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Continuous Operation of Spray-Dried Zinc Based Sorbent in a Hot Gas Desulfurization Process Consisting of a Transport Desulfurizer and a Fluidized Regenerator

Key Words: Hot Gas Desulfurization, Transport Reactor, Attrition Resistance Sorbent, Fluidized Regenerator

1. Introduction

Hot gas desulfurization processes are of several types of fluidized-bed (bubbling and transport), and moving-bed. Bubbling fluidized beds need to operate at a velocity within the bubbling fluidizing regime. Transport reactors can be operated at higher gas velocity, which leads to smaller diameter vessels and lower capital cost. They have the additional advantage of providing better solid/gas contact (less bypassing of bubbles), so less solid holdup is required.

Some reported a fluidized-bed reactor in a batch cyclic mode. For example, Mojtahedi and Abbasian (1995) carried out cyclic tests in a fluidized-bed reactor and Gupta et al. (1992) also performed 175 cyclic tests in a fluidized reactor to identify the chemical reactivity and mechanical strength of zinc ferrite sorbent.

A few reports briefly dealt with the performance results from the continuous operation of a fluidized-bed desulfurizer and a fluidized-bed regenerator. Nakayama et al (1996) reported experimental results obtained at a 200 t/d coal scale and they showed desulfurization performance which was 30 to 70 ppm in effluent sulfur compounds concentration from synthetic gas containing 400 to 800 ppm sulfur compounds with iron oxide sorbent. Kontinen et al. (1996) performed the integrated operation of sulfidation and regeneration at 10 – 20 bar and 500 –750 °C in a pilot scale of 15 MW_{th}. It seemed that there were some operational problems based on the bed temperature swings and the fluctuation of the effluent SO₂ concentration. Our previous research (Yi et al. 2001) has shown the continuous operation results of a HGD system with a fluidized desulfurizer and a fluidized regenerator. Sulfidation reaction, regeneration reaction and the continuous solid circulation of between a desulfurizer and a regenerator were performed at 5 atmospheric pressure for 100 hours to see the long-term reactivity and the attrition resistance of the sorbent.

Little work has been reported in continuous operation of a combination of a transport desulfurizer and a bubbling fluidized regenerator. Only a few batch cyclic test results from a transport reactor were reported. Henningsen et al. (1997) performed cyclic tests of zinc titanate sorbent in a transport reactor test unit. The regeneration was initiated at 538 °C. The initiation temperature of regeneration is not always optimum temperature for the regenerator.

In this study, we demonstrate the continuous integrated operation of a transport desulfurizer and a fluidized regenerator for 160 hours.

2. Objectives & Approach

We see the sorbent reaction performance in a HGD process consisting of a transport desulfurizer and a fluidized regenerator in this study. We have obtained the solid hold-up and solid circulation rate necessary to reach the target desulfurization efficiency. A major obstacle for fluidized- or transport bed sorbent developments is sorbent durability withstanding attrition. Continuous operation only makes similar conditions of real processes such as rapid temperature swing, chemical transformations between sulfidation and regeneration, stresses induced by fluidization and continuous particle circulation between two reactors. Therefore, an integrated system of transport desulfurizer and bubbling regenerator is operated continuously more than 150 hours to see system reliability, sorbent reaction characteristics, sorbent morphology before and after test.

3. Experiments

Figure 1 shows the schematic of the bench scale unit of KIER hot gas desulfurization process. The general features of the system include a mixing zone in the lower part of the desulfurizer, a transport desulfurizer, a fluidized regenerator, a multi-cyclone, and a loopseal. The lower mixing zone is a 0.6 m tall section of 0.035 m inside diameter. The transport desulfurizer above the mixing zone is a 5.2 m tall pipe of 0.025 m inside diameter. Two parts are connected by a reducer of 0.1 m height. A fluidized regenerator is a 1.2 m tall bed of 0.098 m inside diameter. It has a perforated type distributor and a small cyclone. An underflow standpipe and a slide-valve are located below the fluidized regenerator to control the reactor bed level by maintaining the solid circulation rate. The underflow standpipe of 0.025 m inside diameter is located at middle section of the distributor. The bed height is calculated continuously by the data from two differential pressure transducers at the reactor wall. The slide valve is closed automatically when the calculated bed height is smaller than the lower limit of the bed height, and it is open when the bed height is larger than the upper limit. In this way a proper level of sorbent inventory is maintained by just controlling a certain range of bed height. The sorbent from a regenerator is passed through a horizontal pipe and a transport desulfurizer, and is collected in the multi-cyclone. The collected sorbent in the cyclone goes back into a regenerator passing through a loopseal to prevent the gas and sorbents from flowing backward. Solid circulation rate is calculated indirectly by measuring of the bed height change in the reactor after closing one of the slide valves.

