LIQUEFACTION CHARACTERISTICS OF TURKISH LIGNITES

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ABSTRACT.— Samples of Beypazarı, Elbistan and Kangal were hydrogenated in a bomb reactor, in the presence of tetralin and without catalyst, under 395°C temperature and 7MPa cold hydrogen pressure conditions for 17 minutes. Conversion, liquid yield and preasphaltenes-asphaltenes-oil distribution were determined by a standardized procedure. The Beypazarı sample gave higher conversion (91 %) and liquid yield (64 %) in comparison with the others, but the lightest liquid was derived from Elbistan sample. The consistency of the experiment was verified by total mass and sulphur balances, which gave closures around 100 %.

INTRODUCTION

The estimated maximum life of 50 years of world oil reserves (Bayraktar and Özkaplan, 1982) and the reality that oil can be used as a political weapon have rendered coal liquefaction, i.e. the use of coal for the production of petroleum-like liquids, an important issue. Today the only plant which liquefies coal commercially is SASOL in South Africa, where coal reserves are rich and manpower is cheap. Various liquefaction processes are presently under development in the USA, Great Britain, Federal Germany, USSR, and Japan. The commercialization of many of these processes is now dependent almost only on the price of oil.

This report describes the first step of a project undertaken to investigate the liquefaction behaviour of Turkish lignites with reserves greater than 100×10^6 tonnes. At this stage, the amenability to hydroliquefaction under standardized conditions is examined and compared. With regard to reaction approach, the treatment resembles EDS,SRC and CSF processes (Bayraktar and Özkaplan, 1982). Initially, samples from three sites, viz. Ankara-Beypazarı, Maraş-Elbistan and Sivas-Kangal, which have strategic importance because of their geographical locations, were investigated.

EXPERIMENTAL SECTION

1. Preparation of the samples

The samples were freshly taken from the coal face and various representative size fractions were prepared keeping exposure to a minimum (Fig. 1). Optimization between time, labour and particle size homogeneity was done at every stage of the size reduction programme. The —0.2 mm (—200 mm) samples used in the experiments were stored in plastic containers and purged with nitrogen twice a month against oxidation.

2. Characterization of the samples

Unless otherwise stated, it should be understood that all determinations have been made in duplicate and the average reported.

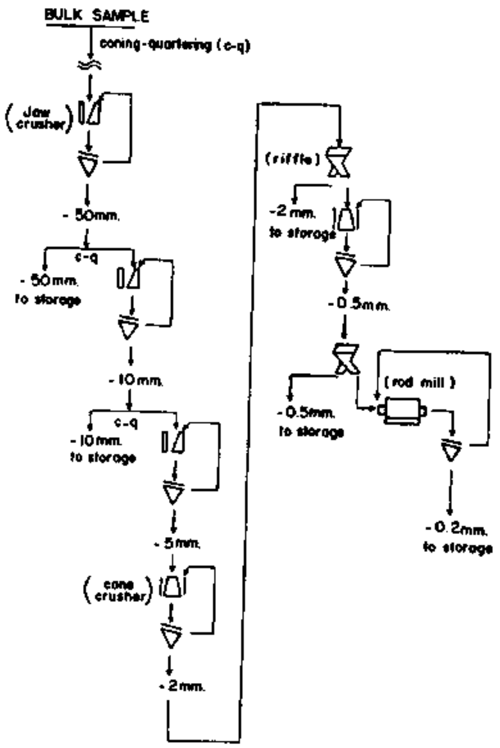

Fig. 1 - Size reduction flowsheet.

a. Particle size analyses were carried out as per ASTM D197. The results are in Table 1. Repeatability of the fraction percentages were better than ±0.5.

b. Chemical, calorific and petrographic data are given in Tables 2,3,4,5.

Moisture was calculated from the weight loss that 1 g of sample underwent at 105-110°C under 0.10 kPa in two hours. Repeatability was better than ± 1 % of the reported value.

Standard ash content was determined as per TS 1042. In order to minimize mineral decompusition and obtain an ash value representing mineral matter more, closely, ash was additionally determined at 520°C. The tolerance of the 520°C-ash was ± 1 %.

