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Thermochimica Acta 277 (1996) 41–52

thermochimica
acta

Thermogravimetric studies of systems pertinent to the in-situ combustion process for enhanced oil recovery.
Part 1. Development of a high-pressure thermobalance

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Received 31 July 1995; accepted 13 October 1995

Abstract

A high-pressure (0–1000 psig) thermobalance experiment has been developed in order to obtain thermogravimetric (TG) and derivative thermogravimetry data (DTG) pertinent to the in-situ combustion process for enhanced oil recovery. Studies on the pressured thermo-oxidative behaviour of 20/80 (W/W) oil/sand and coked sand samples are reported.

In the case of the oil/sand sample under a 100 psig non-oxidising atmosphere, distillation/evaporation occurred below 320°C at which point thermal cracking commenced. Above 520°C only carbon-rich coke material remained. Under an oxidising air atmosphere at 100 psig, the TG curves were more complex. Below 360°C, low-temperature oxidation (LTO) competes with distillation/evaporation; hence the rate of weight loss is significantly decreased whilst fuel deposition occurs. Between 360°C and 475°C, the observed weight loss is believed to be due to a combination of hydrocarbon combustion and visbreaking or coking. Above 475°C, combustion of the remaining organic matter (high-temperature oxidation HTO) occurs.

Experiments conducted using coked sand samples showed that, in air at 100 psig, the sample weight increased up to 250°C. This is attributed to the formation of oxygenated species. Above 250°C, the sample rapidly lost weight due to combustion reactions.

Keywords: Enhanced oil recovery; Heavy oil; High-pressure thermobalance; In-situ combustion; Thermogravimetry

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SSDI 0040-6031(95)02771-8

1. Introduction

Exploitation of the reserves of an oil reservoir typically takes place in three stages, termed primary, secondary, and tertiary recovery. The first two stages may recover up to 40% of the original oil in place. One of the tertiary techniques used to recover the remaining oil is in-situ combustion (ISC). Significantly, this process can also be used as a primary method to recover heavy oil which cannot be recovered by conventional methods. The in-situ combustion process involves igniting the formation, the combustion reaction being sustained by the simultaneous injection of an oxidant gas. The mechanism of this process involves converting some of the oil into a coke which is subsequently burned as fuel. The heat evolved from these combustion reactions reduces the viscosity of the oil thereby improving its mobility and hence recoverability. This heat can also crack some of the heavier fractions to produce an upgraded oil. Thus the fluid produced by an ISC project offers the potential advantage of oil upgrading over alternative methods of oil recovery.

Before implementing an enhanced oil recovery (EOR) technique, an oil company will require evidence of the potential success in terms of the enhanced production available as a result of implementing the procedure. This evidence may be provided by a computer-based simulator. The simulators currently available [1–5] require information regarding the reservoir and oil characteristics, such as the geology of the formation, the composition of the oil and the kinetics of the various combustion processes. Since ISC is generally employed as a tertiary method of oil recovery, the reservoir characteristics will generally be known. The combustion kinetics information will probably not be available and has to be obtained from laboratory investigations.

Two types of laboratory study are used to obtain data for in-situ combustion simulators: (i) combustion tubes, and (ii) thermal analysis techniques [6, 7]. Combustion tubes are basically high-pressure tubular reactors (typically 0.1 m diam × 1 m length). These tubes are filled with reservoir rock (consolidated or ground) and oil. The tube is well instrumented with thermocouples and is run adiabatically using external band heaters. The tube is pressurised to the typical formation pressure and an oxidant gas passed through the tube. The tube contents are ignited at the inlet and the bed is allowed to burn. The temperature profile along the tube is recorded as a function of time, and the exhaust gases and the produced oil are analyzed. This piece of equipment provides information on the ability of the formation to sustain combustion under a particular set of conditions, on the percentage of oil recovered, and on the fluid and heat transfer characteristics of the formation. These experiments are expensive in terms of finance and time. The thermal analysis techniques of TG, DTG and differential scanning calorimetry DSC are complementary to the combustion tube. These techniques may be used to provide information such as heats of reaction (oxidation and combustion) and reaction rates which are relevant to the ISC process. Such experiments have the advantage over large-scale combustion tube experiments, in that they require small samples (20–50 mg) and have relatively short timescales (approx. 1 h), so that a range of conditions can be investigated comparatively quickly. The effect of the surface area of the matrix, catalysts and other additives and the oil composition are some of the factors which may be investigated using these methods. For the experiments to have relevance to the field situation, they must be conducted at pressures equivalent to those experienced in the formation and for this reason high-pressure TG and DSC equipment should be used.

