam, 3 jam dan 5 jam. Berdasarkan kajian yang telah dijalankan, suhu dan masa didapati mempengaruhi komposisi unsur serta tahap penurunan besi.

**KEY WORDS:** Municipal Solid Waste, Reduction of Iron, Biochar, Biomass Ironmaking.

## 1. INTRODUCTION

Municipal solid waste (MSW) is mostly generated from homes, businesses, offices, schools, and other places regularly. It covers all types of non-industrial solid waste, including garbage, street cleanings, decaying organic matter, and medical waste. MSW comprises organic food waste, paper, plastics, metal, and glass. Today, most developing countries use landfills as their final disposal method. This is because landfills are considered the cheapest, easiest, and most cost-effective method of waste disposal. The rising amount of generated MSW is a major cause of environmental problems such as recurring land and water pollution. MSW poses a significant global challenge, with an annual production that exceeds 2 billion tons, which is forecasted to double by the end of the century [1]. As the population grows in Malaysia, an increasing amount of MSW is reportedly deposited at landfills yearly. Foods, papers, and plastics were discovered to be the major composition of Malaysian MSW, which is expected to increase 3.3% annually [2].

However, since the significant components of MSW are found to be organic matter, this makes it a viable candidate to be utilized as a source for bioenergy generation [3]. Pyrolysis is one of the most prominent processes for producing energy from these organic materials. This prominent thermochemical process is conducted at temperatures between 300 and 700°C to thermally convert organic matter into biochar and bio-oil, which can be later used as alternative energy sources [4]. Generally, the temperature for producing biochar is up to 700°C, which would take 20 to 60 minutes. As the temperature rises, the quantity of acidic functional groups, particularly carboxylic functional groups, decreases and causes the development of basic functional groups. In other words, the structure of biochar will change to become graphite carbon at higher pyrolysis temperatures. The function of this biochar is comparable to that of normal coal, which provides an alternative method to coal utilization in iron production [5].

Carbon emissions, mainly from iron mining and steel production, are also increasing due to high market demand in developing countries. As a result, the world is at risk of a worsening global warming crisis. This environmental crisis's risk will affect human health and all life on land and in water. Therefore, prevention is always essential to sustaining this world and all humanity. To overcome these problems, one of the solutions is to utilize other renewable resources, such as MSW, as an alternative to coke.

On the other hand, in Malaysia, iron mines are still operating to extract iron ore. However, Malaysian iron ores are regarded as low-grade iron ores because of the 56 to 59% iron content and a relatively high combined water content. These low-grade iron ores do not fulfill the specifications to be used directly in the blast furnace as a feedstock for pig iron production without pre-reduction treatment [6]. According to the World Bureau of Metal Statistics, in 2021, Malaysia's iron ore production volume was approximately 4.98 million metric tons [7].

Scrutinizing the issues from both perspectives, MSW is a good candidate for biochar production before being utilized as a reductant for the reduction reaction of iron ore. Research on the reduction reactions of iron ore using MSW-derived biochar by solid-state responses is minimal. Therefore, this study was conducted to observe the outcomes.

## 2. METHODOLOGY

MSW was collected from households, and in general, the main components of this MSW in Malaysia are organic waste, plastic waste, and paper waste. The ratios were determined from the proportions of each specific component and were based on 1kg of hematite, Fe<sub>2</sub>O<sub>3</sub>. To obtain MSW-derived biochar that would be used as a reductant for iron ore reduction reaction,

MSW underwent a slow pyrolysis process at 350°C for 2 hours. Based on the chemical reactions, the minimum carbon required to reduce iron ore was calculated, and thus, the ratio for sample preparation could be determined.

For sample preparation, MSW was prepared, with the organic materials, plastics, and papers weighing 60, 20, and 20g, respectively. The samples were oven dried at 105°C for 24 hours to completely remove contact water. Subsequently, MSW was subjected to a heating process from room temperature to 350°C with a 10°C/minute heating rate. This slow thermal decomposition was conducted in an inert atmosphere for 2 hours to produce MSW-derived biochar.

