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Influence of Potassic Additives on Sludge Gasification Under Model Flue Gas Atmosphere

Mi Yan¹ · Jie Lin¹ · Ekkachai Kanchanatip^{1,2} · Sicheng Zhang¹ · Guobin Wang¹ · Dwi Hantoko¹ · Haryo Wibowo¹ · Yanjun Hu¹

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Abstract

In this study, the influence of potassic additives (K_2CO_3 and biomass ash (BA)) on sludge gasification under model flue gases was investigated. Two model gases consisting of $O_2/CO_2/N_2$ and $O_2/CO_2/H_2O/N_2$ were used as gasifying agents. Under $O_2/CO_2/N_2$ atmosphere, the lower heating values (LHV) of produced gas were in a range of 3.07–3.79 MJ/Nm³, and cold gas efficiency (CGE) were in a range of 47–66%. When steam was introduced into the gasification atmosphere, the efficiency of the process significantly improved. LHV of the produced gas under $O_2/CO_2/H_2O/N_2$ atmosphere was in a range of 6.48– 7.18 MJ/Nm³, and CGE was in a range of 73–88%. K_2CO_3 had an obvious catalytic effect on sludge gasification process. Additionally, K_2CO_3 had stronger catalytic effect under steam-free atmosphere and it was better in promoting CO generation when compared with BA. BA exhibited the ability to increase gas yield under steam-free atmosphere, while it only had a slight effect under steam-containing atmosphere. Based on continuous monitoring of gas during gasification process, LHV of the produced gas could be increased by nearly twofold, possibly up to 11.96 MJ/Nm³, by controlling the proper reaction time (10–12 min) to prevent the dilution effect from excessive gasifying agents.

Keywords Gasification \cdot Potassic additives \cdot Sludge \cdot CO₂ \cdot Syngas

Statement of Novelty

Sludge gasification under model flue gas was conducted to assess the feasibility of using flue gas as gasifying agent. It was observed that LHV of the produced gas and CGE were higher under $O_2/CO_2/H_2O/N_2$ atmosphere than those values under $O_2/CO_2/N_2$ atmosphere. In addition, more H₂ was formed in the presence of H₂O. Both K₂CO₃ and BA mainly promoted CO and H₂ production. The additives had stronger catalytic effect under steam-free atmosphere. Moreover, flue gas can be used as a gasifying agent as well as heat carrier for endothermic reaction of gasification. K₂CO₃ works effectively for gasification under both flue gas with and without steam atmospheres.

Introduction

Sewage sludge is a by-product from wastewater treatment plant which enriched by pollutants during wastewater treatment process, so it must be treated in a safe and effective manner [1, 2]. The annual production of mechanically dewatered sewage sludge in China is over 40 million tons with roughly 80% moisture. This large amount of sludge has become a big challenge to the sustainable development of cities. It was also reported that around 80% of sewage sludge was improperly disposed, while the rest was disposed through landfill (13.4%), land application (2.4%), building material (0.24%), and incineration (0.36%) [3]. However, these methods have weaknesses, such as large area occupation, long retention time, and low efficiency [3].

A few studies have reported that calorific value of dried sludge could reach 12 MJ/kg, making it potential to be utilized as energy resource [4, 5]. Thermochemical gasification is the process of converting carbonaceous materials into combustible gases by reacting it with a certain amount of gasifying agent at a high temperature. The main factors determining the outcome of gasification include gasifying

Ekkachai Kanchanatip k_ekk@hotmail.com

¹ Institute of Energy and Power Engineering, Zhejiang University of Technology, Hangzhou 310014, China

² Center of Excellence in Environmental Catalysis and Adsorption, Thammasat University, Pathumthani 12120, Thailand

agent or gasifying atmosphere [6–9], temperature [10], catalyst [11, 12] and type of reactor [13, 14]. This study would focus on two of those factors, namely gasifying agent and catalyst.