Although thermal analysis methods have been widely used in different research areas of the polymer, food and coal industries, their application in research related to the petroleum industry has been limited. However some investigators have obtained both qualitative and quantitative results from thermal curves in order to predict the mechanisms of crude oil combustion.

Tadema [8] seems to have been the first investigator to apply thermal analysis techniques to the study of the thermal effects resulting from combustion of mixtures of various crude oils and sands. Later, Burger and Sahuquet [9] studied the kinetics of the oxidation reactions involved in burning oil by the DTG technique. By analysing the exhaust gases through an evolved gas analyser, three successive oxidation reactions were observed, namely low-temperature partial oxidation, combustion of the crude oil fraction, and combustion of coke. They observed significant differences in the oxidation behaviour of crude oil in the presence of porous media containing clays.

Vossoughi and El-Shoubary [7] investigated the effects of different variables such as specific surface area, oxygen partial pressure and oil content on coke combustion using TG techniques. Each parameter was varied in turn while the others were kept constant. The TG curves and the DTG curves were subjected to kinetic analysis. The rate equation produced indicated that the rate of coke combustion was proportional to the coke content yet to be burned, to the oxygen partial pressure, and to the sand-grain specific surface area.

More recently, Lin et al [10] observed the effect of pyrolysis temperature on Tar Sand using the TG technique. They confirmed that the carbonaceous residue formed at higher pyrolysis temperatures exhibited a higher average activation energy and pre-exponential factor than that formed at lower temperatures.

The above studies were carried out at atmospheric pressure. Since most oil reservoirs are at much higher pressures, it is important that the thermal analysis data be obtained at such pressures in order to be relevant to real conditions. We have previously reported high-pressure DSC data pertinent to the in-situ combustion process [11]. This paper describes further results obtained using a high-pressure thermobalance developed to study this process.

2. High-pressure thermobalance

Commercial balances operating under vacuum or at controlled low pressures of up to one atmosphere have been widely used [12–15]. However, only a few units are available which enable thermogravimetry to be practised at high-pressure under controlled atmospheres [16–18].

The need to develop an instrument capable of handling highly reactive and corrosive gas atmospheres at high temperature and high pressure arose from our laboratory's efforts to study the reaction mechanisms of heavy oil oxidation. In view of the above requirements, various instrumentation problems have had to be overcome, such as the effect of the evolved gas, of buoyancy, and of heat convection/conduction to the balance mechanism. In addition, the possible mass transfer resistance from gas to the sample must also be considered as this is potentially the major effect that can disguise the reaction kinetics [19–21]. These requirements are met by a Sartorious Microbalance model 4406 modified to permit operation at pressures up to 150 bar and temperatures up to 750° C, in corrosive gas atmospheres and under flowing gas conditions. The choice of a microbalance means that the errors due to temperature gradients throughout the sample are minimized [22, 23]. The null-point balance system [24] utilises a light-weight microbalance beam made from a metallized quartz tube to minimise the effect of thermal expansion by heat conduction when the microbalance is located near the furnace and by heat convection of flowing gas from the hot zone. These means ensure that the microbalance beam has adequate long-term thermal stability. The microbalance is housed in a compact high-pressure 316 stainless steel housing wall mounted in a manner designed to protect the weighing system from vibrations. A schematic diagram of the modified balance is given in Fig. 1.