On the other hand, goethite, a low-grade iron ore, was selected as the raw sample for this study. The physical form of this ore was reddish-brown in appearance, consisting of hard clay and hard rock. For sample preparation, iron ore was oven dried at 105°C for 24 hours to remove moisture before being ball milled for 1 hour to break the ore into small particles. The dough was compacted with a single shaft compressor at 1000 psi for 5 minutes. It was then placed in a cylindrical iron mold with water as the binding agent. Each sample weighed approximately 18g with a 2 cm diameter and 2.5 cm thickness. After pelletizing was completed, all pellets were left at room temperature for approximately 20 hours, then the samples were placed in an oven at 105°C for 24 hours to remove moisture inside the pellets.

For iron ore reduction experiments, an electric tubular furnace was used to examine the solid-solid reaction between iron ore bodies and MSW biochar in an argon atmosphere. Iron ore samples were mixed with biochar particles before being placed in enclosed steel baskets in an electric furnace. For the reduction process, samples were prepared according to iron ore to MSW char ratios, which were 8:2 and 8:4. These samples were subjected to experiments with different residence times: one hour, three hours, and five hours. For sample characterization, X-Ray fluorescence (XRF) and X-Ray diffraction (XRD) were utilized to determine the raw iron ore's chemical composition and mineral phases, respectively. The reduction degree was also calculated to observe the reduction reaction of iron.

### 3. RESULTS AND DISCUSSION

The elemental composition of the raw iron ore collected is shown in Table 1. The primary composition of the ore was iron, which is 59.1 wt%. Fig. 1 shows the thermal characteristic curve for this iron ore. At the temperature range of 220 to 350°C, there was a significant weight loss of 3.65%, where an endothermic peak appeared, indicating heat absorption. This was due to the removal of combined water in the form of hydroxyl, which often exists in limonitic ore containing the goethite mineral. This process can also be seen in the endothermic peak on the DTA curve. Heat was absorbed at 220 to 370°C for the iron ore sample, where the weight loss ranged from 1.50% to 3.75%, indicating the occurrence of combined water removal at this temperature range.

Table 1. Elemental compositions of Malaysian iron ore (wt%)

Locality	T-Fe	Si	Al	S	Mn	V	Ti	Ca	T-Fe	Si
3	59.1	0.8	1.3	0.2	0.4	0.5	6.2	0.3	59.1	0.8

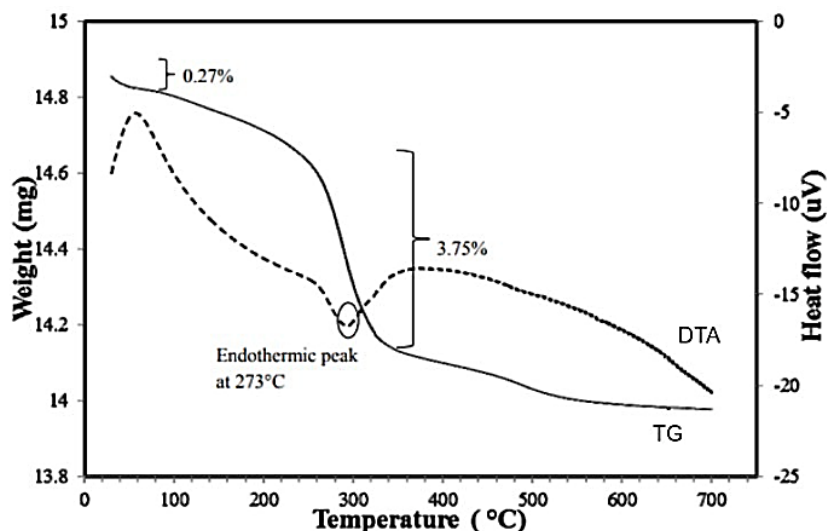


Figure 1. Raw iron ore TG-DTA profile

Fig. 2 shows the raw iron ore's XRD pattern. The primary minerals observed were goethite, an iron oxide hydrate, with the predominant hematite phase. The XRD profile also indicated some quartz as the main impurity.

