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OIL SHALE POTENTIAL IN SERBIA

V. ČOKORILO[(a)*], N. LILIĆ[(a)], J. PURGA[(b)], V. MILISAVLJEVIĆ[(a)]

[(a)] University of Belgrade, Faculty of Mining and Geology, Djušina 7, 11000 Belgrade, Serbia
[(b)] Viru Keemia Grupp, Kohtla-Järve, Estonia

Basic geological exploration in Serbia detected 21 deposits of oil shale with resources of over 4.7 billion tonnes. Oil shale in these deposits is of various qualities and oil content. The biggest deposits of commercial potential are Aleksinac and Vina-Zubetin. Aleksinac deposit has been explored in more detail compared with other deposits, with reserves close to 2.0 billion tonnes with average content of organic matter of approximately 20% and with oil content of approximately 10%. Preliminary analyses and studies showed that oil shale can be mined both by surface and underground mining methods. Initial evaluation of Aleksinac oil shale indicates that basically it can be thermally processed using existing commercial oil shale processing technologies used in Estonia. The company Viru Keemia Grupp AS (VKG) from Estonia established the potential of Aleksinac deposit and showed preliminary interest in mining and processing of oil shale. For this reason VKG established collaboration with Faculty of Mining and Geology of University of Belgrade. Some of the results of this collaboration are presented in this paper.

Introduction

There are numerous basins and deposits of oil shale in Serbia, but they are explored at various levels. Most important basins of oil shale in Serbia are Aleksinac, Vranje, Senonian Tectonic Trench, Valjevo-Mionica, Western Morava, Kruševac, Babušnica, Kosanica, Niš and Levač [2]. Part of Aleksinac Basin and part of Vranje Basin (Goč-Devotin) have been explored in detail, while semi-detailed exploration has been performed at remaining parts of Aleksinac and Vranje (Vlase-Golemo Selo) basins. Other basins of oil shale are explored at basic exploration level, which enables planning of further exploration in order to reliably determine amounts of reserves and quality of oil shale. Serbian oil shale is of sapropel type (Aleksinac, Mionica

* Cornensponding author: e-mail address *cokorilo@rgf.bg.ac.rs*

and Petnica) and sapropel-coaly type (Babušnica, Vranje Basin, Senonian Tectonic Trench, etc.).

Further activities related to geological exploration are of particular importance regarding determination of organic matter and oil content, necessary for planning of future activities in connection to mining, processing, utilization and environmental protection [9].

Overview of oil shale deposits in Serbia

Basic geological exploration detected 21 oil shale basins and deposits of various qualities and potential, but only two deposits are explored in detail. These are Aleksinac deposit and Goč-Devotin deposit. Therefore, most of the oil shale potential in Serbia should be considered in terms of resources instead of reserves. Since Serbian legislation still has not regulated reconciliation of the reporting system, further on, reserves will be presented according to the current Serbian system. Locations of oil shale deposits are presented in Fig. 1, while basic data are given in Table 1.

Aleksinac Basin consists of Aleksinac deposit and Bovan-Prugovac deposit. **Aleksinac Deposit** has been explored in detail over the area of

Fig. 1. Locations of oil shale deposits in Serbia.

1 Aleksinac, 2 Bovan-Purgovac, 3 Goč-Devotin, 4 Vlase-Golemo Selo, 5 Stance, 6 Buštranje, 7 Klenike, 8 Vlaško polje-Rujište, 9 Vina-Zubetin, 10 Podvis-G. Karaula, 11 Manojlica-Okolište, 12 Miranovac-Orlja, 13 Šušeoke-Klašnić, 14 Radobićka strana-Svetla, 15 Pekčanica-Lazac, 16 Parmenac, 17 Odžaci, 18 Raljin, 19 Rača, 20 Paljina, 21 Komarane-Kaludra.