Table 1 - Particle size analyses

Lignite	*Beypazarı*	*Elbistan*	*Kangal*
Moisture (%)	14.0	16.1	12.4
+ 212 μm	4.2	3.6	8.4
— 212 + 150 μm	20.3	20.6	8.6
— 150 + 106 μm	14.2	14.9	18.2
— 106 + 75 μm	18.7	12.5	13.9
— 75 + 63 μm	8.4	5.9	6.8
— 63 μm	34.2	42.5	44.1

Table 2 - Standard characteristics of the samples

% Moisture-free basis	*Beypazarı*	*Elbistan*	*Kangal*
Ash	39.4	32.1	44.4
VM*	31.0	44.0	27.3
Fixed carbon (diff.)	29.6	23.9	28.3
Ash (520°C)	40.4	39.9	45.2
Mineral CO_2	0.66	7.8	0.30
C	42.0	42.0	36.0
H	3.17	2.77	2.93
N	1.17	1.12	0.93
S (total)	4.72	3.16	0.67
O (diff.)**	11.4	11.7	15,2
Sulphate S	0.97	0.21	0.08
Pyritic S	1.53	0.45	0.34
Organic S (diff.)	2.22	2.50	0.25
Gross calorific value (MJ/kg)	16.72	16.15	13.96
H/C (atomic)	0.907	0.791	0.977
"O"/C (atomic)	0.20	0.21	0.32

* Uncorrected for mineral CO_2.

** 100 - C - H - N - S_{org} - ash - mineral CO_2.

Table 3 - Petrographic properties of the samples

Meceral group (volume % , organic basis)	*Beypazarı*	*Elbistan*	*Kangal*
Huminite (vitrinite)	89.0	89.2	72.2
Liptinite (exinite)	10.2	10.0	17.7
Intertinite	0.8	0.8	10.1
% $\overline{R}m$*	0.38	0.30	0.20

* Mean maximum vitrinite reflactance.

Table 4 - Ash analyses of the samples

% in 815°C-ash	*Beypazarı*	*Elbistan*	*Kangal*
SiO_2	48.7	15.6	71.1
Al_2O_3	18.1	7.7	7.5
Fe_2O_3	11.6	4.5	4.8
CaO	4.3	46.6	10.6
MgO	2.7	3.1	2.0
Na_2O	6.3	0.26	0.59
K_2O	1.3	0.38	0.67
TiO_2	0.87	0.87	0.65
SO_3	5.1	19.7	2.5

Table 5 - Rational analyses of the samples

%, Organic basis	*Beypazarı*	*Elbistan*	*Kangal*
Bitumen	4.0	5.7	4.3
Humic acids	14.0	57.2	21.1
Lignin-type constituents	82.0	35.1	74.6
Cellulosics	0.0	2.0	0.0

Volatile matter (VM) was found under the conditions given in TS 711, however on a pelletized sample in order to prevent «sparking» (Montgomery, 1978).

Mineral CO_2, necessary for correction of VM and carbon, was measured by ASTM D 1756 and TS 1044 (11).

Carbon and hydrogen determinations were carried out on a semi-micro scale by a high-temperature method in the Laboratories Dept. of MTA. Carbon was corrected for CO_2 from minerals. Nitrogen and total sulphur were determined as per TS 362 and TS 363 respectively.

In the determination of sulphur forms, BS 1016/11 was followed owing to its simplicity.

Gross calorific value was measured as per ASTM D 2015

Ash analysis was performed on 815°C - ash by wet chemical techniques in the Laboratories Dept. of MTA.

Petrographic and rational analysis was conducted in the Faculty of Chemistry of Hacettepe University (TÜBİTAK project, 1984).

3. Experimental set-up and procedure

Samples of particle size —200 mm were hydrogenated in a reaction bomb, in the presence of tetralin and without catalyst, under 395°C temperature and 6.9 MPa initial hydrogen pressure conditions for 17 minutes. Under these mild conditions the liquefaction performances of the lignites were distinguishable.

a. Apparatus: The liquefaction system is schematically illustrated in Figure 2. The hydrogenations were carried out in two identical PARR 4740 bombs (316 S.S.) of 71 mL capacity. A TECHNE SBL-2 fluidized sand bath with provisions for temperature control was usedas the heat source. The bomb was shaken in the sand bath along its longitudinal axis at 4 cm amplitude and 400 cycles per minute frequency. Upon termination of the experiment, the bomb was immersed into the adjacent cold water bath to accomplish rapid cooling.