The proximate and ultimate analyses of the MSW are shown in Table 2 below. The analysis shows that the contents of volatile matter, fixed carbon, and ash were 69.35, 9.00, and 7.05 mass% of MSW, respectively. Furthermore, the elemental analysis of MSW indicates that it contained 45.00, 6.11, 0.40, 0.12, and 48.37 mass% of carbon, hydrogen, nitrogen, sulfur, and oxygen (by difference), respectively. Table 3 shows that during the pyrolysis process of MSW, 100.0g of the raw material was heated without oxygen, leaving behind an approximate 60% char yield, where 40.5g was lost during the process.

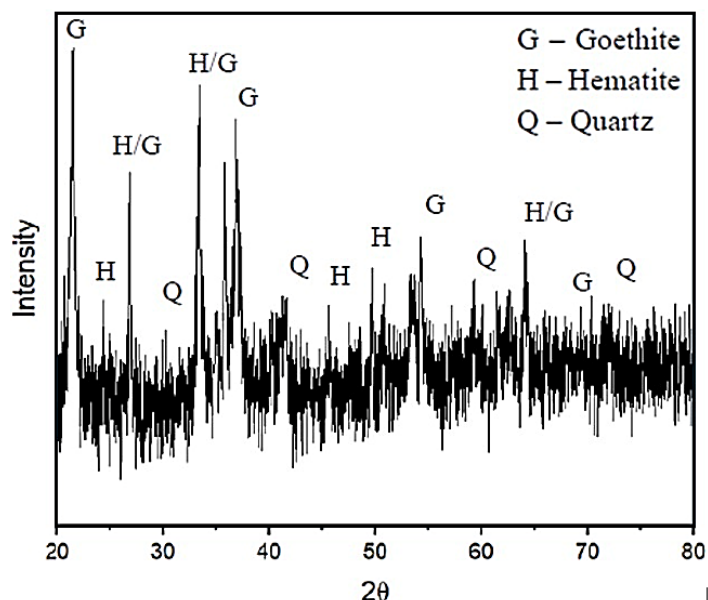


Figure 2. XRD analysis of raw iron ore.

Table 2. Proximate and Ultimate analysis of MSW.

Analysis	Proximate Analysis			Ultimate Analysis				
Elemental Mass (%)	Volatile Matter	Fixed Carbon	Ash	C	H	N	O	S
MSW	69.35	9.00	7.05	45.00	6.11	0.40	48.37	0.12

Table 3. Weight differences of MSW after pyrolysis.

Sample	Weight before (g)	Weight after (g)	Weight loss (g)
MSW	100.0	59.5	40.5

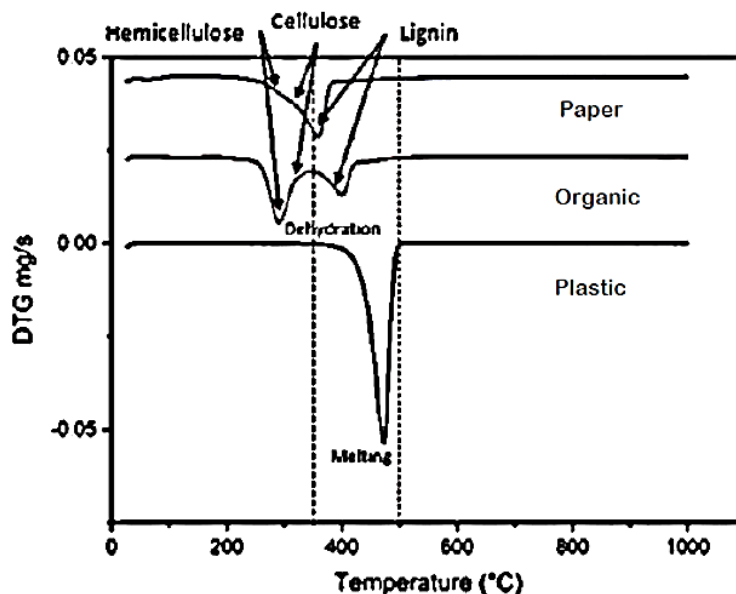


Figure 3. DTG pattern of MSW by component.

