

PRECISION OF OIL SHALE ENERGY RATING AND OIL SHALE RESOURCES

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The main quality indicator of kukersite is calorific value. Determining of this indicator is time and labor consuming. Using simple quality indicators, such as ash content or burning matter range, has not been adequate enough. The current paper shows that in some cases, when mining technology and structure of extracted material is constant, the quality of oil shale can be evaluated by the content of burning matter. In the case of oil shale resource calculations, the specific weight of the mineral is not measured but evaluated by calorific value. This method does not take into account the relationship between clay and lime minerals in the layers. The deviations from calculated resource figures have been noticed in mining practice. The error of this method is evaluated, and recommendations for recalculating oil shale resources are given.

Introduction

The main quality indicator for trade oil shale in Estonia and in Russia is its content of potential energy – heating value (Q , MJ/kg, GJ/t). The same quality criterion, calorific value, is used for oil shale reserve estimation as well. Elsewhere, where oil shale is treated as raw material for oil processing, the oil yield (T , %) is usually the principal quality indicator. Ordinary quality indicators for coal like ash content (A , %) and carbonate carbon dioxide content (CO_2) are seldom used, mainly in the case of oil shale combustion. In addition to the heating value, the essential quality indicator for trade oil shale is the moisture (W , %). Harmful substance content, for example that of sul-

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phur (S, %), is seldom used, because it is not high and does not vary much in kukersite.

All quality indicators noted are used in their different mode, for example lower heating value of dry fuel (Q^d_i), content of organic sulphur of dry fuel (S^d_{o+p}), etc. [1]. In the present article the oil shale quality indicators are treated like in geology and mining science – with respect to dry oil shale, where calorific value $Q = Q^d_b$ is gross calorific or heating value. In this article the particular composition as a quality indicator for trade oil shale is not treated.

Traditionally, oil shale energy content, calorific value, is measured by so-called bomb calorimeter and oil yield in so-called Fischer (aluminium) retort. Both methods are essentially laboratory model experiments: oil shale combustion takes place in the bomb calorimeter and dry distillation in the Fischer retort. In practice at combusting and processing, the real yield of obtainable product (energy and/or oil) correlates well with laboratory results, but is mostly lower than the laboratory yield. Mainly for that reason more specific quality indicators are used for trade oil shale to characterize utilization results better.

For example, in Estonia the principal indicator for trade oil shale is the lower heating value of the fuel as received (Q^d_i). Still, all geological information about oil shale has been collected and saved basing on the dry oil shale energy (calorific value Q^d_b) measured in the bomb calorimeter. That is motivated because the energy of dry oil shale is constant but moisture depends on the mining and trading conditions. Since the calorific value of kukersite oil shale measured in laboratory correlates well with oil yield, the Fischer oil yield is not used as a quality indicator for this kind of oil shale.

The Problem

It is quite usual that geologists, mining engineers, shale oil technologists and power engineers do not understand each other adequately when talking about energy content of oil shale. In most cases it arises from the fact that determination of oil shale calorific as well as heating value is neither exact and simple nor cheap enough.

The quantities of energy and processed shale oil obtained in a real process depend on the content of extraneous rock (limestone) and moisture in sell-up-shale, which depend on mining claim and technology. The geological reserves of oil shale and trade oil shale are different things – both are aggregates of various mineral components, which have not only different calorific value but also different specific weight and other properties. In this paper, the above questions are discussed proceeding from mining aspects as based on our own experience, and methodical recommendations are given to resolve these problems.

Dependence of Calorific Value on Component Content

In principle, the Estonian oil shale consists of three constituents: combustible part (in this article C , %), ash (A , %), and carbon dioxide (CO_2 , %) emitting from carbonaceous minerals.* The combustible part is the component whose combustion produces energy. Kerogen and also sulphur (S , %) are the main components of the combustible part. Dealing only with kukersite oil shale, one can affirm that energy content of the combustible part is relatively constant. According to that, only the content of the combustible part may be used for estimating calorific value of trade oil shale. These kinds of experiments have been done, and Prof. I. Öpik has given a concise synopsis about this work in 1961 already [2]. However, the variability of the non-combustible component part has not enabled to create a sufficiently exact method.

