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SOLID HEAT CARRIER OIL SHALE RETORTING TECHNOLOGY WITH INTEGRATED CFB TECHNOLOGY

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Abstract. *The solid heat carrier (SHC) retorting method, so-called Galoter process, was developed for oil shale processing at the end of the 1940s. Since then the method has undergone several improvements. Nowadays there are different modifications of Galoter process in use – Petroter, Enefit-140 and TSK-500 technologies. The major differences between these technologies are in sizing (throughput), technical solutions and layouts. Recently a shale oil plant based on a new technology, Enefit-280, was commissioned. Enefit-280 is a technology successor of Enefit-140 where the heating of solid heat carrier is accomplished using the circulating fluidized bed (CFB) combustion technology as opposed to the conventional heat carrier combustion technology in Enefit-140. The CFB technology in Enefit-280 was integrated into the process to improve the performance of SHC heating process and reduce the emissions. Operational experience has demonstrated that the modified technology of SHC oil shale retorting has a potential to play a key role in shale oil production with reduced environmental impact.*

Keywords: *Enefit-280, Petroter, shale oil, circulating fluidized bed, solid heat carrier.*

1. Introduction

The composition of oil shale organic matter (kerogen) differs from that of the organic part of the other solid fuels. Oil shale kerogen contains more hydrogen as compared to coal and therefore may be well subjected to thermal conversion into oil and gas, whose yields depend on the hydrogen content of the convertible solid fuel. In that sense the properties of oil shale are very favourable as up to 70% of kerogen is convertible into oil [1].

In Estonia, the thermal processing of oil shale has a relatively long history. Both the low-temperature processing, i.e. semi-coking (retorting) by

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heating oil shale up to 500 °C, and high-temperature processing by heating oil shale up to 1000–1200 °C with the purpose of producing city gas have been commercially used. Today only the low-temperature processing is in use in Estonian oil shale industry. For retorting fine shale (≤ 25 mm) processes with SHC have been developed on a commercial scale. SHC process is based on the effect of high rates of heat transfer between fine-grained heat carrier and fine-grained oil shale due to their direct contact in a rotary drum reactor. One of the processes, Enefit-140, has two thermal processing units with a capacity of 3,000 tonnes of oil shale per day. The same SHC method with a similar capacity is utilized in Petroter I, Petroter II and Petroter III units. The Enefit-280 technology has twice the capacity of Enefit-140 or Petroter technology with improvement related to SHC preparation. The comparison of these technologies is given in Table 1.

Table 1. Characteristics of different SHC based shale oil production technologies [4]

Technology / Parameter	Enefit-140	Petroter	Enefit-280
Consumption			
Raw oil shale, t/h	130	125	280
Oil shale particle size, mm	< 25	< 25	< 6
Electrical power, kWh/t_{OS}	36	39	36
Water, t/$t_{OS,dry}$	0.51 to 0.53	0.16 to 0.23	0.5 to 0.6
Production			
Shale oil, t/h	15	14.3	37
Shale oil, t/t_{OS}	0.115	0.114	0.132
Gas, Nm^3/t_{OS}	33.8	36	34.6
Electrical power, kWh/t_{OS}	–	–	100

OS – oil shale, dry

In retorting processes the primary by-product, semi-coke, is formed. As some portion of carbonaceous residues (up to 9.1 wt% [2]) remains in a solid semi-coke matrix, it can further be used as a heat source/carrier in SHC process. The main and substantial difference between these "older" or "conventional" processes and the new Enefit-280 lies in the technology applied for semi-coke combustion. In the case of the conventional SHC process the so-called lift pipe combustor (in literature known as an aero-fountain combustor (AFC) [3]) has been implemented for burning the semi-coke. In AFC the combustion process occurs under reducing (oxygen deficient) conditions at temperatures between 760 and 810 °C. In Enefit-280, however, the circulating fluidized bed (CFB) technology is integrated into the retorting process for SHC preparation. CFB allows achieving a more complete burnout of the carbonaceous residue already at temperatures below 800 °C, thus enabling the reduction of the environmental impact of shale oil production.

2. Conventional SHC technologies

In Estonia, the shale oil production is mostly based on SHC processing technologies such as Petroter, Enefit-140 (formerly known as UTT-3000) and Enefit-280. The main characteristics of said technologies are given in Table 1 [4].

