**JOURNAL OF FUEL CHEMISTRY AND TECHNOLOGY**
Volume 44, Issue 3, March 2016
Online English edition of the Chinese language journal

**Cite this article as:** J Fuel Chem Technol, 2016, 44(3), 257–262

RESEARCH PAPER

# Shengli lignite liquefaction under syngas and complex solvent

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**Abstract:** The liquefaction of Shengli lignite (SL) were studied in the system of syngas ($H_2$+CO) and complex solvent ($H_2O$+THN) and the system of hydrogen ($H_2$) and tetralin (THN). Asphaltene (AS) and preasphaltene (PA) from the two systems were characterized by FT-IR. The results show that both conversion and oil yield of Shengli lignite liquefaction in the ($H_2$+CO)/($H_2O$+THN) system are significantly higher than those in the $H_2$/THN system. The conversion and oil yield under syngas ($H_2$:CO (volume ratio)=1:1) and complex solvent (THN:$H_2O$ (volume ratio)=1:1) reach respectively 88.79% and 55.47% at 400°C, 4 MPa and 30 min. The differences in the conversion and oil yield between the ($H_2$+CO)/($H_2O$+THN) and $H_2$/THN systems are 8.00% and 7.54%, respectively. This suggests that the water-gas shift reaction can produce active hydrogen in the ($H_2$+CO)/($H_2O$+THN) system, which benefits the hydrogenation of SL and PA. Meanwhile, THN can stabilize the free radicals from SL pyrolysis and dissolve the products (AS and PA) from SL liquefaction. The synergistic effect of two factors results in the improvement of conversion and oil yield. This study shows that it is a new lignite liquefaction technology using the ($H_2$+CO)/($H_2O$+THN) system.

**Key words:** syngas; complex solvent; Shengli lignite; direct liquefaction

Lignite accounts for 13% of total coal reserves in China. Presently, lignite utilizations mainly focus on extraction of humic acid and lignite wax[1,2], gasification[3–5], pyrolysis[6,7], liquefaction[8–10], and preparation of active carbon. Due to its low rank with high H/C ratio, lignite is a suitable feedstock for direct liquefaction with mild conditions, which can result in a low cost for direct coal liquefaction technology. In the world, some direct liquefaction technologies of low rank coals, such as BCL technology (Japan)[8], have been developed, and the engineering data of these technologies have been obtained. If lignite is used as raw materials for the traditional coal liquefaction, there will exist two problems. Firstly, a necessary drying process is adopted for lignite with high water content (the water in ignite is removed difficultly)[11], resulting in an elevated cost. Secondly, high oxygen content of lignite[12] can cause a high $H_2$ consumption to produce a large quantity of unvalued water, resulting in a decreasing economy of direct coal liquefaction. Thereby, it is very imperative to develop a suitable liquefaction technology for lignite utilization.

Based on the above point, this work made a comparison for SL liquefaction between ($H_2$+CO)/(THN+$H_2O$) system and $H_2$/THN system, investigated the effects of CO and $H_2O$ contents in the former system, and made a FT-IR analysis of asphaltenes and preasphaltenes produced in the above two systems, aiming to clarify a feasible replacement of syngas for pure $H_2$ in direct liquefaction of lignite with high water contents.

## 1 Experimental

### 1.1 Samples

Shenli lignite (SL) was from Inner Mongolia Autonomous Region of China. The sample was crushed into below 0.2 mm, dried in vacuum at 80°C for 24 h, and stored for test. Table 1 gives proximate and ultimate analyses of SL.

### 1.2 Liquefaction experiments

The liquefaction experiments were carried out in a 30 mL

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Received: 08-Jul-2015; Revised: 21-Dec-2015.
Foundation item: Supported by the National Natural Science Foundation of China (21476079, 21476080) and the Fundamental Research Funds for the Central Universities (WB1414014).

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micro-reactor[13]. For each run, the reactor was charged with 1 g of SL, 2 mL of solvents (THN or THN+$H_2O$), 3% of catalysts (the mass ratio of Fe and SL), and a certain amount of S (the atomic ratio of S and Fe is 1.2). Next, the reactor was sealed and purged by $N_2$ for three times and subsequently purged by the liquefaction atmosphere ($H_2$ or $H_2$+CO) before pressurizing to the desired initial pressure of 4 MPa, and then put into a salt bath preheated to 400°C. After the liquefaction process was maintained for 30 min, the reactor was quickly quenched to room temperature by cooling water. The gas products were collected by a gas bag for composition analysis by a GC analyzer, and liquid-solid mixtures in the reactor were collected for separation.

