

AN EXPERIMENTAL INVESTIGATION INTO MECHANISM OF HIGH TEMPERATURE STEAM GASIFICATION OF WASTEWATER SEWAGE SLUDGE

by

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High temperature steam gasification experiments were conducted to gasify sludge in an electrically heated tubular furnace at 650, 750, 850, 950, and 1050 °C. The obtained results develop a mechanistic understanding on steam gasification of sludge from starting constituents to final products. The introduction of steam led to a steam reforming of the tar and C_nH_m compounds, and a partial gasification of the solid char and CH₄, which contributed to the generation of rich H₂ and CO. The steam gasification process can be divided into three significant stages. As temperature increased, a decreasing absorbance amount of C-H_{ar} and C-O and a large reduction of C = O were accompanied with an evident reduction of tar yield and an increasing gas yield, especially the increase of H₂ and CO contents. The H₂ and CO were the indicator for the occurrence of tar and C_nH_m steam reforming. In addition, more micro-pores generated in solid char and a sharp increase of Brunauer-Emmett-Teller (BET) surface from 24.2 m²g⁻¹ to 75.4 m²g⁻¹ showed the occurrence of char decomposition. An increasing content of polycyclic aromatic hydrocarbons (PAH) with 2 and 3 rings and a significant decrease of PAH with 4-6 rings in tar showed the tar cracking and reforming of non-condensable intermediates.

Key words: *sewage sludge, gasification, steam, mechanism, intermediates, Fourier transform infrared*

Introduction

Municipal wastewater sewage sludge (SS) is considered an important renewable biomass energy source. Innovative thermochemical processes have attracted considerable scientific interests in many countries. Of the current available thermochemical conversion technologies, gasification treatment of SS are well proven technologies running at close to atmospheric pressure and temperatures from 700 to 1000 °C [1, 2]. It produces low quality but high heating value and high yields combustible bio-gas composed of H₂, CO, CH₄, CO₂, and C_nH_m. The feasibility of producing fuel gas from the gasification treatment of SS has been therefore investigated in some studies, suggesting that very high yields of H₂ and CO under the condition of high temperature and the presence of steam. Of the existing gasification medium, such as air, steam,

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oxygen, or a mixture of steam with either air or oxygen, the main advantages of steam-only gasification are superior carbon conversion, a higher yield of syngas, and a higher gas heating value because the dilution of the gas with nitrogen is avoided [3]. This gas can be utilized power gas engines and gas turbines or as a chemical feedstock to produce liquid fuels [4].

To maximize the production of fuel gases, tars and chars depending on the specific interest, the effect of gasification conditions on SS steam-only gasification have been investigated by some researchers [2, 5]. On the whole, chemical composition, mole ratio of H₂/CO, and heating value of the fuel gas produced from SS gasification are close to typical values obtained from other biomass. This indicates a potential of SS as a raw material for the steam gasification process. However, only a few reports have focused on the pathway of the formation of gasification products and the related mechanism due to the complex chemical reactions among organic compounds, gasifying agent, and non-biodegradable fractions. Nipattummakul *et al.* [5] studied the evolutionary behaviour of syngas characteristics from the steam-only gasification of SS at different temperatures. Roche *et al.* [6] investigated the influence of throughput and steam over the SS gasification products, and found that higher throughputs decreased the H₂ content of the syngas and increased tar production. Gil-Lalaguna *et al.* [7] found that temperature was found to be the most influential factor for most of the response variables analysed in the process of SS gasification. In order to optimize the technique of generating H₂-rich syngas from the steam gasification of SS, some new efforts regarding in-depth investigations into related mechanism are needed.

In this study, a better mechanistic understanding of SS steam gasification process from starting constituents to final products was developed. The aims were to investigate the formation and evolution behaviours of gasification products (syngas, tar, and solid char) obtained at different temperature (650, 750, 850, 950, and 1050 °C), and the gasification process and decomposition mechanism of SS focusing on the variation of organic functional groups and physical structure of products with temperature.