Table 1. **Basic data of oil shale deposits in Serbia**

Basin	Deposit	Thickness of seam, m	Average thickness, m	Average content		Reserves/Resources (10^6 t)		
				Kerogen, vol. %	Oil mass (Fischer assay), %	Shale	Oil	Category
Aleksinac	Aleksinac	54–92	75.5	20.0	10.0	2000	200	A+B+ C_1+C_2
		7–29	20.2	25.0	12.5			
	Bovan-Prugovac	10–33	20.0	12.2	6.0	210	12.6	–
Vranje	Goč-Devotin	10–23	15.0	8.8	4.5	22	1,0	A+B+C_1
		2–6	3.9	5.9	2.1	13.8	0.3	C_1
	Vlase-Golemo Selo	6–13	9.7	5.5	3.4	38.5	1.3	C_1
		3–7	4.4	2.5	1.4	–	–	–
	Stance	–	4.0	5.6	2.6	45	1.2	D_1
		–	6.0	6.2	2.6	–	–	–
	Buštranje	4–13	9.0	8.2	3.4	46	1.6	C_1
		5–9.5	7.0	5.0	1.4	36	0.5	C_1
		4.2–9.0	6.0	5.2	1.4	30	0.4	C_1
	Klenike-Jastina Bara	1.3–13	7.5	5.6	3.4	42	1.4	C_1
		1.5–10	6.0	6.7	3.2	30	1.0	C_1
		9–11	10.0	5.2	1.3	–	–	
	Baraljevac	4–6	5.6	7.3	2.8	8	0.2	C_1
	Drežnica	8–10.7	9.0	8.5	5.1	35	1.8	C_1
		–	7.5	8.4	4.9	30	1.5	C_1
Senonian Trench	Veliko Polje-Rujiš	20–150	72.0	–	0.5	–	–	–
	Vina-Zubetin	20–80	31.0	5.4	2.6	850	22.1	C_2+D_1
	Podvis-Gornji Kar.	2–5	4.0	–	7.5	10	0.1	C_2
	Miran.-Orlja	5–33	12.0	4.5	2.2	70	1.5	C_2
	Man.-Okoliš	10–35	25.0	5.1	2.4	100	2.4	C_2
Valjevo-Mionica	Šuše-Klasnić	5–15	9.0	7.2	3.2	30	1.0	C_2
	Rad. Str. Svet.	4–15	9.0	8.4	3.9	80	3.1	C_2
Western Morava	Pekčanica-Lazac	–	4.4	5.0	1.3	38	0.3	D_1
		–	1.7	3.0	0.9		–	
	Paramenac-Riđage	–	2.3	6.0	1.3	18	0.2	D_1
		–	2.7	3.3	0.8		–	
Kruševac	Odžaci	3–11	7.0	6.8	1.7	20	0.3	D_1
Babušnica	Raljin	24–40	30.0	7.4	3.7	300	9.6	C_2
		9–15	12.0	5.2	2.6			
Kosanica	Rača	4–6	4.4	11.5	5.2	20	1.0	C_2
		1–2	1.4	3.6	1.6			
Niš	Paljina	–	15.0	–	3.2	500	16.5	D_2
		–	6.0	–	3.3			
Levač	Komarane-Kaludra	–	7.0	3.4	0.6	190	1.9	D_1
		–	2.0	3.0	1.4			
						4812.3	**283.8**	

20 km^2 [6]. Calculated reserves of A, B and C_1 category are categorized as non-balanced since no processing technology is defined. Average thickness of roof oil shale is 75.5 m, with average oil yield of 10% by mass, while the thickness of floor oil shale is 26 m, with oil yield of 12.5% by mass. Oil shale dips toward west by 30–50^o, down to the depth of 700 m in the central part of the deposit. Total reserves of oil shale in Aleksinac deposit are calculated 2.0 billion tonnes (Table 1).

Bovan-Prugovac deposit is characterized by floor package of oil shale with average thickness of 20 m with oil yield of 6% by mass. Estimated reserve of C_1 category is 210 million tonnes [8].

Vranje basin consists of several oil shale deposits, where most important one is Goč-Devotin. The thickness of upper oil shale package is 15 m, with average content of organic substance of 8.8% by vol. and average oil yield of 4.5% by mass. Overall reserves of categories A, B and C_1 in upper package are estimated at 22 million tonnes [8]. Average thickness of lower package of oil shale is 3.9 m, with average content of organic substance of 5.9% by vol. and average oil yield of 2.1% by mass. Oil shale reserves in lower package are of C_1 category, and these are estimated at 14 million tonnes [8].

Senonian Tectonic Trench: Significant occurrences of oil shale are located at the proximity of the River Danube, at the north, and due south-east toward the border with Bulgaria. Lamosite type of oil shale is found in most of the deposit.