The pressure at each reactor should be controlled precisely by pressure control valves because the sorbent circulation occurs in high pressure. Figure 2 (a) shows each pressure at a desulfurizer and a regenerator, and the pressure difference between two reactors during 160 hour run. The pressures in the desulfurizer and the regenerator steadily keep at 4.168 kgf/cm² for 160 hour run continuously except that it was stopped around at 31 hr due to careless operation. A control valve at the outlet line of the each reactor automatically controls the pressure in the each corresponding reactor. Small pressure swing always exists between two reactors. If the swing becomes larger, the stable operation in solid circulation would be broken because of the reverse flow of the fuel and oxidizing gases. As shown in Figure 2 (a), the pressure difference between a desulfurizer and a regenerator is continuously maintained between -300 and 200 mmH₂O for 160 hour run. The loopseal and standpipes shown in Figure 1 play important roles to compensate the pressure fluctuations so that the reverse flow of gases does not occur.

The temperature of the reactor is controlled by 4-zone electric furnaces. Figure 2 (b) shows the temperature profiles in a desulfurizer and a regenerator. The temperature in a desulfurizer is maintained at 500 °C continuously, and that in a regenerator is maintained initially at 580 °C and is increased to 630 °C slowly.

Filters are installed in the outlets of a multi-cyclone and a reactor. Data acquisition is

accomplished by a software package FIX MMI of Intellution Inc..

The gas analysis system consists of an online gas chromatograph (H.P. 5890 series II with a flame photometric detector) for H₂S, COS, SO₂ gases, two continuous UV H₂S analyzers (RADAS2 of Hartmann & Braun Co.), a paramagnetic oxygen analyzer (775R of Rosemount Analytical Inc.), a SO₂ analyzer of IR type (URAS4 of Hartmann & Braun Co.), and a H₂ analyzer of TCD type (CALDOS5G of Hartmann & Braun Co.).

The simulated coal gas composition of 11.7% H₂, 19% CO, 10% H₂O, 6.8% CO₂, 0.5% H₂S, 52% N₂ based on an air-blown KRW gasifier is used for sulfidation reaction. All of the simulated gases except H₂S are fed to horizontal tube, H₂S is fed to the bottom of transport desulfurizer above the mixing zone to see the desulfurizing characteristics only in transport desulfurizer, excluding desulfurizing reaction in horizontal tube. The gas composition of 10% O₂ and 90% N₂ is maintained for regeneration reaction. Electronic mass flow controllers are used to feed various gases. The fluidizing superficial gas velocities is maintained at 3 m/s in a transport desulfurizer, and 0.05 m/s in a fluidized regenerator because the minimum fluidizing velocity of the used particles is about 0.01 m/s. The temperature in a desulfurizer is 500 °C and that in a regenerator is maintained from 580 °C to 630 °C, and the pressures of both reactors are 5.065x10⁵ N/m². Initial sorbent inventory is 7 kg.

The spray-dried, zinc oxide based ZAC-32SU sorbent was supplied from Korea Electric Power Research Institute (KEPRI). The average particle size of sorbent is 82 μm. The skeletal, particle, and bulk densities of the sorbent are 4.10, 1.57, and 0.91 g/ml, respectively. The BET surface area is 16.3 m²/g. Mercury pore volume, average pore diameter based on (4V/A) are 0.395 ml/g, 35 nm respectively. The 5 hour attrition loss at 10 slpm flow rate based on ASTM-D 5757-95 is 28.9% (Attrition Index, AI), that shows a little bigger attrition loss than AI, 19% of the calcined Akzo FCC catalyst.

4. Results and Discussion

4-1. Sulfidation Characteristics in a transport desulfurizer

The reaction in the desulfurizer is as follow;



The sulfur capture availability in the transport reactor depends mainly on the extent of regeneration. Regeneration temperature was varied from 580 °C to 630 °C to keep the effluent H₂S concentration below 40 ppm. For example, we increased regenerator temperature to promote the regeneration of the sorbent as the outlet H₂S concentration was increased.

Figure 3 shows the H₂S, COS, SO₂ concentration profile in the exit of the transport

desulfurizer for 160 hour run. In the transport desulfurizer, the total sulfur compounds concentrations of H_2S , COS , SO_2 at exit are consistently maintained around 40 ppm for 160 hours with a simulated coal gas containing 5500 ppm inlet H_2S gas. It corresponds to 99.2 % desulfurizing efficiency. It is shown that SO_2 emits intermittently for initial 70 hours and COS emits about 8 ppm continuously. It is considered that a part of metal oxides become metal sulfate in the regenerator and the metal sulfate reacting with reducing gas emits SO_2 in the desulfurizer.