Experimental

Samples preparation

The SS samples were collected after mechanical dewatering using a filter press in a municipal wastewater treatment plant, Hangzhou, China. Sludge sample with around 80% of moisture content was firstly dried in open air for 2 days to remove most moisture content, and then were dried in a lab-scale air convection oven. The dried sludge was kept in an airtight container to prevent re-absorption of moisture before experimentation. The contents of ash, moisture, combustible matters, and fixed carbon of sewage sludge were 48.2%, 3.8%, 46.3%, and 1.7%, respectively, which were determined according to the National Testing Standard of Proximate Analysis of Coal [8].

Gasification procedures

A lab-scale tube furnace with heating area of 40 mm in diameter and 600 mm in length was used to simulate sludge gasification. Figure 1 shows the schematic diagram of SS gasification treatment. The furnace is constructed with quartz and is heated by a programmable temperature control, and can be adjusted from room temperature to 1100 °C. High temperature steam at atmospheric pressure was generated from a steam generator. The obtained steam was delivered by a quartz tube with the electrically heating band so that the input temperature of steam could be well controlled. High temperature steam then was introduced into the gasification reactor containing a

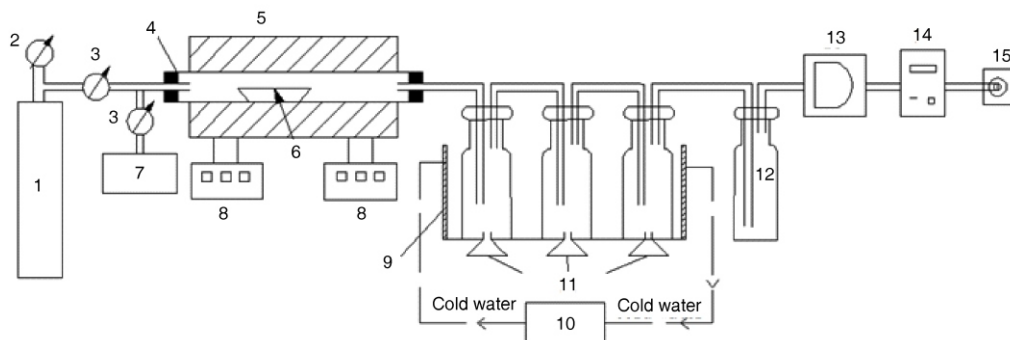


Figure 1. The schematic presentation of the experimental apparatus; 1 – nitrogen, 2 – pressure gauge, 3 – flow meter, 4 – quartz tube, 5 – furnace, 6 – quartz container, 7 – high temperature steam generator, 8 – programmable temperature controller, 9 – water tank, 10 – pump, 11 – condensed oil container, 12 – gas dryer, 13 – gas filter, 14 – flow meter, 15 – gas collecting bag

known amount of SS sample, 35 g (dry basis). For each experiment, the steam flow rate was set for 2.5 g/min and reactor temperature was varied as 650, 750, 850, 950, and 1050 °C. The evolved volatiles from the reactor were swept and passed through three consecutive condensers immersed in three ice baths, by which the condensed liquid fraction (tar) was collected. The non-condensable gas then passed through two filters which were filled with absorbent cotton and silica-gel desiccant in order to remove the residual moisture and other impurities. Finally, the clean gas was collected in a tedlar sample bag. The flow rate of the gas was measured by a flow meter. The solid char was collected and directly weighted. After each gasification experiment, the condenser and connection tubes were washed with methylene chloride. All the extraction liquid was dehydrated by anhydrous sodium sulphate and filtered to remove the fine particles.