Fig. 3 depicts the DTG curve for the MSW sample by component. As the temperature increased to 300 °C, the weight of MSW steadily decreased as cellulose, hemicellulose, and lignin decomposed. The weight of the samples does appear to significantly decline over 600°C, indicating that the decomposition of hydrocarbons has completed. This demonstrates that certain elements, such as CO, H<sub>2</sub>, CO<sub>2</sub>, and hydrocarbons, were released. The DTA curve shows apparent peaks or valleys between 300 and 500 °C, representing reactive devolatilization in the materials [8]. The pyrolysis process in this experiment was conducted at 350°C so that during the reaction, there would be some diffusion of gaseous compounds, which would help reduce iron [9]. Fig. 4 shows the weight loss of the iron pellets after each sample was reduced with different reduction times within the range from one hour to five hours, with different iron ore to MSW char ratios, to study the effectiveness of the reduction process. The weight loss of pellets significantly increased with time, and the highest weight loss was obtained when twice the mass of MSW char is used to reduce iron ore at a temperature of 900°C. As the reduction temperature increases to 900°C, the rate of iron ore reduction also increases [10]. At this temperature, MSW char was gasified into CO and H<sub>2</sub>, which act as a reduction agent for the reduction reaction of iron oxide in the ore. Excess carbon and hydrogen in the mixture would also help to proceed with the reduction reaction further, as shown in equations 1 to 6. Therefore, higher carbon and hydrogen contents in the biomass promote more oxygen removal from iron species, which causes greater weight loss of iron ore pellets.

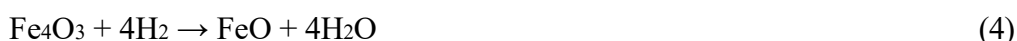
Reducing gases are formed during the reduction process through the reaction of chemicals between iron oxide and carbonaceous materials. The Boudouard reaction (Eq. 7) and the water

gas reaction (Eq. 8) produced syngas, which would react with iron oxide to convert iron ore to metallic iron. In the lower oxide and iron direction, simultaneous and continuous reduction processes occur from hematite to magnetite, from magnetite to wustite, and from wustite to metallic iron. The following sequence summarizes the main reduction processes of iron from hematite to iron (Fe):

