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Solar Extractive Metallurgy for the Production of Mg and Zn from Carbothermal Reduction of MgO and ZnO at Low Pressure in a Solar Thermochemical Reactor

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Abstract. Solar thermochemical pyro-metallurgical process is an attractive prospect for the sustainable conversion of both metal oxides and sunlight into value-added chemicals. In this work, a comparative study of carbothermal reduction of ZnO and MgO was performed in a 1.5 kW, directly-irradiated solar reactor utilizing concentrated sunlight. Important operating parameters were studied during the experiments including the type of carbon reducing agent (activated charcoal and carbon black) in batch and continuous operating modes under both reduced (0.11-0.40 bar) and atmospheric (0.90 bar) pressures in the operating temperature range of 600-1600°C, demonstrating the solar reactor flexibility, reliability, and robustness. As a result, regarding batch tests, a decrease in the total pressure promoted the conversion of ZnO and MgO above 78% and 99%, respectively, which in turn increased Zn and Mg yields. Nevertheless, an increase in the CO2 with decreasing total pressure was observed, especially in the case of ZnO reduction, due to reduced gas residence time. In contrast, CO2 yield remained negligible in the case of MgO. Regarding continuous tests, an increase of gas production yields as well as reaction extent through the increase of reactant feeding rate was highlighted. Employing activated charcoal showed higher conversion of both ZnO and MgO, resulting from the higher available specific surface area for chemical reactions. Finally, high-purity Zn and Mg content in the solar-produced powders were achieved.

INTRODUCTION

Solar energy is the most abundant renewable energy resource available on Earth and it can meet the global energy consumption demand. However, the direct utilization of solar energy is limited because of intermittency and dilution. Solar thermochemical conversion processes are attractive for converting intermittent and dilute sunlight into liquid/gaseous fuels and chemical commodities. Among them, the solar metallurgical carbothermal reduction process represents a promising route for producing metals and carbon monoxide (CO that is a key component of syngas) in a single reaction. The stoichiometric carbothermal reduction reaction of metal oxide to metal and CO is represented in Equation 1.

\[ \text{M}_\text{x}\text{O}_\text{y} + \text{yC} \rightarrow x\text{M} + y\text{CO} \]  

In this approach, solid carbon is employed as a reducing agent to both extract oxygen from metal oxides and lower the thermodynamic barrier, as compared to the thermal-only dissociation of metal oxides. Regarding metal oxides, they can be classified into two groups according to the presence or absence of phase change. Non-volatile oxides such as iron oxides (Fe2O3/Fe3O4, ferrites [1]), perovskites (e.g., La1-xSr_xMnO3-δ), and ceria (CeO2-δ) [2] deal with solid state reactions over the entire process. Therefore, only oxygen is released from the metal oxide structure, thereby bypassing the recombination issue. Non-volatile oxides are commonly used as oxygen carriers in H2O/CO2...
splitting redox cycle systems [3] and chemical looping reforming [4]. However, their drawbacks are related to their physicochemical characteristics such as sintering (iron oxides) [1] and non-stoichiometric reactions (case of ceria or perovskites). Volatile metal oxides deal with a solid-to-gas/liquid phase transition of the products (either gaseous: ZnO/Zn [5] and MgO/Mg, or liquid: SnO2/Sn) in the reduction step. The reduced product species first vaporize, and when temperature decreases, they condense in the form of fine solid particles. In contrast to non-volatile oxides, volatile metal oxides exhibit high oxygen exchange capacity as they can be completely reduced to their metallic elements. However, volatile metal oxides display a recombination issue with O2, which can be alleviated by gas quenching. In this work, two attractive volatile oxides candidates regarding ZnO and MgO are studied for solar carbothermal metallurgical processes towards Zn/Mg and CO products. The overall carbothermal reduction (CTR) reactions of ZnO and MgO are represented according to Equation 2 and Equation 3.