Analysis methods

The dried SS and solid chars were grounded to fine particles for further analysis. Fourier transform infrared (FTIR) analysis was employed to identify the chemical functional groups in the original SS and solid chars after SS gasification treatment. To obtain observable FTIR spectra, the sample was mixed with KBr powder to prepare pellets. The mass ratio of the sample to KBr powder was 1:70. The spectra of the samples were measured using a Thermo Nicolet6700 Spectrometer, Thermo Nicole Corporation, USA. Infrared absorbance data were obtained for wave numbers in the range of 400-4000 cm^{-1} . The surface morphology of the original SS and solid chars were analysed by SEM (Hitachi S-4700). Microscopic surface area and distribution of pore volumes of the samples were determined from nitrogen adsorption data obtained from a surface area and porosity analyser, ASAP 2020. The BET method was used to calculate the surface area based on selected N_2 adsorption data within the p/p_0 range of 0.025-0.3.

Chemical composition of non-condensable gas was detected by a gas chromatograph GC-9790 equipped with thermal conductivity detectors (TCD). Nitrogen was used as a carrier gas at a constant flow of 20 mlmin^{-1} . The TCD was calibrated with a standard gas mixture at periodic intervals. Chemical composition of the pure tar was identified by GC-MS CP3800/saturn 2000. Around 10 g of the tar was sampled and diluted with dichloromethane solvent with a volume ratio of 1:1 and then centrifuged in order to separate residues. The column used in the GC was a 30 m \times 0.25 mm HP Ultra 2 with a thickness of 0.24 μm . The following temperature program was adopted: oven temperatures rose from 60 °C to 230 °C at a rate of 10 °C/min, where it

was held for 5 min, and then increased from 230 °C to 300 °C at a rate of 3 °C/min, where it was held for 0.67 min. One microliter of the sample was injected into the capillary column with an auto-sampler. The injector and ion source temperatures were both set at 310 °C. The carrier gas was N₂ of 99.9% purity and was provided with a flow rate of 1.0 mLmin⁻¹. The chromatographic peaks were identified with the help of NIST mass spectral data library.

Results and discussion

Gasification products distribution

The experimental results about the distribution of SS gasification products are presented in tab. 1. Because of the high ash content in the used SS (48.2%), the solid char is an important by-product in the gasification process. The solid yield moderately decreased from 54.8% to 47.5% as the temperature increased from 650 and 1050 °C. As demonstrated by other researchers, the decrease of solid fraction yield could be due to the devolatilization of the solid hydrocarbons and the partial gasification of the carbonaceous fraction in the char at a high temperature [1, 2, 9, 10]. The carbon content in the solid char reduced from 11.5 to 4.9% by increasing gasification temperature from 650 and 1050 °C, which was analysed using a Leco Truspec Micro Element Analyser. The fact could show that carbon reactions with steam are sensitive to temperature variation. In addition, some data of the solid yields are even below the original ash content in SS. It suggests that inorganic ash compounds could have been released to the gas during the gasification process. The transformation of ash compounds to gas phase fraction during thermochemical processes have been shown in some studies at a high enough temperature [7, 11]. The gas yield data from SS gasification varied between 0.66 and 1.95 m³ kg⁻¹ SS, which are close to the typical ones found in some previous studies for similar operation conditions of SS gasification [6, 7, 10]. The increase of the fuel gas yield as temperature increased could be due to the complex chemical reaction process during SS gasification that are favoured by higher temperature, such as cracking and steam reforming of tars and endothermic reactions of char gasification [12, 13]. The average tar content contained in the gas product ranges between 14.4 and 33.4 g/m³ gas. The rise in the gasification temperature from 750 to 850 °C resulted in a large reduction in tar production, which may be due to the enhancement of tar cracking and reforming reactions. As the temperature increased further from 850 to 1050 °C, tar content moderately decreased.