The gasifying agents play an important role on the performance of gasification in term of syngas yield, syngas composition, and cold gas efficiency (CGE). The commonly used gasifying agents are air, steam, and oxygen [15–17]. Air is the cheapest gasifying agent and widely used in biowaste gasification due to its abundant availability. However, the produced syngas has relatively lower calorific value due to the dilution effect of nitrogen [15]. The use of CO_2 and H_2O offers the higher heating value of product gas compared with that obtained from air gasification [16]. Introduction of steam in biomass gasification enhances the concentration of H₂ in syngas since steam plays key role as a hydrogen source for gasification [17]. Recently, some studies have been focusing on the use of CO₂ as gasifying agent in gasification process [18]. The use of CO₂ provides several benefits such as reducing greenhouse gas emission, allowing high carbon conversion, reducing soot formation, and enhancing CO production in syngas [9, 16, 19]. Sadhwani et al. [20] conducted CO₂ gasification of southern pine wood in a benchscale atmospheric bubbling fluidized bed gasifier. The effect of temperature and CO₂/C ratio have been studied. It was observed that CO₂ gasification produced high microporous char and enhanced the conversion rate. The introduction of CO₂ on biomass gasification resulted in higher concentration of CO and decreased H_2 [9, 19, 21].

In chemical reactions, catalysts help promote or inhibit the formation of certain reaction products without itself being consumed as a reactant. Catalysts in gasification process mainly include alkali metals, metals based catalyst and minerals [22]. Alkali metals are the most commonly used catalysts in gasification industry due to their relatively low cost. It has been shown that K_2CO_3 is the most effective catalyst for gasification compared with Na_2CO_3 , CaCO₃, CaO, and MgO [23].

Dual-bed gasifier technology has advantages in terms of operational stability and material adaptability [24, 25]. Figure 1 shows the proposed process diagram of dual-bed gasifier which consists of a gasifier and a combustion chamber. The high heat recovery from flue gas can be achieved and gas component in the flue gas can be utilized as gasifying agent. The existing boilers such as in coal power plants or municipal solid waste incinerators could be utilized as the combustion furnace. This would allow co-disposal of sludge and conventional fuel, which will reduce the cost of sludge disposal. In order to adjust the atmosphere of the gasifier, hot air could be introduced into the gasifier to control the concentration of oxygen.

This study investigated the influence of potassic additives (K₂CO₃ and BA containing alkaline metals, mainly



Fig. 1 Dual bed gasification system

ie %, air dried basis)
%, air dried basis)
9
4
, dry basis)
2
9

 Table 1
 Properties of sludge sample

potassium) on syngas production under model flue gas atmosphere. The results and observations are expected to provide theoretical guidance for optimizing the operation of gasification under flue gas atmosphere.

Methodology

Material

Sludge sample was collected from Chengxi Sewage Treatment Plant in Hangzhou, China. The sludge was mechanically dewatered (81.58% moisture content), dried (5.76%moisture content), and ground to pass 100-mesh sieve (<0.15 mm). The proximate analysis and ultimate analysis of the dried sludge sample are presented in Table 1. A large amount of sludge sample was collected in one time to ensure homogeneity of sample and its abundance for the whole experiments.
 Table 2
 Chemical compositio

 of BA (wt%)
 Page 100 (wt%)

nposition	Element	K	Si	Mg
	Content	27.77	11.60	6.27





Cl

3.09

Р

1.95

S

1.50

Al

0.98

Potassium carbonate (K_2CO_3) and biomass ash (BA) were used as catalysts for sludge gasification. K_2CO_3 (purity: 99.99%) was purchased from Shanghai Lingfeng Chemical Reagent Co., Ltd and used without any further purification. BA was obtained by burning palm oil shell at 800 °C in a muffle furnace. The chemical composition of BA was examined by X-ray fluorescence spectrometer, and the results are presented in Table 2. The dominant composition of BA was

potassium (27.77%). Alkaline metals in the BA are expected

to positively affect the gasification process [26].

Experimental Set-up

A downdraft fixed bed gasifier was used in the experiment, the main part of the installation consists of a quartz fixed bed tubular gasifier with an inner diameter of 30 mm and length of heating section of 300 mm and an electric furnace with PID temperature controller. The experimental sludge gasification system is illustrated in Fig. 2. In this study, model flue gas was used as a gasifying agent. The flow rate and the composition of model flue gas were individually controlled by mass flow controllers. All experiments were conducted at 950 °C with the total gasifying agent flow rate of 300 ml/ min. The composition of different model flue gases are shown in Table 3.

