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REVIEWS

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DEVELOPMENT OF OIL SHALE PROCESSING INDUSTRY IN ESTONIA BEFORE WORLD WAR II

The Baltic oil shale (kukersite) possesses unique properties due to a high oxygen content of kerogen ($\approx$ 10 %). Long experience of commercial oil shale processing in Estonia has demonstrated that these properties become most evident when kukersite is used for the production of shale oil. Since oxygen-containing compounds prevail in the oil, it is possible to use the latter as feedstock for the manufacture of products not easily obtainable from petroleum, coal and oil shales other than kukersite.

During the early years of development of the oil shale industry in Estonia several methods of retorting were used (Table 1). A series of long experiments in different units (Geissen, Rolle, Pumpherston, etc. type retorts) demonstrated their complete unfitness for retorting kukersite [1—5]. On the other hand, test runs in a Pintsch-designed experimental retort with a throughput of 7—8 tonnes per day erected at Kohtla-Järve in 1921 showed promising results. An oil yield of 18 % from the feed shale was attained. Taking into account, however, that at that period mostly high organic oil shale was used for retorting (Table 2), the oil yield obtained could not be regarded as sufficiently high (77 % of Fischer assay oil).

Based on positive results obtained by testing the retort, already in 1921 "J. Pintsch A/G" began to design the first oil shale retorting plant at Kohtla-Järve, which consisted of 6 retorts with a throughput of 33 t/day each (later named GGS-1). The Pintsch-type retorts where the retorting zone was separated from the gasifier by a narrowed shaft at the mid-height were selected for the first plant. The air needed for processing was drawn in through the lower part of the retort by vacuum maintained within the whole reaction volume.

By the end of 1924 the construction of GGS-1 was completed with the start-up on December 24, 1924. The first production was obtained two days later. In December 1926 GGS-1 was commissioned into full commercial operation. A two-week balance test run gave the following results [3—4]: average throughput of oil shale — 33.4 t/day, the oil yield — 17.3 % from feed shale (68 % of Fischer assay oil), specific gas yield — 690 m^3/t^* (incl. 20.6 g/m^3 C_5^+ hydrocarbons) with a calorific value of 4.86 MJ/m^3. The characteristics of the feed shale used and the spent shale discharged during the test run are given in Table 3.

The demand for liquid fuels increased considerably in the early 1930's as a result of the beginning economic crisis. The State Oil Shale Works began to intensively search for opportunities of introducing new retorting capacities as fast and least

*All data given at 20 °C and p=101.3 kPa.

Table 1. **Start-up of Oil Shale Processing Units in Estonia before World War II**

Plant	Unit	Through-put rate, t/day	Number of retorts/ ovens	Start-up	Cease of operation
Kohtla-Järve	Experimental vertical retort (generator)*	7—8	1	3.08.21	December, 1924
	GGS-1	33	6	24.12.24	30.07.85
	GGS-2	40	8	31.03.36	30.07.85
	GGS-3	40	16	28.05.38	
	GGS-4	45	20	1943	
Kiviôli	Experimental tunnel oven section		1	1926	In the 1930's
	Commercial-scale experimental tunnel oven	75	1	1927	
	Commercial tunnel oven	250	2	1931	The last oven was closed down in 1975
		350	2	1935	
Sillamäe	Commercial tunnel ovens	270	1	1928	Most likely during World War II
		500	1	1938	
Kohtla-Nômme	Davidson horizontal rotary retorts	25	4	1931	1961
		25	4	1934	1961

* In the Baltic oil shale basin a mode of direct heated vertical retorts is historically referred to as "oil shale generators".

expensive as possible. Under these circumstances local specialists led by K. Luts developed a new "Kohtla-Järve" design of a cylindrical retort without the narrowed shaft for an oil shale throughput rate of 37.5 t/day. The units GGS-2 and GGS-3 were equipped with such retorts. On GGS-1 and GGS-2 the spent shale residue was removed from the units by small steel trucks (initially driven by horses, later by electric locomotive), but at GGS-3 a steel conveyer was used, replaced by belt conveyer in 1953. Analogous belt conveyers were installed on GGS-1 and GGS-2 in 1956.