Gas composition

Syngas evolution vs. different temperature intervals

In order to study the evolution characteristics of the syngas during SS gasification process, gas samples were collected at different temperature intervals. Based on our previous study about TG and DTG thermal analysis [14], five temperature intervals were proposed, including the

Table 1. Yields of solid, gas, and tar products at different temperature

Production distribution	650 °C		750 °C		850 °C		950 °C		1050 °C	
Solid yield (g solid/100 g SS)	54.8	2.3	51.5	1.9	48.3	1.1	47.9	1.8	47.5	2.1
Carbon content in the solid product [wt.%]	11.5	0.8	9.6	0.7	5.6	0.5	5.1	0.3	4.9	0.4
Gas yield (m ³ /kg SS)	0.66	0.06	0.82	0.05	1.28	0.1	1.75	0.2	1.95	0.2
Tar content (g/g gas)	33.4	2.1	28.2	3.5	18.8	1.7	15.2	1.3	14.4	1.2

first stage: room temperature to 160 °C, the second stage: 160-410 °C, the third stage: 410-700 °C, the fourth stage: 700-850 °C residence time for 7 minute at 850 °C and the fifth stage: remaining residence at 850 °C for 20 minute. The main compositions of the gas collected at different temperature intervals were shown in fig. 2. The C_nH_m represents summation of produced gaseous hydrocarbons, such as ethylene, ethane and acetylene.

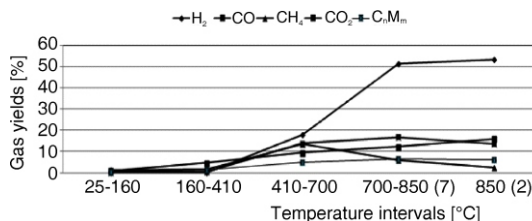
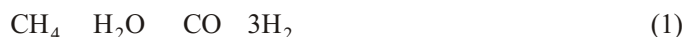


Figure 2. The gas composition at different temperature intervals

Two obvious enhancements were found for the volume content of H₂, which increased from 0.08% at second stage to 17.9% at the third stage, and from 17.9% at the third stage to 51.5% at the fourth stage. However, CO content increased almost linearly from 0.97% to 15.9% as the temperature intervals varied from the first stage to the fifth stage. The H₂ and CO contents reached their maximum values at the fifth stage, which were 53.5% and 15.9%, respectively. However, for the maximum value of CH₄ content were 13.5% in the gas collected from the temperature interval of 410 -700 °C. After 700 °C, the content of CH₄ was largely decreased. For CO₂, when remaining solid char existing in the gasifier longer such as under the temperature of 850 °C for 20 minute, its content obviously decreased. The result could be resulted from the chemical reaction between C and CO₂ to produce more CO and a secondary reaction from tar cracking favored high temperatures. But it can be noted that the first stage (room temperature to 160 °C) and secondary stage (160-410 °C) also contributed relatively higher contents of CO₂, CO, and CH₄ compared with the production of H₂. This maybe because pyrolysis reaction is more dominant reaction before 410 °C compared with gasification reaction during SS gasification.

In addition, it can be observed that long residence time and high temperature, such as the fifth stage for remaining residence at 850 °C for 20 min, would result in some active reactions between the steam and the intermediate products. High temperature intensified the dehydrogenation reaction, led to an increase in H₂ content that released from heavy hydrocarbon compounds. The H₂ can also be a good indicator for secondary cracking of tar. A steam-rich atmosphere was kept at high temperature could give rise to the endothermic reaction between the steam and gasification products, which also significant to H₂ production. The participation of steam at high temperatures can give rise to the steam reforming of the volatile compounds and the partial gasification of the solid carbonaceous char. The main reactions are steam reforming reaction. In addition, the composition of the collected gas samples was not only the result of tar cracking or char and tar gasification, but also the non-condensable products could undergo gas phase reactions with each other, such as the chemical reaction happened as followed steam gasification reaction in eqs. (1) and (4):



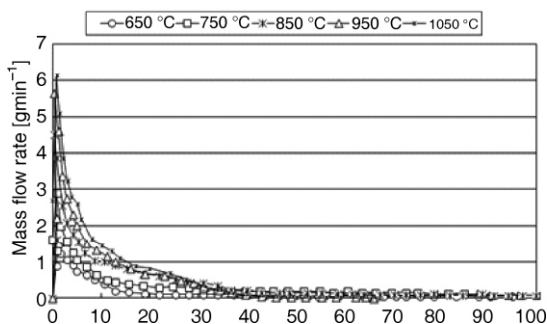


Figure 3. Gas-flow rates from SS gasification at different temperature vs. reaction time