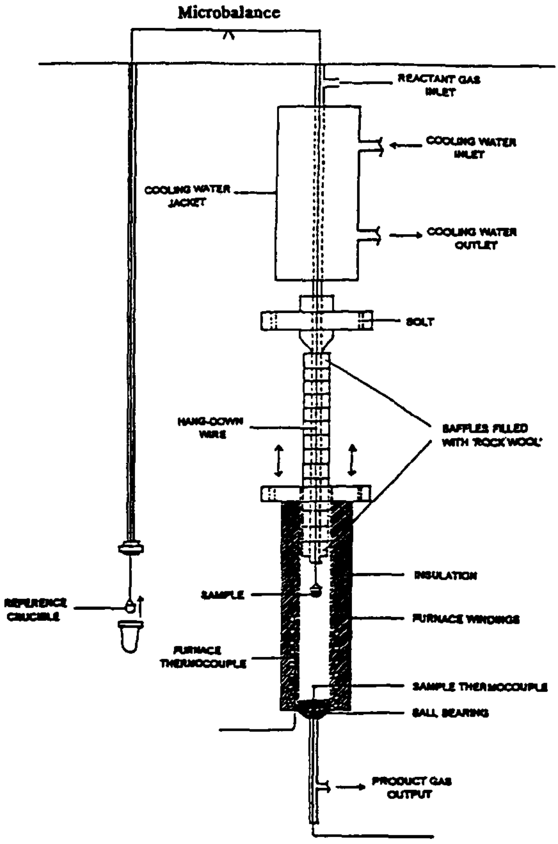

Fig. 1. A schematic diagram of the pressurised thermobalance system.

Other methods have been applied to minimise heat transfer effects to the microbalance beam. These include placing a cooling jacket between the microbalance and the furnace, and maintaining an adequate gap between them. Furthermore, the use of a flowing atmosphere instead of a static atmosphere from microbalance to furnace also minimises heat transfer effects to the microbalance beam. A downward flow direction of the gas atmosphere also minimises any evolved gas reactions with the sample, and will sweep volatile products away from the microbalance mechanism and prevent corrosion. Gas flow onto the bed of the sample is another major improvement made. Estimates of the mass transfer coefficient under the conditions of our experiments indicated that the external mass transfer resistances are negligible [25].

The sample side of the thermobalance is in two sections; an upper section attached to the microbalance, and a lower reactor section which can be raised or lowered when loading the sample. The microbalance was mounted above the reactor, the sample holder being freely suspended via a single piece of very fine nickel–alumel wire attached to the microbalance arm. The suspending wire passed down a narrow stainless steel tube within the reactor in the heated zone. A cooling jacket was located above the reactor, as shown in Fig. 1, in order to reduce conductive and convective heat transfer to the thermobalance.

The reactor section contains the furnace and thermocouples. This section is also constructed of chromium–nickel steel and measured 290 mm length × 25 mm O.D. and 21 mm I.D., with a narrower section of a diameter 6 mm × 100 mm length at the lower end of this section as a gas outlet line. Two calibrated sheathed chromel–alumel thermocouples of 1.0 mm diameter were used. One placed just below the sample crucible, recorded the sample temperature and was brazed into the 6 mm O.D. tube at the reactor outlet, while another thermocouple was placed close to the heater to control the heating rate. The validity of the sample temperature as measured by the thermocouple positioned under the sample holder was assessed using the decomposition of copper sulphate pentahydrate ($CuSO_4 \cdot 5H_2O$) as a standard material [26]. The procedure showed the sample thermocouple temperature measurements to be satisfactorily accurate to within ±2°C and also confirmed the reproducibility of the data. Great care was taken in construction to prevent a disturbance by this sample thermocouple during the weighing procedure.

The convection effects resulting from the cold flowing gases can play havoc with temperature homogeneity in the hot zone of the reactor. In order to maintain a uniform temperature, the bottom of the reactor was packed with ball bearings which were held in place with fine mesh gauze. This bed reduced the total flow rate so that heat dispersal via convection was reduced and also served to centralise the sample thermocouple. Flow of gases through the system is controlled via a mass flow controller, Brooks model 5878.

3. Sample holder

The shape of the sample crucible is one of the most important factors in the design of the sample holder. Sample holders should be light enough compared with the maximum load of the balance, to produce good balance sensitivity. Furthermore the sample holder should allow intimate contact between the sample and the surrounding atmosphere. This interaction of the sample with the atmosphere will be completely achieved if a large interface is created, e.g. by spreading the sample thinly over a wide area on an open sample holder. But the area and shape of such a sample holder is limited by the dimensions of the furnace tube. In addition, a shallow holder may be unsuitable for samples such as oils which sputter or froth on reaction. Thus for our work, a basket-shaped crucible is preferred. The quartz cylindrical crucible used was 8 mm in diameter, 9.0 mm in depth and 1 mm in wall thickness. This was suspended by a very fine nickel–alumel wire from the microbalance assembly hanging down into the heated zone of the reactor.