The process flow scheme of Enefit-140 technology is depicted in Figure 1. The design capacity of Enefit-140 is 3000 tonnes of raw oil shale per day with an oil shale particle size below 25 mm. Currently two Enefit-140 units are in operation at full capacity at Eesti Energia AS. The process is characterised by an oil yield of 85–90% on the Fischer assay basis, which is a standard method for characterizing shale oil yields [5]. The elemental composition of shale oils from Enefit-140 and Enefit-280 is given in Table 2.

The efficiency of Enefit-140 calculated based on the chemical energy of oil shale and useful products produced is about 80% [4]. The technological set-up consists of the following main processes: (1) raw oil shale drying and preheating in a lift-type dryer by using heat from flue gas before venting it into the atmosphere (after purification); (2) thermal decomposition (pyrolysis) of oil shale kerogen in a rotary kiln type retort mixed with hot ash

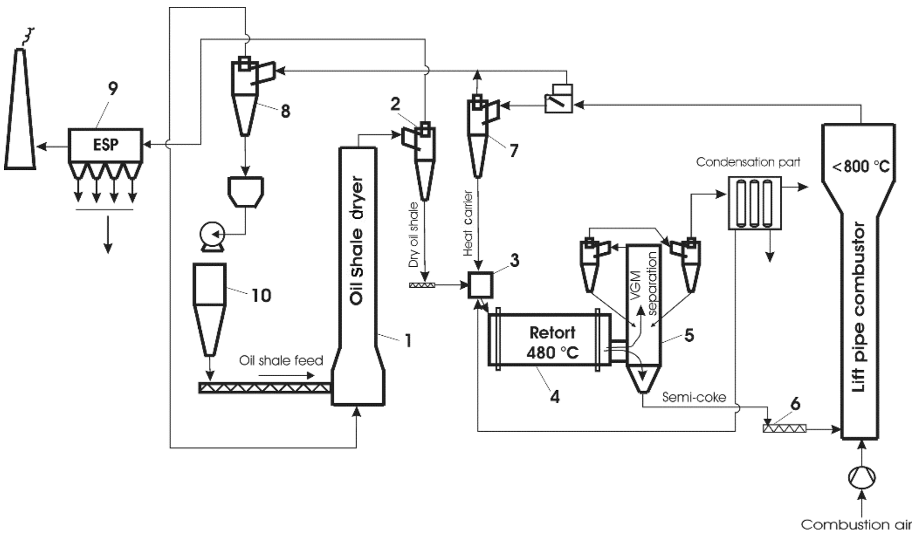

Fig. 1. Layout of Enefit-140 technological process (adopted from [4, 6]): 1 – lift pipe dryer; 2 – dry oil shale cyclone; 3 – dry oil shale and heat carrier mixer; 4 – rotary-type reactor; 5 – separation chamber; 6 – semi-coke feed screw conveyer; 7 – heat carrier cyclone; 8 – fly ash cyclone; 9 – flue gas purifier; 10 – electrostatic precipitator.

Table 2. Elemental composition of shale oils [4]

Element, wt% \ Technology	Enefit-140	Enefit-280
C	83	79.4
H	9.5	12.1
N	0.3	0.3
O	6.5	7.7
S	0.7	0.5
Cl	0.018	0.02
C/H ratio	8.73	6.55
C/O ratio	12.8	10.3

(SHC); (3) combustion of carbonaceous residue (semi-coke) in a lift pipe combustor (AFC) releasing the greater part of heat during combustion to be used as the energy source in pyrolysis at temperatures in the range of 480–520 °C, and liberating the rest of the heat to be then used for raw oil shale drying; (4) purification of the vapour-gas mixture generated in retorting from dust in gravitational and cyclone separators and direction into the condensation section to obtain different fractions (heavy oil, middle oil, gas turbine fuel oil and gasoline) during multistage condensation.