\[
\begin{align*}
ZnO + C & \rightarrow Zn + CO & \Delta H^0 = 370 \text{ kJ/mol} & T_{\text{equilibrium}} = 1220 \text{ K} \\
MgO + C & \rightarrow Mg + CO & \Delta H^0 = 638 \text{ kJ/mol} & T_{\text{equilibrium}} = 2130 \text{ K}
\end{align*}
\]

In addition, the solid-gas side reactions during ZnO reduction are possible, according to Equation 4 and Equation 5. In case of MgO reduction, only the side reaction associated with Equation 5 is possible as the MgO reduction with CO is not thermodynamically favorable within the considered operating temperature range.

\[
\begin{align*}
ZnO(s)+CO(g) & \leftrightarrow Zn(g)+CO_2(g) \\
C(s)+CO_2(g) & \leftrightarrow 2CO(g)
\end{align*}
\]

MgO and ZnO conversion (\(X_{MgO}\) and \(X_{ZnO}\)) is defined as the net fraction of MgO or ZnO converted to Mg or Zn (also corresponding to Mg or Zn yield), and is obtained from an oxygen balance according to Equation 6.

\[
X_{MgO}(X_{ZnO}) = \frac{n_{CO}+2n_{CO_2}}{n_{MgO}(n_{ZnO})}
\]

Where \(n_{CO}\) and \(n_{CO_2}\) indicate the mole amounts of produced CO and CO2 obtained by time integration of their production rates over the reaction duration.

Generally, Mg product can be used in various applications such as magnesium-based alloys [6], while Zn product can be used as material for several industrial sectors such as galvanizing and electrical batteries. Furthermore, both Mg and Zn can be employed as oxygen carriers in a H2O/CO2 splitting redox cycle [7]. Conventionally, Mg is produced by Pidgeon Magnetherm and electrolytic methods [8], while Zn is extracted from ores via smelting and refining processes [9]. Such conventional approaches thus result in CO2 emissions. As seen in the Equation 2 and Equation 3, differences in the enthalpy change of reaction and temperature at equilibrium (i.e., \(\Delta G^0\) equals to zero) between Zn and Mg are noticeable. On the one hand, MgO+C reaction displays a \(\Delta H^0\) significantly higher than that of ZnO+C, implying high potential for the solar energy storage at the expense of higher heat requirement. On the other hand, the lower \(\Delta H^0\) for ZnO+C leads to a lower heat requirement, thus pointing out a simpler reactor operation system. Hence, the different versatility between ZnO and MgO has motivated the comparative study of CTR of ZnO and MgO for producing metals and CO. Most previous works [10] were focused on vacuum CTR of MgO and ZnO, which was investigated both thermodynamically and experimentally. The studies reported that operating at reduced pressure enhanced the reduction extent as well as lowered the reduction temperature, at the expense of pumping energy requirements and increased solar energy consumption. Nevertheless, no prior work has been conducted on an experimental comparative study of CTR of ZnO and MgO in a solar chemical reactor.

The present work aims to further explore the on-sun experimental study and comparison of solar CTR of ZnO and MgO in a metallurgical solar reactor. Experiments were conducted with different reducing agents including activated charcoal (AC) and carbon black (CB) in batch and continuous modes under both vacuum and atmospheric pressures to demonstrate flexibility, reliability, and robustness of this metallurgical process for Zn and Mg production. In addition, a parametric study of CTR of ZnO and MgO regarding the influence of pressure, reducing agents (AC and CB), and C/MgO or C/ZnO molar ratio on metal oxides conversion and yields (CO and Mg/Zn) was conducted.
EXPERIMENTAL SETUP AND METHODS

Figure 1 shows the operating principle of the CTR of ZnO and MgO in a 1.5 kW\textsubscript{th} prototype solar vacuum reactor driven by highly concentrated sunlight, delivered by a 2 m-diameter parabolic concentrator located above the reactor, with a solar concentration ratio up to 10,551 suns (0.85 m focal distance, peak flux density of ~10.5 MW/m\textsuperscript{2} for a Direct Normal Irradiation DNI of 1 kW/m\textsuperscript{2}). More details about this solar reactor have been previously described [11], and the additional information is reported here. The reactor can be operated in both batch and continuous modes under vacuum and atmospheric pressures at various reduction temperatures (transient and isothermal temperatures, in the range 600-1600°C). One pressure sensor is employed to measure the pressure in the reactor cavity (P), and one temperature measurement (B-type thermocouple) is mounted inside the cavity (T\textsubscript{1}). A solar-blind pyrometer located at the center of the facedown parabolic concentrator also measures the temperature inside the cavity receiver to compare with T\textsubscript{1}.