Figure 4 shows differential pressure fluctuation profiles measured at four different zones in the transport desulfurizer. The average values of DPs for 160 hours, designated DP1, DP2, DP3, and DP4 from bottom to top position are 28.2, 16.0, 28.2, and 55.1 mmH_2O , respectively. Estimated voidages based on the respective DP data are 0.986 for DP1, 0.989 for DP2, 0.989 for DP3, and 0.975 for DP4. It is clear that such voidages, sensitive to the solid circulation rate and gas velocity, provide enough solid/gas contact to maintain below the required sulfur level in clean gas as shown in Figure 3. The lowest voidage of DP4 at the top position in the transport reactor is observed since some particles hitting the top of the transport reactor flows downwardly.

Compositions of simulated coal gases were changed after 108 hours. When H_2 was increased from 13 % to 25 % and CO_2 was decreased from 10 % to 6%, the effect of inlet gas compositions on the desulfidation efficiency was examined. As shown in Figure 5, total effluent concentration of sulfur compounds is maintained continuously around 40 ppm regardless of inlet gas compositions.

ZAC-32SU sorbent was very sensitive to the temperature of regenerator and O_2 concentration. When the outlet H_2S concentration was increased, desulfurization reaction was also improved by manipulating regeneration temperature and inlet O_2 rate in regenerator, which was the fast way to improve the desulfurization.

4.2 Regeneration Characteristics of ZAC32SU Sorbent

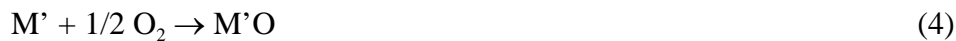
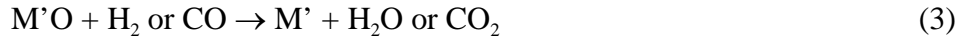
While sorbent is coming from a loopseal and is leaving to transport desulfurizer via a standpipe and a horizontal tube, the fluidized regenerator is maintained at 580 °C and 5 atm with continuous sorbent circulation. Figure 6 shows the effluent SO_2 and O_2 concentration from regenerator and the temperature profile at the center of the regenerator bed. For initial 20 h period the effluent SO_2 concentration increases up to 1.8% when the regenerator temperature is 580 °C. Considering the inlet H_2S concentration of 0.5 %, the sizes of two reactors and gas velocities in reactors, the effluent SO_2 concentration should be close to 2.1% based on the stoichiometry. Therefore, regenerator temperature is increased little by little to make SO_2 to be matched with stoichiometric value. After near 100 h, average SO_2 concentration reached to about 3 % SO_2 , which is obviously greater than a stoichiometric value, It seems that accumulated SO_2 emits because of higher regeneration temperature. Outlet O_2 concentration trend in figure 6 is increased slowly to about 3 % although it shows

some swings. It is unusually lower if it is considered that inlet O₂ concentration is 10 %.

There are several possible reactions including main reaction like the reaction (2)

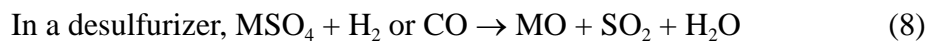


The first case will be the reduction of active metal oxide in desulfurizer followed by oxidation in a regenerator:



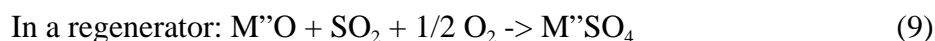
The reduced metal (M') in a desulfurizer reacts with H₂S at first. Therefore when H₂S concentration is high in reducing gases, the chance for the reducing side reaction is very low. In our experiment, there are some possibilities of metal oxide reduction because the reducing mixed gases except H₂S are fed to the horizontal tube and H₂S is fed to the following transport reactor.

Secondly,



If a lot of MSO₄ is formed, SO₂ in a desulfurizer should be emitted following equation (8) and COS is also formed. When SO₂ concentration is high in figure 3, COS concentration is also high, and outlet H₂S concentration is also high; however, the absolute values of SO₂ and COS are not so high because the SO₂ concentration is only around 10 ppm to 80 ppm in figure 3 and COS is maintained around 10 ppm through 160 hours. Therefore, we can conclude that active metal component is converted to metal sulfate just a little bit.

Thirdly, there may be a possibility of metal sulfate formation of inert metal oxide components.



The decomposition of the inert metal sulfate thus formed would not occur at the current desulfurizer temperature. At the initial stage, it was observed that O₂ was consumed more than expected possibly because reaction (9) and (10) were dominated. So, most inert metal oxide became metal sulfate as operation proceeded and reaction (2) will be dominated.

ZAC-32SU sorbent shows different regeneration aspects, compared with zinc titanate sorbent (Yi et al, 2001). More planned experiments are needed to confirm the validity of the claims and to analyze the extent of reactions quantitatively.