Recently Petroter I, II, III units were commissioned at VKG AS. The Petroter technology design is based on the SHC method for processing 140 tonnes of fine oil shale (< 25 mm) per hour. All units are currently in operation at full capacity. In 2017, Petroter III unit demonstrated high operational availability as it was in operation for 331 days with an oil production of 149,300 tonnes [7]. The scheme of Petroter technology is depicted in Figure 2. Being based on the same SHC method as the Enefit-140 described above, the main process steps of both technologies are basically the same. The major difference with Petroter is the more complete use of released heat and additional treatment of flue gas before venting it to the atmosphere. The heat recovery boiler is integrated into the process downstream of the fly ash cyclone from the flue gas side. In this boiler the afterburning of carbon monoxide and hydrogen sulphide is arranged. SO_2 formed during H_2S oxidation is then partially captured in a semi-dry desulphurization system (NID). Heat recovered in the heat utilization boiler and semi-coking gas (the composition is given in Table 3) are used for electricity production in the neighbouring conventional power plant. Additionally, heat is recovered from hot ash separated in the fly ash cyclone and is used for district heating. All this increases the overall efficiency of shale oil production, reducing at the same time environmental impact.

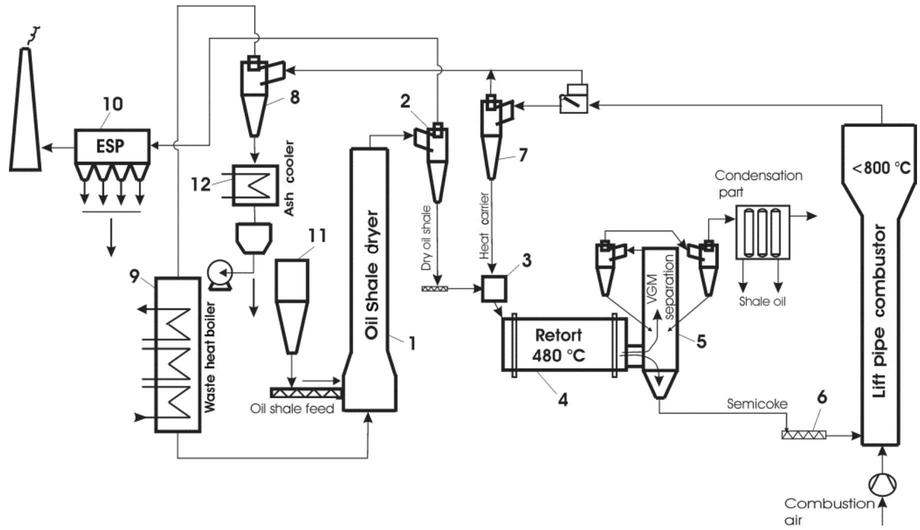

Fig. 2. Layout of Petroter technology (adopted from [4]): 1 – lift pipe dryer; 2 – dry oil shale cyclone; 3 – dry oil shale and heat carrier mixer; 4 – rotary-type reactor; 5 – separation chamber; 6 – semi-coke feed screw conveyer; 7 – heat carrier cyclone; 8 – fly ash cyclone; 9 – flue gas heat utilization boiler; 10 – electrostatic precipitator; 11 – raw oil shale bin; 12 – ash heat exchanger.

Table 3. Semi-coking (pyrolysis) gas composition [4, personal communication]

Technology / Component, vol%	Enefit-140	Petroter	Enefit-280
H_2	13.3	14.4	10.0
CO	9.5	9.6	8.4
H_2S	2.5	2.0	1.1
CO_2	9.5	8.9	11.9
N_2	1.1	11.2	10.1
O_2	0.2	0.2	0.1
Saturated hydrocarbons	33.5	11.9	27.5
Unsaturated hydrocarbons	29.4	20.1	24.9
Unidentified	1.0	21.7	6.0

3. CFB technology for semi-coke combustion

One of the significant drawbacks of the existing conventional shale oil production processes is the formation of semi-coke containing organic compounds and toxic heavy metals. Semi-coke from the operating vertical retorts (gaseous heat carrier) is mainly disposed of in the landfill, which consequently creates the risk of environmental contamination and is also accompanied by a significant energy loss. This problem is partly solved in SHC process where the lift pipe combustor has been implemented for semi-coke combustion, allowing partial recovery of some heat which is used then as the energy source in the pyrolysis process. Owing to the need to achieve high energy efficiency, great attention has been paid to energy recovery from semi-coke. Therefore it is agreed that the most effective way of semi-coke utilization is its combustion by applying the fluidized bed (FB)/CFB technology [8, 9]. Essentially, semi-coke combustion in FB/CFB is not a new idea as the first attempts to combust semi-coke under FB conditions were made already in the 1950s [8]. Due to its low organic content, co-combustion of semi-coke with oil shale or the other by-products generated in the retorting process or some other fuels was considered in different studies [10–12]. Laboratory scale studies of semi-coke combustion under FB/CFB conditions demonstrated a high potential of this technology in respect of both environmental and thermodynamic aspects.