MgO (particle size: 1-2 \(\mu\text{m}\), 99.8% chemical purity, PROLABO) and ZnO (particle size: 1-5 \(\mu\text{m}\), 99.0% chemical purity, PROLABO) and two types of solid carbon; namely activated charcoal (AC) and carbon black (CB) with different physical properties (Table 1) were used. AC and CB were provided from Sigma Aldrich and Asahi Carbon (Japan), respectively. Metal oxides powders (either MgO or ZnO) and solid carbon were mechanically mixed with C/metal oxides molar ratios of 1.5 and 2, which represents 50% and 100% excess carbon, respectively, to favor CO production and complete conversion of MgO or ZnO. In batch mode, a given amount of metal oxide and carbon blend was loaded directly inside the cavity and the reaction occurred during solar heating under non-isothermal conditions. Concerning the continuous experiments at a given temperature (isothermal), a mixture of ZnO and CB was fed into the reactor cavity receiver via a particle delivery system, mainly consisting of a hopper, screw feeder, and electrical motor (Figure 1).

The solar vacuum reactor was positioned at the focus of the vertical axis parabolic dish solar concentrator. It was heated with highly concentrated solar energy to the desired temperature. Regarding vacuum operation, once reaching the temperatures of ~650°C (for CTR of ZnO) and ~900°C (for CTR of MgO), the reactor was sucked with a rotary vane vacuum pump (Alcatel) to the targeted pressure (0.11-0.40 bar). Note that prior to each experiment, vacuum leak testing was performed several times. The solar reactor was evacuated via pumping to a preset pressure level and the reactor pressure kept stabilized, confirming that the set-up was airtight. During the reactor heating...
under concentrated solar radiation in batch mode, the reduction reaction occurred from ~750°C (ZnO) and ~1000-1200°C (MgO depending on total pressure) during temperature rising, as reflected by CO and CO₂ formation detected from an online gas analyzer. Solar heat supply rate was kept constant (~50% shutter opening for CTR of ZnO and 100% shutter opening for CTR of MgO). It could also be varied by adjusting the shutter opening to stabilize the targeted temperature in case of isothermal experiments (continuous tests).

### TABLE 1. Physical properties of solid carbonaceous materials.

<table>
<thead>
<tr>
<th>Material</th>
<th>Purity (%)</th>
<th>Average primary particle size (μm)</th>
<th>Specific surface area (m²/g)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Activated charcoal (AC) Darco®</td>
<td>99.9</td>
<td>&lt;149</td>
<td>732</td>
</tr>
<tr>
<td>Carbon black (CB), SB905 powder</td>
<td>99.9</td>
<td>15.10⁻³</td>
<td>210</td>
</tr>
</tbody>
</table>

During reactor heating, N₂ protective gas flows (2 NL/min) were supplied for protecting the window and preventing the back-flow of hot gases through the screw path (0.5 NL/min) in the case of continuous reactant particle injection. A small stream of product gases was sampled by a secondary membrane pump to the on-line syngas analyzer to continuously analyze product gas species (CO, CO₂). The syngas yields (mmol per gram metal oxides, mmol/(g_ZnO or g_MgO)) were calculated by time integration of the gas species production rates over the experiment duration. Finally, the condensed products contained in the removable outlet reactor components (zone A and zone B) were collected and then analyzed by calibrated X-ray diffraction (XRD) for phase identification. Particle morphology analysis was carried out using a field emission scanning electron microscope (FESEM).

