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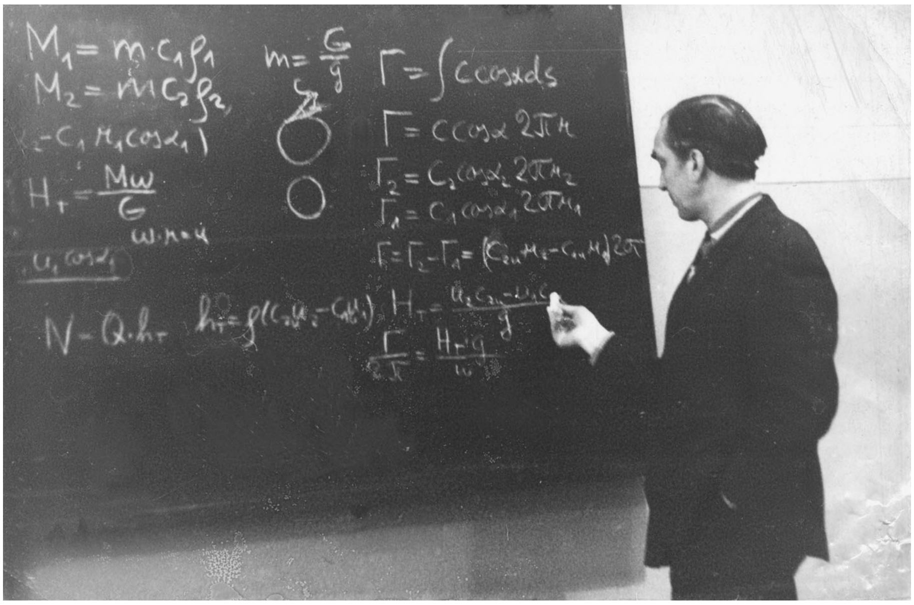
In memoriam

Investigation of the physical properties of oil shale of the Estonian deposit

In memoriam Heino Aruküla



Edited by Enno Reinsalu*

This is Assistant Professor Heino Aruküla's presentation at the UN Oil Shale Symposium in Tallinn in 1968. The text was published in a very limited edition. Oil shale natural properties have not changed during fifty years. What has changed a little is the terminology. However, interest in these properties of oil shale has been increasing. The presentation deserves attention because no significant studies have been made in this area hitherto.

Introduction

The physical properties of the oil shale bed in the Estonian deposit were investigated at the Mining Department of Tallinn University of Technology

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in 1964–1967. This paper is aimed at establishing the initial data to solve the following problems pertaining to the utilization of oil shale:

1) The choice of optimal methods for breaking and preparing oil shale for combustion at heat and power plants and for treatment at shale oil plants.
2) The choice of the optimal method and parameters for concentrating the rock mass.

The above processes are noticeably influenced by the strength, density, specific volume, electric properties, and millability of the rock to be tested, as well as its mineralogical and petrographical characteristics.

The physical properties of the rock in the developed area of the Estonian oil shale deposit were determined. The samples were taken at the mines of Kukruse (the northern part of the deposit, 59°23' 27°23', closed 1967), Kiviõli (the western part of the deposit, 59°20' 26°57', closed 1987) and Viru (the central part of the deposit, 59°18' 26°20', closed 2103), and in the eastern part of the deposit's open-casts at Sirgala (59°18' 27°46') and Viivikonna (59°19' 27°40'). The number of sampling points at each mine as well as the number of samples or portions for determining the average rock strength with a sufficient reliability was selected on the basis of the permissible coefficient of variation, 20%. At each mine the samples were taken from 2–4 places in each layer of oil shale (6) and interlayer of limestone (4) of the bed. The weight of one sample was from 20 to 50 kg. The samples were subjected to a complete analysis, to determine the strength, density and volumetric mass of the rock, as well as its mineralogical and petrographical features.

The rock strength was determined for all samples. The data on strength were obtained for a particular mine field and the entire deposit. Based on these data the samples were selected to estimate the other physical properties of the rock.

An arithmetic mean of the data for the layers, sampling points, mine and deposit, and the mean of squared deviations and coefficients of variation were accepted as the basic characteristics for a set of representative physical properties. Distribution curves were drawn and correlation relationships derived for certain main indices (rock strength, specific volume).

The oil shale bed in the developed part of the Estonian deposit has a quiet, nearly horizontal stratum with a persistent total thickness. The thickness of particular oil shale layers and the rock constituting the oil shale bed is also comparatively uniform, although individual layers (layer B at Kiviõli mine, layer C at Kukruse mine and layer F at Viru mine) have a somewhat smaller average thickness than the similar layers at the other mines.