Fuel gas evolution vs. reaction time

A comparison among gas-flow rates from SS gasification at 650, 750, 850, 950, and 1050 °C is presented in fig. 3. Results show that the influence of temperature on gas evolution rate is more obvious in the first 30 minutes for the 750, 850, 950, and 1050 °C cases than the rest of the gasification period, while in the first 17 minutes for the 650 °C cases. About 43% of gas yield was obtained at this stage. The prolonged reaction evolution at the 850, 950, and

1050 °C cases could be attributed to the intensified steam reformation reaction during the residence time at higher temperature. In addition, it was further confirmed that the pyrolysis reaction was more dominant reaction before 400 °C, and a large share of gas release in the first few minutes of gasification is due to pyrolysis. Some relative pyrolysis reactions are as eqs. (5) and (6),



The composition variation of the gas was influenced by two main stages, including the decomposition of the volatile functional groups in SS and the gasification of tar and char at high temperature. It can be observed that the decomposition of SS ended within around 35 minutes at gasification temperature 650 °C, while the process ended within approximately 85, 76, 65, 45, minutes at 750, 850, 950, and 1050 °C, respectively. The peak gas-flow rate occurred approximately between t the 7th and 12th minute for all cases. The maximum gas-flow rate was 6.1 gmin⁻¹, which was obtained at 1050 °C run. In addition, at all cases a process time of more than 40 minutes indicated a slow conversion. Comparing with the time duration of SS gasification with Nipattummakul's [5] studies, suggest a higher reaction rate in our case of SS. More complete chemical reactions among organic compounds could be related to more production of gas products. This could be dependent of the used reactor and a lower ash content in SS used by them.

Gas composition at different temperature

The results in tab. 2 indicate that SS steam gasification at different temperature produces a gas with a higher H₂ concentration and a lower CH₄ concentration. The H₂ content increased almost linearly from 40% to 52% as temperature increased from 650 °C to 1050 °C, which the result is basically consistent with other studies [5, 6]. In addition, the CO content increased significantly from 10% to 20%, and the concentration of CO + H₂ and H₂/CO ratio (mole/mole) reached their maximum value (72 vol.%) at 1050 °C, respectively. The high release of H₂ could be resulted from the presence of steam as the gasifying agent and the promoted secondary reaction by the increased temperature. Char gasification reactions were promoted by the increase in temperature to support high yield H₂ mole fraction. The increase in CO content with temperature increase was attributed to the reaction (1), (3), (5) and the Boudouard reaction, which the similar behaviours have been observed by other researchers [3, 7, 13]. Hydrocarbons

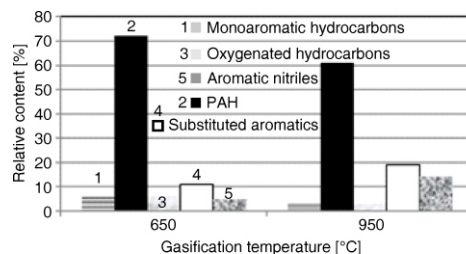
Table 2. The main composition of gas from gasification at different temperature

	Average content of main composition in the fuel gas [%]				
	H ₂	CO	CH ₄	CO ₂	C _n H _m
650 °C	40.3	11.2	6.0	19.7	22.9
750 °C	47.5	13.9	4.5	16.3	17.7
850 °C	51.0	14.8	3.2	15.7	15.3
950 °C	52.6	15.3	2.8	15.2	14.2
1050 °C	52.4	17.2	2.8	14.9	12.7