**RESULTS AND DISCUSSION**

**Gas Species Production Yield**

Figure 2 shows the CO and CO₂ yields along with metal oxides conversion as a function of total pressure and reducing agents (AC and CB) for CTR of ZnO (Figure 2a) and CTR of MgO (Figure 2b) in batch mode. Concerning CTR of ZnO, decreasing pressure lowered the CO yield because of concomitant increased CO₂. For instance, CO and CO₂ yields for AC were 4.52 and 1.85 mmol/g ZnO at 0.40 bar compared to 4.22 and 2.59 mmol/g ZnO at 0.15 bar, respectively. This can be explained by the fact that the reaction mechanism proceeds via two solid-gas reactions in which ZnO is first reduced by the produced CO to form both Zn and CO₂ (Equation 4), and the produced CO₂ is then reduced by C (Equation 5). However, when decreasing the pressure, the gas residence time decreases as well. Thus, the CO₂ reduction with C may be kinetically limited, thereby leading to increased CO₂ in the output gas. Within the reduced pressure range 0.15-0.4 bar, the net ZnO conversion (X_ZnO) was in the range 72.6-78.2% for CB, and 66.8-76.5% for AC. As observed in Figure 2a, X_ZnO trends were not obvious even though their evolutions tended to increase. This is because, at the pressure conditions of 0.15 bar (CB) and 0.40 bar (CB), the solar power input fluctuated during experiments because of clouds’ passage. The impact of the reducing agents between AC and CB on CO and CO₂ yields was not significantly different, although using AC led to superior CO yield, possibly thanks to the favorable physical properties of AC, according to Table 1.

Regarding CTR of MgO (Figure 2b), in contrast to CTR of ZnO, the CO yield rose significantly with decreasing total pressure regardless of the C/MgO molar ratios. For example, at AC/MgO molar ratio = 2, CO yield increased from 21.21 mmol/gMgO at 0.16 bar to 24.59 mmol/gMgO at 0.11 bar, in turn resulting in MgO conversion (X_MgO) increasing from 86.8% to 99.9%. In addition, CO₂ yields were found to be negligible (e.g. 0.10-0.17 mmol/gMgO for AC/MgO molar ratio of 2). The low formation of CO₂ may be attributed to the Boudouard equilibrium because the solid–gas reaction between MgO and CO is not thermodynamically favorable within the considered temperature range (MgO+CO→Mg+CO₂, T(ΔＧ°m=0) > 2000°C), whereas the solid–gas side reaction of ZnO with CO (Equation 4) is thermodynamically favorable. Thus, the MgO reduction reaction chiefly occurs via the solid–solid reaction (Equation 3). Therefore, the difference in the reduction behavior between CTR of ZnO and CTR of MgO was noticeable and evidenced from experimental results. Besides, the effect of excess C/MgO molar ratios (between 1.5 and 2) on MgO conversion was found to be insignificant. Using AC as reducing agent exhibited better MgO conversion, similar to CTR of ZnO. In comparison, the total gas yields obtained from CTR of MgO (Figure 2b) were much higher than those obtained from CTR of ZnO (Figure 2a). This can be explained by the fact that the
discharged oxygen is in the form of mainly CO for MgO; in contrast, a significant portion is discharged in the form of CO$_2$ in addition to CO for ZnO. In addition, the molecular weight of MgO is around half of that of ZnO (40.30 g/mol (MgO) vs. 81.38 g/mol (ZnO)). In addition, the lower $X_{\text{ZnO}}$ than $X_{\text{MgO}}$ was possibly due to the higher Zn re-oxidation with CO$_2$ to ZnO in comparison with Mg re-oxidation, given that the CO$_2$ yield obtained from the CTR of ZnO was significantly higher compared to CTR of MgO.

![Figure 2](image.png)

**FIGURE 2.** CO and CO$_2$ yields of carbothermal reduction with AC and CB in batch mode as a function of total pressure: (a) ZnO (C/ZnO=1.5) and (b) MgO (C/MgO = 1.5 and 2).