Solid residue or ash A and gaseous residue CO_2 are the main non-combustible constituents. Ash is formed mostly from clay and sand minerals, and CO_2 from lime minerals. The diagram in Fig. 1 illustrates the ratio of components in oil shale. The samples have been taken from different sources: from all mines and opencasts of the Estonia deposit, from all layers and interlayers, separately and together, from run-of-mine oil shale, from all products of mineral processing. The main statistical data are presented in Table 1.

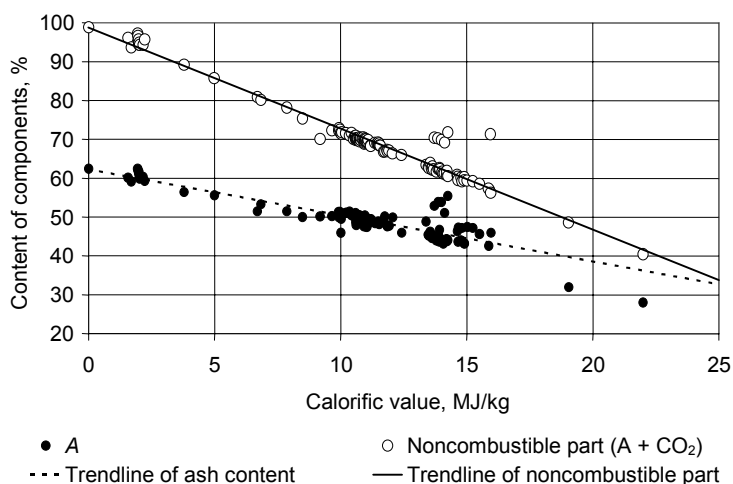


Fig. 1. Component content of oil shale versus calorific value (119 samples). The points mark sample parameters and the lines describe linear trends (correlation coefficients $r(A, Q) = 0.85$ and $r(\text{CO}_2, Q) = 0.95$). Lines must be treated as average limits of the content of three main components by any calorific value

* We fully agree that such a characterization is simplified. In this article, we discuss oil shale as a consumer item, not as a subject of academic research.

As seen in Fig. 1, the correlation between the ratio of oil shale components and calorific value is not uniform enough. There are several samples in the middle of the quality area ($13 < Q < 17$ MJ/kg) where the ash content is higher than the trend. At the same time the ash content of high calorific value samples ($Q > 18$ MJ/kg) is below the trend line, but the content of carbonate component is above it. In the first case clay and sand minerals prevail in the non-combustible part of the samples, and in the second case, on the contrary, lime-minerals prevail in it. Insufficient correlation between calorific value and content of the non-combustible component part is also demonstrated by studies on oil shale combustion [1]. A linear regression and correlation between combustible part and calorific value in our case was determined as $Q = 0.369C$ (R Squared = 0.94) or $Q = 0.3631C + 0.1904$ (R Squared = 0.94).

Table 1. Main Statistical Data of Samples

Quality indicator	Minimum	Maximum	Mean	Standard deviation
Ash A	28.8	62.4	49.1	5.1
Carbon dioxide CO_2	10.3	36.4	20.6	5.4
Combustible part C	4.2	59.7	31.2	9.4
Calorific value of dry oil shale in bomb Q	1.57	22.0	11.2	3.7