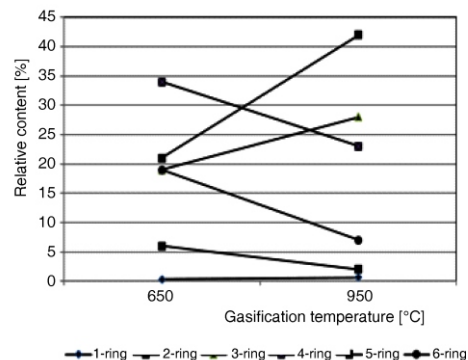
C_nH_m were decreased from the increase in temperature through steam reformation reactions. However, the contents of CH₄ and CO₂ decreased gradually with temperature increased. According to a previous references, the release of CO₂ at a relatively lower temperature (650 °C) is mainly caused by the breaking of carbonyl and carboxyl functional groups in sludge. But a slight increase of CO₂ at 850 °C can be observed at tab. 2, which was attributed to the promoted water-gas shift reaction at a higher temperature, reaction (4).

Tar composition at different temperatures

Based on the total typical ion count chromatograms of the tars obtain from the gasification of SS at 650 °C and 950 °C. Here, two cases (tars at 650 and 950 °C) were taken as the representatives to observe the composition variation of the tar generated as temperature increased. It was noted that the main qualitative components of the tars were almost the same at the different scopes, but the peak areas (their relative proportions) for the identified components present the evident difference. The results could indicate that the gasification temperature has a significant effect on the content of each component in tar. In this work, the main components qualitatively identified in the tars were divided into five groups: monoaromatic hydrocarbons, PAH, oxygenated hydrocarbons, substituted aromatics, and aromatic nitriles. In the two selected cases, most of the tar components were qualitatively identified by summing up the peak areas of each compound. The peak area of the same species were summed up and normalized to 100% to represent the relative contents of the main compounds. Therefore, the relative content of the five groups in the tars is presented in fig. 4(a). It can be seen that the group with the maximum content was PAH, which decreased evidently from 71.6% to 60.7% as temperature increased.



(a) The content of the five groups in the tars



(b) Variation of different rings of PHS

Figure 4. Main composition of the tar from SS gasification at 650 and 950 °C

In addition, the relative contents of substituted aromatics and aromatic nitriles increased drastically from 11.1% and 5.4% to 18.9%, and 13.8%, respectively. However, the oxygenated hydrocarbons decreased from 6.2% to 2.7%, and the relative contents of monoaromatic hydrocarbons at 950 °C were decreased more than 55% compared with that at 650 °C. This could be attributed to the secondary cracking and steam reformation reactions of tar under a higher gasification temperature. The results previously demonstrated that the increase of temperature was conducive to the reduction of the species in the tar. In particular, it is notable to concern with the distribution of PAH in the tar, which is necessary to further study.

The distribution of PAH was shown in fig. 4(b). It can be observed that the most abundant PAH in the two cases were relatively low molecular weight PAH (2-5 rings), such as naphthalene, biphenylene, phenanthrene, and fluoranthene and pyrene. Some similar observations have been found by other researchers [15]. As temperature increased from 650 °C to 950 °C, the PAH with 5 and 6 rings decreased obviously from 25% to 9%. The relative content of PAH with 4 rings decreased by 29% as well. In contrast, the PAH with 2 and 3 rings presented a major increase as the temperature increase. This is probably because a higher gasification temperature promoted the completion of steam reforming reaction of heavy hydrocarbons and parts of tar, reaction (1) and (2).

Surface chemistry and micro-pore structure of solid char

Figure 5 shows that FTIR spectra of the chars obtained from SS gasification at different temperature. The comparison between the surface chemistry of solid chars is targeted to better understand a relationship between the chemical structure of solid char and the composition of other gasification products. The FTIR spectra revealed that remarkable changes in chemical texture of the chars happened during SS gasification. The bands within 3000-3750 cm^{-1} assigned to the O-H and N-H stretching vibration tend to disappear since 850 °C, but only remained a slight peak at 650 °C and 750 °C. The two peaks at 2927 cm^{-1} and 2712 cm^{-1} disappeared and left only an imperceptible vestige, indicating that the decomposition of C-H bonds completely finished when reached a high enough temperature, such as 650 °C. Moreover, the large reduction of C = O stretching vibrations at 1649 cm^{-1} for all chars at different temperature demonstrates the breaking of the C = O bond with the generation of large amount of CO and CO₂. As gasification temperature increased further from 750 °C to 1050 °C, the absorbance

