



Full Length Article

Effect of air and oxyfuel staged combustion on oil shale fly ash formation with direct in-furnace limestone addition for sulphur retention



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ABSTRACT

El-Lajjun oil shale fly ash formation and S retention during in-furnace limestone injection were investigated in this work. Staged air-firing and 27% O₂/CO₂ combustion was performed at 1200 °C in a vertical once through reactor. Ash samples were collected at 2.5 m far from the burner and from the conductive section of the reactor. Limestone was added with oil shale before burning in both combustion modes. Ultimate, proximate, and XRF analyses were done for all collected samples, while XRD and SEM analyses were performed for some of them. Sulphur retention in the convective section increased by 13% compared to the radiative section of the reactor. Sulphur retention in the collected ashes ranged from 22 to 32% and 35 to 45% at 2.5 m from the burner and at the candle filter, respectively. The combustion mode had a slight effect on the collected ashes chemical composition. In both combustion modes, the ash samples were enriched with lime and depleted in sulphur, which proved sulphur capture. Fly ash particles were spherical in shape with some aggregates. XRD analysis showed that the main crystalline phases formed in the produced fly ashes were the same regardless the combustion mode and environment, some differences in the relative intensities of anhydrite and lime peaks were present.

1. Introduction

Oxyfuel process is a promising option for carbon capture and storage technologies. Oxygen with a purity of more than 95 vol% is diluted with recycled flue gas, which consists mainly of CO₂ with and without H₂O, and is used for fossil fuel combustion during oxyfuel process. Flue gas recirculation ensures a proper heat transfer without major changes in the layout of the boiler and controls the flame temperature. The recovery of CO₂ becomes feasible with a lower-efficiency penalty since the concentration of carbon dioxide in the exhaust gas is significantly increased. Oxyfuel combustion affects the combustion process, heat transfer and other processes due to the variation in the oxidant and consequently the in-furnace gas environment compared to the conventional air-firing [1].

Jordan realizes that oil shale utilisation is one of its future energy sources. Oil shale combustion results in serious global air pollution. Jordanian oil shale contains high sulphur content which is considered as one of the greatest challenges for its utilisation. Organic and inorganic sulphur forms exist in oil shale [2]. Inorganic sulphur exists

mostly in the form of pyrite and/or marcasite, together with small amounts of sulphates [3]. The organic sulphur exists either in aromatic rings or in aliphatic functional groups, which is categorised as mercaptans, aliphatic and aryl sulphides, disulphides and pyritic sulphur [4].

The influence of O₂/CO₂ combustion on the ash particle formation was studied by Sheng et al. [5–7]. Sheng et al. [6,7] generated ashes under various combustion conditions in a drop tube furnace by burning three typical Chinese coals. The size distribution of the residue ash particles remained the same under O₂/CO₂ combustion. The O₂/CO₂ combustion affected significantly the mass and composition size distributions of both the submicron particles and the fine fragmentation particles, but the formation mechanisms of the submicron particles and the fine fragmentation particles is not affected. Sheng and Li [5] studied the ash formation and mineral transformations of four Chinese thermal pulverised coals in a drop tube furnace. Ashes were generated using O₂/N₂ and O₂/CO₂ environments at 1200 and 1400 °C. They reported that O₂/CO₂ combustion did not change the mineral phases formed in the residue ashes significantly, but the relative amounts of the mineral

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phases was affected. The differences observed in the produced ashes in the two atmospheres were due to the combustion temperatures of char coal particles. During oxyfuel combustion the particle combustion temperature is higher since the oxygen concentration in the oxidant is elevated (around 30% by volume). The higher particle combustion temperature affects the associated vaporization of elements which often serve as a bonding agent for ash deposits [6,8].

Using a 20 kW vertical furnace, Al-Makhadmeh et al. [9] studied the effect of oxyfuel conditions on Jordanian oil shale ash formation during unstaged combustion. The minerals present in oil shale were mainly calcite and quartz with minor kaolinite and fluoroapatite phases. The oil shale ashes contained anhydrite, lime, quartz, calcium phosphate, calcium aluminum silicates, several calcium silicate phases, and hematite. They reported that the main crystalline phases formed in residue ashes were almost the same in OF27 combustion and air-firing. Some differences in the relative intensities of the XRD peaks for some formed phases in both combustion modes were observed. They also studied the effect of direct limestone injection during unstaged combustion on desulphurisation. The S content of the collected ashes increased with limestone addition and as the molar Ca/S ratio increased in both combustion modes, the sulphur content in the collected ashes increased [9].

Staging technique is used for NO_x reduction. Al-Makhadmeh et al. [10] investigated this technique during Jordanian oil shale combustion and found a significant reduction in NO_x emission in both air-firing and OF27, 27% O₂/73% CO₂ environment combustions. Al-Makhadmeh et al. [11] performed simultaneous control for NO and SO₂ emissions. They mixed limestone with oil shale before staged combustion and used gas concentration at the end of the reactor in their analysis. They found that limestone addition lowered SO₂ emissions significantly during staged air-firing and OF27 combustions. The desulfurization efficiencies ranged from 32 to 61% and 25 to 60% during the staged air-firing and staged OF27 combustion, respectively.