Table 2. **Properties of Feed Shale Processed at the Kohtla-Järve Plant**

Year	Moisture W^r, %	Content, %			Fischer assay oil yield, %	Calorific value (bomb calorimeter), MJ/kg
		Mineral carbon dioxide, $(CO_2)_M^d$	Ash A^d	Conventional organic matter*		
1922	18.2	11.6	42.8	45.6	30.1	17.1
1923	18.8	11.9	45.6	42.5	28.1	16.0
1924	18.3	11.9	45.6	42.5	28.1	16.0
1925	19.0	12.2	46.0	41.8	27.6	15.7
1926	16.0	12.6	42.7	44.7	29.5	16.8
1927	12.3	12.2	40.1	47.7	31.5	17.9
1928	13.2	12.0	41.9	46.1	30.4	17.3
1929	12.5	12.6	43.1	44.3	29.2	16.6
1930	12.7	12.3	41.3	46.4	30.6	17.4
1931	11.8	12.1	40.4	47.5	31.3	17.8
1932	11.6	12.0	40.4	47.6	31.4	17.9
1933	11.6	12.0	40.3	47.7	31.5	17.9
1934	11.2	14.0	44.5	41.5	28.6	16.3
1935	11.0	15.2	46.7	38.1	26.3	15.0
1936	11.0	17.5	45.9	36.6	24.4	13.9
1937	11.0	19.4	46.6	34.0	23.3	13.3
1938	10.6	17.9	45.7	36.4	25.1	14.3
1939	10.6	18.7	46.6	34.7	23.6	13.4
1940	10.7	19.1	47.5	33.4	23.2	13.2
1941	10.7	19.3	48.2	32.5	22.7	12.9

* $100 - (CO^2)_M^d - A^d$.

Table 3. **Properties of Feed Shale Used and Spent Shale Discharged in Test Run on GGS-1 in 1926**

Properties	Oil shale	Semi-coke*	Spent shale
Moisture, %	13.2		
Proximate analysis (dry basis), %:			
carbon dioxide, $(CO2)_M^d$	12.5	18.0	13.2
ash, A^d	39.6	62.4	75.7
organic matter	47.9	19.6	11.1
Fischer assay product yield, %:			
shale oil	29.3	-	-
pyrogenetic water	2.2	-	-
semicoke	59.1	-	-
gas and losses	9.4	-	-
Calorific value (bomb calorimeter), MJ/kg	17.2 5	6.15	3.43

* Sample of semicoke taken from the narrowed shaft.

Table 4. **Oil Shale Processing on Retorting Plants in Estonia**

Year	Kohtla-Järve			Kiviôli		
	Oil shale processed	Produced		Oil shale processed	Produced	
		crude oil	motor petrol		crude oil	motor petrol
1921	1013	115				
1922	1668	259				
1923	2368	361				
1924	2196	337				
1925	18746	2652				
1926	39601	5784				
1927	31621	4237				
1928	42412	6595				
1929**	32454	5453				
1930	37959	6318				
1931	43842	6829				
1932	52503	9001				
1933	61200	10404				
1934	64888	11031				
1935	69165	11758				
1936	133703	22868	1179	116393	22926	3569
1937	178205	30008	968	284782	55563	8852
1938	301926	48977	1041	328876	65079	10034
1939	370198	60545	1443	340185	70004	11515
1940	386341	59701	946	354530	68195	10772

* Calcualted by difference.
** 1929-1933 - the years of world economic crisis.

The daily throughput of these cylindrical retorts was 41—42 tonnes of oil shale, the shale oil yield being 14.0—14.5 % on feed shale or 60—65 % of Fischer assay oil. The gas yield was 720—750 m^3/t.

The construction of a new plant (GGS-4) was started before the World War II, the Pintsch-type retorts with a design capacity of 37—40 t/day were selected again, but this time with more complete control of the process which enabled to obtain higher oil yields. As on GGS-3, for removal of ash residue a steel conveyer was also installed on GGS-4 which in 1955 was replaced by conveyer belt.