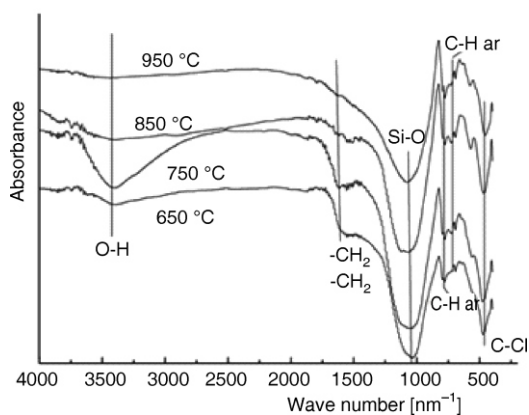


Figure 5. Surface chemical of different solid chars

amount of C-O and C-H_{ar} decreased sharply, indicating that cracking and reforming of aromatics have been intensified by high temperature, and the decomposition of volatile matter under the participation of high temperature steam to thereby generated large amount of H₂, CH₄, and C₂ hydrocarbons. It could be drew a similar conclusion to previous studies that H₂ yield could be significantly as temperature increase, and this is also consistent with the data results regarding H₂ production as temperature increased.

The fed steam can penetrate the internal pore of the SS to develop a large proportion of microspores. The pores on the surface of the solid char displayed reduced smooth

faces with increasing temperature, and the pore development was then well established. Meanwhile, the BET surface area of the chars increased from $26.5 \text{ m}^2\text{g}^{-1}$ to $37.9 \text{ m}^2\text{g}^{-1}$. It was also reported that the surface area of the solid char generated from SS pyrolysis increased with increasing reaction temperature by many researchers, which could be due to changes in the chemical structure of the fed SS during pyrolysis [16, 17].

Conclusions

The SS steam gasification process targeted to produce syngas can be divided into three stages: the dominant pyrolysis reaction at a relatively low temperature ($<410 \text{ }^\circ\text{C}$), the breaking of C-H bonds mainly led to evolution of CO_2 , CH_4 , and C_nH_m , the dominant steam gasification reaction ($410\text{-}750 \text{ }^\circ\text{C}$), the release of CO , CO_2 , and H_2 evolved mainly as the result of the decomposition of C-H and C = O bonds, and the intensified steam gasification and reformation reaction and the Boudouard reaction after $750 \text{ }^\circ\text{C}$, a large amount of H_2 and CO released as the result of the breaking of the C = O bond and the diminishing absorbance amount of O-H, C-H, and $-\text{CH}_2$ ($-\text{CH}_3$). As the temperature increased, the decreasing absorbance amount of C-H_{ar} and C-O and the large reduction of C = O was accompanied by an evident reduction of tar yield and an increasing gas yield, in particularly the content of H_2 and CO . The H_2 and CO were also the indicator for the occurrence of tar and C_nH_m steam reforming. The main components in the tars were monoaromatic hydrocarbons, PAH, oxygenated hydrocarbons, substituted aromatics, and aromatic nitriles. The group with the maximum content was PAH, which decreased evidently from 71.6% to 60.7% as temperature increased from 650 to $1050 \text{ }^\circ\text{C}$. Moreover, PAH with 2 and 3 rings presented a major increase while 4-6 rings presented a major decrease, demonstrating that high temperature favoured the secondary cracking and reforming of non-condensable intermediates. In addition, more micropores generated in solid char and a sharp increase of BET surface from $24.2 \text{ m}^2\text{g}^{-1}$ to $75.4 \text{ m}^2\text{g}^{-1}$ showed the occurrence of char decomposition.

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