In this paper, direct limestone injection is performed during staged air-firing and OF27 combustion to study the effect of both measures on ash composition and formation. In addition, sulphur retention is analyzed in the collected ashes at the end of the reactor and in the conductive section of the furnace.

2. Material and methods

In this study, Jordanian oil shale from El-Lajjun area was used. Oil shale samples were characterized by proximate and ultimate analyses, heating value, and ash composition. The analysis results were listed in Tables 1 and 2. Oil shale ash was mainly composed of calcium oxide (44.5%) and silica (25.85%) with other trace elements, such as Zn, Sr, Cr and Mo. The particle size was D₉₀ = 35.3 μm (D₉₀ represents that 90% by volume of the particles that have a diameter equal to or less than D₉₀). The minerals present in oil shale were calcite and quartz as major phase with minor presence for kaolinite and fluoroapatite. Limestone sample with 96 wt% CaCO₃ from Kalk-Laden and Kalk-Schule was used. The chemical composition of the sample was determined using RTG fluorescence and the analysis results were listed in Table 3 [9].

El-Lajjun oil shale combustion was performed in a 20 kW vertical furnace shown in Fig. 1, which consists of a ceramic tube reaction zone of 200 mm diameter and 2500 mm length. The primary and secondary combustion air was injected through annular clearances. To enable constant wall temperature, the furnace was electrically heated around the reaction zone [12].

Oxidant staging was applied for air-firing and OF27 combustion. All the combustion experiments were performed at a wall temperature of 1200 °C. Staged combustion input parameters, listed in Table 4, were the same as in Al-Makhadmeh et al. [9]. Direct in-furnace sorbent injection was applied for both staged combustion modes for simultaneous SO₂ and NO emissions control at three burner oxygen ratios (λ) of 0.75,

Table 1
Characterization of El-Lajjun oil shale.

<i>Proximate Analysis</i>	
Moisture content (as received,%)	1.08
Ash (moisture free,%)	54.20
Volatile (moisture ash free,%)	99.06
Fixed carbon (moisture ash free,%)	0.85
<i>Ultimate Analysis</i>	
C (moisture ash free,%)	55.68
H (moisture ash free,%)	4.27
N (moisture ash free,%)	0.87
S (moisture ash free,%)	8.30
O (by difference,%)	30.88
Low Heating Value (water ash free, kJ/kg)	19585.15
<i>Size Distribution (μm)</i>	
D ₁₀	1.25
D ₅₀	9.86
D ₉₀	35.3
<i>Main crystalline phases</i>	
Calcite	
Quartz	

Table 2
Ash main and trace elements contents of El-Lajjun oil shale.

Ash main elements (oxide form,%)%5% [wf, %]	(%)	Ash trace elements (mg/kg)	(mg/kg)
Al ₂ O ₃	5.70	As	11.5 ± 0.3
BaO	0.007	Ba	52.7 ± 1.5
CaO	44.50	Cd	49.5 ± 0.5
Fe ₂ O ₃	1.65	Cr	36.4 ± 1
K ₂ O	0.561	Cu	73.5 ± 3
MgO	1.076	Mn	23.3 ± 2.8
Na ₂ O	0.009	Mo	235 ± 9
Mn ₂ O	0.193	Ni	198 ± 3
P ₂ O ₅	3.67	Hg	n.b.(0.085 ± 0.016)
SO ₃	16.44	Pb	0.555 ± 0.181
SiO ₂	25.85	Sb	10.2 ± 5.4
SrO	0.151	Se	34.6 ± 1
TiO ₂	0.189	Sr	824 ± 6
		V	167 ± 4
		Zn	599 ± 18

Table 3
Main and trace elements contents of limestone.

Main elements (oxide form) [wf, %]		Trace elements [mg/kg]	(mg/kg)
TOC (%)	0.062	As	18.3
TIC (%)	10.98	Cd	< 0.5
CO ₂ (%)	40.3	Co	4.08
Al (mg/kg)	3920	Cr	42.5
Ba (mg/kg)	26.1	Cu	39.7
Ca (g/kg)	395.1	Hg	< 0.4
Fe (mg/kg)	235	Mo	3.9
K (mg/kg)	276	Ni	18.1
Mg (mg/kg)	1490	Pb	33.9
Na (mg/kg)	60.4	Sb	24.7
Mn (mg/kg)	191	Se	20.6
P (mg/kg)	115	Sr	402
S (mg/kg)	94.1	Tl	8.43
Si (g/kg)	16.6	V	29.5
Sr (mg/kg)	34.5	Zn	22.7
Ti (mg/kg)	16.5		

0.85 and 0.95. Limestone was mixed with El-Lajjun oil shale at three different molar ratios of Ca/S (1, 2 and 3). Three different positions (1, 1.5 and 2 m) of the burnout oxidant probe from the burner were also tested. To ensure repeatability and reproducibility of the experiments before any staged test, unstaged air-firing or OF27 combustion tests with limestone addition were performed and almost the same gas

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