RETORTING OF FOUR KINDS OF SMALL SIZE OIL SHALE WITH AN INCLINING TYPE RETORT

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ABSTRACT

In preparing shale feed stock for a vertical retort, most popular type of above-ground retort, powder shale of below about 5 mm in diameter should generally be removed to obtain smooth shale flow. However, the removed powder shale has to be retorted also to make full utilization of oil shale resources.

Aiming at using a multi-tube heat exchanger as a retort, we carried out preliminary study on a single tube retort set at 45 degree to test its efficiency and practicability using oil shales from Colorado, Mae sot (Thailand), Condor (Australia) and Maoming (China).

The retort could treat oil shales consisting of smaller particle size than 5 mm in diameter giving 90 % of oil yield at the most, though in some cases plugging-up occurred depending on kind of shale and operating conditions.

Burning of carbon deposit on retorted shale with a combustion unit equipped at the bottom of the retorting section was also tested and it could work depending on particle size distribution of feed shale.

1. Introduction

Various type of oil shale retort such as Union, Petro-six, Paraho, etc have been designed and tested. With a vertical shaft type retort, generally most popular type, oil shale having particle size above 5 to 10 mm can be retorted, but fine particles below 5 mm have to be removed from the feed. TOSCO and Lurgi retorts can treat the powder shale though the systems are rather complex. It will be of advantage, if a multi-tube heat exchanger, popular in refinery operations, can be used for retorting oil shale powder.

From this point of view we carried out a series of studies using an inclining tube set at 45 degree as a retort. A clearance formed

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between the inner wall of the tube and the upper surface of oil shale layer made it easy for gas and oil shale to flow uniformly.

2. Experimental

Sources and preparation of Colorado and Thai oil shales, equipments and analytical procedures have already been described in previous papers.[1) 2)] Preparation of condor and Maoming shales has been conducted similarly to result in particle size distribution shown in Table 1.

Table 1 Particle size distribution

Kind of shale	Colorado		Condor		Maoming		Thai	
wt% / mm D.	Powder	Granule	P.	G.	P.	G.	P.	G.
-0.053	4.3		0.2		1.4		0.5	
0.053-0.105	6.2		4.3		8.8		3.9	
0.105-0.250	11.7		15.8		21.2		13.5	
0.250-0.500	13.6		18.2		15.5	0.4	20.0	0.1
0.500-1.00	25.6		27.8	0.2	24.2	0.5	32.6	1.0
1.00-2.00	38.0	3.9	33.0	13.1	29.0	1.4	27.4	14.3
2.00-4.76	0.6	80.9	0.7	81.5		94.3	0.1	79.1
4.76-9.65		15.2		5.1		3.3		5.5

Oil shale feeds were dried at 100°C for 24 hours. Powder here means shale having particle size of below 2 mm in diameter.

Schematic diagram of the inclining retort is shown in Fig.1a and 1b, and oil recovery section in Fig. 2. The basic layout is similar to the vertical one previously reported,[1)] except that the pyrolysis tube (inner diameter 90, length 1350 mm) was set at 45 degree. As shale flows in the tube, large particles move toward the upper surface of the shale layer forming a passage permitting the gas containing oil vapor to flow smoothly. A sweeping device (7) was used after the end of a run, only if necessary. Almost all runs started with the spent shale remaining in the pyrolysis tube from previous run. Since Thai shale was apt to form lumps, a mill (17) was needed to crush them, but not needed for the other shales. Therefore, a vertical tube with a cooling jacket, shown in Fig. 1b, was equipped instead of the mill.

For several runs, the spent shale down from retort tube was burned to remove carbon deposit by heating it with a burner and then by

stopping fuel gas. A part of the flue gas was introduced into the retort tube, while the other part was released after collecting shale mist with a cyclone. Oil was recovered from three separators and the mist collector, and then mixed them together forming crude shale oil samples.

3. Result and Discussion

Selected data are shown in Table 2 and Table 3. Temperatures were controlled at 4 sections divided approximately equally the retort tube, keeping 540, 560, 580 and 600°C from the top.

Three runs using the spent shale burner are shown in Table 2. With increase in powder content, quantity of fly ash increased, causing difficulty in igniting spent shale containing powder of above 35%. Since the density of Colorado shale is so high that the powder hardly flew resulting in low content of solid in oil. For granular shale alone fed (CO-4), the combustibility was so good that almost white spent shale was obtained.