4. Furnace design

In the present work, the furnace winding was placed outside the reactor tube and electrically insulated with Dalfratex high-temperature sleeving which was in turn insolated from the stainless steel reactor tube with Dalfratex tape. A long furnace winding was used to create a long hot zone which reduced the heat loss due to convection currents to negligible levels. The furnace was wound non-inductively, with the windings carrying current in opposite directions so that their magnetic fields cancelled, thus avoiding magnetic interference with the weighing mechanism. The spacing of the winding was decreased towards the ends of the furnace to compensate for heat loss. The whole winding, including the furnace thermocouple which was placed between the reactor tube and the furnace windings, was contained within a Fiberfrax high-temperature heat insulation blanket.

The choice of material for the furnace winding was governed by the maximum required working temperature. Up to 1100°C, nichrome wire is a suitable material.

A model Micro 96TP5 Newtronic temperature controller was used to power and control the furnace. This was used to program the start temperature, the rate of temperature rise, and the end temperature of each experiment.

5. Data acquisition and recording equipment

The Sartorius 4406 microbalance is equipped with three auxiliary measuring units, a model 7085021 Digital Voltameter/DVM, a Model 7091 Amplifier and a Model 7078 Digital/Analogue Convertor/DAC, which provide a signal proportional to the mass. A CIL analogue-to-digital convertor of 15 channels was used to record data from both sources. The CIL reads data in the range of -1.0 V to $+1.0$ V from each of the two BNC sockets on its front panel and then transmitted them via an IEEE-488 interface to a BBC microcomputer as 12-bit values (0–4095).

A BBC BASIC program was written to convert the signals into the true value of both sample temperature and weight which were stored on a floppy disc. This data, in BBC format, was then converted to IBM PC format using Kermit file transfer supplied by the University of Colorado, USA, operative on both machines with a serial cable interface. All calculations and interpretations of the data were subsequently performed on a 486 PC. The latter can output the data as weight vs. temperature plots and, with suitable calculations, as differential thermogravimetric plots. This computer also performs all calculations of kinetic parameters.

Fig. 2 presents a schematic block diagram of the data acquisition system. The signal from the digital temperature indicator, in the range of 0–10 V, was reduced using a potentiometer in potential divider configuration in order to reduce the voltage input to the CIL/IEEE interface range 0–1 V. The signal from the Sartorius microbalance 7078 amplifier was sent via a differential amplifier (buffer) to the CIL/IEEE interface connected to the BBC microcomputer.

The data from the balance must be requested by the BBC. This is performed by the BBC computer which initiates a relay to activate the external trigger pins on the rear of the balance. At this point the value currently on the balance display is transmitted to the CIL/IEEE/BBC. This value is not updated despite changes on the balance display until the relay is next opened and reclosed.

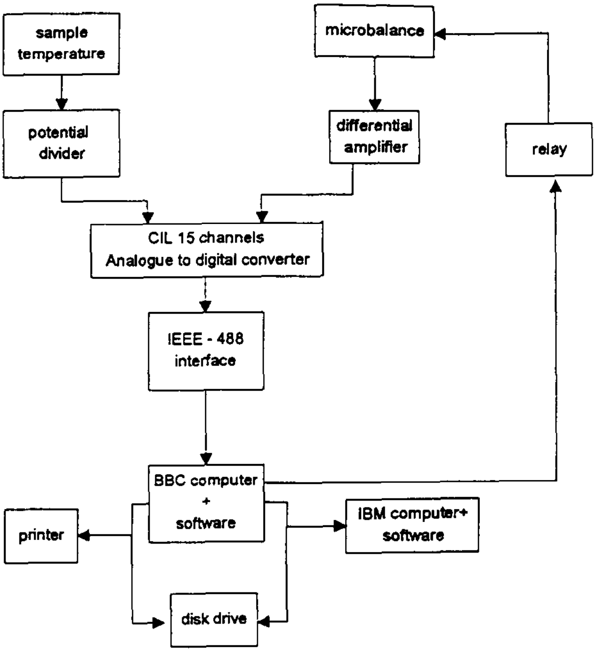

Fig. 2. A schematic block diagram for data acquisition.