Continuous CTR of ZnO with CB was experimentally investigated by varying the reactant feeding rate (0.5 and 1.0 g/min) at a constant temperature of 950°C. The cavity was thus empty initially and the ZnO/C mixture was continuously fed to the cavity. Note that the temperature of 950°C was chosen to avoid damaging the screw feeder tip that was directly inserted into the cavity receiver. Before on-sun experiments, the screw feeder was first calibrated for the mixture of ZnO+C to precisely control its mass feeding rate. A homogeneous mixture of reactant powder with a fixed CB/ZnO molar ratio of 1.5 (12 g in total mass) was prepared and fed into the cavity receiver. The reactor was operated isothermally under atmospheric pressure (0.90 bar). Figure 3 shows the CO and CO$_2$ yields along with $X_{\text{ZnO}}$ as a function of reactant feeding rate. It was found that both CO and CO$_2$ yields increased with increasing reactant feeding rate, from 2.08 and 1.68 mmol/g$_{\text{ZnO}}$ at 0.5 g/min to 2.25 and 1.77 mmol/g$_{\text{ZnO}}$ at 1.0 g/min, thereby resulting in $X_{\text{ZnO}}$ increasing from 44.3% to 47.1%. From these results, it can be pointed out that increasing the feeding rate hastened the reactant consumption that promoted gas yields. In addition, excessively low feeding rate results in low gas production rate and inefficient utilization of solar energy input, whereas excessively high reactant feeding rate may cause reactant accumulation. Therefore, for a given temperature, the reactant feeding rate must match the ZnO reduction reaction rate to avoid the issue of reactant accumulation. In comparison, $X_{\text{ZnO}}$ values obtained from continuous tests were much lower than those obtained in batch tests (44.3-47.1% for continuous tests vs. 66-78.2% for batch tests). This is because of the fact that the kinetics during isothermal operation at a constant temperature of 950°C was lower (950°C for continuous test vs. 623-1248°C for batch test), thereby leading to lower $X_{\text{ZnO}}$. 

130002-5
Characterization of Zn and Mg Products

Figure 4 shows XRD patterns of the collected solid products as a function of the operating pressure for CTR of ZnO (Figure 4a,b) and CTR of MgO (Figure 4c,d). As shown in Figure 1, the outlet components where the solid products deposited were separated into two zones: (i) zone A represents the collected product from both the outlet alumina tube and connector, and (ii) zone B represents the collected product from the ceramic filter. Concerning CTR of ZnO, neither zone A (Figure 4a) nor zone B (Figure 4b) showed traces of ZnO; thus, only Zn pattern with high peak intensity was evidenced as compared with the commercial pure Zn reference pattern, thereby demonstrating high-purity Zn production in both zones. Besides, the impact of pressure on solid products composition was not significant. In contrast to CTR of ZnO, traces of MgO were observed in both products collected from zone A (Figure 4c) and zone B (Figure 4d), indicating partial Mg oxidation during products collection (and/or recombination with CO). Additionally, Mg intensity was greater at lower operating pressure (0.11 bar). Note that, during collecting and transferring the solid products, Mg oxidation with air was noticed with a potential pyrophoric issue at ambient temperature due to the very fine and dispersed reactive powder (composed of agglomerates of nanoparticles). This issue directly caused an increase in the MgO content in the solid products due to contact with air (thus not caused by the reactor operation), which explains the presence of MgO phase in all the XRD patterns of samples from zones A and B. Nevertheless, net MgO conversion values up to 97.8% (Figure 2b) proved that MgO conversion was completed, and high Mg production yields were produced.