### 1.3 Products separation

Figure 1 shows the separation process of liquefaction products. After completion of liquefaction, the liquid-solid mixtures were recovered from the reactor by washing with THF and transferred into a cleaning flask. They were separated into THF-insolubles (THFI) and THF-solubles by an automatic Soxhlet extraction with THF. The THF-insolubles after being dried in vacuum were termed as THFI. The THF-solubles after removing THF by rotary evaporation were precipitated by *n*-hexane, and then filtered to produce filtrates and precipitates. The precipitates were again extracted by an automatic Soxhlet extraction with *n*-hexane to obtain *n*-hexane-insolubles and *n*-hexane-solubles. The mixtures of *n*-hexane-solubles and filtrates after removing *n*-hexane by rotary evaporation were termed as the *n*-hexane-soluble fraction (HS). The *n*-hexane-insolubles were extracted with toluene to produce toluene-solubles and toluene-insolubles. The toluene-solubles after removing toluene were termed as the *n*-hexane-insoluble and toluene-soluble fraction which was so-called asphaltene (AS). The toluene-insolubles were termed as the toluene-insoluble and THF-soluble fraction which was so-called preasphaltene (PA). The gas yield was calculated approximately according to the Ideal Gas State Equation, and the conversion and yields of other products were calculated according to Equations (1)–(4).

$$w_{AS}=\frac{M_{AS}}{M_{coal}}\times 100\% \tag{1}$$

$$w_{PA}=\frac{M_{PA}}{M_{coal}}\times 100\% \tag{2}$$

$$w_{con}=(1-\frac{M_{THFI}-M_{cat}-M_{ash}}{M_{coal}})\times 100\% \tag{3}$$

$$w_{oil}=w_{con}-w_{gas}-w_{AS}-w_{PA} \tag{4}$$

Where $w_{oil}$, $w_{AS}$, $w_{PA}$, $w_{con}$ and $w_{gas}$ are respectively oil yield (including water), AS yield, PA yield, conversion and gas yield, and $M_{AS}$, $M_{PA}$, $M_{THFI}$, $M_{cat}$, $M_{ash}$ and $M_{coal}$ are the respective masses of AS, PA, THFI, catalyst, ash (in raw coal), and coal (on the dry and ash-free basis).

Table 1 Proximate and ultimate analyses of SL

| Sample | Proximate analysis *w*/% | | | | Ultimate analysis *w*/% | | | | H/C (atomic ratio) |
|---|---|---|---|---|---|---|---|---|---|
| | $M_{ad}$ | $A_d$ | $V_{daf}$ | $FC_{daf}$ | $C_{daf}$ | $H_{daf}$ | $N_{daf}$ | $S_{t,d}$ | |
| SL | 20.77 | 17.14 | 46.96 | 53.04 | 70.18 | 5.31 | 1.09 | 1.82 | 0.91 |

$S_{t,d}$: total sulfur on dry basis

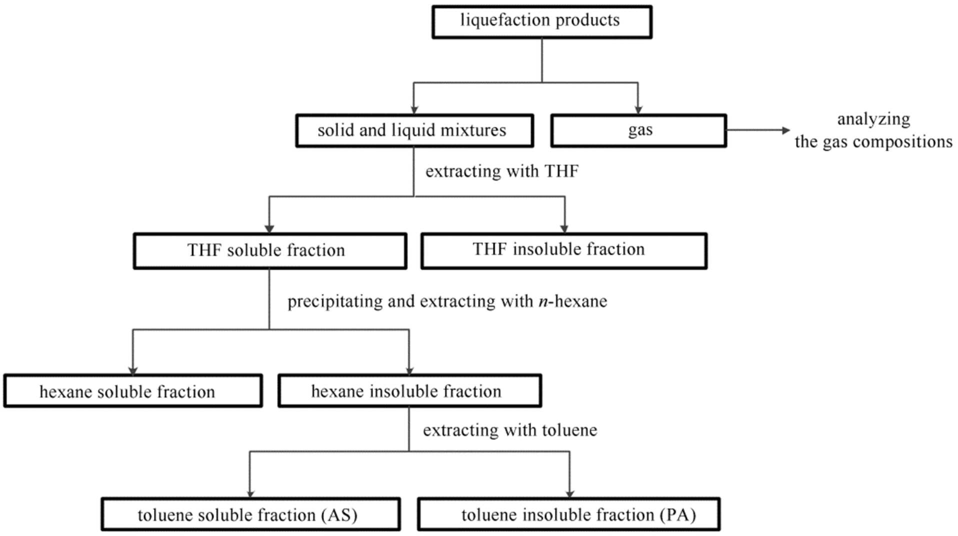

Fig. 1 Schematic diagram of product separation