4. Enefit-280 technology

The layout of Enefit-280 retorting technology implemented at Eesti Energia is depicted in Figure 3 [13]. The major distinguishing feature of Enefit-280 compared to conventional SHC [3] is related to the combustion of semi-coke in an adiabatic CFB combustor. In addition, the new Enefit-280 technology is scaled-up as it is designed to raw oil shale feed rate of about 280 tonnes per hour. This is about twice the rate in the existing Petroter and Enefit-140 units (Table 1) [13]. Nevertheless, changes in combustion technologies have a relatively low effect on shale oil or semi-coke (pyrolysis) gas composition (Table 3).

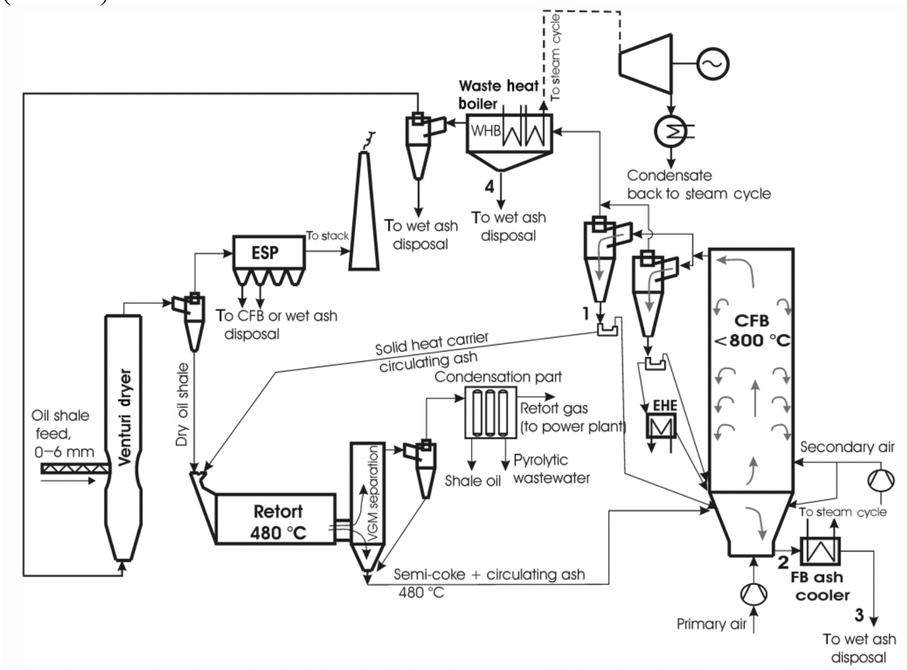

Fig. 3. Scheme of Enefit-280 technology.

As a first step, after leaving the retort at temperatures around 480 °C the mixture of semi-coke and circulating ash is separated from the vapour-gas flow followed by feeding them into the CFB combustor having adiabatic walls. Most of the heat released during combustion of the low calorific semi-coke is transferred to the circulating ash which serves as SHC in this process. The rest of the heat generated in the combustion process is distributed as follows. Part of it is transferred to steam by the circulating CFB ash through the external heat exchanger (EHE). The physical heat stored in the CFB bottom ash is recovered in fluidized bed coolers and is used for steam production. The combustor has a staggered combustion air supply at three levels without its preheating as is generally implemented in conventional CFB boilers.

Hot flue gases leaving CFB pass firstly through a waste heat boiler (WHB) transferring heat to the water-steam cycle, followed by the removal of particulate matter (ash particles) from the gas flow. Further, the enthalpy of the flue gas stream is used for drying the raw oil shale in a Venturi type dryer. Dried oil shale particles are separated from flue gas in the cyclone and fed into the retorting reactor (retort).

Gas separated from dried oil shale in the cyclone is passed through the electrostatic precipitator (ESP) for removing fine oil shale and ash particles still retained in the gas flow followed by venting them into the atmosphere at temperatures around 155 °C. Particulate matter (ash particles) captured in ESP is directed back into CFB and/or disposed of in the landfill. The chemical and mineralogical compositions of selected ash samples (sampling locations shown in Figure 3) are given in Table 4 and Table 5, respectively.

