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STUDY OF HYDRO-LIQUEFACTION TECHNOLOGY OF WUCAIWAN COAL FROM XINJIANG, CHINA

Негізінен Wucaiwan көмірінің гидросұйылту технологиясы қарастырылды. Үш фактор мен екі кезеңнен тұратын тікбұрышты қондырмадағы гидросұйылту нәтижесінде бастапқы сутек қысымы, реакция уақыты және еріткіш пен көмір (Е/К) қатынасы зерттелді. Нәтижелер гидросұйылту үшін ең тиімді жағдайларда алынды. Олар бойынша 400 ℃ температура, қысым 6,0 МПа, реакция ұзақтығы 45 мин және Е/К = 2 кезінде максималды конверсия 76,14 % құрайды. Қытайдың Wucaiwan көмірі гидросұйылту үшін жарамды болып табылды.

Изучена технология гидросжижения Wucaiwan-углей. Вследствие гидросжижения в прямоугольной конструкции с тремя факторами и двумя уровнями были проведены исследования по влиянию начального водородного давления, времени реакции и отношения растворитель–уголь (Р/У). Были получены результаты оптимальных условий для гидросжижения угля. Результаты показывают, что при температуре 400 ℃, давлении 6,0 МПа, длительности реакции 45 мин, и Р/У = 2 максимальная конверсия составляет 76,14 %. Wucaiwan-уголь из Китая подходит для гидросжижения.

Introduction

Reserves of coal in Xinjiang, China, are abundant. It has been said that forecast reserves is about 2,19 billion ton, [1] among about 90 % coal belongs to low metamorphose one being easy to liquefy. This article is to investigate the optimum conditions to hydro-liquefaction for Wucaiwan coal.

1 Experimental

1.1 Coal and solvent. The coal sample came from Wucaiwan in Zhungeer basin, Xinjiang, China. The sample was grinded to the size of less than 200 mesh, dried at 105 °C under vacuum for 4 hours and stored in a brown glass bottle. The analysis results of proximate and ultimate of the sample are shown in Table 1. The data were provided by coal science institute of Xinjiang. Extraction solvents have *n*-hexane, toluene and tetrahydrofuran (THF). Accommodate hydrogen solvent is 1,2,3,4-tetrahydronaphthalene (THNP). All solvents were chemical purely.

Table 1

Analysis of proximate and ultimate of the sample

Proximate analysis /%			Ultimate analysis daf/%					H/C
M_{ad}	A_d	V_{daf}	C	H	O*	N	S	
5,00	2,64	37,62	71,25	4,23	22,89	1,07	0,56	0,71

*By difference.

1.2 Hydro-liquefaction conditions. There are three factors such as initial H_2 pressure, reaction time and ratio of S/C. Orthogonal table of L_8 (2^7) with three factors and two levels was chosen. Detailed conditions are shown in table 2.

Table 2

Orthogonal experimental conditions of the sample

A(P) / MPa	B(T) /min	C(S/C)
5,0; 6,0	45, 60	1:1, 2:1

1.3 Process of hydro-liquefaction and separating products

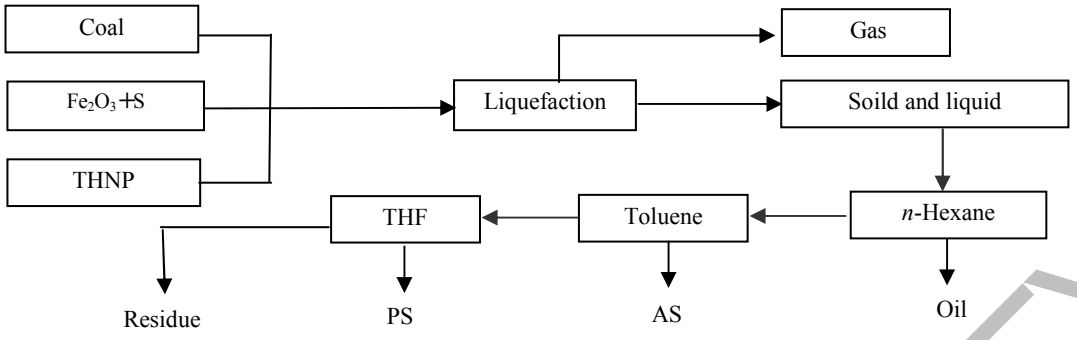

Fig. 1. The diagram of experimental process

Experimental process is shown in figure 1. Experiment was carried out in a reactor of CJF-1.0L batch autoclave with magnetic stirring. Catalyst consists of 1,2 g of Fe_2O_3 and 0,3 g of sublimation sulfur. In each running, 30 g of the sample, THNP and the catalyst were mixed, and then put into the reactor. Reaction temperature was 400 °C. Hydro-liquefaction products were separated by Soxlet extractions with *n*-hexane, toluene and THF in turn. The fractions of *n*-hexane soluble, *n*-hexane insoluble but toluene soluble, toluene insoluble but THF soluble and THF insoluble were defined as oil(this oil contains THNP put as a solvent before), asphaltene (AS), preasphaltene(PS) and residue, respectively. Calculating yields were similar to article [2]

2 Results and discussion

2.1 Experimental results. Table 3 shows the yields of hydro-liquefaction reactions. Article [3] has been reported that if oil yield reaches up to 30,3 %, the sample can be suitable to hydro-liquefaction. As shown in table 3 not only all of yields are more than this value, but also the max reaches up to 56 %, so this coal adapts to as hydro-liquefaction material.

