Catalytic Gasification of Sewage Sludge in Supercritical Water: Influence of K₂CO₃ and H₂O₂ on Hydrogen Production and Phosphorus Yield

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ABSTRACT: In this work, the catalytic gasification of sewage sludge in supercritical water was investigated in a batch reactor (460 °C, 27 MPa, 6 min), and the separate and combined effects of the catalyst on the H₂ production and phosphorus yield were investigated. The experimental results indicated that K₂CO₃ alone improved the H₂ yield, gasification efficiency (GE), and carbon gasification efficiency (CE). The largest H₂ yield of 54.28 mol/kg was achieved, which was approximately three times that without a catalyst. Furthermore, the inorganic phosphorus (IP) yield increased with the addition of K₂CO₃. However, when H₂O₂ was added, the H₂ yield quickly decreased with increasing H₂O₂ coefficient, and more than 97.8% of organic phosphorus (OP) was converted into IP. The H₂ yield increased with the addition of various K₂CO₃/H₂O₂ ratios, whereas the IP yield decreased.

1. INTRODUCTION

Sewage sludge is an inevitable product of wastewater treatment. The amount of sewage sludge has increased with the construction and expansion of wastewater treatment plants while stricter policies have limited its disposal.¹ Sewage sludge contains complex organic materials mainly consisting of carbohydrates (~14%), proteins (~40%), lipids (~10%), lignin (~17%), and ash (30−50%).² Various techniques for converting sewage sludge into useful secondary energy have been developed.³−⁵ One of the methods for energy recovery is the gasification of sewage sludge in supercritical water.⁶ Supercritical water gasification (SCWG) is regarded as an emerging economical and environmentally friendly technology for sewage sludge treatment and hydrogen production.⁷ In recent years, many research studies on the supercritical water gasification of sewage sludge have been reported.⁸−¹¹ To improve the hydrogen yield, a small amount of oxidant was added in the SCWG process. Both hydrogen yield⁵,¹² and removal efficiency of organic matter¹³ in sewage sludge could be improved with the addition of oxidant (H₂O₂) in the SCWG of sludge.

The gasification reaction could be accelerated with the addition of suitable catalysts in SCWG, resulting in an increase in hydrogen production. These catalysts include metals, carbon, metal oxides, and alkalies (e.g., KOH, NaOH, K₂CO₃, and Na₂CO₃). Alkali catalysts are widely employed for high hydrogen production and inhibition of tar/char formation.¹⁵−¹⁷

When the K₂CO₃ catalyst was added in the SCWG process, the gasification efficiency (GE) was enhanced.⁷ Gong et al.⁸ also reported that the hydrogen yield was higher when a combined catalyst with different Ni/NaOH ratios was used. A multicomponent catalyst consisting activated carbon and Na₂CO₃ has been reported to enhance production and the removal of organics.⁵ Phosphorus is essential for the growth of life. As phosphate resources have gradually become scarce, it is necessary to look for a sort of recyclable, environmentally friendly phosphorus resource.¹⁶ Sewage sludge has a high phosphorus content, approximately 8% (w/w), which makes it a potential source of the P nutrient.¹⁷ Phosphorus recovery from sewage sludge using different methods (e.g., chemical oxidation, subcritical water process, and SCWG) has been investigated.¹⁸−²³ Also, the gasification of sewage sludge in supercritical water for hydrogen production and phosphorus recovery has been investigated.¹¹,²⁴,²⁵ However, systematic studies on the effects

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of K₂CO₃ and H₂O₂ on hydrogen production and phosphorus recovery during SCWG of sludge have not been reported.

In this work, the catalytic gasification of sewage sludge in supercritical water with the addition of K₂CO₃ and H₂O₂ was studied, and the effects of the catalyst separately and the combined effects of K₂CO₃ and H₂O₂ on hydrogen production and phosphorus recovery were investigated.