Fig.1b

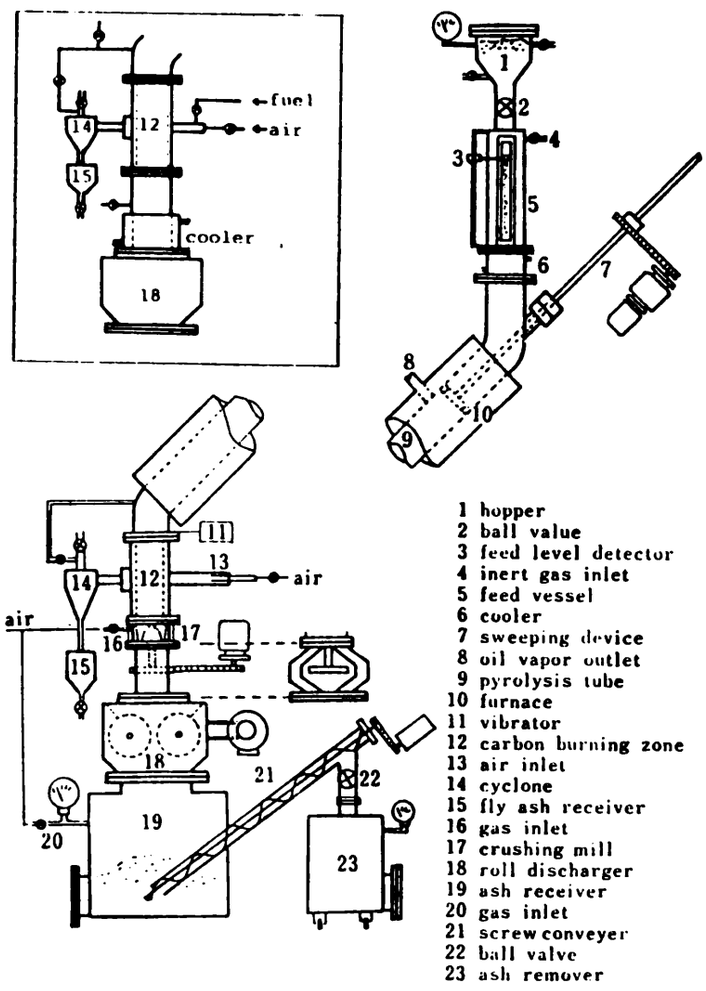


Fig.1a,1b Schematic Diagram of the Inclining Retort

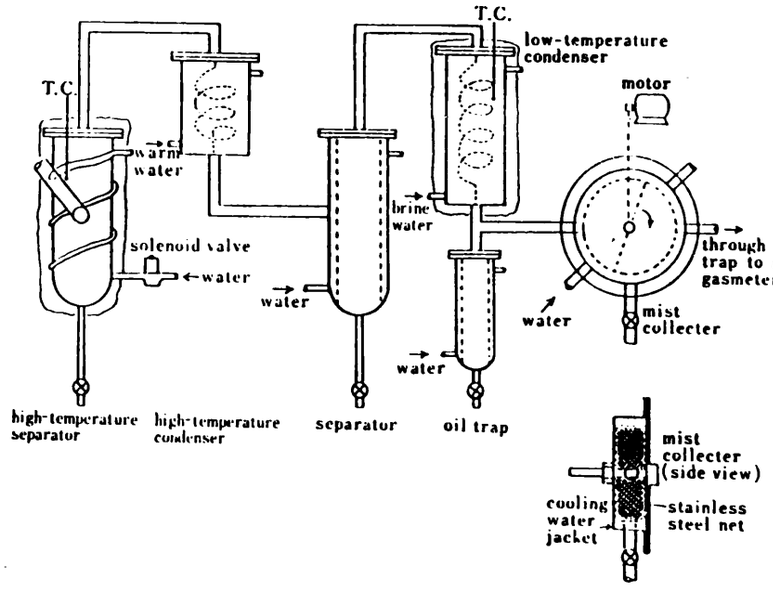


Fig. 2 Oil Recovery Section

Table 2 Summerized Data on Retorting

Exp. No.	CO-4	CO-6	CN-7	CN-10	ME-3	ME-7	TH-1	TH-6
Kind of shale	Colorado		Condor		Maoming		Thai	
Operating mode								
Sp. shale burner	used	used				used		
position of air inlet	burner	burner	burner	bottom of retort		burner	burner	burner
Inlet air rate l/min	30	26	5	3	1.5	26	5.6	5.7
Pressure cm-water	25	25-50	80-120	53-80	30-98	35-60	25	39
Shale F.A. wt%oil	9.9		4.8		6.0		22.2	
Shale rate kg/hr	5.05	5.93	8.97	5.44	5.75	5.21	5.22	5.40
Powder wt%	0	35	50	50	50	30	38	50
Oil yield F.A base %	95	85	54	73	77	83	76	73
Product yield wt%								
Oil	9.4	8.4	2.6	3.5	4.6	5.0	16.8	16.1
Water	2.1	2.0	3.6	6.2	7.5	7.2	4.3	3.9
Gas	4.2	3.5	2.3	2.5	2.4	3.1	7.4	6.6
Spent shale	75.7	73.2	88.0	84.8	81.4	81.6	71.5	73.4
Properties of oil								
Sp. gr. 50°C/4°	0.89	0.88	0.86	0.87	0.87	0.88	0.85	0.85
Viscosity 50°C cst	6.5	5.8	3.7	4.2	3.5	5.3	4.9	5.4
Pour Point °C	25	22.5	25	27.5	22.5	27.5	32.5	35
N content wt%	1.7	1.6	1.3	1.3	1.1	1.1	1.1	1.1
S wt%	0.56	0.57	0.33	0.33	0.40	0.43	0.26	0.28
C wt%	83.7	84.0	85.1	84.5	85.1	85.1	79.3	80.5
H	12.3	13.1	13.1	13.1	11.6	13.0	13.4	13.5
Weight ratio C/H	6.8	6.4	6.5	6.5	7.4	6.5	5.9	6.0
Solid in oil g/kg-oil	0.03	0.02	0.14	0.04	0.21	3.5	0.24	0.20
Distillation 0-200 °C	14.1	15.2	16.2		18.7	12.7	16.5	15.5
wt% 200-300	20.1	19.8	30.5		29.9	27.7	20.7	19.6
300-400	23.2	24.5	26.0		25.6	27.4	27.0	26.5
400-500	26.1	27.2	18.9		17.6	21.2	23.4	39.4
500-	14.3	11.2	6.8		5.9	9.6	11.5	
Gas composition vol%								
H_2	21.9	15.9	15.1	19.7	39.9	9.7	12.4	11.6
CO	7.2	13.9	1.6	1.8	2.1	2.0	2.9	2.5
CO_2	28.2	26.4	23.4	22.5	10.6	14.4	12.3	12.1
CH_4	15.4	10.3	5.6	6.8	10.0	2.3	10.0	8.9
C_2 - C_5	14.8	11.0	6.1	5.8	8.0	1.7	17.3	15.5
N_2	12.4	22.1	47.6	42.9	29.0	69.0	44.1	47.7
O_2	0.1	0.4	0.7	0.6	0.4	1.0	0.4	1.2
Spent shale								