6. Results and discussion

The applicability of the high-pressure TGA system to provide information pertinent to the in-situ combustion process will be illustrated by an investigation of the thermo-oxidative behaviour of an oil/sand mixture. The oil/sand mixture used was a heavy oil from Marguerite Lake, Canada, with gravity of 10.28 API and a Croxden sand of 90–100 mesh, the ratio being 20 wt% oil and 80 wt% sand; 300 mg samples were taken for each experiment.

It is important to note that the crude oil is composed of many components and a range of different mechanisms may occur during any combustion process. It was not intended to study or even detect these individual mechanisms. However, the aim of the present work is to group the major components of a given crude oil with similar mechanisms (distillation, combustion/cracking) by studying appropriate TG curves and to produce average kinetic data for these groups.

The raw weight (W) vs. temperature (T) data obtained from an experiment are fitted first to third-order piecewise polynomials to obtain smoothed TG curves. The deviations between experimental and fitted values of W were within the range of $\pm 2.5\%$. From these polynomials, the W values were then differentiated to produce the rate values ($\mathrm{d}W/\mathrm{d}t$) required.

Although heavy oils are complex mixtures with widely varying physical properties, they undergo similar reactions when heated and have similar thermal characteristics. TG and DTG curves of a heavy oil/sand heated in nitrogen and air are plotted in Figs. 3 and 4 respectively. Both the TG and DTG curves are plotted as a function of temperature and are presented as percentages of the initial sample weight. These thermal curves were produced at a heating rate of 5°C min^{-1} from 25 to 625°C and a gas flow of 100 ml min^{-1} at a total pressure of 100 psig.

In a non-oxidising atmosphere, as shown in Fig. 3, the only possible mechanisms for weight loss are distillation and thermal cracking of the medium/heavy components into lighter and more volatile components. The weight loss below 325°C is due primarily to distillation/evaporation with thermal cracking becoming the predominant process at higher temperatures. Above 520°C, only a carbon-rich coke material remains.

In an oxidizing environment, as shown in Fig. 4, TG and DTG curves are more complex and three reaction regions can be distinguished. The first region occurred between 25 and360°C, which is the low-temperature oxidation (LTO) region and competes with distillation. LTO significantly decreases the rate of weight loss and increases the fuel deposition. Under nitrogen, oil loses more than 25 wt% up to 360°C, as observed from the TG curve of Fig. 3 compared with less than 15 wt% loss in air as shown in Fig. 4.

The second transition of crude oil takes place between 360 and 475°C. This is believed to be due to the combustion of liquid hydrocarbons which compete with the thermal cracking reactions (visbreaking or coking). Reasons for the second region being a combination of two reactions can be seen in the TG curves for crude oil oxidation in which the rate of weight loss in this region is shown by the steepness of the curved portion with respect to the temperature. A possible explanation is that as the temperature is increased, the C–C, C–H and C–heteroatom bonds are broken produc-

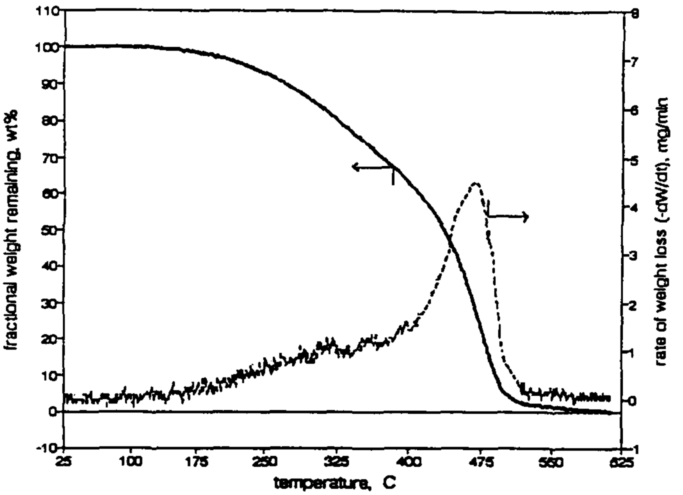

Fig. 3. TG (——) and DTG (.......) curves of heavy oil/160 μm sand with a nitrogen purge of 100 ml min^{-1}, total pressure of 100 psig, and heating rate of 5°C min^{-1}.