2. RESULTS AND DISCUSSION

2.1. Effects of K₂CO₃ and H₂O₂ Individually on Gas and Phosphorus Yield. Figure 1 illustrates the effects of varying the amount of the K₂CO₃ additive on the H₂ gas and phosphorus yields from the SCWG of sludge at 460 °C and 27 MPa after 6 min. As shown in Figure 1a, an increase in H₂ yield was obtained as the K₂CO₃ concentration increased. When the K₂CO₃ concentration increased from 0 to 8 wt %, the H₂ yield increased from 19.86 to 54.28 mol/kg, which was approximately three times that without catalyst. Unlike the H₂, the CO₂ yield increased from 7.13 to 12.57 mol/kg and then reduced to 10.65 mol/kg as the K₂CO₃ increased. An inflection point at a concentration of 6 wt % for K₂CO₃ was observed. Meanwhile, the CO and CH₄ yields hardly changed with an increase in the K₂CO₃ concentration. These results agreed with previous reports, which showed slight changes in the CO and CH₄ yields with increasing K₂CO₃.[26,27] The comparison of this study with previous literature[28–35] is shown in Table 1.

As shown in Figure 1b, similar to the change in the CO₂ yield, the GE and CE values increased from 49.78 and 257.34% to 80.8 and 368.09%, respectively, with an increase in the amount of the K₂CO₃ additive. As the K₂CO₃ concentration increased from 0 to 8 wt %, the H₂ yield increased from 19.86 to 54.28 mol/kg, which was approximately three times that without catalyst. Unlike the H₂, the CO₂ yield increased from 7.13 to 12.57 mol/kg and then reduced to 10.65 mol/kg as the K₂CO₃ increased. An inflection point at a concentration of 6 wt % for K₂CO₃ was observed. Meanwhile, the CO and CH₄ yields hardly changed with an increase in the K₂CO₃ concentration. These results agreed with previous reports, which showed slight changes in the CO and CH₄ yields with increasing K₂CO₃.[26,27] The comparison of this study with previous literature[28–35] is shown in Table 1.

Figure 1. Effect of the amount of K₂CO₃ on the gas and phosphorus yields from the SCWG of sludge at 460 °C and 27 MPa after 6 min: (a) gas yield, (b) GE and carbon gasification efficiency (CE), (C) inorganic phosphorus (IP) and organic phosphorus (OP) yields, and (d) total phosphorus (TP) yield in the liquid sample and solid residue.
the K2CO3 concentration increased. Several previous studies demonstrated that the K2CO3 catalyst is to improve the water gas shift reaction (eq 1) as shown in Figure 2a. The H2 yield quickly decreased from 3.37 to 0.14 mol/kg with an increase in the H2O2 coefficient from 0.5 to 2.5, whereas a H2 yield of 19.86 mol/kg was obtained without the addition of the oxidant. A similar decrease in the CH4 content from 8.16 to 0.15 mol/kg was also observed. However, the CO2 yield increased directly from 7.13 to 29.77 mol/kg as the H2O2 coefficient increased from 0 to 2.5.

As shown in Figure 2b, when the H2O2 coefficient was 1.5, the GE was higher than 100%. It increased from 114.86 to 131.6% as the H2O2 coefficient increased from 1.5 to 2.5. In addition, the CE also increased from 333.54 to 502.08% with the addition of H2O2. As shown in Figure 2c,d, when the H2O2 coefficient was 2.5, over 97.8% of OP was converted to IP. The amount of TP in liquid production decreased as the H2O2 coefficient increased from 0 to 2.5, whereas the concentration of TP in the solid residue increased. The total mass balance of phosphorus was between 97.7 and 98.8%.

It has been reported that a large amount of oxidant can enhance the oxidation reaction and convert combustible gases into H2O and CO2, thereby leading to a decreased H2 yield.28 Our experimental results coincided with these published results. The experimental results indicated that a greater number of carbohydrates and phosphorus organic compounds were decomposed with the addition of H2O2. As the H2O2 coefficient increased, the oxidation reaction became dominant in the supercritical water. H2 and CH4 were converted to CO2 and H2O, with an excess of H2O2, which led to a decrease in the H2 yield. The maximum H2 yield was obtained without the addition of H2O2. In addition, an increase in the decomposition of organic compounds and the combustion of gas containing carbon with the addition of an oxidant to the SCWG process contributed to a higher CO2 yield, as well as increased GE and CE values.

The organic phosphorus contained in sewage sludge is composed of a collection of phospholipids, phosphonic compounds, nucleic acids, phosphoproteins, etc. In SCWG, the sufficient H2O2 primarily promotes the oxidation and decomposition of organics in sludge into small molecule intermediate products at the initial and middle stages. The P–C bond is promoted to crack with the addition of H2O2 and OP was converted completely to IP, resulting in an increase of the IP yield and TP yield in the solid residue.