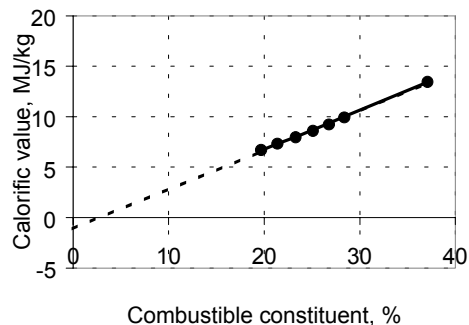
As the common variability in the content of clay and lime minerals does not allow finding an universal formula for calculating calorific value on the basis of combustible and non-combustible part, it is of interest, if it could be possible, when the mineral content of non-combustible parts is approximately constant. The non-combustible part is homogenous when oil shale is produced from the same mine using the same technology. For modeling that kind of situation, three series of laboratory experiments were made. The mixtures of two components – oil shale and extraneous rock (limestone waste) were studied. The initial components (Table 2) for mixtures were prepared, and calorific values and content of combustible part were measured in the Central Laboratory of Oil Shale Mining Company (*AS Eesti Põlevkivi*).

Table 2. Initial Components of Mixtures

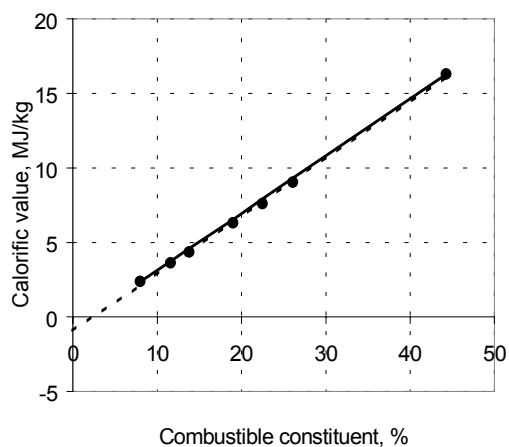
Series	Component	Q , MJ/kg	C , %
1. Two low-grade components	Waste rock, low kerogen content	6.66	19.7
	Clayey oil shale	13.43	37.1
2. Low-grade component and kerogen-rich component	Waste rock, limestone	2.37	8.0
	Oil shale	16.30	44.3
3. Two kerogen components	Waste rock, moderate kerogen content	10.54	29.6
	Oil shale	16.30	44.3

Series 1

$Q = 0.389C - 1.10$ MJ/kg
 R Squared = 0.990
 Standard error of estimated Q
 $s_Q = 0.097$ MJ/kg
 Calorific value of kerogen
 if $C = 100\%$
 $Q_c = 37.8$ MJ/kg
 Standard error of estimated Q_k
 $s_{Q_k} = 0.69$ MJ/kg
 Calorific value of noncombustible
 if $C = 0\%$
 $Q_0 = -1.10$ MJ/kg

**Series 2**

$Q = 0.385C - 0.92$ MJ/kg
 R Squared = 0.999
 Standard error of estimated Q
 $s_Q = 0.157$ MJ/kg
 Calorific value of kerogen
 if $C = 100\%$
 $Q_c = 37.6$ MJ/kg
 Standard error of estimated Q_k
 $s_{Q_k} = 0.53$ MJ/kg
 Calorific value of noncombustible
 if $C = 0\%$
 $Q_0 = -0.92$ MJ/kg

**Series 3**

$Q = 0.391C - 1.08$ MJ/kg
 R Squared = 0.998
 Standard error of estimated Q
 $s_Q = 0.086$ MJ/kg
 Calorific value of kerogen
 if $C = 100\%$
 $Q_c = 38.0$ MJ/kg
 Standard error of estimated Q_k
 $s_{Q_k} = 0.72$ MJ/kg
 Calorific value of noncombustible
 if $C = 0\%$
 $Q_0 = -1.08$ MJ/kg

