



Co-liquefaction behavior of corn straw and Shengli lignite



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ABSTRACT

In this study, the co-pyrolysis and co-liquefaction properties of corn straw (CS) and Shengli lignite (SL) were investigated. Individual liquefaction and co-liquefaction of CS and SL were performed and the characterization of co-liquefaction products was tested. The synergetic effect was discussed. The pyrolysis of SL can be obviously promoted by addition of CS, resulting in the larger weight loss of their blends compared to calculated values of their individual pyrolysis. The individual liquefactions of CS and SL show that CS gives much higher liquefaction conversion than SL at the same liquefaction conditions. During the co-liquefaction of CS and SL, there exists an obviously synergistic effect, which may be attributed to that free radicals or intermediates produced by thermally degradation of CS at low temperature aid the depolymerization of SL, and that the new-formed free radicals or intermediates from co-liquefaction can be easily decomposed and hydrogenated. The characterization of asphaltene and pre-asphaltene shows that the heteroatom-contained structure in asphaltene and the aliphatic structures in preasphaltene prefer to convert into lighter product compared to individual liquefaction of CS and SL. The largest enhancements in conversion of 8.67% and oil yield of 6.46% were obtained at 4/6 and 6/4 blend ratio of CS/SL among different blends, respectively, with 375 °C temperature, 30 min reaction time and 4 MPa initial pressure.

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1. Introduction

With the quickly growth of energy demand and the reduction of petroleum reserves, the imbalance between supply and demand of petroleum products will become an increasingly prominent issue. Countries in the world vigorously develop new technologies about alternative liquid fuels.

China has abundant coal resources, including low rank coal reserves which account for about 13% of Chinese total coal reserves. The ineffective utilization of low rank coal is due to its properties about high moisture content, low calorific value and other short-comings. It is necessary to produce clean liquid fuels instead of petroleum products through direct coal liquefaction. Meanwhile, China is a developing country with abundant agricultural wastes, such as straw, rice husk and other biomass solid waste every year. Currently, these agricultural wastes commonly are generally used to incineration and landfill, which cause amount of serious environmental pollution and result in a waste of resources. According to the resources distribution, the development of co-liquefaction technology of coal and biomass is not only fits China's energy security but also benefits the utilization of resources.

In recent years, co-liquefaction technology of coal with biomass has been rapidly developed. Plenty of literatures showed that there exist a positive synergetic effect in the co-liquefaction of coal and biomass. The synergetic effect could promote conversion and increase oil yield under mild condition. Coughlin et al. [1,2] reported that liquefaction of bituminous coal could occur at temperatures as low as 300 °C when the lignin was present in the reaction mixture and believed that phenoxy radicals from the pyrolysis of lignin at relatively low temperatures

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could aid the depolymerization of coal through promoting the scission of aliphatic C-C bonds in the coal. Altieri et al. [3] showed that co-liquefaction of coal and lignin produced more benzene soluble fraction and pentane soluble oil, compared to the case of reacting coal and lignin separately. Lalvani et al. [4] reported that there existed synergistic in co-liquefaction of coal with lignin and significantly improved the quality and yield of the liquid products under moderate pressure and temperature conditions. Jung et al. [5] found that the conversion of coal increased with addition of paper, but the yield of oil and gas remained unchanged. Shui et al. [6] showed that the synergistic effects in co-liquefaction of sub-bituminous coal and sawdust attributed to the promotions of the pyrolysis and liquefaction of coal by free radicals or intermediates formed from pyrolysis of sawdust at low temperatures. Guo et al. [7] investigated the co-liquefaction of lignite and sawdust under syngas and found that there was a positive synergetic effect during co-liquefaction. Thus, there exist promotion effects on the co-liquefaction of coal and biomass under coal liquefaction condition. It depends on the species of coal and biomass, the ratio of blends and the reaction conditions.

In this study, the effect of different liquefaction condition (the ratio, the reaction temperature, the hydrogen initial pressure and the reaction time) in co-liquefaction of lignite and corn straw was investigated. The TG analysis of the blends of lignite and corn straw was performed under nitrogen atmosphere. The characteristic of co-liquefaction products was also tested. The synergetic effect in co-liquefaction of lignite and corn straw was discussed.

2. Experiment

2.1. Materials

Shengli lignite (SL) and corn straw (CS) were ground to pass through 80 meshes sieve as received, then they were dried under vacuum at 80 °C for 12 h before used. The proximate and ultimate analysis of CS and SL samples are given in Table 1. The liquefaction solvent is tetralin in all experiments. The solvents are commercial pure chemical reagent (purity higher than 99.5%) without further purification. The purity of hydrogen is 99.9%.

2.2. Liquefaction procedure

The total weight of the blends of CS and SL was kept constant at 1 g. 2 ml of solvent (tetralin), 5 wt.% (based on coal) of Fe-based catalyst, and elemental sulfur (S/Fe = 1.2, atomic ratio) were also added into a reactor (30 ml). Before the liquefaction experiment, the reactor was sealed and flushed three times with hydrogen, followed by pressuring the system to the desired initial pressure with hydrogen. The reactor, agitated vertically at 120 times per min, was submerged into a eutectic salt bath, which had been heated to the desired temperature, and maintained at that temperature for desired reaction time. Then the reactor was quenched to ambient temperature in a water bath, and the overhead pressure in the reactor was released slowly. The gaseous products were analyzed by GC. The liquid and solid products were divided into soluble and insoluble by washing with THF. The water content in THF soluble fraction was measured by Karl Fischer titration (GB/T 11146-2009). THF soluble fraction was precipitated with n-hexane. Then the n-hexane insoluble fraction were divided into soluble fraction (asphaltene, AS) and insoluble fraction (preasphaltene, PA) with toluene. The THF insoluble fraction (THFI) was dried at 80 °C for 12 h in a vacuum oven before weighing [6].

The yields of products are defined as follows:

$$w_{Gas} = \frac{M_{Gas}}{M_{daf}} \times 100\%$$

$$w_{Water} = \frac{M_{Water}}{M_{daf}} \times 100\%$$

$$w_{AS} = \frac{M_{AS}}{M_{daf}} \times 100\%$$

$$w_{PA} = \frac{M_{PA}}{M_{daf}} \times 100\%$$

$$w_{Residue} = \frac{M_{THFI} - M_{cat} - M_{ash}}{M_{daf}} \times 100\%$$

$$w_{Conversion} = 100\% - w_{Residue}$$

Table 1
Proximate and ultimate analysis of CS and SL.

Sample	Proximate analysis (wt.%)				Ultimate analysis (wt.%)			
	M _{ad}	A _d	V _{daf}	FC _{daf}	C _{daf}	H _{daf}	N _{daf}	S _{t,d}
SL	20.77	17.14	46.96	53.04	70.98	7.21	1.02	1.01
CS	6.82	4.27	75.21	24.79	49.00	5.47	0.40	0.15

daf: dry and ash-free base; M_{ad}: moisture on air dried base; A_d: ash on dry base; V_{daf}: volatile matter on dry and ash-free base; S_{t,d}: total sulfur on dry base.

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