### 2.2. Effect of the Combination of K2CO3 and H2O2 on Gas and Phosphorus Yields

The effect on the gas and phosphorus yields by simultaneously adding K2CO3 and H2O2 was investigated at 460 °C and 27 MPa after 6 min.

We first assumed that the catalytic effects of H2O2 and K2CO3 were independent. When they were added in different proportions, the calculated value of hydrogen production for the mixture was assumed to be the sum of the results from H2O2 and K2CO3 catalyization separately.

As shown in Table 2, there were large differences between the theoretical values and experimental data, indicating that H2O2 and K2CO3 have a synergistic effect on the reaction. The magnitude of the difference between the theoretical values and

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### Table 1. Comparison of Hydrogen Yield Obtained from This Study with the Literature (460 °C, 27 MPa, 6 min)

| Feed            | Catalyst        | Hydrogen Yield | Refs |
|-----------------|-----------------|----------------|------|
| Sludge          | KOH, K2CO3, NaOH, Na2CO3 | 15.49 mol/kg  | 28   |
| Dewatered sewage sludge | H2O2/Ni | 0.79–1.28 mol/kg of organic matter | 25 |
| Sewage sludge   | none | 38.5–39.4 vol % | 24 |
| Dewatered sewage sludge | NaOH, K2CO3, Na2CO3, Ca(OH)2 | 3.45 mol/kg of organic matter | 15 |
| Dewatered sewage sludge | Formic acid | 10.07 mol/kg of organic matter | 29 |
| Dewatered sewage sludge | NaOH and Ni | 4.8 mol/kg of matter | 30 |
| Horse manure    | Na2CO3 | 5.31 mmol/g | 31 |
| Semicoke        | K2CO3 | 85.90 mol/kg | 32 |
| Timothy grass  | KOH | 30.6 mol/kg | 33 |
| Bagasse         | KOH | 75.6 mol/kg | 34 |
| Landfill leachate | NaOH | 70.05 mol/kg | 35 |
experimental data first increased and then decreased, which indicated that the K$_2$CO$_3$:H$_2$O$_2$ ratio also affected the reaction.

To investigate this further, both K$_2$CO$_3$ and H$_2$O$_2$ were added to the SCWG of sludge at various ratios. As mentioned above (shown in Figure 2), when the H$_2$O$_2$ oxidation coefficient was greater than 1.0, oxidation rather than gasification dominated in the supercritical water. To investigate the synergistic effect of K$_2$CO$_3$ and H$_2$O$_2$ on the SCWG reaction, the amount of H$_2$O$_2$ in the additive mixture (K$_2$CO$_3$ + H$_2$O$_2$) was below the theoretical demand for oxidation. When the K$_2$CO$_3$:H$_2$O$_2$ ratio was 1:1, the amount of K$_2$CO$_3$ was approximately 2 wt %. Five different K$_2$CO$_3$:H$_2$O$_2$ ratios were investigated. As shown in Figure 3a,b, the H$_2$ yield increased from 5.47 to 29.06 mol/kg as the K$_2$CO$_3$:H$_2$O$_2$ ratio increased from 1:4 to 4:1. The H$_2$ yield obtained with a K$_2$CO$_3$/H$_2$O$_2$ ratio of 1:1 was greater than 21.5 mol/kg, which was obtained with 2 wt % K$_2$CO$_3$. The CO$_2$ yield decreased from 20.35 to 8.39 mol/kg and then increased to 11.4 mol/kg. The inflection point of the K$_2$CO$_3$/H$_2$O$_2$ ratio was observed at 1:1. The trends of the GE and CE were similar to that of the CO$_2$ yield.

The effect of the combination of K$_2$CO$_3$ and H$_2$O$_2$ on the phosphorus yield was also determined, as shown in Figure 3c,d.