Table 4. Chemical composition of selected ash samples obtained by XRF analysis, %

Sample \ Compound/element		SiO_2	TiO_2	Al_2O_3	Fe_2O_3	MnO	MgO	CaO	Na_2O	K_2O	P_2O_5	Cl
Location	No.											
1	1	11.80	0.170	2.91	3.01	0.06	5.08	40.93	0.09	1.13	0.13	0.064
	2	11.40	0.172	2.82	3.16	0.08	9.09	38.60	0.15	0.92	0.13	0.072
	3	10.32	0.150	2.55	3.19	0.10	11.42	37.40	0.13	0.84	0.13	0.072
	Average	11.17	0.16	2.76	3.12	0.08	8.53	38.98	0.12	0.96	0.13	0.07
2	1	6.27	0.090	1.53	1.84	0.06	4.19	46.35	0.11	0.41	0.09	0.022
	2	6.26	0.090	1.48	2.29	0.09	9.42	40.85	0.09	0.40	0.09	0.018
	3	5.71	0.079	1.32	2.34	0.09	9.53	40.58	0.12	0.35	0.07	0.014
	Average	6.08	0.09	1.44	2.16	0.08	7.71	42.59	0.11	0.39	0.08	0.02
3	1	5.87	0.084	1.41	2.06	0.05	3.82	46.74	0.09	0.38	0.09	0.031
	2	5.93	0.089	1.43	1.88	0.06	4.50	46.08	0.07	0.44	0.09	0.029
	3	5.92	0.086	1.40	2.12	0.08	8.21	41.95	0.05	0.46	0.09	0.017
	Average	5.91	0.09	1.41	2.02	0.06	5.51	44.92	0.07	0.43	0.09	0.03
4	1	29.09	0.424	7.26	4.11	0.04	3.91	29.66	0.09	3.46	0.13	0.015
	2	31.20	0.450	7.64	4.13	0.06	5.70	27.05	0.07	3.71	0.14	0.011
	3	30.20	0.438	7.43	4.05	0.06	6.75	27.29	0.11	3.54	0.14	0.009
	Average	30.16	0.44	7.44	4.10	0.05	5.45	28.00	0.09	3.57	0.14	0.01

Table 5. Mineralogical composition of selected ash samples obtained by XRD analysis, %

Sample \ Mineral		Calcite	Anhydrite	Periclase	Dolomite	Orthoclase
Location	No.	$CaCO_3$	$CaSO_4$	MgO	$CaMg(CO_3)_2$	$KAlSi_3O_8$
1	1	46.6	8.5	4.2	6.9	5.7
	2	37.2	9.1	6.6	13.1	5.6
	3	37.4	10.0	8.3	15.0	5.6
	Average	40.40	9.20	6.37	11.67	5.63
2	1	66.3	3.4	1.4	13.6	3.4
	2	44.3	3.8	3.4	32.7	3.4
	3	43.2	2.7	2.8	29.9	3.3
	Average	51.27	3.30	2.53	25.40	3.37
3	1	65.2	4.6	1.6	9.5	2.5
	2	64.5	4.4	1.6	13.3	2.7
	3	49.8	3.8	2.5	28.1	2.9
	Average	59.83	4.27	1.90	16.97	2.70
4	1	28.1	7.0	2.5	4.9	14.3
	2	21.0	6.4	3.9	6.9	14.9
	3	18.9	6.8	4.7	6.9	15.8
	Average	22.67	6.73	3.70	6.23	15.00

5. Semi-coke utilization through its combustion

At present time it could be quite unambiguously stated that implementation of CFB process for oil shale combustion in power generation has resolved numerous technical and environmental issues associated with high temperature pulverized combustion (PC), such as intensive fouling of heat transfer surfaces, high temperature corrosion, SO_2 and NOx emissions, reduced extent of dissociation of carbonate minerals, etc. [14]. The decreased combustion temperature in the combustor chamber as well as the significantly increased residence time of Ca-rich ash particles in the CFB system have resulted in a substantial decrease of SO_2 emissions from 1500–2000 mg/Nm^3 to practically down to zero level at all boiler loads. This has also eliminated the need to install a desulphurization unit in order to meet the requirements of SOx regulations [15]. NOx emissions, due to the relatively low nitrogen content in oil shale and low combustion temperature, were naturally reduced twice and hence complied with the limits on NOx emissions levels from power industry set by current regulations. Low combustion temperature and increased fuel particles size have caused the decrease of the extent of carbonate minerals decomposition up to 70%, resulting in lower CO_2 emissions and providing higher heat gain per 1 kg of oil shale.