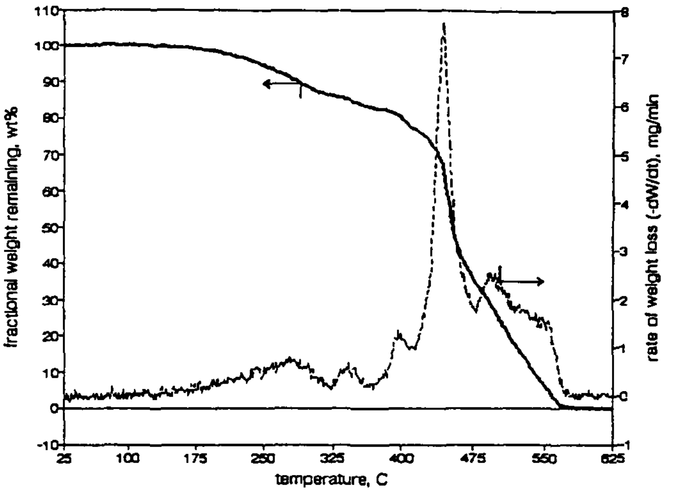

Fig. 4. TG (——) and DTG (.......) curves of heavy oil/160 μm sand with in air atmosphere, flow rate 100 ml min^{-1}, total pressure of 100 psig, and heating rate of 5°C min^{-1}.

ing reactive free radicals. These radicals can either crack further or condense, with the separation of a low H/C ratio carbonaceous material; these two processes are responsible for the distillate and coke production [27]. Drici and Vossoughi [28] reported that during crude oil oxidation, CO and CO_2 production was observed in the temperature range of 345–470°C, which indicates that combustion of the oil is taking place. Furthermore, they reported that the H/C atomic ratio of the produced gas was high at the start of the reaction and dropped continuously during the run until it reached a minimum of 0.77. Around the end of the gas analysis period, the H/C atomic ratio again started increasing and reached a maximum value of 2.24, which suggested the occurrence of two reaction regions.

The final reaction region was observed above 475°C. Here the weight loss corresponds to the combustion of the remaining organic material (high-temperature oxidation (HTO)). From analysis of the exhaust gas of heavy oil/sand oxidation at 460°, Tadema [8] found that mainly CO_2 and some CO are formed and little water was produced, which indicates that a combustion reaction occurred. Vossoughi et al. [29] showed that between 500 and 600°C the instantaneous hydrogen/carbon ratio of the effluent gas during crude oil oxidation was much lower towards the end of the run. This indicates that a carbon-rich residue (coke) existed which exhibited a relatively high exothermicity compared with the other oxidation reactions of the crude. This has been confirmed by Indrijarso et al. [11] using high-pressure differential scanning calorimetry (DSC) with samples of heavy oil/sand, where the DSC curves showed that the second peak (considered as the HTO reaction) produced a much more exothermic curve compared with the first peak (LTO reaction). The corresponding values of heat released were 1290 J g^{-1} and 850 J g^{-1} respectively.

Since deposited coke is considered as the main fuel in in-situ combustion, it is important to study the kinetics of coke oxidation in detail. For this reason the pressurised thermobalance has also been used to study the oxidation of the coking residue deposited on the sand during the pyrolysis of crude oil. The coked sand samples are obtained by pyrolysis of a crude oil/sand mixture in a small separate ancillary high-pressure reactor [25]. Coked sand samples studied were produced at 5 different pyrolysis temperatures, namely, 300, 350, 400, 450, and 500°C, in order to evaluate the effect of the pyrolysis temperature on the subsequent combustion oxidation process. The pyrolyses were carried out under nitrogen at a pressure of 250 psig, and a gas flow-rate of 100 ml min^{-1}. Oil/sand samples were 2 g in weight and the coking time was 2.5 h.