Since the construction ofthe bomb prohibited the use of an internal thermocouple, the inside temperature was calibrated against the bomb surface temperature. In calibration experiments the charge was simulated with tetralin/coke mixtures. Eventually the temperatures and durations of heat-up, reaction, and cool-down were estimated with good reproducibility.

b. Procedure: The procedure was developed to obtain as many data as possible in a short time. Typically, a test is completed in ten hours.

All chemicals were used as supplied. Unless otherwise stated, the sensitivity of the weighings was 0.01 g.

15 g (W_T) of tetralin and a quantity of—200 mm air-dried lignite sample, corresponding to 7.5 g (W_C) on a moisture-free (mf) basis, are transferred into the bomb. The bomb is assembled, purged twice with hydrogen, and pressurized to 6.9 MPa with hydrogen. After a pressure test of at least two hours, it is immersed into the sand bath preheated to about 450°C and shaking is initiated. The temperature of the sand bath is continuously monitored, by addition of cold sand or adjustment of the temperature controller, according to the temperature calibrations. On completion of the appointed reaction time, the bomb is quickly quenched to room temperature by immersion into the cold water bath. Typical heat-up reaction, and cool-down times are 10, 17, and 10 minutes respectively.

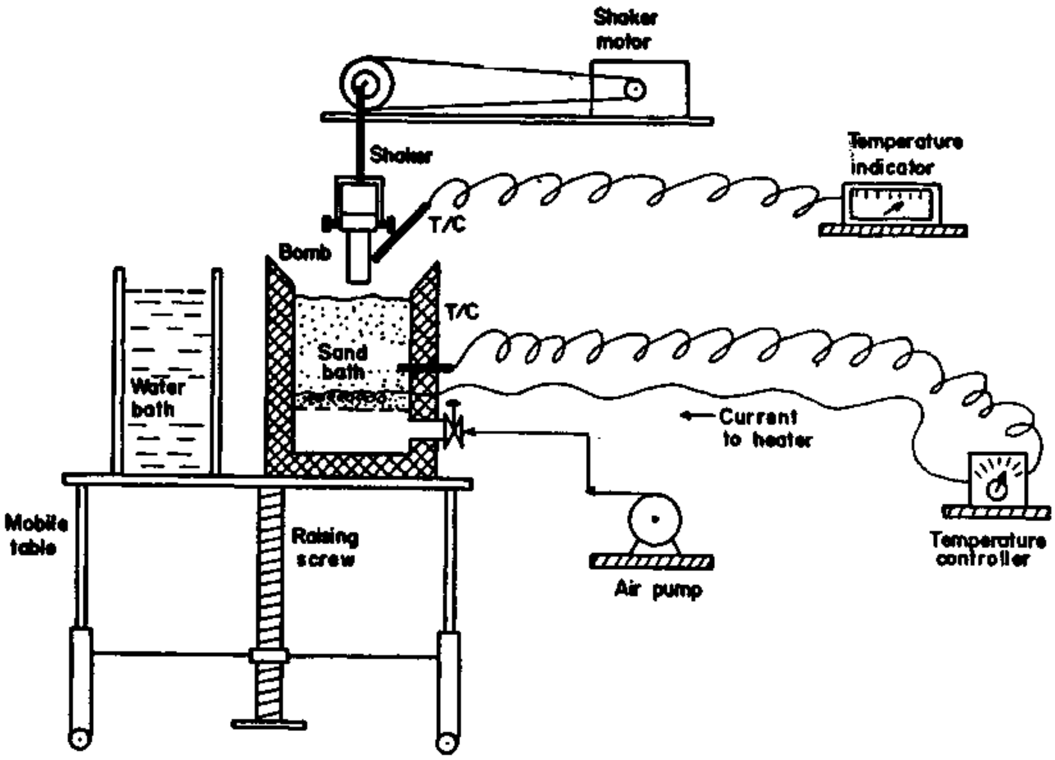

Fig. 2 - Liquefaction system.

Final bomb pressure is measured. If total mass and sulphur balances are to be made, the gas is passed through an absorption train for characterization (see Addendum); otherwise it is discarded.

The bomb contents are washed quantitatively into a beaker with fresh THF (tetrahydrofuran). The mixture is preserved under nitrogen in the refrigerator overnight.

The succeeding operations are shown in the form of a flowchart in Figure 3. The slurry is filtered through Whatman no: 42 or 44 paper (pore size: 2.5 mm) in a Buchner funnel. The residue is dried at 105-110°C and 0.10 kPa for 2 h and weighed (W_R) for calculation of total conversion (C_t). Soxhlet extraction has been found superfluous after a number of comparative experiments.