The thickness of oil shale seam in Vina-Zubetin deposit is 30 m, with organic substance content of 5.4% by vol. and oil yield of 2.6% by mass. Reserves of C_2 category are estimated at 150 million tonnes, while D_1 category at 700 million tonnes. Oil shale from the immediate roof of coal seam at Podvis and Tresibaba deposits is characterized by oil yield from 4 to 11% by mass, which makes these deposits very interesting. However, seams are thin, mainly below 1 m [1].

Aleksinac oil shale deposit

Aleksinac oil shale deposit is located in proximity of Aleksinac town, in the area north-east from the town. Most of the oil shale deposit is being explored simultaneously with exploration of coal deposit, although part of exploration activities was directed only to oil shale. Oil shale is located in the roof and in the floor of the coal seam, while the deposit encompasses the area of Aleksinac coal mines, the area between South Morava and Moravica rivers, and spreads some 10 km in length from Aleksinac town in direction south – south-west. Aleksinac oil shale deposit, as already mentioned, is divided into three parts: Dubrava, Morava and Logorište.

Depth of oil shale series varies from out-crops along the whole length of the deposit, down to the depth of 700 m. Dipping angle varies from 0^o (horizontal) to 90^o (vertical). As an illustration of this varying dipping and folding structure, one of the profiles is shown in Fig. 2. Raw oil content in oil shale is 6–20% or approximately 10% on average. Floor oil shale seam

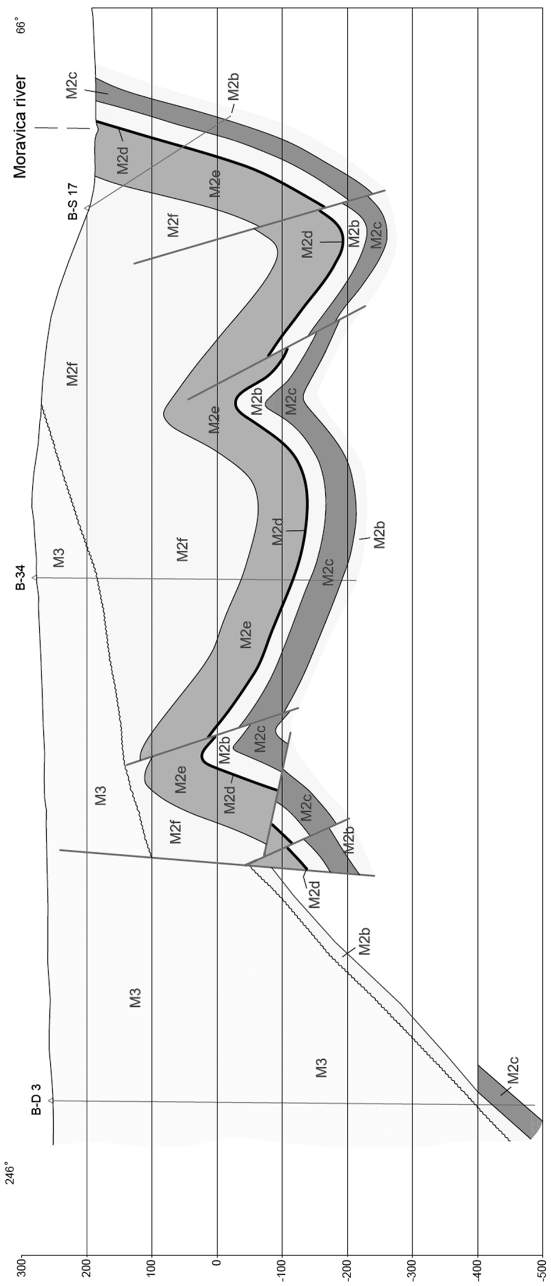

Fig. 2. Profile of Aleksinac oil shale deposit, direction 246° – 66°.

contains thin coal seams, with thicknesses from 0.2 to 0.8 m. General spreading direction of oil shale seams is north-northwest – south-southeast. Dipping angle increases with depth. The deposit is divided into numerous blocks by faults, both transversal and diagonal, with significant horizontal and vertical movement. Bituminous marlstone, marlstone with intrusion of sandy clay and rarely marly sandstone are located above oil shale seam.

Aleksinac oil shale quality and reserves

Oil shale quality was determined by physical and chemical analyses. Sampling of oil shale for examination in laboratory was performed in roadways and by core sampling during exploration drilling from the surface.