Hematite to Magnetite



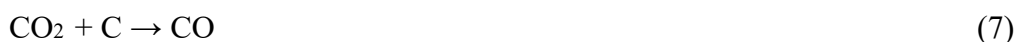
Magnetite to Wustite



Wustite to Iron



Boudouard reaction



Water-Gas reaction

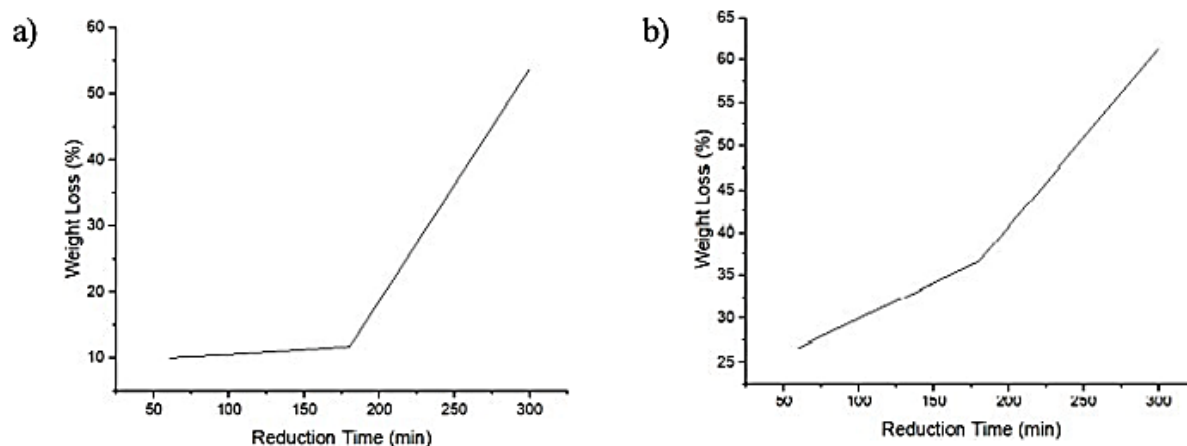


Figure 4. Effect of time on weight loss during iron reduction at 900°C for:  
(a) 8:2 ratio (b) 8:4 ratio pellets.

As iron ore was reduced, the characterization using XRD was conducted to observe the transformation phase of iron ore according to the reduction time. A longer reduction time allows hydrocarbons and gases to diffuse and thus reduce the iron ore. Samples with 8:2 ratios were exposed to reduction reaction with a holding time of 1 hour (Sample 1), 3 hours (Sample 3), and 5 hours (Sample 5). Samples with an 8:4 ratio were also exposed to the same residence time; those were 1 hour, 3 hours, and 5 hours for Sample 2, Sample 4, and Sample 6, respectively.