Integration of CFB combustion of semi-coke into the SHC process-based shale oil plant has reduced the environmental impact of oil shale production as it has allowed an almost complete burnout of carbonaceous residues in semi-coke. The maximum unburnt carbon content of 0.6% has been measured in WHB ash. Obviously there is some unburnt carbon in bottom ash, however, due to favourable conditions in bottom ash coolers, such as high temperature and long residence time, a complete carbon burnout is achieved. Semi-coke combustion in CFB occurs under adiabatic conditions without addition of some other higher calorific fuel.

At the same time, as regards shale oil plant, CFB should not only provide efficient energy recovery from semi-coke and lower environmental impact, but also produce SHC (circulating ash) that has properties which comply with certain requirements in order to facilitate the pyrolysis process in the retort. The parameters that affect the yield and quality of oil obtained in the retort process are, for example, particle size distribution (PSD) of oil shale, chemical composition and temperature of SHC (circulating ash), and char content [16–18].

In Enefit-280 technology the particle size of oil shale fed into the process was limited due to the implementation of CFB to a maximum value of 6 mm as compared to the 25 mm used in the conventional SHC process. Operational experience has shown that the implemented CFB system is quite sensitive to solids particle size which consequently should be kept in the specified range.

Another PSD-related effect to be considered in the case of kukersite oil shale is that during the grinding of oil shale, which is naturally heterogenic fuel, the distribution of softer components such as kerogen and sandy-clay minerals into finer particles occurs. Harder components, such as carbonate minerals, which have lower grindability, will be distributed into coarse particles. This distribution process can be clearly seen in Figure 4 which illustrates the results of chemical analysis performed for oil shale with different particle size ranges.

The heat transfer inside the retort is affected by the tendency of segregation of solid particles with non-homogeneous PSD. Inside the retort PSD would determine the heating rate of oil shale particles. Heating rate is one of the parameters that can affect the yield and quality of shale oil yield [17, 19–22] and the volatile product distribution through intraparticle mass transport resistance [23]. Higher heating rates can provide even a higher shale oil yield compared with the Fischer assay's due to the rapid release of volatiles, which avoids secondary decomposition reactions [24].

Considering the chemical composition of circulating ash, it was experimentally shown that one of the main parameters affecting oil yield is CaO content. Namely, an increase in CaO content in circulating ash can lead to the decrease of oil yield by up to 20 wt% [20]. In this context, CFB process has several advantages over AFC that enable achieving better process performance [13]. These advantages are realized as the temperature in the CFB combustor integrated into the oil plant CFB during operation is maintained below 800 °C. Low combustion temperature allows a significant reduction of dissociation of carbonate minerals, mainly calcite $CaCO_3$, which naturally exist in Estonian oil shale in significant amounts. As a result, in Enefit-280 technology, the free lime (CaO) content of ash in WHB ash discharge ports is below 2% [22].