The filtrate is concentrated following a temperature programme (maximum: 80°C) in a HEINO S rotary evaporator under nitrogen sweep. It has been experimentally verified that water in the filtrate is removed practically completely as water-THF azeotrope under these conditions. Evaporation is continued until a concentrate of ca. 50 % THF is obtained, and this concentrate is weighed (W_{CON}).

The THF content of the concentrate is estimated chromatographically. The operating characteristics are as follows:

Chromatograph :PACKARD-BECKER Model 419
Column :2 m X 1/4", 15 % Apiezon-L on 80-100 Chrom W
Detector :TC, 240°C, 250 mA
Carrier gas :He, 20 mL/min (at 20°C)
Injection temperature :240°C
Sample quantity :5.00±0.05 mL
Attenuation :64
Temperature programme :100°C - 240°C, stepwise

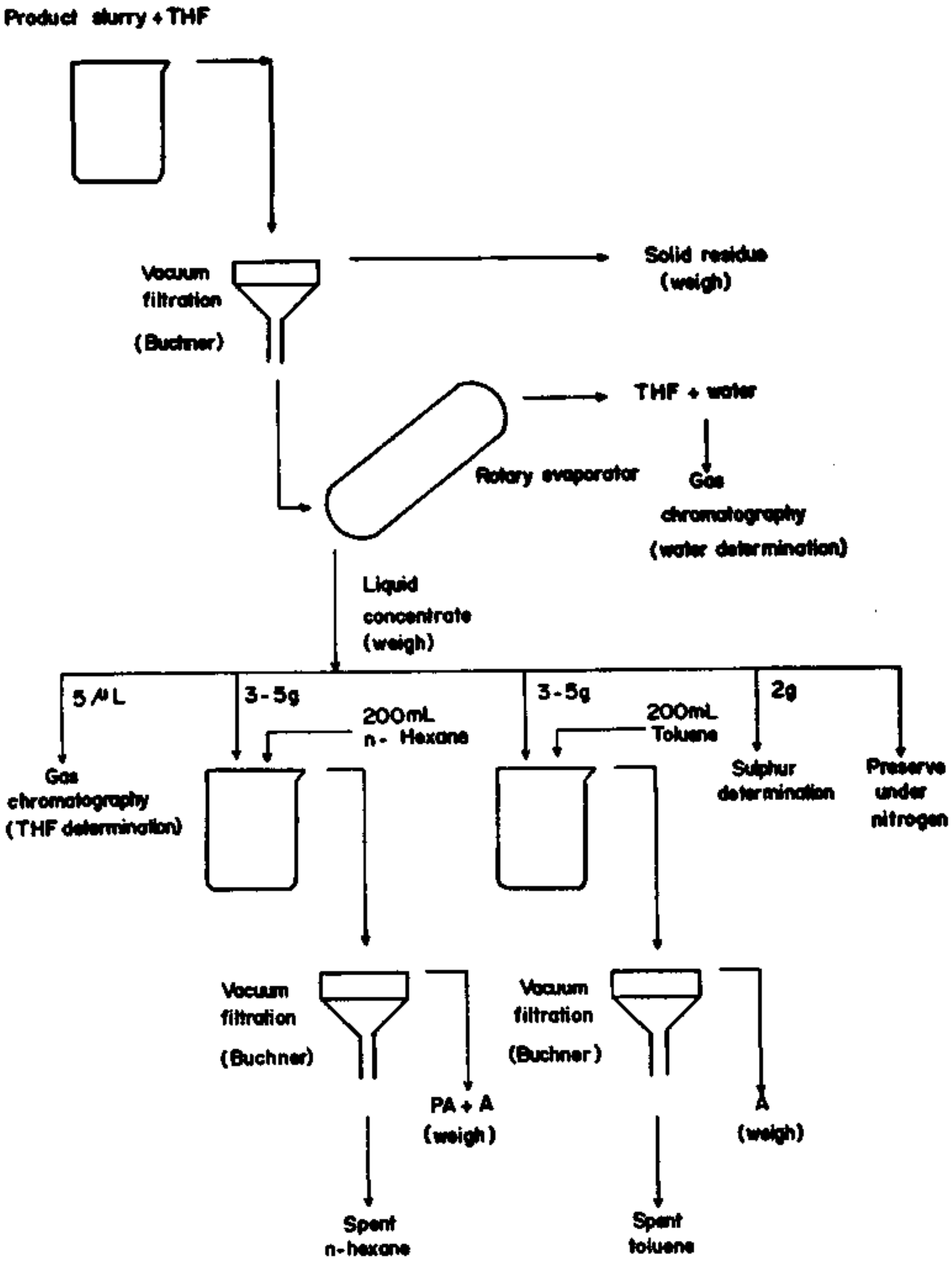

Fig. 3 - Product work-up.