These examinations were comprehensive regarding number and type of samples, and the purpose was to determine quality of oil shale, one of major parameter for economic evaluation of the deposit. Examination of oil shale quality regarding oil content was performed by analyses according to Fischer method at oil refinery in Pančevo [7], Serbia, laboratories at Faculty of Mathematics and Natural Sciences in Belgrade and Institute for Chemistry, Technology and Metallurgy (IHTM) in Belgrade.

Examination results showed alternating richer and poorer seam parts. Generally, roof oil shale can be divided by thickness at upper and lower part. The upper part mainly without dirt intrusions is of better quality (contains mostly 12 to 14% of oil). The lower part is usually inwrought with dirt intrusions, with content of oil ranging between 5 and 7%.

Actual thickness of roof oil shale at the north part of Morava field is between 45 and 70 m (57 m on average), while thickness considerably varies in the south part (between 34 and 77 m), although average thickness is similar (56 m). Waste intrusions are at the lowest level due north (3%), increasing due south up to 7%. Average oil content is almost the same, 9.8% at north and 9.7% at south. Thickness of floor oil shale is less consistent (3 to 33 m), as well as regarding the number of oil shale seams.

Results of complete chemical analyses of three composite samples of oil shale are given in Table 2. Samples were taken from each of three fields of the deposit. Analyses were performed at Mining Institute in Belgrade.

Ash from Logorište location oil shale is categorized as moderately smelting ash, with diffusion temperature of 1300 °C. Ashes from Morava and Dubrava are categorized as more easily smelting ash with diffusion temperatures of 1170 °C and 1200 °C.

Oil shale from Aleksinac deposit is a fuel with low moisture as delivered (1.2–6.4%), very high ash content (63.66–64.80%), high carbonate content (8.33–13.33%), moderate content of volatiles (28.29–34.27%) and combustibles (29.90–34.47%) and relatively low calorific value (5.57–9.35 MJ/kg). Also, contents of carbon (16.22–20.47%) and hydrogen (1.90–2.56%) are low, while the content of N+O varies in a wide range (5.72–13.48%). C/H ratio is 8 to 8.5. Content of total sulphur varies significantly from 1.41 to 4.87%. According to performed chemical analyses, oil shale mineral part is mainly alumina-silicate, with moderate content of iron oxide, while calcium oxide content varies in a broad range, from 8.04 to 24.03%, and with significant content of alkali metals. Ash from Aleksinac oil shale is categorized as easily smelting to smelting ash.

According to the level of performed geological exploration, roof oil shale reserves are classified into A, B, C1 and C2 categories, while floor oil shale are classified into B, C1 and C2 categories. Overall reserves at Aleksinac deposit are given in Table 3 [3].

Aleksinac oil shale deposit contains significant reserves. These reserves have average oil content of 9.78%. Once the balancing of reserves is performed and mining method is selected, evaluation of oil shale mineable reserves, as well as content of raw oil could be estimated more precisely. At this moment production losses could be estimated at level of 10 to 30%. Oil

Table 2. **Chemical and technical analyses of oil shale**

	Sample I Logorište	Sample II Morava	Sample III Dubrava
Moisture, as delivered, %	6.40	5.30	1.20
Ash, %	63.66	64.80	64.33
Sulphur, total, %	2.30	4.50	0.41
Sulphur in ash, %	1.11	1.15	0.23
Sulphur combustible, %	1.19	3.35	0.18
Coke, %	65.31	65.55	64.53
C-fix, %	1.65	0.75	0.20
Volatiles, %	28.29	29.15	34.27
Combustibles, %	29.94	29.90	34.47
Upper calorific value, kJ/kg	9,550	6,220	7,410
Lower calorific value, kJ/kg	8,810	5,660	6,870
CO_2, %	8.33	11.78	13.33
Carbon, %	20.47	16.22	18.55
Hydrogen, %	2.56	1.90	2.26
N + O, %	5.72	8.43	13.48
Raw oil content, acc. Fischer			
Raw oil, %	12.70	7.60	12.20
Ash composition:			
SiO_2, %	59.87	47.73	42.57
Fe_2O_3, %	14.21	11.02	9.50
Al_2O_3, %	7.27	5.69	11.58
CaO, %	8.04	24.03	18.30
MgO, %	2.30	2.79	9.26
SO_3, %	4.56	5.45	0.88
P_2O_5, %	0.31	0.52	0.46
TiO_2, %	0.66	0.62	1.06
Na_2O, %	1.15	0.80	3.00
K_2O, %	0.50	0.50	2.20
Reaction	very acid	poorly acid	poorly acid
Ash smelting:			
Start of sintering, °C	900	890	920
Temperature of softening, °C	1,140	1,100	1,130
Temperature of semi-sphere, °C	1,260	1,160	1,190
Temperature of diffusing, °C	1,300	1,170	1,200