Table 2. Comparison of Calculated Values and Experimental Data of Hydrogen Yield from SCWG of Sludge (460 °C, 27 MPa, 6 min)

| hydrogen yield (mol/kg) | with only H$_2$O$_2$ (wt %) | with only K$_2$CO$_3$ (wt %) | calculated value | experimental data with K$_2$CO$_3$ and H$_2$O$_2$ |
|------------------------|-----------------------------|----------------------------|------------------|----------------------------------|
| 19.86 | 19.86 | 39.72 | 5.47 |
| 3.37 | 21.50 | 24.87 | 8.75 |
| 2.29 | 37.81 | 40.10 | 22.65 |
| 0.59 | 47.52 | 48.11 | 23.70 |

Figure 2. Effect of the amount of H$_2$O$_2$ on the gas and phosphorus yields from the SCWG of sludge at 460 °C and 27 MPa after 6 min: (a) gas yield, (b) GE and CE, (C) IP and OP yields, and (d) TP yield in the liquid production and solid residue.
The IP yield decreased from 95.9 to 90.49% as the K₂CO₃/H₂O₂ ratio increased. This result coincided with those for K₂CO₃ and H₂O₂ separately. However, OP increased as the ratio increased. The fraction of TP in the solid residue first decreased from 71.94 to 46.78% and then increased to 50.23%. When the amount of H₂O₂ increased, OP was almost completely converted to IP.

As shown in Figure 3a, the mixing ratio of K₂CO₃ to H₂O₂ affected the H₂ production and phosphorus yield. When the ratio of K₂CO₃ to H₂O₂ was less than 1:1, the amount of H₂O₂ was greater than that of K₂CO₃ and incomplete oxidation dominated in the SCWG process. Xu et al.²⁷ reported that H₂ and CO will be fully oxidized to H₂O and CO₂ in case of excess of oxidant, which results in a decreased H₂ yield. However, because of the incomplete oxidation with insufficient H₂O₂, CO will be generated from hydrocarbons in sludge. The production of CO promoted the water gas shift reaction (eq 1), enhancing the H₂ yield. With K₂CO₃, the yield of hydrogen is higher than that without K₂CO₃ (Table 3). The K₂CO₃ alkali catalyst had more effect on the water gas shift reaction. In contrast, the effect of K₂CO₃ on gasification was much stronger than that of H₂O₂ when the ratio was greater than 1:1. In the SCWG process, H₂ was mainly derived from the reforming reactions of small intermediates.⁴¹

In the absence of K₂CO₃ or H₂O₂, it is difficult for high-molecular-weight organic compounds to break down into low-molecular-weight compounds. When H₂O₂ was added, the gasification products mainly consisted of low-molecular-weight linear compounds. Meanwhile, complex organic matters such as cyclic compounds were enhanced to open the ring, resulting in the generation of low-molecular-weight linear compounds. Further gasification of these intermediates will be promoted.

Figure 3. Effect of the ratio of K₂CO₃ to H₂O₂ on the gas and phosphorus yields from the SCWG of sludge at 460 °C and 27 MPa after 6 min: (a) gas yield, (b) GE and CE, (C) IP and OP yields, and (d) TP yield in the liquid production and solid residue.
with the addition of the K$_2$CO$_3$ catalyst. The compounds in the raw sewage sludge solution and the liquid gasification products are shown in Table 3. As shown in Table 3, the main compounds in the raw sewage sludge solution are dimethoxybutane and other complex carbohydrates, with small amounts of nitrogen and phosphorus compounds. In the chromatogram, the ratio of the peak area of dimethoxybutane is 13.25%, which is approximately twice the area of any other compounds before gasification. After gasification in supercritical water, the main compounds in the liquid products are phenolic compounds and a few nitrogen compounds with small molecular weights. Methylbenzaldehyde is the main compound in the liquid products, with a 10.98% peak area ratio in the chromatogram.

3. CONCLUSIONS

Few studies have attempted to simultaneously recover phosphorus and generate syngas from sludge using SCWG. However, the influence of alkali catalysts on phosphorus recovery in the SCWG of sludge has not been reported. The effect of the combination of K$_2$CO$_3$ and H$_2$O$_2$ on the hydrogen production and phosphorus yield was investigated for the first time in this work. When K$_2$CO$_3$ was used as a single additive, GE and CE were improved with the increase of K$_2$CO$_3$. Hydrogen yield was approximately three times that without a catalyst. The OP in the sewage sludge could be eliminated and converted to IP by increasing the K$_2$CO$_3$ concentration. However, when H$_2$O$_2$ was added separately to the SCWG process, the H$_2$ yield sharply declined and carbon dioxide was the dominant gaseous gasification product. Furthermore, more than 97.8% of the OP was converted to IP. H$_2$O$_2$ and K$_2$CO$_3$ have a synergistic effect on the gasification. With the increase of the ratio of K$_2$CO$_3$ to H$_2$O$_2$, the K$_2$CO$_3$ catalyst had more effect on the water gas shift reaction, resulting in improvement of the hydrogen yield. Meanwhile, under the catalysis of K$_2$CO$_3$, more organic phosphorus can be completely converted into inorganic phosphorus (IP), resulting in a decrease of the TP yield and an increase of the IP yield.