Ignition loss Gr.	3.3	6.0	8.4	5.0	10.8	6.3	15.5	
wt% 600°C Pd.	3.0	6.1	9.2	5.5	12.5	9.5		
Wt. loss in Gr.	0.0	0.0	1.0	0.2	0.7	0.0		
TG anal. wt% Pd.	0.0	0.0	1.9	0.2	1.2	0.3		

On the other hand, Maoming shale became bulky when crushed into fine powder and, hence, was easy to fly resulting in high solid content in oil. The combustibility, especially of powder, was relatively bad judging from the result of spent shale ignition loss. In this run (ME-7), about half of flue gas, calculated from nitrogen content in product and flue gas, was estimated to be introduced into the retort tube. Therefore, this run was considered to be under the most severe operating condition, compared with runs CO-4 and 6 in which about 3 % of flue gas went to the retort tube.

Table 3 Properties of Oil Fractions

Run No.	°C	0-200	200-300	300-400	400-500	500-
CO-4	C wt%	81.3	81.9	83.9	83.7	
Colorado	H	14.2	12.7	12.7	11.9	
	N	0.67	1.2	1.8	1.8	2.4
	S	0.53	0.60	0.58	0.60	0.64
	C/H	5.7	6.5	6.6	7.1	
CN-7	C	82.2	81.9	85.0	84.3	
Condar	H	11.9	12.6	13.0	11.6	
	N	0.94	1.0	1.3	1.4	2.0
	S	0.32	0.29	0.33	0.37	0.47
	C/H	6.9	6.5	6.5	7.1	
ME-3	C	80.7	82.9	84.9	84.3	85.9
Maoming	H	12.7	11.6	12.6	11.7	10.5
	N	0.77	0.85	1.2	1.3	1.9
	S	0.39	0.36	0.41	0.44	0.58
	C/H	6.4	7.1	6.8	7.2	8.2
TH-1	C	80.0	81.6	80.5	81.6	81.5
Thai	H	14.0	14.4	14.1	13.7	12.0
(Maesot)	N	0.61	0.82	1.0	1.2	1.9
	S	0.34	0.25	0.25	0.27	0.30
	C/H	5.7	5.7	5.7	6.0	6.8

Runs (CN-10, ME-3) were carried out introducing air exactly at the bottom of the retort tube to observe the pressure drop through the retort tube. Condor and Maoming shales generated a lot of retort water and became bulky when crushed into fine powder, which made the pressure drop high and apt to fluctuate. Generation of a lot of water in retorting zone heightened gas flow rate excessively, preventing stable operation.

High powder content made the clearance between the shale layer and inner wall of the tube narrow resulting in increase in pressure drop.