Table 3

Hydro-liquefaction yields of coal sample/%

№	Oil	AS	PS	Conversion
1	31,69	21,55	2,67	61,89
2	55,34	4,44	3,96	71,72
3	37,14	18,30	6,14	62,75
4	55,30	9,46	1,34	71,50
5	44,93	16,60	4,91	69,21
6	54,39	8,16	8,08	75,21
7	53,02	11,15	7,87	71,89
8	56,16	11,73	2,56	76,14

2.2 Orthogonal and variance analysis of oil yields. Figure 2 shows that oil yields increase with three factors rising. The influence of S/C on difference between two oil yields was the maxima, whereas reaction time was the minimum, namely, the conditions of P = 6,0 MPa, θ = 60 min and S/C = 2:1 is optimized.

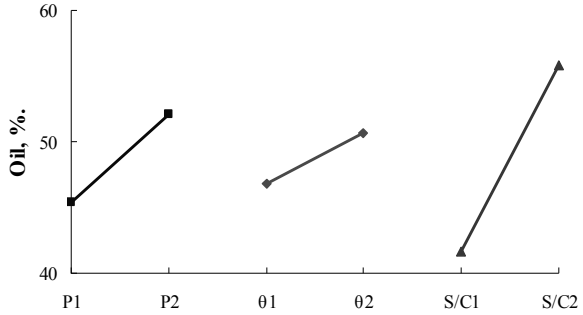

Fig. 2. The effect of three factors on oil yields

Table 4 shows results of variance analysis. It is known $F_{0,01}(1,4) = 21,2$, $F_{0,05}(1,4) = 7,71$ and $F_{0,1}(1,4) = 4,54$. Consequently, $F_{0,05} < F_C < F_{0,01}$, $F_A < F_{0,1}$ and $F_B < F_{0,1}$. Namely, the effect of S/C is highly significance, initial H_2 pressure and reaction time may be neglected. In fact, initial H_2 pressure and reaction time have effect on hydro-liquefaction. Experiment results may be owing to initial H_2 pressure in the range of insensitive low-pressure. As a result if oil yield is as an investigated target, feasible hydro-liquefaction condition is temperature = 400 °C, initial H_2 pressure = 6,0 MPa, reaction time = 60 min, and S/C = 2.

Table 4

Variance analysis results of oil yields

Factor	Sum of warp square	*f*	Warp sum of average	F_{ratio}	Significance
A	91,33	1	91,33	2,58	
B	29,15	1	29,15	0,82	
C	397,76	1	397,76	11,22	○
Error(e)	141,76	4	35,44		

Note. $F_{0,01}(1,4) = 21,2$, $F_{0,05}(1,4) = 7,71$, $F_{0,1}(1,4) = 4,54$. ○ — Highly significance.

2.3 Orthogonal and variance analysis of conversions. As shown in figure 3, the effect of three factors on conversion is $C_2 > A_2 > B_2$, that is, same with 2,2.

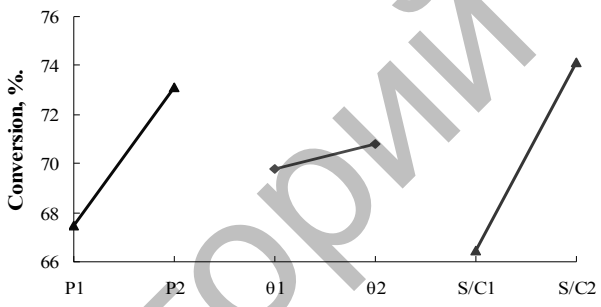

Fig. 3 The effect of three factors on conversion

Table 5 shows results of variance analysis. The effect of S/C, initial H_2 pressure, and reaction time on yield of conversion is highly significance, middle and much smaller, respectively because there are $F_C > F_{0,01}$, $F_{0,05} < F_A < F_{0,01}$ and $F_B < F_{0,1}$. As a result if conversion is as an investigated target, feasible hydro-liquefaction conditions are reaction temperature = 400 °C, initial H_2 pressure = 6,0 MPa, reaction time = 60min and S/C = 2, as the same with 2,2.

Table 5

Variance analysis results of conversion

Factor	Sum of warp square	*f*	Warp sum of average	F_{ratio}	Significance
A	63,79	1	63,79	16,47	○
B	2,26	1	2,26	0,58	
C	118,81	1	118,81	30,67	※
Error(e)	62,10	4	15,50		

Note. $F_{0,01}(1,4) = 21,2$, $F_{0,05}(1,4) = 7,71$, $F_{0,1}(1,4) = 4,54$; ○ — highly significance, ※— middle.

3 Conclusions

1. Wucaiwan coal is fit for hydro-liquefaction.
2. Results of oil yields and conversion are the same by orthogonal and variance analysis.

3. Optimized hydro-liquefaction conditions for Wucaiwan coal are reaction temperature = 400 °C, initial H_2 pressure = 6,0 MPa, reaction time = 45 min and S/C = 2. At these conditions, the maximum oil yield reaches up to 56,16 %, conversion 76,14 %.

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