5 g of dried sludge were used in each experiment. The potassic additives were added into sludge at 6% by weight. The reaction time was set to 20 min. The gas evolution profile during gasification process was examined by collecting the produced gas sample periodically in gas sampling bags. Afterward, the composition of produced gas was analyzed by gas chromatography (Fuli GC-9790, China) with thermal conductivity detector and equipped with TDX-01 column. High-purity helium (99.99%) was used as the carrier gas at a flow rate of 30 ml/min. The total volume of produced gas was measured by water displacement method. The tar composition was analyzed by using GC-MS (JEOL-JMS, Q1050GC Master Quad) with DB5 column (30 m × 0.25 mm × 0.25 µm). Every experiment has been carried out with two replications and the data presented were the average values of two experiments.

Data Interpretation

The total gas yield, combustible gas yield, lower heating value (LHV), and CGE were determined to evaluate the process efficiency. All calculations were based on the gas volume at [normal temperature and pressure (NTP), taken as 20 °C and 1 atm] The total gas yield was defined as the total gas volume obtained from gasification process per one kilogram sludge and was calculated using Eq. (1).

Total gas yield
$$\left(\frac{L}{kg}\right) = \frac{V_{gas}}{m_{sludge}}$$
 (1)

The combustible gas yield was defined by the volume of H_2 , CO and CH_4 in the obtained gas per 1 kg sludge and was calculated using Eq. (2).

Na

0.85

Table 3The experimentalconditions

Model flue gas com-	Experiment no.								
position	NS1 ^b	NS2	NS3	WS1 ^c	WS2	WS3			
$\overline{O_2^a}$	16	16	16	16	16	16			
CO ₂	20	20	20	20	20	20			
H ₂ O	0	0	0	40	40	40			
N ₂	64	64	64	24	24	24			
Additives	None	K ₂ CO ₃	BA	None	K ₂ CO ₃	BA			

^aGas composition are in volume percentage (vol%)

^bModel flue gas atmosphere without steam $(O_2/CO_2/N_2)$ is denoted as NS

^cModel flue gas atmosphere with steam (O₂/CO₂/H₂O/N₂) is denoted as WS

Combustible gas yield $\left(\frac{L}{kg}\right)$ = $(\phi H_2 + \phi CO + \phi CH_4) \times Total gas yield$ (2)

The LHV of the gas was calculated based on the content of the gas components that have combustion value (H_2 , CO, CH₄) using Eq. (3) [27].

LHV_{gas} (MJ/Nm³)
=
$$\frac{(107.98 \times \varphi H_2 + 126.36 \times \varphi CO + 358.18 \times \varphi CH_4)}{1000}$$
 (3)

CGE is the ratio of the energy content in the gas to the energy content in sludge feedstock. CGE was calculated according to Eq. (4).

$$CGE = \frac{LHV_{gas} \times V_{gas}}{LHV_{sludge} \times m_{sludge}}$$
(4)

where V_{gas} (L) is the volume of the obtained gas at NTP, m_{sludge} (kg) is the mass of sludge, ϕH_2 , ϕCO , and ϕCH_4 are volume fractions of H₂, CO and CH₄, respectively, at NTP, LHV_{gas} (MJ/Nm³) is lower heating value of gas at NTP, LHV_{sludge} (MJ/kg) is lower heating value of sludge.

Result and Discussion

The experimental results of sludge gasification under model flue gases are shown in Table 4. There were notable differences in the produced gas components and gas yields between two different flue gas conditions (NS and WS). N₂ concentrations were maintained consistent among steamfree (NS) atmosphere series (64%) and with steam (WS) atmosphere series (24%). N₂ is inert so it does not involve in the reaction. However, it will affect the LHV of producer gas as dilution effect, which can be seen that the volume of total gas in NS was higher than that of WS atmosphere but the heating value of total gas was lower. Under flue gas atmosphere without steam (NS), the combustible gas yields were in a range of 255.1–363.8 L/kg, the LHV of produced gases were in a range of 3.07–3.79 MJ/Nm³, and the CGE were in a range of 47.02-65.56%. In contrast, under flue gas with steam (WS), the combustible gas yields were in a range of 407.2-508.9 l/kg, the LHV of produced gases were in a range of 6.48–7.18 MJ/Nm³, and the CGE were in a range of 73.03-87.98%.