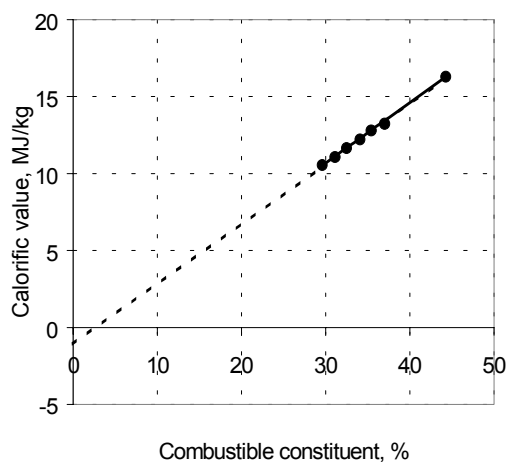


Fig. 2. Relationship between combustible constituent content and calorific value of tested mixtures: ● measured, - - - regression method, — ratio method

Three series of five mixtures with waste rock content of 50–90 % were made from every pair of initial components. So, each series included five mixtures and two initial components, seven tests in all. The main results are demonstrated in Fig. 2.

The relation between the content of combustible part C and calorific value Q in the mixture was determined by three ways:

1. Measured in laboratory.
2. Calculated on the basis of partial ratio:

$$Q = c_1 \times Q_1 + (1 - c_1) \times Q_2$$

$$C = c_1 \times C_1 + (1 - c_1) \times C_2$$

where c_1 is the share of waste rock in the mixture;

Q_1 is calorific value of the waste rock;

Q_2 is calorific value of the oil shale used in the mixture;

likewise C_1 is combustible constituent in waste rock;

C_2 is combustible constituent in the oil shale used in the mixture.

3. A linear regression equation was calculated by regression method on the basis of laboratory measurements:

$$Q = a + b \times C$$

where a (MJ/kg) and b (MJ/100 kg) are equation parameters.

Theoretically $a = 0$, because the non-combustible part does not give heat, and b should express the calorific value Q_c of the combustible part, as if $C = 100$, $Q = a + 100 = Q_c$.

In fact the charts in Fig. 2 and regression equations demonstrated that $a < 0$. This is simply explicable with the fact that some of the non-combustible parts of mineral components decompose at burning and absorb the energy released from the combustible part. More exactly, in the mixture with a very low calorific value, the heat capacity of endothermic processes exceeds the heat capacity supply of exothermic processes. It has been thoroughly described by I. Õpik already [2].

The following conclusions can be made basing on the tests:

- Calorific value as oil shale energy content can be determined with sufficient precision on the basis of the ratio of combustible/non-combustible part if mineral content of oil shale non-combustible part is constant.
- In heating value calculations of oil shale mixtures, calorific value of the non-combustible part (waste rock, limestone from interlayers and concretions) must be considered negative.

According to the first conclusion, a simple method for determination of oil shale energy value for steady vendors/customers can be composed.

The second conclusion may cause correction in evaluation of Estonian oil shale resources.

Problems of Energetic Potential of Estonian Oil Shale Deposit

Energy rating (E , GJ/m²) and weighted average calorific value of oil shale bed are the principal indicators for oil shale reserves. The quantity of mineable reserve is determined on the basis of energy rating. Calorific value of the bed is the basic indicator for estimating heating value of run-of-mine oil shale. The mentioned indicators of the bed are calculated by the following formulas. Calorific value of the bed

$$Q_{OS} = \Sigma(Q_i \times h_i \times D_i) / \Sigma(h_i \times D_i) \text{ (GJ/t)}$$

Energy rating for every layer forming the bed (oil shale layer, limestone interlayer)

$$E_i = Q_i \times h_i \times D_i \text{ (GJ/m}^2\text{)}$$

Energy rating of the bed is the sum of energy ratings of the layers:

$$E = \Sigma E_i \text{ (GJ/m}^2\text{)}$$

where Q_{OS} is calorific value of the oil shale bed, GJ/t (MJ/kg);

Q_i is calorific value of the layer, GJ/t (MJ/kg);

h_i is layer thickness, m;

D_i is volumetric weight, t/m³ (kg/dm³);

E_i is energy rating of the layer, GJ/m²;

i is index of the layer.