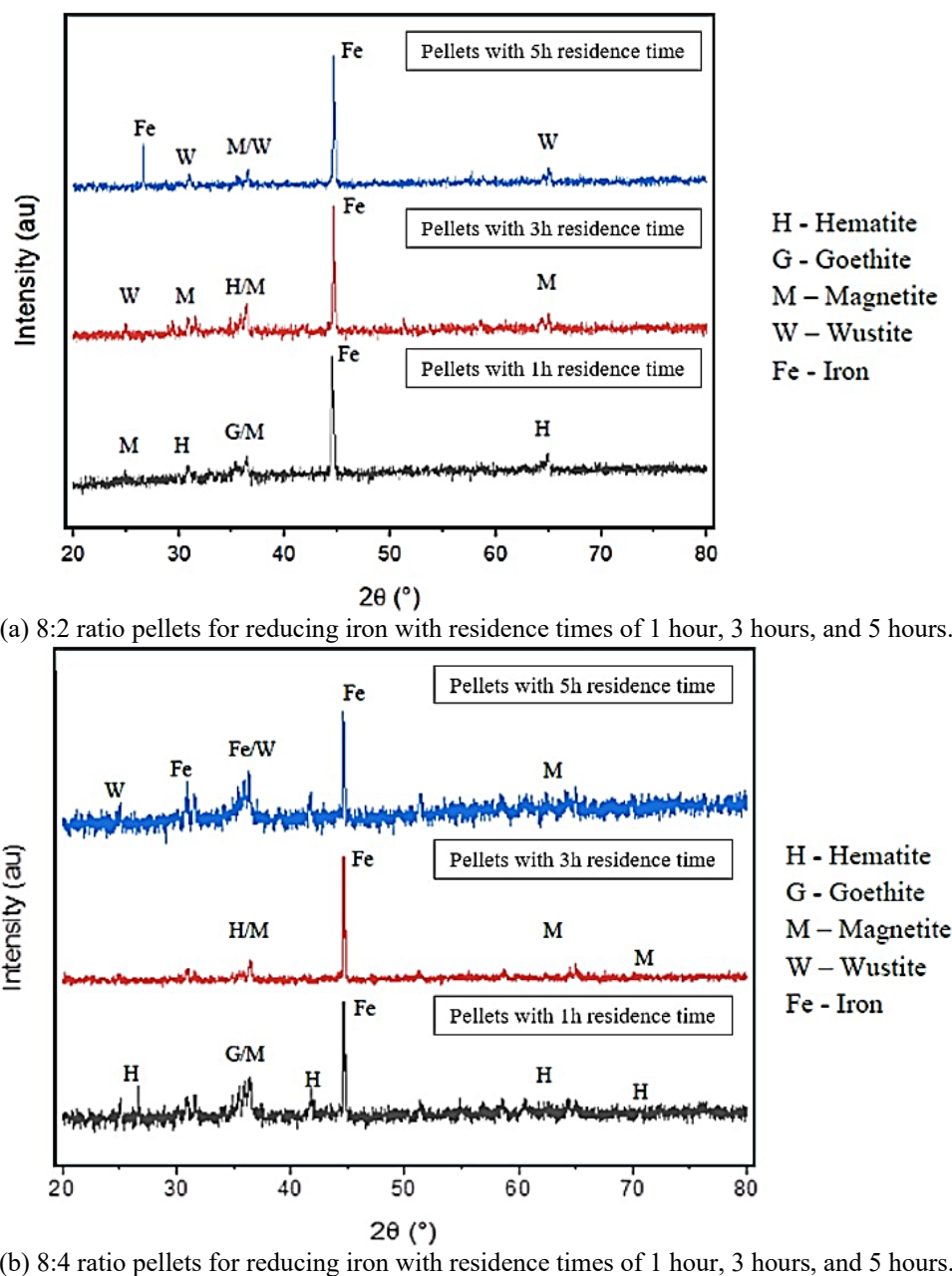


Figure 5. XRD Pattern

Both reduction reactions for samples with 8:2 and 8:4 ratios indicate the presence of iron, as the minimum temperature to obtain iron is 900°C, as shown in Fig. 5 (a) and Fig. 5 (b) below. One hour is sufficient to reduce hematite to iron, but only a small number of ores can be reduced directly to iron. Samples 1 to 4 show the presence of magnetite, hematite, and some iron-Fe. Samples 5 and 6 illustrate the presence of mainly wustite and iron with little magnetite. From this trend, it can be deduced that by increasing time and adding more biochar, there is a possibility for iron ore to be reduced further.

The degree of reduction was calculated depending on the mass loss of the iron ore. A similar pattern can be observed in the mass change of the pellets after the reduction process. The degree of reduction increases with a reduction time of up to five hours. Maximum reduction has been achieved when a 16:2 ratio of the weight of the MSW was used. As the reduction continues, the biomass gasifies to produce more reducing gas in the chamber.

Diffusion of CO and H<sub>2</sub> through the pellets resulted from the gasification of MSW, which was facilitated by the reduction reaction of iron oxide.

As the weight loss percent of both ratios of iron to MSW shows an increasing pattern with time, the percentage of Fe content is also increasing, as shown in Table 4. The 8:2 ratio has an increment of 23.9% in total, starting from one hour of reduction time up to five hours, while the 8:4 ratio shows an increment of 27% of Fe produced. This indicates that the weight loss due to the char element diffusion would lead to reduced iron. The mass of MSW biochar could also affect the percentage of Fe increment.

This is because, as the mass of MSW increases, the iron oxides can be expected to fully reduce into iron when exposed to a longer residence time. A short residence time would limit the degree of reduction reaction. From the weight loss data of iron ore, ore pellet, and MSW, the reduction degree was calculated, and it was found that as the residence time increased, the percentage of Fe increment also increased, showing that MSW can be used as a reducing agent for iron ores.

Table 4. Percent of Fe increment with time.