Effect of additives on the sludge gasification under O₂/CO₂/N₂ atmosphere

The effect of different flue gas atmosphere and catalyst addition on the gas yield and gas composition is presented in Fig. 3.

Table 4	Experimental results
of sludg	e gasification under
different	t conditions

No.	Volume fraction (%)				Total gas	Combustible	LHV (MJ/Nm ³)	CGE (%)	
	H ₂	СО	CH_4	Other ^a	yield (l/kg)	gas yield (l/kg) ⁶			
NS1	8.86	8.85	2.78	79.51	1245	255.1	3.07	47.02	
NS2	8.65	14.32	2.94	74.09	1404	363.8	3.79	65.56	
NS3	8.77	12.48	2.98	75.77	1376	333.4	3.59	60.78	
WS1	23.74	15.11	5.61	55.54	916	407.2	6.48	73.03	
WS2	24.39	21.65	5.06	48.90	996	508.9	7.18	87.98	
WS3	23.61	16.21	5.67	54.51	946	430.3	6.63	77.13	

^aOther gas components are mainly N₂, CO₂, and O₂

^bGas components that have combustion value, namely H₂, CO, and CH₄



Fig. 3 Effect of flue gas atmosphere and catalyst on **a** gas yield and **b** combustible gas fraction

According to experimental data under steam-free atmosphere (NS1–NS3), it was found that the additives enhanced the process efficiency on sludge gasification. Both gas yield and LHV of produced gases were obviously increased when K_2CO_3 and BA was added. The results listed in Table 4 showed that the CGE of the non-catalytic experiment was as low as 47.02%, and it was increased to 65.56% and 60.78% with the addition of K_2CO_3 and BA, respectively. As a result, the improvement of CGE catalyzed by K_2CO_3 (NS2) and BA (NS3) accounted for 39.43% and 29.26%, respectively, when compared with the non-catalytic reaction (NS1).

The results from Fig. 3a showed that K_2CO_3 (NS2) produced the highest yield of H_2 (121.4 l/kg), CO (201 l/kg), and CH₄ (41.3 l/kg) compared with other experimental conditions. CO yield increased about 82.7%, after K_2CO_3 was added. The BA had comparable effects to K_2CO_3 on both H_2 and CH₄ yields, though, the enhancement on CO yield was smaller. Figure 3b showed that the CO fraction in NS2 and NS3 were higher than that without additives (NS1) due to a large increase in CO yield. These results indicated that both K_2CO_3 and BA could promote the generation of CO.

The catalytic mechanism of K_2CO_3 in gasification has been investigated by many studies with different kinds of feedstock. The reaction mechanism is recognized as follows [28]:

$$K_2CO_3 + C \rightarrow 2K + CO + CO_2 \tag{R1}$$

$$2K + CO_2 \rightarrow K_2O + CO \tag{R2}$$

$$K_2O + CO_2 \to K_2CO_3 \tag{R3}$$

Under steam-free atmosphere, K_2CO_3 mainly promotes the generation of CO, and the presence of CO₂ is also beneficial to improve the catalytic effect of K_2CO_3 on gasification. These results are in good agreement with those obtained from catalytic study of K_2CO_3 in CO₂ gasification of biomass [29].

On the basis of produced gas composition, the gas evolution profiles of sludge gasification under $O_2/CO_2/N_2$ were evaluated, as shown in Fig. 4. The generation rate of combustible gases reached a maximum value around 2 min reaction time for H₂ and CH₄, while 5 min for CO, under three experimental conditions. CO generation also took a longer time to complete than that of H₂ and CH₄ in all profiles. The overall gas generation rate increased significantly with the addition of K₂CO₃ and BA. The maximum CO generation rate greatly increased from 86.1 to 182.3 ml/min (K₂CO₃) and 133.1 ml/min (BA), respectively. The addition of catalyst caused the waveform of CH₄ generation to become narrower. The generation rate of CH₄ increased from 81.4 ml/ min (NS1) to 114 ml/min (NS2) and 115.7 ml/min (NS3). The peaks of H₂ also became higher and sharper in both NS2 and NS3 (Fig. 4b, c), the generation rate of H₂ increased from 124.9 ml/min (NS1) to 218.9 ml/min and 148.4 ml/min in NS2 and NS3, respectively. In addition, the completion of CO and H₂ generation was slightly delayed when catalyst was added, while CH_4 shifted a little forward. According to the monitoring of gas evolution, it was found that the gasification reactions had mostly completed after 10 min. By optimizing the reaction and gas collection time, the dilution of the gasifying agents could be avoided. The fraction of combustible gases in the total gas could be enlarged and the heating value would be proportionally increased.