The change in the thickness of the layers of the oil shale bed is shown in Table 1.

Table 1. Layer thickness at sampling points, cm

Layer	Minimum	Maximum	Average
A	20	30	24
A/B	10	23	17
B	22	68	50
B/C	4	21	12
C	33	49	42
C/D	19	34	23
D	6	13	10
E/D	4	15	9
E	15	53	32
F	67	109	96
Bed	261	335	309

Mineralogical and petrographical investigations

The mineralogical and petrographical investigations of the thin sections made from samples from all mines and layers were conducted with the aid of the Hi-8 model polarizing microscope. Main attention was given to the structural and textural features with the aim of elucidating the effect of rock structure on its strength.

In the oil shale layers the organic matter is represented by two varieties:

1) Slightly rounded somewhat flattened dark and dense accumulations of small ball-shaped bodies; the diameter of accumulations and that of separate balls are 0.07–0.1 and 0.005 mm, respectively.
2) Light-brown homogeneous mass into which these accumulations are submerged.

The bulk of the carbonate matter in the oil shale layers is composed of the skeletal remains of various organisms, consisting as a rule of micro-crystalline calcium, sometimes of fibrous structure. Recrystallized fragments of organisms are more common in layers C and F. In some places, the carbonate matter surrounds as a thin film particular accumulations of organic matter.

The rock in the oil shale layer possesses a rather clear-cut microstratified texture. The thinnest layer of oil shale is distinguished by the presence of clayey material and angular grains of quartz.

As compared to the other oil shale layers, layer F contains less organic matter, especially in the form of accumulated dark balls, while the carbonate skeleton manifests itself better and is practically uninterrupted, which increases the mechanical strength and brittleness of the layer.

The limestone interlayers consist mainly of finely crystallized calcite (grain size 0.01–0.03 mm), recrystallization of which has produced larger (up to 0.5 mm), not quite homogeneous grains, usually of an elongated form. The organic matter is represented by accumulations of black balls immersed into a light-brown amorphous mass. Such accumulations can be mostly found in interlayers A/B and E/D (> 15%), and less frequently in interlayers

B/C (< 10%) and C/D (3%), which correspondingly affects the rock strength of interlayers.

The thin sections from interlayers B/C and E/D reveal a non-uniform distribution of organic matter, while in interlayer C/D its accumulations have an irregular shape.

The organic portion of the samples from Kukruse mine from the zones of karst dislocations and belts of weathering exposes the traces of oxidation (a darker amorphous mass), which may also affect the mechanical properties of oil shale layers.

Volumetric mass, density and porosity of rock

The volumetric mass of rock was determined by hydrostatic weighing, density by the pycnometric method and porosity by calculations. The volumetric mass and density were found to an accuracy of 0.01 g/cm^3. The results are presented in Table 2.

The maximum mean density and porosity of the bed rock were observed at Kiviõli mine. This can be attributed to the great total thickness of limestone interlayers and a higher amount of concretions in the oil shale layers.

For individual mines, the mean values of the density, volumetric mass and porosity of oil shale layers were observed to vary to a much greater extent than the same indices for the limestone interlayers.

Table 2. Density, volumetric mass and porosity rock of the mineable oil shale bed

Oil shale layer and limestone interlayer	Volumetric mass, g/cm^3			Density, g/cm^3			Porosity, %		
	min	max	average	min	max	average	min	max	average
A	1.29	1.61	1.44	1.70	1.92	1.78	16.4	24.8	19.4
B	1.21	1.34	1.27	1.44	1.62	1.54	13.5	22.0	17.5
C	1.52	1.73	1.62	1.62	2.00	1.85	10.6	24.2	14.5
D	1.45	1.61	1.55	1.80	2.02	1.91	10.5	28.0	18.3
E	1.24	1.38	1.31	1.44	1.62	1.52	13.3	15.0	14.1
F	1.29	1.51	1.41	1.89	1.98	1.93	23.7	31.8	27.0
Oil shale	1.35	1.46	1.41	1.78	1.88	1.82	19.3	24.3	21.1
A/B	2.28	2.34	2.32	2.50	2.54	2.51	7.0	9.01	7.8
B/C	2.21	2.34	2.28	2.49	2.53	2.50	5.6	13.7	8.8
C/D	2.39	2.57	2.48	2.62	2.76	2.71	5.0	12.6	8.0
D/E	2.12	2.21	2.18	2.28	2.35	2.34	6.0	11.5	8.0
Limestone	2.32	2.39	2.35	2.54	2.57	2.55	7.0	9.6	8.3
Bed	1.57	1.63	1.60	1.91	2.02	1.96	17.0	20.5	18.2