At the Kiviôli and Sillamäe plants tunnel ovens were selected for processing of oil shale, where the retorting process was carried out in steel oven-trucks. The tunnel ovens enabled to obtain oil with a higher content of gasoline fractions in the oil (up to 20 Vol. %). Owing to the use of the gas and oil vapours formed in the tunnel oven process as circulating gaseous heat carrier heated in heat exchangers

before World War II, tonnes

Sillamäe			Kohtla-Nômme*			Total		
Oil shale processed	Produced		Oil shale processed	Produced		Oil shale processed	Produced	
	crude oil	motor petrol		crude oil	motor petrol		crude oil	motor petrol
	-			-	-	1313	115	
	-			-	-	1668	259	
	-			-	-	2368	361	
	-			-	-	2196	337	
	-			-	-	18746	2652	
	-			-	-	39601	5784	
	-			-	-	31621	4237	
	5271			-	-	69951	11866	
	3549			-	-	61896	10776	910
	-			-	-	49466	10066	251
	-			1586	154	106674	17149	1223
-	-			4124	678	198261	36595	4209
	-					202099	37617	4992
	-			8248	1356	243465	46876	5899
	-					250866	47273	6217
43893	8019	1342	49391	9643	1649	343380	63456	7739
81452	15703	2969	54220	10619	1612	598651	11893	14401
81904	14938	2403	54498	10637	2582	767204	139631	15160
204443	36944	7623	54639	11397	2034	969465	178890	22615
196989	35197	6719	73374	10936	2063	1011234	174029	20500

(superheaters), the plant oil yield reached that of the Fischer assay. In addition, about 1 % (on the initial feed shale) of light gasoline was recovered in a refrigerating plant. The calorific value of the product gas with a specific yield of 20—30 m^3/t was about 33.5 MJ/m^3 (excl. 20—25 g/m^3 of C_5^+ hydrocarbons which remained in the gas after light gasoline separation).

At Kohtla-Nômme the low-capacity Davidson horizontal rotary retorts were operated for a relatively short period. The plant consisted of 4 rotating horizontal drums, heated externally with flue gases, generated by burning of their own semicoke. The oil vapours were withdrawn through offtake pipe. The oil yield was 19—20 % from the feed shale, i.e. about 90 % of Fischer assay oil. The gas yield was within a range of 80—120 m^3/t, its calorific value (excl. C_5^+ hydrocarbons) being about 3,500 kcal/m^3.

Table 5. **Export of Estonian Oil Shale Fuel Products**

Year	Crude oil				Motor petrol		
	Tonnes	Thousand kroons	Average price, kroons/tonne	Export, per cent of total production	Tonnes	Thousand kroons	Average price, kroons/tonne
1923	8.1	0.34		2.5			
1924	9.3	1.31		2.7			
1925	72.8	4.96		4.1			
1926	11.2	1.67		1.9			
1927	863.3	93.60		20.8			
1928	2516.8	244.30	97.0	21.3			
1929	1866.5	132.0	74.5	16.7	209.5	37.2	187
1930	1568.9	104.3	67.0	15.7	227.8	39.4	173
1931	2353.9	125.1	53.2	13.8	518.3	48.4	271
1932	3939.9	220.8	60.5	10.0	753.3	188.3	250
1933	6210.7	320.0	51.5	16.8	1573.5	437.4	278
1934	13967.4	691.7	49.8	30.0	2078.4	685.9	330
1939	90000.0			50.3			

Table 6. **Properties of Feed Shale and Basic Operational Data for Semicoking in Retorts of Different Design**

Characteristics	Tunnel ovens		Davidson retort Dec. 12, 1960	Retorts GGS-3 October 21—24, 1963
	Kivióli December 1973	Kohtla-Järve February 1961		
Test number	1	2	3	4
Oil shale				
Moisture, %	8.3	9.0	8.0	8.8
Conventional organic matter, (dry basis), %	31.2	35.0	30.0	34.0
Fischer assay oil yield, %	21.6	23.6	20.2	23.1
Calorific value (bomb calorimeter), MJ/kg	11.97	13.44	11.22	13.15
Yield of products				
Shale oil, %				
Plant yield (raw shale basis)	19.0	20.8	16.5	13.9
Yield of Fischer assay oil	97.6	97.0	89.0	66.0
Specific gas yield, m^3/t	20	25	100	585
C_5^+ hydrocarbons in product gas, g/m^3	22	22	no data	24
Operational data				
Feed shale throughput rate, t/day	384	388	23	47
Temperature of oil vapours at the retort offtake, °C	460	480	450	210
Final gas temperature after condensation system, °C	25	30	20	63