Effect of Additives on Sludge Gasification under O₂/ CO₂/H₂O/N₂ Atmosphere

According to the results of LHV and CGE in Table 4, the sludge gasification performed better under $O_2/CO_2/H_2O/N_2$ atmosphere than under $O_2/CO_2/N_2$ atmosphere in both with and without potassic additives. Moreover, the potassic additives, especially K_2CO_3 , had obviously positive influence in the gasification under $O_2/CO_2/H_2O/N_2$ atmosphere. The addition of K_2CO_3 increased the CGE from 73.03 to 87.98%. The ratio of combustible gases in produced gas



Fig. 4 Evolution rate curves of combustible gas products from sludge gasification under a NS1, b NS2, and c NS3

increased from 44.46 to 51.10% and the LHV of produced gas increased from 6.48 to 7.18 MJ/Nm³. The CO yield also significantly increased from 138.4 to 215.6 l/kg with addition of K₂CO₃, which accounted for 55.8%. The experimental results are similar to those of K₂CO₃ in lignite gasification under steam atmosphere [30]. In contrast, BA additive only had a slightly positive effect on sludge gasification under O₂/CO₂/H₂O/N₂ atmosphere. The improvement in CGE and LHV were < 6%, indicating that BA might not be a suitable additive for gasification under flue gas with steam atmosphere. By considering different gasification atmosphere, the enhancement of CO generation catalyzed by K_2CO_3 under steam atmosphere (55.8% in WS2) is weaker than under steam-free atmosphere (82.7% in NS2). Under flue gas with steam atmosphere, the catalytic reactions of K_2CO_3 are as follows [31]:

$$K_2 CO_3 + 2C \rightarrow 2K + 3CO \tag{R4}$$

$$2\text{KOH} + 2\text{H}_2\text{O} \rightarrow 2\text{KOH} + \text{H}_2 \tag{R5}$$

 $2\text{KOH} + \text{CO} \rightarrow \text{K}_2\text{CO}_3 + \text{H}_2 \tag{R6}$

$$2C + 2H_2O \rightarrow 2CO + 2H_2 \tag{R7}$$

Combustible gas evolution profiles over a time period of sludge gasification under O₂/CO₂/H₂O/N₂ atmosphere are shown in Fig. 5. Compared with NS1-NS3 conditions, the gas generation was slightly delayed with the addition of steam. The highest generation rate of all gases could be found at around the 3rd min. When K₂CO₃ and BA were added, the peaks of curve notably shifted. With BA additive (WS3), the times of H₂ and CO peaks occurred at the 4th min instead of the initial 3rd min in WS1. The H₂ gas had highest formation rate in all cases. The maximum generation rate of produced gases was ranked in the order of $H_2 > CO > CH_4$. The gas evolution profiles showed that the gasification reactions had completed after approximately 12 min. Hence, the reaction time could be shortened from 20 to 12 min to minimize the dilution effect from gasifying agents. The LHV of produced gas could consequently be increased to 11.96 MJ/Nm³ in the case of K₂CO₃ additive (WS2).

Composition of Tar from Gasification of Sewage Sludge under Different Model Flue Gas Atmospheres

The composition of tar from the gasification under two model flue gases, namely $O_2/CO_2/N_2$ and $O_2/CO_2/H_2O/N_2$ with



Fig. 5 Evolution rate curves of combustible gas products from sludge gasification under a WS1, b WS2, and c WS3