Rock strength

The rock strength factor *f* [1], according to Prof. Protodyakonov's scale, is the most universal index which characterises the relative resistance of various rocks to destruction. A number of methods are in common use for determining the rock strength factor: compression of regular or irregular specimens, pounding method, etc. The most widespread method, compression of regular specimens, is rather laborious because the specimens must be of fixed dimensions with their surfaces thoroughly ground.

During the first stage of the work, the investigators aimed at determining the strength of rock by all the above methods in order to obtain a comparative estimate of the results under conditions of the Estonian oil shale deposit. The samples were taken from Sirgala open-cast. The results are shown in Table 3.

According to the data listed, the strength factor *f* determined by compressing regular specimens is proportional to the strength factor *f* found from both the compression of irregular specimens and their pounding. However, the absolute values of the factor *f* obtained from the regular specimens compression are 1.1–1.5 (in some layers such as A, C and D even 2.1–2.5) times higher as compared with the two latter methods, which yielded practically the same results. The factor *f* was increased because many of the prepared regular specimens proved unsuitable for the tests (cracks, peeling).

The strength of rock can be determined by compressing regular specimens and by pounding more accurately than by compressing irregular specimens because the coefficients of variation obtained by the latter method are much higher. The pounding method likewise involves less work for preparing specimens for the tests, and for this reason this method was

Table 3. Mean rock strength factors at Sirgala open-cast

Oil shale layer and limestone interlayer	Regular specimens	Irregular specimens	Pounding
A	2.7	1.8	2.0
B	3.4	2.3	2.1
C	2.7	1.3	1.2
D	3.3	1.7	1.5
E	3.4	2.8	2.4
F	3.5	3.1	2.7
Oil shale	3.3	2.4	2.0
A/B	4.4	3.2	3.2
B/C	4.8	3.3	3.6
C/D	6.2	5.2	4.9
D/E	4.3	3.3	3.7
Limestone	5.4	4.3	4.3
Bed	3.8	2.8	2.5

[1] $f \cong$ UCS / 10 where UCS is uniaxial compression strength, MPa (ed.)

accepted as the principal one during the second stage of investigations. For the sake of comparison the factor *f* was determined also by compressing the specimens of irregular shape.

The data on the strength factor and variation factor for three mines are presented in Table 4. In the pounding method, the relative measurement error was 1.2%, and in compressing irregular specimens, 2.1%.

The mean strength factor of oil shale and limestone and the bed as a whole were determined as a massed average, account being taken of the thickness of the layers. In the pounding method, the mean factor of rock strength in the oil shale bed was 2.0, and in compressing irregularly shaped specimens, 1.7.

The mean strength of oil shale and limestone for various mines differs negligibly. Only at Kukruse mine, a certain reduction in the oil shale strength for the samples taken from both the belt of weathering and the zone of karst dislocations can be observed. An appreciable decrease in the strength of oil shale in the layers has been shown by means of Student's t-criterion.

The stronger interlayers should be taken into account when designing the machines and equipment, and the softest layers when problems of the stability of pillars are to be solved. It is advisable to use for this purpose the rock strength distribution curve (Fig.) drawn on the basis of 569 tests. Investigation of the relationship between rock strength and volumetric mass revealed the following correlation ratio f = 1.61+1.85 with the correlation factor r = 0.8.