Figure 5 shows the particle morphology of the solid products at the reactor outlet (zone A) and filter (zone B), characterized by FESEM for CTR of ZnO (Figure 5a,b) and CTR of MgO (Figure 5c,d). Concerning CTR of ZnO, at zone A (Figure 5a), Zn product grew in plane layers as Zn vapor condensed when encountering the cold surface thanks to the alumina water-cooled outlet tube. In contrast to zone A, a clear hexagonal crystal structure of condensed Zn particles was highlighted at zone B (Figure 5b). Besides, a small amount of fine solid carbon (entrained particles) in both zone A and zone B in contact with the condensed Zn particles was observed (Figure 5a,b). Concerning CTR of MgO, a difference of Mg morphology in both zone A (Figure 5c) and zone B (Figure 5d) was not clearly visible. This is because of the issue of Mg oxidation with air during the solid product collecting process. The powder was composed of fine agglomerated particles (~100 nm) in spherical shape. In comparison, the Mg particle morphology displayed a more homogeneous distribution in contrast to that of Zn, and the Zn particles’ size was bigger than Mg.
FIGURE 4. XRD patterns of the collected solid products in zone A and zone B as a function of total pressure: (a,b) ZnO (AC/ZnO = 1.5) and (c,d) MgO (AC/MgO = 1.5).

FIGURE 5. FESEM micrographs of solid products from the outlet tube (zone A) and filter (zone B): (a,b) ZnO (AC/ZnO = 1.5) and (c,d) MgO (CB/MgO = 1.5).
CONCLUSION

Solar carbothermal reduction of ZnO and MgO has been performed in a prototype solar reactor with different reducing agents including activated charcoal (AC) and carbon black (CB), in either batch or continuous operation modes under both vacuum and atmospheric pressures, thus demonstrating flexibility, reliability, and robustness of this solar metallurgical process for Zn and Mg production. The experimental outcomes related to CO and CO\textsubscript{2} yields, metal oxides (ZnO and MgO) conversion, Zn and Mg production yields were experimentally evaluated.

Concerning batch tests, a decrease in total pressure promoted the ZnO conversion, thus enhancing Zn production yield; however, CO yield was decreased while CO\textsubscript{2} yield was increased due to the reduced residence time. The maximum value of ZnO conversion up to 78.2\% was accomplished at 0.40 bar. In contrast to ZnO reduction, decreasing pressure for MgO reduction significantly enhanced CO yield with the highest value of 24.59 mmol/gMgO obtained for activated charcoal at the lowest pressure (0.11 bar). The CO\textsubscript{2} yield from MgO reduction was negligible regardless of pressures, denoting the noticeable differences of reaction mechanism between MgO and ZnO carbothermal reduction. MgO conversion up to 99.9\% was achieved at 0.11 bar up to 1600°C. No significant impact of excess C/MgO molar ratios (between 1.5 and 2) on MgO conversion was noticed. Utilizing activated charcoal as reducing agent showed better conversion of both MgO and ZnO than using carbon black thanks to its larger specific surface area, which led to a favored solid–solid reaction. Regarding continuous tests at isothermal temperature, an increase in the reactant feeding rate accelerated ZnO consumption, which in turn promoted ZnO conversion to 47.1\% at 1 g/min. However, a possible reactant accumulation was noticed when reactant feeding rate was higher than reactant consumption rate. In comparison, batch tests reached significantly higher reaction extents than continuous tests; nevertheless, they induced higher heat losses resulting from non-isothermal operation (longer operating duration). Reducing the total pressure is essential to upgrade ZnO and MgO conversion, and a reduced pressure of 0.40 bar is recommended for both ZnO and MgO reduction. Regarding the characterization of Zn and Mg products, high-purity condensed Zn production was demonstrated, and Zn morphology clearly displayed hexagonal crystal structure in micrometric particle size, whereas Mg fine powder (~100 nm) was produced. In addition, Mg powder was highly reactive with air, thereby resulting in low Mg content after exposure to air based on ex-situ solid products analysis. The continuous carbothermal reduction of ZnO and MgO under vacuum condition should be further investigated to both enhance metal oxides conversion while producing metallic powders in continuous mode. The prototype vacuum reactor was demonstrated to be flexible in processing different volatile metal oxides (MgO and ZnO) for co-production of metallic Mg/Zn and CO in batch and continuous modes under both vacuum and atmospheric conditions.

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