Sample	Holding Time (hour)	Fe Content (%)
8:2 Ratio	(Raw)	59.1
	1	68.6
	3	76.2
	5	83.0
	(Raw)	59.1
8:4 Ratio	1	80.9
	3	83.4
	5	86.7
	(Raw)	59.1

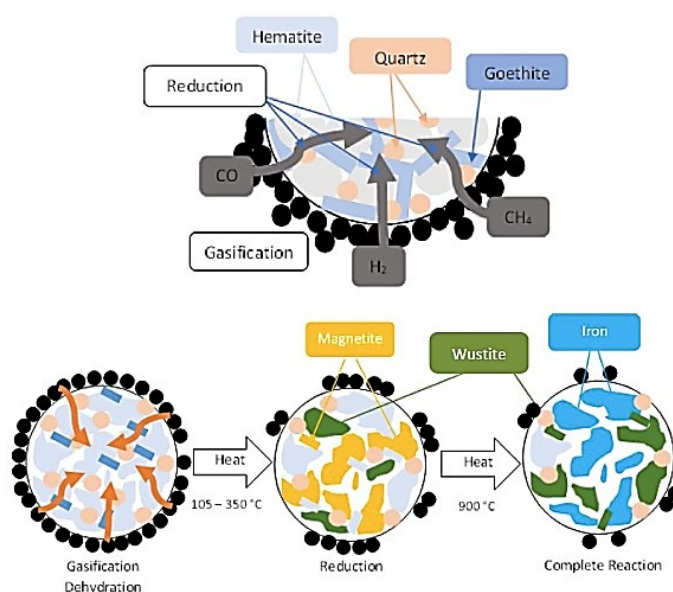


Figure 6. Solid-solid reaction mechanism of MSW biochar with iron ore.

Fig. 6 illustrates the reduction mechanisms in the reduction process of iron pellets with MSW biochar as reducing agent via solid-solid reaction. As the pellets were heated with bedded char around them, the temperature inside the pellet increased due to heat transfer from the outside to the inside of the pellet. In this experiment, combined water removal from iron ore occurred when heated to a low temperature by releasing the hydroxyl chain, creating many



voids and generating cracks on the iron ore surface with a large specific surface area. Gases produced from MSW char gasification also diffused into the pellets of biochar-iron ore mixture because of the formation and volume expansion of syngas occurring at this location. Therefore, the hydrocarbons and gases produced a large amount of heat, spreading throughout the bodies of the pellet. Biomass gasification produces syngas such as CO and H<sub>2</sub> that can be utilized as reducing agents in reducing iron ore. As these gases are being produced and diffused in the inner area of the pellets, they also diffuse outwards to reduce the iron oxide species in the outer part of the pellets. For the iron reduction, iron ore was first reduced by H<sub>2</sub> and hydrocarbon gases generated from MSW biochar, because reduction by H<sub>2</sub> is a faster process than that of other gases. However, the reaction process is very complicated because many elemental reactions can take place in this process. During MSW biochar gasification, syngas and hydrocarbon gases were produced as a potential reduction agent for iron oxide, and the decomposition of hydrocarbon gases into carbon to inhabit the formed pores on the surface of iron oxide. On the other hand, gases reacted together along with ore oxides, reducing Hematite to Magnetite, Magnetite to Wustite, and Wustite to metallic iron phase.

#### 4. CONCLUSION

Low-grade iron ore with 59.1% of Fe was used as the raw material for this study. MSW was selected to be converted into biochar, a carbon material that acts as the reductant and/or energy source in reducing iron. A sample with an 8:2 ratio of iron to MSW showed an increasing weight loss pattern. A sample with an 8:4 ratio presented the same pattern but with more weight loss because of the extra carbon particles. As the holding time increased, the degree of reduction also increased.

After the experiment, the XRD results of each sample indicated the presence of different iron species such as magnetite, wustite, and iron-Fe. During the heating process, MSW-derived biochar produced reductant gases that would assist in reducing iron from goethite to iron-Fe. By doubling the mass of the MSW-derived biochar, the reaction towards iron ore particles is highly active, with an increasing percentage of total iron in the sample, and more weight loss, which indicates the removal of oxygen atoms. This study promotes the reduction of MSW at landfills, which has become a troubling issue in Malaysia year by year. MSW can be a new alternative source of energy for metal smelting.

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