As a result of such operation, THF and tetralin yield tall and sharp peaks, and probable near-boiling materials such as hexane, benzene, toluene and decalin, naphthalene are nicely separated.

The THF content of the concentrate (X_{THF}) is determined by matching with similar standard mixtures, and the quantity of coal liquid (W_L) and liquid yield (L) are calculated therefrom.

Two aliquots of 3-5 g (T and H) are pipetted from the concentrate, and 200 mL of toluene and 200 mL of n-hexane are added respectively. After 30 minutes of standing, the two mixtures are filtered as in the case of bomb washings and the precipitates washed copiously with the respective solvents. The «preasphaltene» precipitate (t) from toluene precipitation and the preasphaltene+asphaltene' precipitate (h) from n-hexane precipitation are dried at 105-110°C and 0.10 kPa for one hour and weighed to a sensitivity of 0.1 mg. The preasphaltene-asphaltene-oil distribution is then computed.

Sulphur content of the coal liquid is determined on a 1 g sample of concentrate in a Leco Analyzer in the Laboratories Dept. of MTA.

c. Calculation of results

Conversion: Cold THF solubles+gases

$$C_t = \frac{W_C - W_R}{W_C(1-a)} x100$$

W_C : Quantity of lignite sample charged (mf)

W_R : Quantity of solid residue (mf)

a : Fractional ash content of lignite (mf)

C_t : Conversion (% , maf)

Involved error is ± 0.5 % of the value.

Liquid yield: Product which is soluble in cold THF and boils above 80°C

$$W_L = W_{CON} (1-X_{THF})-W_T$$

$$L = \frac{WL}{W_C(1-a)} X \ 100$$

W_{CON} : Quantity of concentrate

X_{THF} :Fractional THF content of concentrate

W_T :Tetralin charged

W_L :Quantity of coal liquid obtained

W_C :Quantity of lignite sample charged (mf)

a :Fractional ash content of lignite (mf)

L :Liquid yield (%, maf)

Here it is assumed that tetralin gives no gaseous product, i.e. that itself and all its reaction products remain in-liquid. It has indeed been reported that deromposition of tetralin to gases is insignificant under these conditions (Charlesworth, 1980). Besides it is a precondition for a viable process that generates as much solvent as it consumes.

Error in liquid yield arises mainly from the quantity of chromatography sample, which bears the largest theoretical error. The error is ±7 % of the liquid yield for an injection of 5 ± 0.05mL.

Distribution of preasphaltene-asphaltene-oil: Preasphaltene, liquid component soluble in THF and insoluble in toluene; asphaltene, liquid component soluble in THF and toluene and insoluble in n-hexane; oil, liquid component soluble in all three

$$PA = \left(\frac{t}{T}\right)\left(\frac{W_{CON}}{W_L}\right) \times 100$$

$$A = \left(\frac{h}{H}\right)\left(\frac{W_{CON}}{W_L}\right) \times 100 - PA$$

$$\O = 100 - PA - A$$

t :Quantity of precipitate from toluene precipitation

T :Quantity of sample for toluene precipitation

W_{CON} :Quantity of concentrate

W_L :Quantity of coal liquid obtained

h :Quantity of precipitate from n-hexane precipitation.

H :Quantity of sample for n - hexane precipitation

PA, A,f : Preasphaltene, asphaltene, oil (% of liquid)

The theoretical errors are ± 7,16 and 27 % of the respective values of PA, A and f. These values are affected by the high theoretical error involved in liquid yield.

RESULTS AND DISCUSSION

1. Characteristics of the samples

Particle size analyses (ASTMD 197) of the samples (nominal —200 mm) used in the experiments are given in Table 1.