4. MATERIALS AND METHODS

4.1. Materials. Sewage sludge was provided by the Shuangqiao Sewage Treatment Plant in Zhengzhou, China and was stored in a preservation box at less than 4 °C prior to use. The compositions of the sewage sludge are given in Table 4. The K$_2$CO$_3$ catalyst and oxidant H$_2$O$_2$ (30 wt %) were purchased from Tianli Chemical Reagent Co., Ltd. and Tianjin Fuchen Chemical Reagent Co., Ltd., respectively. All of these reagents were analytically pure.

4.2. Supercritical Water Gasification System. The SCWG of the sewage sludge was performed in an HC276 stainless steel batch reactor, which was purchased from the Huaan Supercritical Extraction Co., Ltd., Nantong, Jiangsu, China. The reactor was described in detail in an earlier study. In a typical experiment, 3.0 g of sewage sludge with deionized water (100 mL) was added in the reactor. Different amounts of K$_2$CO$_3$ catalysts were mixed with the sewage sludge. Then, this mixture was loaded into the reactor. The reactor was electrically heated at an average heating rate of 3 °C/min. Once the reaction temperature (460 °C) was reached, the reaction was maintained for 6 min. At the end of each reaction, the reaction was quenched with deionized water to stop the reaction.
experiment, heating was stopped and the reactor was rapidly cooled to room temperature with cooling water. After the reactor had cooled, gas and liquid samples from the reactor were collected and submitted for analysis. Methods for the collection and separation of solid and liquid products have been described previously in detail.42

4.3. Analytical Methods. An Elementar Vario-III was used to analyze the organic elemental of sewage sludge. Furthermore, Agilent 890A/5975C was used to analyze the gasification liquid product, which was equipped with a DB-SMS column (30 m × 0.25 mm id, 0.25 μm film thickness) using high-purity helium as the carrier gas. Mass spectrometry was performed in a selected ion-monitoring mode. The mass spectrometry (MS) transfer line temperature was 270 °C, and the ion source temperature was 220 °C. The column temperature was initially held at 60 °C for 5 min, increased to 270 °C at a rate of 30 °C/min, and held at this temperature for 5 min. The injection volume was 10 μL. The total flow rate of the carrier gas was 25.0 mL/min, and the split flow rate was 20.0 mL/min.

The gas yield (G), H2, CH4, CO2, and CO concentrations. Furthermore, Agilent 890A/5975C was used to analyze the gasification liquid product, which was equipped with a DB-SMS column (30 m × 0.25 mm id, 0.25 μm film thickness) using high-purity helium as the carrier gas. Mass spectrometry was performed in a selected ion-monitoring mode. The mass spectrometry (MS) transfer line temperature was 270 °C, and the ion source temperature was 220 °C. The column temperature was initially held at 60 °C for 5 min, increased to 270 °C at a rate of 30 °C/min, and held at this temperature for 5 min. The injection volume was 10 μL. The total flow rate of the carrier gas was 25.0 mL/min, and the split flow rate was 20.0 mL/min.

The gas yield (G), the TP yield. The OP yield was obtained by subtracting the IP yield from the TP yield.

The OP yield was obtained by subtracting the IP yield from the TP yield.

The oxidation coefficient was defined as:

\[
\text{oxidation coefficient} = \frac{\text{the actual H}_2\text{O}_2 \text{ added}}{\text{the theoretical H}_2\text{O}_2 \text{ demand}}
\]

(8)

The gas yield (G_{H2}, G_{CH4}, G_{CO2}, and G_{CO}), GE, and CE have been described in detail in an earlier study.42

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Notes

The authors declare no competing financial interest.

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