Consequently, the calorific value of the bed is the weighted average value of oil shale and limestone layers, which takes into co

nsideration their various thickness and volumetric weight of various layers. Energy rating takes into consideration energy, thickness and mass of all the layers as well. Geological methodology makes two simplifications:

- Calorific value of limestone with very low kerogen content is omitted without measuring, i.e. equated to zero.
- Volumetric weight is calculated on the basis of calorific value.

The non-combustible part of oil shale bed absorbs heat as mentioned above. Resulting from this and from the first simplification, calorific value and energy productivity of oil shale bed given in the geological data are greater than factually. This problem will be discussed once more at the end of this article.

The second simplification is not correct either. Using the laboratory method for measuring the volumetric weight of kukersite oil shale was ended 20 years ago. The volumetric weight is now calculated as a function of calorific value. Mining engineer H. Sits worked out an exact method for that, and it was taken into use by the Estonian Branch of Skotchinski Institute of Mining [3, 4]. However, this method is complicated. Score geologist V. Kattai worked out a simpler formula for computing volumetric weight [5]:

$$D_i = 1.383 \exp(-0.0003Q_i) + 0.89 \text{ (t/m}^3\text{)}$$

This formula was implemented officially.

It can be mathematically derived from the V. Kattai formula that the calorific value of combustible part of oil shale is $Q_c = 37.2$ MJ/kg, which is in good accordance with Fig. 2.

We have found the official method not to be exact enough [6]. The component ratios of the non-combustible part A/CO_2 are different in all oil shale layers and interlayers. This has not been taken into consideration. In the same article [6], we acquainted the exact calculation method worked out by H. Sits, which was free from this deficiency. The Estonian Geological Survey has not considered this up to now, and the Estonian oil shale reserve and quality parameters have been calculated by the simple official method.

At calculating the balance of Estonian mineral resources for 1997, it appeared that in several oil shale mines the in-place tonnage of the bed was lower than recorded in the State Mineral Resources Register. At first glance, it seemed doubtful because the smaller used reserve means smaller mineral resources tax for the mine. After the detection of this difference, we compared the data of all reserve blocks in the working mines to know how the volumetric weight determination method affects calculation of the bed rating. It became clear that the official method gives greater volumetric weight for oil shale layers and smaller for limestone layers as compared with the exact one. As most of the bed contains oil shale, in all the cases the official method gives greater bed rating as compared to the exact one. The differences are presented in Table 3.

Table 3. The Difference in Energy Rating and Tonnage of Bed Depending on Calculating Method of Volumetric Mass

Reserve blocks of mine fields	Difference in bed energy rating, %	Difference in oil shale tonnage, %
Good blocks with energy rating above 42.21 GJ/m ²	3.80	3.22
Poor blocks with energy rating below 42.21 GJ/m ²	3.59	2.79
All the blocks, average energy rating 42.21 GJ/m ²	3.69	3.03

Consequently, the difference depends on the quality of oil shale bed: the difference in energy rating as well as in oil shale tonnage calculated by different methods is greater for blocks with higher energy rating. This is in accordance with the above-mentioned different trends of differences in oil shale and limestone volumetric weights. The same is illustrated in Fig. 3.

We made calculations avoiding the mentioned methodical mistakes. The calorific value of an oil shale seam was estimated to be 7–10 % higher. The error is higher in higher-calorific blocks. The energy rating of the bed was estimated to be 7–11 % higher. This error is greater in lower-calorific blocks (Fig. 4).