Although size reduction parameters were optimized, comminution to fines inevitably occurred to some extent. However, it is established that particle size at these levels does not influence the liquefaction reaction (Curran et al., 1967; Pastor et al., 1970; Ruether, 1977; Neavel, 1976; Whitehurst et al., 1980). Tables 2,3,4, and 5 contain the results of the standard characterizations. All the three samples are of high ash, but the ash compositions differ.

The high sulphur contents of Beypazarı and Elbistan samples are prominent. Half of Beypazarı sulphur and three-quarters of Elbistan sulphur are organic in nature, therefore high-sulphur liquids would be expected from these coals.

The atamic H/C ratios are typical for lignites. The ratio is the highest in Kangal lignite, showing agreement with the high exinite content of this sample. Since all the three samples are rich in reactive macerals (vitrinite + exinite), amenability of liquefaction can be expected.

The ash and sulphur contents of the samples are similar to the respective reserve averages (MTA, 1983), but for the extraordinarily low sulphur content of the Kangal sample.

2. Liquefaction findings

The experimental conditions and the main results presented in Table 6. Table 7 contains the results of statistical evaluation.

Table 6 - Liquefaction conditions and results

Sample	Experiment code	Charge lignite (g) /moisture (%) tetralin (g)	Conditions T_r (°C) P_i, P_s (MPa)	Conversion (% maf, 520)	Liquid yield (% maf 520)	PA, A, Ø (% of liquid)
Beypazarı	TARD 6	8.51/11.9 15.00	397 6.91, 5.53	90.3	57.1	47, 33, 20
	TARD 7	8.46/11.3 15.06	391 6.75, 5.59	89.4	64.2	43, 32, 25
	TARD 11	8.42/10.9 15.00	396 6.93, 5.78	92.5	69.6	42, 30, 25
	TARD 16	8.42/10.5 15.01	393 7.01, 5.83	90.0	63.4	43, 37, 20
Elbistan	TARD 9	8.92/15.6 15.05	392 6.78, 6.59	86.8	57.0	19, 29, 52
	TARD 10	8.92/15.6 14.98	396 6.91, 6.73	78.9	51.7	21, 24, 55
	TARD 15	8.59/12.7 15.00	391 7.15, 6.62	78.9	52.3	19, 31, 50
Kangal	TARD 1	8.46/11.8 15.01	398 7.08, 7.02	82.0	43.1	41, 46, 13
	TARD 3	8.46/13.9 15.01	398 7.18, ?	78.4	40.4	49, 39, 12
	TARD 12	8.71/11.9 15.01	399 6.82, ?	83.8	52.6	36, 27, 37
	TARD 13	8.51/11.9 15.00	393 6.85, 6.67	86.7	58.4	32, 28, 40
	TARD 14	8.51/11.6 15.00	392 6.96, 6.84	85.4	46.4	40, 35, 25

T_r - Reaction temperature; P_i - Initial hydrogen pressure (normalized to 20°C);
P_s - Post-reaction pressure (normalized to 20°C); PA- Preasphaltene;
A- Asphaltene; Ø- Oil — reaction time: 17 min.

In the experiments tetralin-to-coal ratio was kept constant at 2:1 on a moisture-free basis. Since the effect of moisture level on charge volume was found negligible, hydrogen to charge ratio should remain nearly constant. Although the difference in moisture levels would give rise to slight differences in hydrogen partial pressures during reaction, this would probably not affect conversion and liquefaction characteristics at this pressure level and in the presence of sufficient hydrogen-donor (Morita et al., 1979; Kamiya et al., 1978).

In expressing the experimental results on a moisture- and ash-free basis, the 520°C-ash values are used. At this ashing temperature carbonates would remain intact but the majority of ashing reactions would reach completion. As can be seen in Table 8, use of the standard high-temperature ash gives rise to artificially low results, particularly for samples such as Elbistan which contain hgh proportions of unstable minerals.