Table 7. **Properties of Oil Produced by Semicoking Oil Shale**

Properties	Test number (see Table 6)			
	1	2	3	4
Density at 20 °C, g/cm³	0.9644	0.9650	0.9490	1.0031
Viscosity at 75 °C, °E	1.76	1.4	1.5	2.9
Flash point, °C	10	25	9	104
Distillation, Vol. %:				
Initial boiling point, °C	91	65	60	183
80 °C	-	2	4	-
100	1	3	6	-
120	3	4	9	-
140	4	5	12	-
150	6	7	14	-
160	8	9	15	-
180	11	11	18	-
200	15	14	22	3
220	19	16	24	5
240	23	20	27	9
250	25	22	30	11
260	27	24	30	13
280	32	28	34	18
300	39	35	39	24
320	45	40	48	31
340	56	55	53	40
350	64	73	60	49
360	79	-	68	58
Calorific value (bomb calorimeter), MJ/kg	40.19	40.11	40.11	39.27
Phenolic compounds, %	23.3	22.3	23.1	26.7
Molecular mass, M	276	-	-	280
Elemental composition, %:				
C	82.1	82.6	81.5	83.1
H	9.9	10.4	9.9	9.7
S	0.8	0.9	1.1	0.8
O + N (by difference)	7.2	6.1	7.5	6.4

Table 8. **Chemical Group Composition of Light-Middle Fractions of Shale Oil, wt. %**

Fractions and compounds	Test number (see Table 6)			
	1	2	3	4
Fraction boiling up to 200 °C				
Alkanes and cycloalkanes	23	7	20	16
Alkanes	50	77	57	34
Aromatic hydrocarbons	15	11	13	30
Neutral oxygen compounds	10	3	9.5	17
Phenols	2	2	0.5	3
Fraction yield, %	11.9	12.8	17.5	4.4
Fraction 200—350 °C				
Alkanes and cycloalkanes	11	9	11	9
Alkanes	14	17	13	13
Aromatic hydrocarbons	29	28	33	32
Neutral oxygen compounds	22	22	20	24
Phenols	24	24	23	22
Fraction yield, %	36.6	27.4	29.5	35.1

Table 9. **Properties of C_5^+ Light Gasoline Fraction Recovered from Gas**

Indices	Tunnel ovens Kivilôli, 1963				Retorts GGS-3 1.—5. november, 1963
	Two ovens 26.—29.11. I and II lines		Three ovens 10.—11.12. II line		
	prior to recovery	after recovery	prior to recovery	after recovery	
Test number	5	6	7	8	9
C_5^+ hydrocarbons, g/cm³	270	22	287	22	24
Density at 20 °C, g/cm³	0.6569	0.6764	0.6445	0.6528	0.7160
Refraction index, n_D^{20}	1.3840	1.3865	1.3752	1.3870	1.4140
Molecular mass	75	-	-		89
Distillation, Vol. %					
Initial boiling point, °C	25	-	21		31
30	8	-	17		-
40	31	-	37		2
50	53	-	56		8
60	67	-	71		16
70	78	-	78		25
80	84	-	82		36
90	92	-	83		48
100	-	-	84		59
110	-	-	-		70
120	-	-	-		79
130	-	-	-		86
140	-	-	-		92
150	-	-			93
Calorific value (bomb calorimeter), MJ/kg	-	-	48.15		45.80
Elemental composition, %					
C	-	-	-		85.4
H	-	-	-		13.4
S	-	-	-		0.8
O + N	-	-	-		0.6

As can be seen from Table 4, by 1940 the annual oil shale processing capacity reached 1 million tonnes from which 170—180 thousand tonnes per year of oil were produced. The oil was refined to produce 20—22 thousand tonnes of motor petrol annually. Up to 50 % of the oil and a portion of motor petrol were exported (see Table 5) [5]. The motor petrol was of low quality (octane number 66—68), however, at that time it met the needs of home market.