different additives are listed in Table 5. The major components detected in all tars were the heavy-hydrocarbon with C_{10} - C_{20} . These were produced by the rupturing of aliphatic side chains non-aromatic rings which were then released as part of tar [32]. Three compounds, namely caprolactam ($C_6H_{11}NO$), tetradecane ($C_{14}H_{30}$), and octadecane ($C_{18}H_{38}$), were detected in all tar samples. The proportions of these three compounds were in a range of 34.85-63.92% of the total composition. Tetradecane $(C_{14}H_{30})$ was the main component in tar from most of the conditions, except WS3. In contrast, the dominant compounds in tar from WS3 was hexadecane (33.88%) followed by azulene (14.84%). Hexadecane ($C_{16}H_{34}$) was the main component in tar from WS3, accounting for 33.9%, followed by azulene (14.8%) and dodecane (10.4%). Phenol was found only in tar from NS2 (11.81%) and WS1 (7.74%). Butylated hydroxytoluene and 1,2-bezenedicarboxylic acid were found only in tar from the conditions with addition of K₂CO₃ (NS2 and WS2). In addition, it is noteworthy that cyclopentasiloxane (C10H30O5Si5) was found only in NS3 and WS3, which BA was added in the gasification. This compound may be formed by Si contained in BA.

Conclusions

In this study, K_2CO_3 and BA were added to sludge gasification process under model flue gas and the following results were obtained:

- Under O₂/CO₂/N₂ atmosphere, the combustible gas yields were in the range of 255.1–363.8 l/kg, and the dominant component was CO. LHV of produced gas was in a range of 3.07–3.79 MJ/Nm³, and CGE was in a range of 47.02–65.56%. Under O₂/CO₂/H₂O/N₂ atmosphere, the combustible gas yields were in the range of 407.2–508.9 l/kg, the dominant component was H₂. LHV of produced gas was in a range of 6.48–7.18 MJ/Nm³ and CGE was in a range of 73.03%–87.98%. The potassic additives mainly promoted CO generation in both flue gas atmospheres.
- Both K₂CO₃ and BA had more obvious catalytic effect under steam-free atmosphere (O₂/CO₂/N₂) than the atmosphere with steam (O₂/CO₂/H₂O/N₂). Under O₂/ CO₂/N₂ atmosphere, CGE increased by 39.4% and 29.3% with the addition of K₂CO₃ and BA, respectively. While under O₂/CO₂/H₂O/N₂ atmosphere, BA only slightly improved the cold gas efficiency Therefore, BA

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RT (min)	Components	Area (%)					
		NS1	NS2	NS3	WS1	WS2	WS3
6.45	Phenol	_	11.81	_	7.74	_	_
6.51	Dodecane	13.13	-	7.89	-	_	10.45
8.07	5,7-Dimethyl-undecane	-	7.25	-	9.35	13.71	-
9.21	Cyclopentasiloxane	-	-	11.44	-	-	7.45
10.16	Azulene	8.89	-	8.22	5.71	_	14.84
11.09	Caprolactam	12.04	8.84	9.56	11.97	11.61	10.04
11.29	Tetradecane	31.28	19.37	17.63	21.51	27.73	7.55
11.48	Benzeneaceticacid, a-3,4-tris[(tri- methylsilyl) oxy]-trimethylsilylester	-	-	11.37	-	7.6	7.93
13.58	6-Methyloctadecane	-	12.40	9.52	12.83	-	7.86
14.19	Hexadecane	20.59	12.37	7.67	13.72	17.61	33.88
14.32	Butylated hydroxytoluene	-	10.86	-	-	9.13	-
16.50	Octadecane	14.07	8.42	16.70	9.32	-	-
18.00	Dibenzo[b,e]-7,8-diazabicyclo[2.2.2]octadiene	-	-	-	7.85	_	-
20.22	1,2-Benzenedicarboxylic acid	-	8.68	-	-	12.61	-

Table 5 Tar composition fromsludge gasification

is not a suitable additive for the gasification under flue gas with steam.

- 3. Based on the combustible gas evolution profiles, the gasification reactions had finished since the 10th–12th min of reaction time. The gasification process could be improved by setting a shorter reaction time to reduce the dilution from gasifying agents. LHV of the produced gas could be increased by nearly twofold. The maximum LHV of 11.96 MJ/Nm³ could be achieved with K_2CO_3 additive under $O_2/CO_2/H_2O/N_2$ atmosphere.
- 4. Although the increase in percentage of CO generation in WS2 (55.8%) was lower than in NS2 (82.7%) but the volume of generated CO in WS2 (215.63 l/kg) was still higher than in NS2 (201.05 l/kg). Moreover, the yield of all combustible gases in WS2 were higher than in NS2, resulting in both higher LHV and CGE. By considering the results, gasification under flue gas with steam will give better performance than without steam.

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