Figs. 5 and 6 present typical results obtained from the combustion of coked sands using pressurized TG at 100 psig in the presence of oxygen partial pressures of 20, 40, and 60 psig respectively. The fractional weight of carbonaceous residue remaining as a function of temperature for the coke sand produced during pyrolysis at 450°C is presented in Fig. 5, while that of the coke sand produced at 500°C is shown in Fig. 6. These curves were obtained, as in the case of the oil sand sample, by fitting the data of weight loss of coke versus temperature to third-order piecewise polynomials. The deviation between experimental and fitted values of weight loss were within the range of $\pm 2\%$.

The TG curves show that in the low-temperature region up to 325°C, a weight increase was observed, especially at higher oxygen partial pressures. This is thought to be due to the formation of oxygenated species by a low-temperature oxidation mechanism, or possibly gas adsorption. The reaction between coke and oxygen is heterogeneous: the oxygen diffuses from the bulk gas stream to the coke interface; the oxygen is then adsorbed and reacts with the coke.

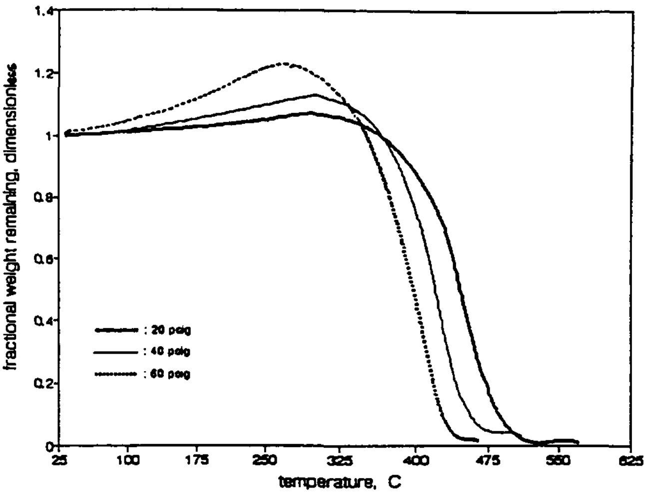

Fig. 5. TG curves as a function of oxygen partial pressure for coked sand formed at 450°C. Total pressure 100 psig, flow rate 100 ml min^{-1}, heating rate 5°C min^{-1}.

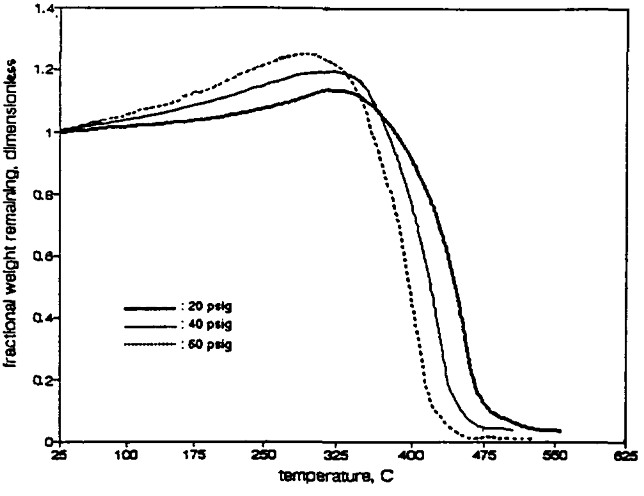

Fig. 6. TG curves as a function of oxygen partial pressure for coked sand formed at 500°C. Total pressure 100 psig, flow rate 100 ml min^{-1}, heating rate 5°C min^{-1}.

The steepness of the curved portion indicates that weight loss with respect to temperature starts in the region of 350–400°C. This weight change is due to the combustion reactions. The combustion reaction was completed at the point of the inflection of the curve. The higher the concentration of oxygen employed, the faster the combustion reaction that occurred. At the temperature of inflection, the carbonaceous residue was virtually exhausted.

In addition to the above experiments, a sequence of runs was carried out to study the sensitivity of pressurized thermogravimetric analysis (TGA) procedures to changes in the main operating variables. The variables studied were; heating rate (5, 7, 10, and 12°C min^{-1}), gas flow rates (50, 100, and 200 ml min^{-1}), total pressures (100, 200, 300, 400 psig), oxygen partial pressures (20, 40, and 60%) and sand grain-sizes (380, 160, and 70 μm). The results obtained from these runs together with a kinetic analysis will be presented in subsequent parts of this series.

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