For complete characterization of the oil shale retorts operated in Estonia in the pre-war period, in Tables 6—11 the physical and chemical properties of the retort products are presented. The analyses were performed after World War II by the Oil Shale Research Institute when systematic investigation of oil shale processing plants was carried out.

Table 10. **Chemical Composition of C_5^+ Light Gasoline Fraction Recovered from Gas, wt. %**

Hydrocarbons	Test number (see Table 9)			
	6	7	8	9
Alkanes				
n-propane	-	-	-	0.7
n-butane	6.0	5.9	8.1	3.8
n-pentane	17.3	16.6	14.4	6.5
n-hexane	10.9	11.8	9.1	6.4
n-heptane	2.9	2.0	2.7	6.2
n-octane	1.0	0.7	2.0	3.6
higher paraffins			1.0	
Total	38.1	37.0	37.3	27.2
Alkenes				
propene-1	-	-	-	-
butene-1	2.8	3.4	7.3	4.7
trans-butene-2	2.5	2.0	4.9	1.8
cis-butene-2	1.3	1.2	2.8	1.2
1,3-butadiene	0.3	0.3	0.6	-
pentene-1	12.2	12.4	9.9	6.0
trans-pentene-2	5.9	6.1	4.4	3.0
cis-pentene-2	2.8	2.8	2.2	1.9
2-methylbutene-2	1.1	1.2	0.9	-
cyclopentene	1.3	2.5	1.4	-
trans + *cis*-1,3-pentadiene	4.8	5.9	4.4	-
hexene-1	11.6	12.7	8.9	7.7
hexehe-2	3.9	4.1	3.4	2.2
heptenes	6.9	4.7	4.7	8.7
octenes	1.5	1.0	1.2	4.6
isoolefins	-	-	-	1.1
diolefins	-	-	-	10.1
cycloolefins	-	-	-	1.8
Total	58.9	60.3	57.0	54.8
Aromatics				
benzene	1.0	0.8	1.9	7.1
toluene	1.3	0.6	1.3	4.6
Total	2.3	1.4	3.2	11.7
Identified	99.3	98.7	97.5	93.7
Not identified	0.7	1.3	2.5	6.3

Table 11. **Characteristics of Retort Gas**

Indices	Tunnel ovens [6]		Davidson retorts [6]	Retorts GGS-3
	Kiviõli	Kohtla-Järve		
Content of components, vol. %				
CO_2				
H_2S	21.0	22.7	12.6	18.3
C_nH_m	9.0	12.9	4.7	0.5
Including:	9.9	10.6	5.5	1.3
C_2H_4				
C_3H_6	3.5	3.3	1.9	0.8
C_4H_8	4.6	4.2	2.0	0.4
O_2	1.8	3.1	1.6	0.1
CO	1.2	1.2	3.2	2.0
H_2	7.4	8.9	4.4	5.6
C_nH_{2n+2}	5.6	5.6	5.5	6.2
Including:	24.5	32.9	13.1	4.6
CH_4				
C_2H_4	9.6	15.0	5.5	3.5
C_3H_8	8.8	11.1	4.5	0.8
C_4H_{10}	4.8	5.0	1.9	0.2
N_2	1.3	1.8	1.2	0.1
Content of H_2S, g/m^3	21.4	5.2	51.0	61.5
	130	186	28	8
Calculated gross calorific value				
(without C_5^+ hydrocarbons), MJ/m^3	27.26	33.70	15.20	3.8

REFERENCES

1. *Winkler H.* Der estländische Brennschiefer. Untersuchung, Gewinnung und Verwertung. Reval, 1930. 350 S.
2. *Zeidler R.* Neue Wege der Verwertung des Ölschiefers und seiner Umwandlung in Öle. Reval, 1933. 194 S.
3. *Luts K.* Der estländische Brennschiefer Kukersit, seine Chemie, Technologie und Analyse. Tallinn, 1934. 356 S.
4. Riigi Põlevkivitööstus 1918—1928. - Tallinn: Riigi põlevkivitööstuse kirjastus, 1928.
5. *Кузнецов Д.Т.* Очерки развития сланцевой промышленности Эстонской ССР. Л, 1960. С 200.
6. *Баршевский М.М., Безиогин Э.С. Шапиро Р.Н.* Справочник по переработке горячих сланцев.- Л., 1963. С .238.

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