Table 7 - Evaluation of liquefaction results (means and standard deviations)

	Beypazarı	*Elbistan*	*Kangal*
Tetralin : coal (mf)	2.00 ± 0.01	2.00 ± 0.01	2.00 ± 0.04
Tetralin : coal (maf, 520)	3.36 ± 0.01	3.32 ± 0.01	3.66 ± 0.07
T_r (°C)	394 ± 3	393 ± 3	395 ± 4
P_i (MPa)	6.90 ± 0.11	6.95 ± 0.19	6.96 ± 0.12*
P_s (MPa)	5.68 ± 0.15	6.65 ± 0.07	6.84 ± 0.18*
ΔP (MPa) **	1.22 ± 0.11	0.30 ± 0.20	0.12 ± 0.06*
Conversion (%, maf, 520)	90.6 ± 1.4	81.5 ± 4.6	83.3 ± 3.2
Liquid yield (%, maf, 520)	63.6 ± 5.1	53.7 ± 2.9	48.2 ± 7.3
Gas (diff. %, maf, 520)	27.0 ± 4.4	27.8 ± 1.7	35.1 ± 5.0
PA (% of liquid)	44 ± 2	19 ± 1	40 ± 6
A (% of liquid)	33 ± 3	28 ± 4	35 ± 8
Ø (diff., % of liquid)	23 ± 4	52 ± 3	25 ± 13
Tetralin recovery (%)	95 ± 1	94 ± 2	93 ± 2

* Only three experiments have been considered; the post-reaction pressures of the other two were not determined.

** $\Delta P = P_i - P_s$

Table 8 - Effect of ash determination temperature on liquefaction data

	Ash determination emperature (°C)	*Beypazarı*	*Elbistan*	*Kangal*
Conversion (%, maf)	520	90.6	81.5	83.3
	815	89.0	72.2	82.1
Liquid yield (%, maf)	520	63.6	53.7	48.2
	815	62.5	47.5	47.5

Table 9 - Total mass and sulphur balances

	Beypazarı (TARD 16)		*Elbistan (TARD 15)*	
	Mass (g)	*Sulphur (g)*	*Mass (g)*	*Sulphur (g)*
Input				
Coal (mf)	7.54	0.356	7.50	0.256
Water (as moisture)	0.88	—	1.09	—
Tetralin	15.01	—	15.00	—
Hydrogen	0.30	—	0.30	—
Total	23.73	0.356	23.89	0.256
Output				
Solid residue (mf)	3.48	0.193	3.94	0.071
Water	1.47	—	1.29	—
Coal liquid, Tetralin Tetralin derivatives (liq.)	17.86	0.120*	17.36	0.145
CO_2	0.36	—	0.61	—
H_2S	0.045	0.043	0.054	0.051
Other gases	0.39	—	0.56	—
Total	23.61	0.356*	23.81	0.267
Closure (%)	99.5	100*	99.7	104

* Calculated assuming 100 % closure.

The weight loss involved in the conversion of pyrite to pyrrhotite would have an elevating affect on the measured total conversion. Assuming full transformation, the «increase» in total conversion percentages of Beypazarı, Elbistan and Kangal would be 1.3, 0.4, and 0.3 respectively. These maximum expectable values do not exceed the experimental tolerances (Table 7).

In this stage of the research programme, only general comparisons are made. More detailed correlation of lignite properties with liquefaction behaviour is reserved until screening tests on samples from other important sites are completed.

The greatest consistency (i.e. the lowest standard deviations) is associated with Beypazarı with respect to conversion and with Elbistan with respect to liquid yield. All the samples, Beypazarı in particular, give high conversions under these relatively mild reaction conditions. The highest liquid yield is also obtained from Beypazarı sample. The high pyrites content of this lignite may be partly responsible for this superior performance. Liquid selectivity, which can be represented by the percentage ratio of liquid yield to conversion and is, desired to be high, is 70 for Beypazarı, 66 for Elbistan, and 58 for Kangal. Both liquid selectivity and liquid yield follow the same order with mean reflectance values and the reverse order with «oxygen» content. Thus, liquid yield increases with degree of coalification for these three lignites. Gas (+ water), expressed as the difference between conversion and liquid yield, shows the reverse trend. The greater the oxygen content, the more gas the coal would give during liquefaction (Whitehurst et al., 1980; Cudmore, 1977/78).

Beypazarı and Kangal liquids are similar on the basis of preasphaltene -asphaltene-oil distribution. Elbistan liquid, on the other hand, is completely different; it is remarkably «light» with its oil content of 52 %. Preasphaltenes and asphaltenes are composed of undistillable products, contain large proportions of heteroatoms (O,N,S) pose difficulties in catalytic processing,and negatively influence the storage properties of liquids. In short, they are undesirable. It appears that a quarter of Beypazarı and Kangal liquids and one half of Elbistan liquid are potentially distillable. The tendency of Elbistan lignite to yield light liquids was also observed by Bayraktar in an earlier study (Bayraktar, 1981). This would perhaps imply that the molecular structure of Elbistan lignite is made up of small building blocks. Unless the high oil yield is realized at the expense of high hydrogen consumption, Elbistan lignite can be specified as quite amenable to liquefaction. However, the sulphur content of Elbistan liquid is high.