Table 3. **Overall reserves at Aleksinac deposit**

Category	Reserves, t		
	Non-disturbed	Disturbed	Geological
Dubrava field			
A	10 486 570	54 912 260	65 398 830
B	119 533 010	16 541 880	163 074 890
A+B	130 019 580	71 454 140	201 473 720
C1	134 051 380	43 354 250	177 405 630
A+B+C1	264 070 960	114 808 390	378 879 350
C2	30 000 000	-	30 000 000
A+B+C1+C2	294 070 960	-	408 879 350
Morava and Logorište fields			
A	11 613 190	-	11 613 190
B	100 929 880	-	100 929 880
A+B	112 543 070	-	112 543 070
C1	179 613 370	125 563 100	305 176 470
A+B+C1	292 156 440	125 563 100	417 719 540
C2	665 808 190	450 415 730	1 116 223 930
A+B+C1+C2	957 964 630	578 978 830	1 533 943 470
Total for deposit			
A	22 099 760	54 912 260	77 120 020
B	220 462 890	16 514 880	237 004 770
A+B	242 562 650	71 454 140	314 016 790
C1	313 664 750	168 917 350	482 582 100
A+B+C1	556 227 400	240 321 410	796 598 890
C2	695 808 190	450 415 730	1 146 223 920
A+B+C1+C2	**1 252 035 590**	**690 787 220**	**1 942 822 810**

shale occurs together with brown coal, in clay-marly-sandy Tertiary series. Most of oil shale is at the middle of production series in the roof of the main coal seam, thus referenced as roof oil shale.

Floor oil shale, below the main coal seam, occurs in the form of seams or groups of the seams, with intrusions of clay and sand sediments.

Average thickness of roof oil shale is:

- Dubrava field: 72.5 m (68.5 without waste);
- Morava and Logorište fields: 56.6 m (53.5 without waste).

Characterization of Aleksinac shale oil

Two samples of Aleksinac shale oil produced by different pyrolysis processes were characterized, using TBP distillation besides other methods. The sample obtained by the Galoter process gave 90% by volume of distillate below 550 °C, while the other produced by the Kiviter process gave only 56%. Sample components included *n*-paraffins, 1-*n*-olefins and alkenyl-benzenes.

Shale oil sample K was produced in Estonia by the Kiviter retorting process in 1977. About 300 kg of sample K spent six years in closed barrels before being analyzed. Shale oil sample G was also produced in Estonia by a pilot plant solid heat carrier retorting process which simulated the Galoter process. About 24 kg of oil was kept for six months in a closed metal vessel prior to being analyzed. Physicochemical properties of shale oil samples are given in Table 4 [4]. Samples K and G were homogenized at 50 °C, dried over $MgSO_4$ or Na_2SO_4 and filtered to remove mechanical parts.

TBP distillation was accomplished in an apparatus which consisted of a 6 dm^3 distillation flask, an insulated fractionating column 400 mm long × 25 mm i.e. filled with glass spirals 6 × 3 mm and a distillation head with automatic regulation of the reflux ratio. The theoretical plate number was 4-5 and the reflux ratio was 5. Vacuum distillation was performed at 40 to 90 Pa, and a usual chart was used to calculate boiling points at atmospheric pressure. The atmospheric TBP distillation ended at 190 °C and the vacuum TBP distillation at 456 °C. Finally, distillation was continued from a Claisen flask up to 560 °C. Thus, fraction 456-560 °C was not a TBP fraction. Results of TBP distillation of samples K and G are given in Table 5 showing a very pronounced difference between the two ones. Sample G gave as much as a 89.5 vol.% of distillate below 550 °C, while only 55.5 vol.% was obtained from sample K.

It is important to mention that this is achieved by RECYCLING of heavy oil back to the process in the Galoter system. Recycled heavy oil is subject for secondary cracking resulting in formation of lighter compounds. It is the same in commercial process as well. In the Kiviter process there is no need for recycling, because heavy oil from distillation is used in coking process.