Ignition loss, meaning the weight reduction in burning spent shale in air at 600°C for 8 hours, was higher for powder than granule, which suggested that coking was easier to occur for powder during oil generation.

Weight loss in TG analysis, corresponding to remaining organic matter, was also higher for powder, suggesting that granular shale was

more easily heated with the gas flowing in upper shale layer. This was probably the reason why oil yield decreased with increase in powder content, as shown in Fig. 3.

Fig. 3 Effect of Powder Content on Oil Yield

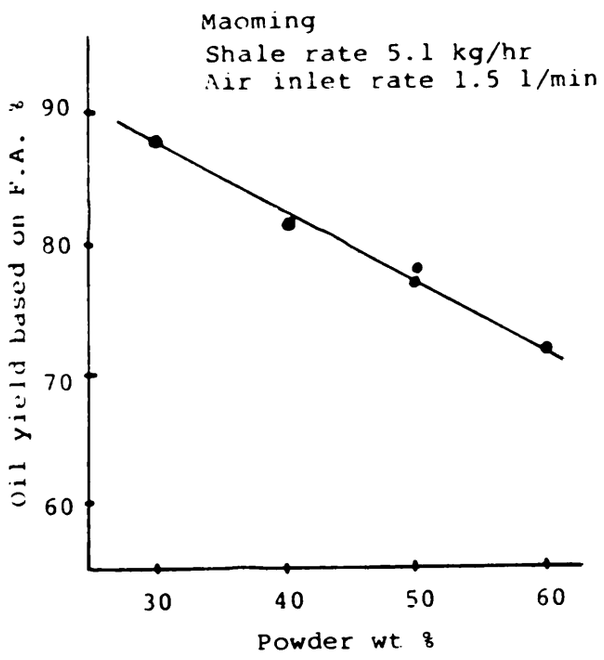
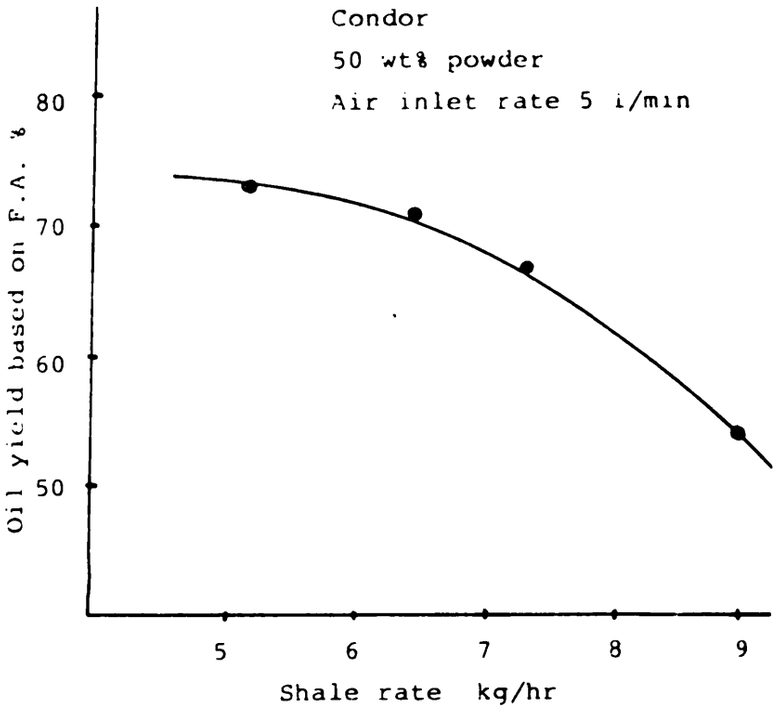


Fig. 4 Effect of Shale Feed Rate on Oil Yield



Effect of oil shale feed rate on oil yield is shown in Fig. 4, in which residence time ranges from about 60 to 100 minutes.

Data on Thai shale were obtained using the retort with a crushing mill (17) shown in Fig. la. In case of Thai oil shale having high Fischer Assay value, retorting granular shale alone caused agglomeration resulting in plugging-up, but by mixing it with 38 to 50 wt% of powder shale, smooth operation could be performed, about which we reported previously.[2)]

4. Conclusion

A series of experiments using an inclining tpye continuous retort clarified that it could retort four kinds of oil shales from Colordo, Condor, Maoming and Thailand, though its oil yield was 90 % at the most and suitable retorting condition varied depending on kind of shale. In order to promote operation stability, preliminary removement of water, reduction of powder content (except for Thai oil shale) and enlargement of maximum granular size (possibly 10 mm

in diameter) are considered to be effective.

References

1) Enomoto,M., Sato,S., Takahashi,S., Matsuzawa,S., Sekiyu Gakkaishi Vol 28 No2, 126 (1985)

2) Enomoto,M., Sato,S., Takahasi,S., Sekiyu Gakkaishi Vol 30 No 3, 166 (1987)