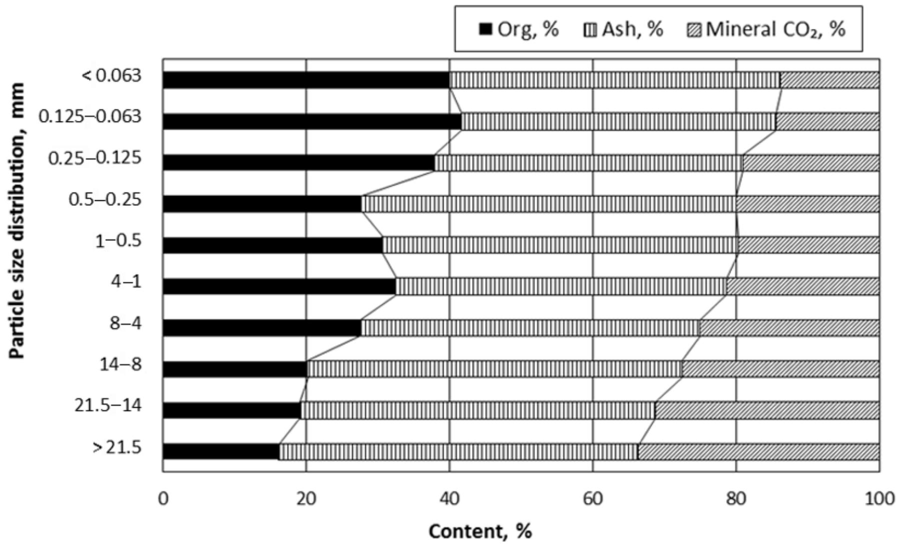

Fig. 4. Distribution of different components in oil shale of different particle size ranges (vertical axis particle size in mm). (The abbreviation used: Org – organics.)

CFB processes are due to low combustion temperatures characterised by NOx emissions formed mainly due to the oxidation of fuel nitrogen. Generally, besides the nitrogen content in the fuel, the factors contributing to NOx formation in CFB technology are combustion temperature, excess air coefficient, staging of combustion air, catalytic effects of limestone and PSD. Therefore, it has been reported that an effective optimization of the CFB combustion process and fluid dynamics by applying a fluidization state specification (FSS) design principle allowed achieving NOx emissions levels even below 50 mg/Nm^3 for coals with a nitrogen content of up to 1.7% [25]. For the CFB combustion of kukersite oil shale a typical NOx emissions value is around 150 mg/Nm^3 at 6% O_2 [26]. During retorting at temperatures around 500 °C most of the nitrogen in oil shale kerogen remains in semi-coke [27], being thus the main source of NOx in the CFB combustion unit. The results of Enefit-280 flue gas composition measurements downstream ESP are shown in Figure 5 and Figure 6. From Figure 5 it can be seen that Enefit-280 is characterized by relatively low NOx emissions, below 200 mg/Nm^3 at 6% O_2. In Figure 6, the concentrations of water, CO_2 and SO_2 in flue gases are shown.

During the thermal processing of oil shale in the retort, the greater part of combustible sulphur (pyrite, organic sulphur) remains in semi-coke [28]. In the conventional SHC process combustion of semi-coke in the AFC combustor under oxidizing conditions (air to fuel ratio 0.8), 75–77% of the sulphur in oil shale remains in the ash residue as sulphide. Increasing the combustion temperature above 790 °C will initiate the formation of CaO due to the partial decomposition of $CaCO_3$, which results in the formation of calcium sulphide through binding with hydrogen sulphide [28]. 7–8% of the

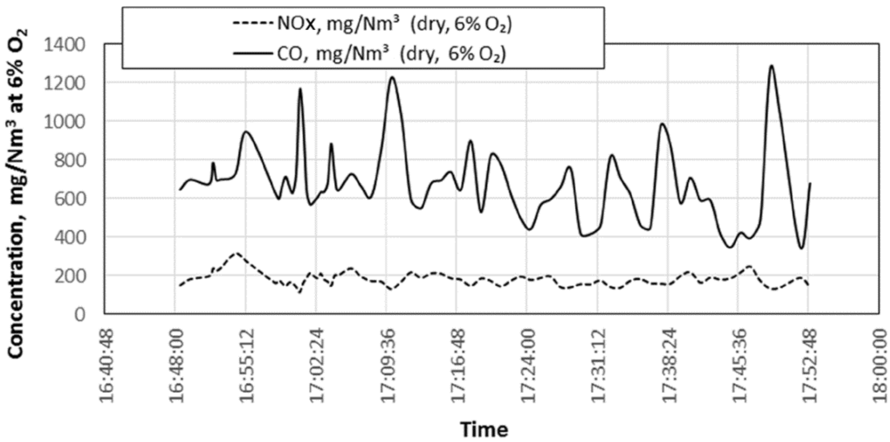

Fig. 5. Normalized NOx and CO concentrations in flue gas downstream ESP.