Fig. 3. The difference in bed tonnage calculated by two methods depending on bed calorific value: ● high-quality reserve blocks, ○ low-quality reserve blocks

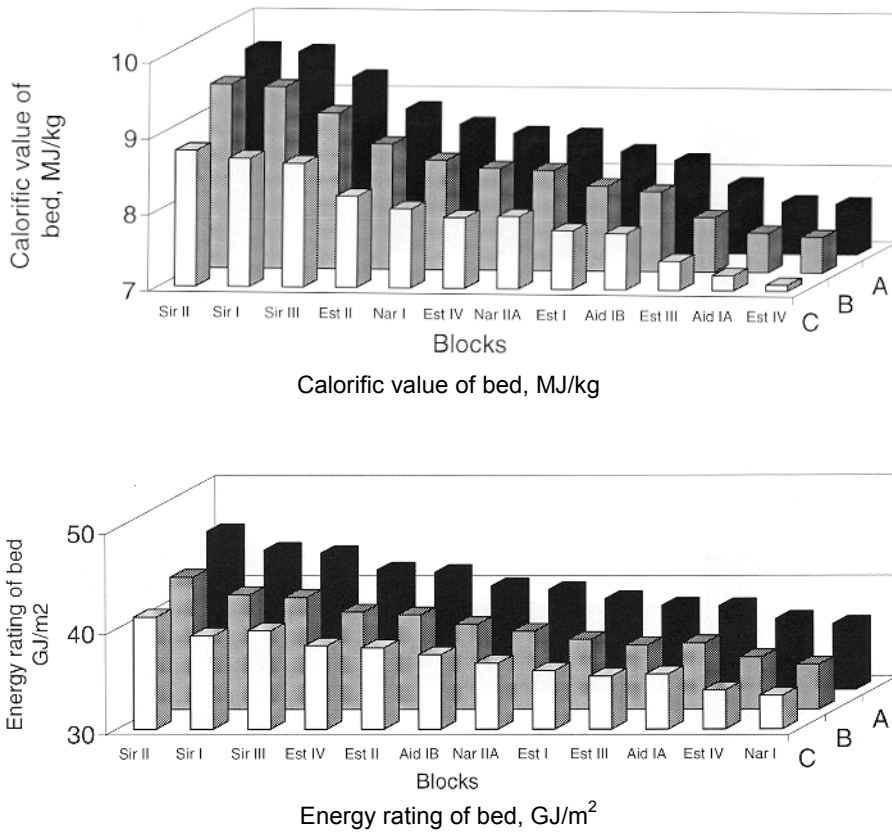
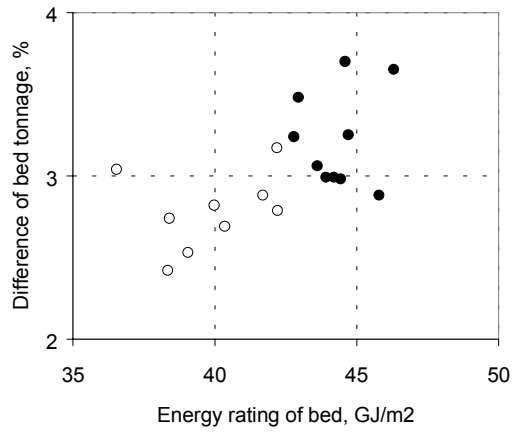


Fig. 4. The impact of the methods on the quality indicators of oil shale bed: A - official method; B - volumetric mass method considering different mineral content of the noncombustible part; C – additional consideration of energy absorption to decompose lime minerals. Mine field index for reserve blocks: Sir – *Sirgala* opencast, Est – *Estonia* mine, Nar – *Narva* opencast, Aid – *Aidu* opencast

Calculation results let us conclude that the official calculation method of oil shale bed quality shows higher heating values and energy ratings than the precise method proposed by us. The difference can reach 10 % depending on the quality of the bed.

In spite of the fact we do not recommend to re-evaluate oil shale reserves as:

- First, as long as selective mining or preparation is used to produce trade oil shale, the weighted heating value and energy rating of the bed are not essential in business relationships.
- Second, the cut-off-grade for proved and probable reserve are relative, not absolute.
- Third, the study of potential energy of Estonian oil shale is going on and could correct the principles.

Acknowledgements

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