Table 4. Rock strength factor *f* at different mines

Oil shale layer and limestone interlayer	Kukruse mine				Viru mine				Kiviõli mine				Average for three mines	
	f_p	f_v	f_c	f_v	f_p	f_v	f_c	f_v	f_p	f_v	f_c	f_v	f_p	f_c
A	1.2	15	0.5	56	1.2	9	0.6	30	1.3	13	1.8	50	1.2	1.0
B	2.0	20	1.6	38	1.8	15	1.0	26	1.8	18	1.6	36	1.9	1.4
C	1.4	31	1.4	46	1.2	11	0.8	57	1.3	17	1.8	59	1.3	1.3
D	1.3	14	1.1	50	1.6	9	1.0	23	1.3	20	1.2	28	1.4	1.1
E	1.9	24	1.6	34	1.4	6	1.0	57	1.6	8	1.5	19	1.6	1.4
F	1.8	26	1.6	63	1.5	8	1.3	35	1.7	12	1.2	44	1.7	1.4
Oil shale	1.7		1.4		1.5		1.1		1.6		1.4		1.6	1.3
A/B	2.8	41	2.8	32	2.4	7	2.4	29	2.8	12	2.8	19	2.7	2.7
B/C	4.1	18	3.0	26	3.8	20	3.0	25	3.4	22	3.6	17	3.8	3.2
C/D	3.9	18	3.2	26	4.0	9	3.4	26	4.2	14	3.6	20	4.0	3.4
D/E	3.0	14	3.1	29	2.4	8	2.1	34	2.8	10	2.6	22	2.7	2.6
Limestone	3.4		3.0		3.2		2.8		3.4		3.2		3.3	3.0
Bed	2.0		1.7		1.9		1.5		2.0		1.8		2.0	1.7

f_p – strength of rock determined by the pounding method
f_c – strength of rock determined by compressing irregular specimens
f_v – coefficient of variation, %.

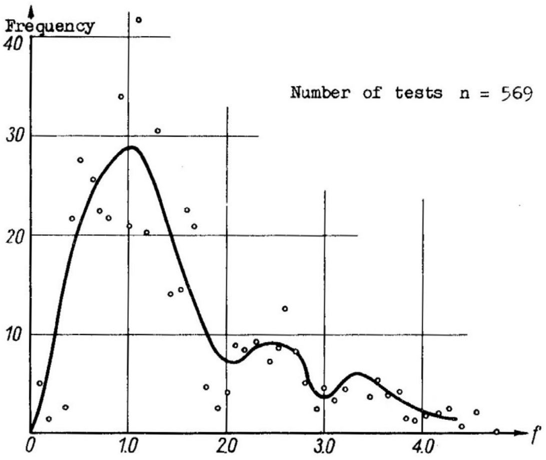

Fig. Rock strength distribution curve.

Millability of rock

The resistance of the bed rock to breaking was estimated using the millability factor obtained by the methods of TsKTI [2] employed for rock samples from Sirgala open-cast (Table 5). The representative samples of oil shale were taken from separate layers of the bed on the basis of profiles.

To determine the millability factor the oil shale samples of 230 cm^3 (by volume) were taken and weighed on a second-class technical scale. The fineness of the sample was 0.630–0.088 mm.

The millability factor was determined by the average amount of the fines (g/min) formed during the crushing operation on a drum-ball mill and passed through a 0.088 mm screen sieve.

The determination of the millability factor of oil shale samples from Sirgala open-cast has shown that milling affords high selectivity. At the same time, as in the case of coal, two or three milling operations are sufficient to obtain stable operating conditions (10% passage through a

Table 5. Mean millability factors at Sirgala open-cast

Sampling site	Bed	Bed without interlayer C/E
1st area	2.96	3.86
3rd area (southern)	2.72	3.67
3rd area (northern)	2.98	4.03
Mean value	2.89	3.86

Maximum deviation from the average value was 5.9%.

[2] The method used in Russia (ed.)

88-mesh screen during the same period of time), the oil shale required seven or eight preliminary crushings. This was governed by the different resistance to milling offered by the micro-components of the combustible kukersite oil shale and limestone. In such conditions, it will be more advisable to take the effect of selectivity into account and develop a new more efficient method for determining the millability.

Electric characteristics of rock

The methods employed at the present time for determining the quality of oil shale at both the mines and electric power plants require much effort and time, and the sampling representativeness is not reliable.

Thus, at definite time intervals, both the differential and industrial bed samples are taken by slotting from the walls. These samples are used to determine by laboratory analysis the density, and the content of moisture, ash and organic matter.

The qualitative indices of oil shale may also be assessed indirectly by electrical methods. The most promising is the radio-wave method in which the permittivity ε and the loss angle tangent $\tan\delta$ are determined in high-frequency electromagnetic fields depending on moisture content, and granulometric and mineralogical composition.

The oil shale from selected layers has been studied for ash and moisture content at Dnepropetrovsk Mining Institute by E. I. Arsh and G. R. Nosov.

The tests did not show any noticeable correlation between the electrical parameters ε and $\tan\delta$ and the ash content of oil shale. The study of the electric properties of moist oil shale (loose material) has shown that the moisture content may be controlled by either permittivity or the loss angle tangent.