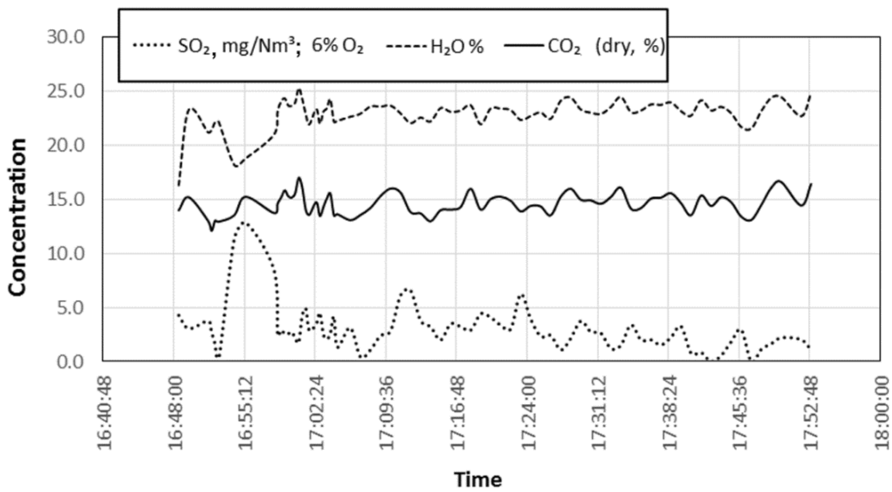

Fig. 6. Normalized SO_2, H_2O and CO_2 concentrations in flue gas downstream ESP.

remaining initial sulphur is transferred to hydrogen sulphide in the retorting process, which is subsequently oxidized to sulphur dioxide. In practice, downstream of AFC in some SHC technologies the utilization boiler is used, which allows afterburning H_2S to SO_2.

In the SHC method with integrated CFB sulphur in semi-coke is oxidized to SO_2 (obviously initially through H_2S) followed by its binding through indirect and direct sulphate binding reactions:

$$CaO + SO_2 + {}^{1}\!/_{2}\,O_2 \rightarrow CaSO_4$$

$$CaCO_3 + SO_2 + {}^{1}\!/_{2}\,O_2 \rightarrow CaSO_4 + CO_2$$

Due to the much higher molar content of calcium to sulphur, in the CFB combustion environment SO_2 is practically fully captured, resulting in a near zero level of SO_2 emissions (see Fig. 6). The comparison of flue gas compositions for different shale oil processing technologies is presented in Table 6.

Table 6. Comparison of concentrations of flue gases from different technologies

Technology / Flue gas, mg/Nm^3	Enefit-140	Petroter	Enefit-280
SO_2	130	935	< 5
NOx	240	540	190
CO	16400	1760	700
H_2S	475	< 75	~0

The heat released during semi-coke combustion in Enefit-280 technology is partly used for pyrolysis of dry oil shale in the retort through the action of SHC. The other part of heat is recovered by external recycling of ash through the ash heat exchanger, bottom ash fluidized bed coolers, and WHB. The heat given to steam is used for electricity generation. The conversion of useful heat to electricity is performed by applying a conventional steam Rankine cycle with relatively low steam parameters: pressure 41 bar and temperature 450 °C. This certainly reduces operational and capital costs; however, it also significantly reduces conversion efficiency, which is quite low compared to the efficiency obtained in conventional power units generally operating with much higher steam parameters. Pyrolysis gas as a by-product obtained in the retorting process is currently burned in separate power units with a higher thermodynamic efficiency of the steam cycle. This has a positive effect on the shale oil production efficiency.

An important advantage of CFB integration into shale oil production in Enefit-280 technology is that CFB might provide favourable conditions for implementation of oxy-fuel combustion for carbon capture. This could provide an opportunity to transform shale oil production to CO_2 neutral.

6. Conclusions

Integration of circulating fluidized bed into shale oil retorting technology based on the solid heat carrier method allows a more complete conversion of energy stored in semi-coke into useful heat through its combustion. As a result, analysis of ashes from different discharge ports in Enefit-280 plant showed a relatively low content of unburnt carbon. This extracted energy is used as the heat source in the oil shale pyrolysis process as well as for electricity generation, although currently at low efficiency. Moreover, the integration of CFB has also resulted in a significant reduction of gaseous pollutant emissions. Due to the high molar content of calcium to sulphur in oil shale and its semi-coke an almost complete in-situ SO_2 desulphurization has been achieved. NOx emissions are below 200 mg/Nm^3 at 6% O_2, which is generally characteristic of CFB combustion. All this together provides an effective utilization of semi-coke with reduced environmental impact. On the other hand, there is enough potential for enhancing the efficiency and reliability of the considered Enefit-280 oil shale retorting technology together with the potential for improving the sustainability through implementing novel carbon capture technologies like oxy-fuel combustion.

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