Table 4. **Physicochemical properties of shale oil samples**

Characteristics	Sample	
	K	G
Density d_4^{20}	0.9145	0.8832
Pour point, °C	30	11
Flash point, °C P.M.	94	28
Viscosity, 50 °C mm^2/s	16.3	3.8
Moisture, % wt	0.10	0.15
Conradson C.T., % wt	2.5	2.2
Ash, % wt	0.43	0.01*
Molar mass	348	242
Iodine number, g/100 g	32.9	61.4
Asphaltenes, % wt	1.4	0.9
Elementary analysis, % wt		
C	83.71	84.55
H	11.69	11.69
N	1.52	1.13
S	1.11	0.49
O	2.13	2.27
V, mg/kg	0.5	1
Ni, mg/kg	3.0	5
As, mg/kg	33.9	< 5

* Pilot facility data - From industrial Galoter plant raw oil has significantly higher ash content (because of solid heat carrier ash).

Table 5. **Results of TBP distillation of samples, % by volume**

Temperature	% of distillate						
	Sample	0 °C	100 °C	200 °C	300 °C	400 °C	500 °C
+0 °C	K		1.2	11.0	28.7	41.0	51.6
	G			20.5	35.5	61.0	84.5
+10 °C	K		1.4	12.9	29.8	42.0	52.3
	G			23.0	38.0	65.0	86.0
+20 °C	K		1.7	14.9	31.2	43.2	53.0
	G		4.5	26.5	39.5	67.0	87.0
+30 °C	K		2.0	16.9	32.5	44.5	54.0
	G		6.0	29.5	41.0	70.0	88.0
+40 °C	K		2.5	18.9	33.7	45.7	54.6
	G		7.5	31.5	45.0	75.0	89.5
+50 °C	K		3.1	20.5	35.0	46.8	**55.5**
	G		9.0	31.5	45.0	75.0	**89.5**
+60 °C	K		4.5	22.2	36.4	47.4	
	G		11.0	32.0	48.0	78.0	90.0
+70 °C	K		5.7	23.5	37.4	48.4	
	G		13.0	32.5	51.5	80.0	
+80 °C	K	1.0	7.4	25.3	38.7	49.4	
	G		16.5	33.0	54.0	81.5	
+90 °C	K	1.1	9.0	26.8	39.6	50.3	
	G		19.0	33.5	57.0	83.0	

As result, coke and light coke distillate form, both are good commercial end-products. So from commercial standpoint the result depends on the whole value-chain, and probably combination of the two processes will be the best solution.

There is a significant difference in the quantitative chemical composition of samples K and G, which is mostly the result of different oil shale pyrolysis conditions. Sample G represents a shale oil of better quality, as it has lower N, S, and As concentrations and produces 21.8% by vol. of distillate below 205 °C and 48.0% by vol. of distillate below 360 °C compared to 12.0% and 36.4% by vol. of sample K, respectively. This can be partially explained with Galoter technology as mentioned in the previous paragraph. Both samples contained *n*-paraffins, 1-*n*-olefins and alkyl benzenes which could be valuable feeds and intermediates for the chemical industry. The identification of the most abundant polyaromatic hydrocarbons, hetero- and unsaturated compounds was important to define hydrotreating parameters such as: catalyst type, space velocity, temperature and pressure of hydroprocessing, as well as to calculate theoretical hydrogen consumption.

Conclusions

Geological reserves of oil shale in Serbia are large. However, except for Aleksinac and Goč-Devotin deposits, most of the oil shale deposits have been explored at the level of basic geological exploration, what means that more detailed explorations are necessary. This activity is of particular importance regarding determination of organic matter and oil content, necessary for planning of future activities related to mining, processing, utilization and environmental protection.

Aleksinac oil shale deposit is explored by surface drilling and underground workings, since underground coal mine operated within the same deposit for more than hundred years, between 1883 and 1990. Having in mind that last examinations of oil shale were performed during the 1980-ies, it is necessary to verify the results obtained at that time. In any case, Aleksinac deposit, with almost two billion tonnes of oil shale reserves represents high potential energy resource for Serbia and an opportunity for potential investors having in mind that the Government of Serbia is ready to support this endeavor.

Certain activities related to examination of oil shale exploitation possibilities at Aleksinac deposit have been undertaken by Estonian company Viru Keemia Grupp AS – world second largest shale oil producer today. VKG has extensive experience in the field of oil shale processing and oil upgrading. According VKG's evaluation Serbia's Aleksinac deposit possesses several advantages for development compared with other potential oil shale deposits of the world. It is expected that these activities continue in the future.

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