Tetralin recoveries were estimated with reasonable accuracy by gas chromatography. The results indicate that consumption of hydrogen-donor solvent remains low.

Total mass and sulphur balances are given in Table 9. The additional operations required are explained in Addendum.

Attainment of full closure in total mass balances despite the small scale of experimentation signifies the reliability of the present techniques and results. The elements missing from the balance sheet are water left in solid residue and ammonia. It has been found that only very little water can be retained in the residue since water is removed almost quantitavively by THF. Even if all coal nitrogen were converted to ammonia, 0.11 gof ammonia would result; in fact only one-third undergoes such transformation (Bayraktar,1981). Hence significant erros would not ensue when ammonia is neglected; it is very difficult to determine in such minute quantities.

The sulphur balance around one Elbistan experiment also gave good closure. Here the greatest error would be associated with the sulphur of the liquid since it compounds errors from i) determination of coal liquid, ii) sampling a volatitle liquid, and iii) multiplication of the specified precision of the Leco determination (± 2 %) with a large factor. Nevertheless practically full closure of the sulphur balance shows that sulphur content of coal liquid can be calculated reliably from a forced sulphur balance. So was the sulphur in the Beypazarı concentrate calculated in Table 9.

The sulphur contents of the Beypazarı and Elbistan liquids can be calculated as 4.2 and 6:1 % respectively. Such high-sulphur liquids are unlikely to be used without radical desulphurization. The distribution of sulphur into the products are shown in Table 10. In the case of Beypazarı, more than half of the sulphur remains in the residue as expected from lignite of high sulphate and pyritic sulphur. Whereas in Elbistan, whose sulphur is over three-quarters organic more than half of the sulphur reports to the liquid. A similar distribution was found for Elbistan lignite in a previous study (Bayraktar, 1981). A direct linear relationship was observed between organic sulphur in feed coal and sulphur in product liquid (Whitehurst et al., 1980). Pilot-scale experience indicates that sulphur in liquid varies exponentially with hydrogen consumption (Gorin, 1981). In this work, 72 and 68 % respectively of organic sulphur in Beypazarı and Elbistan appear in coal liquid.

Table 10 - Distribution of sulphur into products

	% of lignite S	
Product	*Beypazarı (forced balance)*	*Elbistan*
Solid residue	54	27
Coal liquid	34	54
Gas	12	19

Yields of carbon dioxide are 8 and 14 % for. Beypazarı and Elbistan respectively. Thus 30 and 50 % of lignite "oxygen" are removed in the form of carbon dioxide.

CONCLUSION

Lignite samples from three inland deposits (Ankara-Beypazarı, Maraş-Elbistan, Sivas-Kangal) were hydrogenated under mild conditions so as to differentiate between liquefaction properties. Favourable conversions and liquid yields were obtained. The «screening» procedure was devised after a number of preliminary tests and shown to be consistent by total mass and sulphur balances. The highest liquid selectivity was observed with Beypazarı and the «lightest» liquid with Elbistan. However, the liquids derived were of high sulphur.

In the next stage, samples from other substantial deposits will be subjected to the standard screening test, qualitative and quantitative relationships between lignite properties and liquefaction performance will be sought, and three or four samples will be selected for more detailed autoclave experiments.

ADDENDUM

Experiments of total mass and sulphur balances

For total mass and sulphur balances additional determinations are made, viz. product gas is collected and characterized, and water recovered as mixture with THF in evaporation is quantified.

The gas is bubbled through two absorbers containing excess 2N sodium hydroxide and the sweet gas is collected over water. Gas volume is measured as displaced water. Gas density is determined by the method of Regnault (Yarzab et al., 1980).

The contents of the absorbers are combined. Absorbed carbon dioxide is determined by acidification followed by absorption in soda-asbestos (Given and Yarzab, 1978). Trapped hydrogen sulphide is determined by wet oxidation and subsequent precipitation as barium sulphate.

Water carried over with THF in evaporation is estimated chromatographically in a 2.5 m x 1/8" Porapak Q column with TCD detection (Guin et al., 1977).

Quantity of input hydrogen is calculated by the ideal gas law.

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