Jo et al (2002) showed the regeneration characteristics of a ZAC-C sorbent in a batch fluidized reactor which regeneration was initiated at 500 °C and the peak temperature of sorbent bed in the reactor reached up to 750 °C due to the vigorous exothermicity of regeneration reaction with neat air. We have known only initiation temperature for the regeneration reaction because most of researches have been done in batch cyclic test in which temperature rises suddenly by exothermic heat of reaction and drops slowly; however, the optimum regeneration temperature for continuous system have not been reported in the open literature. It is evident from our 160 hour continuous operation that regeneration temperature ranges from 600 °C to 630 °C represents the reasonable regeneration operation temperature of ZAC-32SU in continuous solid circulating mode. If regeneration of active components in a sorbent were incompleting in a regenerator section, sulfided components would be accumulated in sorbent, which would result in losing H₂S sorption capacity.

4-3. Solid Circulation Rate and Solid Residence Time

Solid circulation rate is an important parameter to calculate the heat balance and the sulfur balance in the whole HGD system. Partially sulfided sorbent from the transport desulfurizer is captured by multi-cyclones and sent to a loopseal. Sorbent passing a loopseal to a fluidized regenerator is regenerated with oxygen and it is leaving to transport desulfurizer via a standpipe and a horizontal tube. The more sorbent is circulated, the more reaction occurs giving higher desulfidation efficiency because the solid hold-up is increased in the transport reactor. However, the more sorbent is circulated, the more it is difficult to maintain heat balance between two reactors.

Sorbent circulation rate is estimated from the measurement of the bed height change in a reactor after closing one of the slide valves. Estimated solid circulation rate is 54.6 kg/h, that is 30.9 kg/m²/s based on the cross section of transport desulfurizer. Because sorbent inventory in each reactor is estimated around 3.4 kg, the solid residence time in each reactor becomes 0.06 hours based on the estimated solid circulation rate, which is much faster solid circulation than that of fluidized desulfurizer-fluidized regenerator system.

4-4. Sorbent Characteristics and Sulfur Analysis

Table 1 shows the physical characteristics of fresh and used sorbents in a regenerator and a desulfurizer after 150 hour run. Bulk densities of fresh sorbent, used sorbent in a desulfurizer, and used sorbent in a regenerator are 0.91, 1.38, and 1.40 g/ml, respectively and the corresponding particle densities are 1.57, 2.43, 2.34 g/ml, respectively. It is considered that density is increased because of the sintering at the regeneration temperature during 150 hour run.

BET surface area of fresh sorbent, 16.30 m²/g is decreased to 11.00 and 8.00 m²/g of used sorbents during 150 h run. Mercury pore volumes of the respective desulfurizer and regenerator are 0.135 and 0.161 ml/g, compared with 0.395 ml/g of fresh sorbent. Pore volumes of the used sorbents are decreased. Average pore diameter is changed from 35 nm of fresh to 16 nm of a desulfurizer and 34 nm of a regenerator sorbent. From these results, we can conclude that fresh sorbent was sintered at high temperature during the repeated sulfidation and regeneration reactions.

The 5 hour attrition loss of fresh sorbent at 10 slpm flow rate based on ASTM-D 5757-95 was 28.9% (Attrition Index, AI), that compared with AI, 22% of the Akzo FCC catalyst. After 150 h test, AI of the sorbent in the regenerator was 9.2, showing much improved attrition resistance. It also seems to be the evidence of sintering of sorbent.

Sorbent in a desulfurizer and a regenerator was sampled after 150 hour run. Table 2 shows particle size distribution and sulfur content at each size class of sorbent remained in a desulfurizer and a regenerator. The particle size distribution of sorbent in a desulfurizer was not measured because the sampled quantity was not enough for the screen analysis. The surface mean diameter of sorbent remained in a regenerator is 80 μm. The sulfur contents of sorbent in a desulfurizer are almost same within the range from 5.31 to 5.69. Sorbent in a regenerator also has same tendency within the range from 4.10 to 4.88. Average sulfur content of a desulfurizer and a regenerator is 5.51 wt%, and 4.63 wt%, respectively, which indicates the sorbent is partially sulfided and partially regenerated. In fact, sulfur contents and their difference between two reactors strongly depends on the solid circulation rate. It is expected that their values will be increased with the decrease of solid circulation rate.

Figure 7 shows particle size distributions of fresh sorbent, sorbent remained at the regenerator and captured sorbent in a regenerator cyclone after 160 hour run. The particle size distribution of reacted sorbent is almost same as that of fresh sorbent. Most captured particles in a regenerator cyclone are smaller than 50 μm.

5. Conclusion

We have demonstrated for 160 h that the transport-fluidized process was simpler and easier to access to the steady state operation relative to the fluidized-fluidized HGD process with spray-dried ZAC 32SU sorbent supplied by KEPRI.

In the transport desulfurizer, the total sulfur compounds concentrations of H₂S, COS, SO₂ at exit are consistently maintained around 40 ppm for 160 hours with a simulated coal gas containing 5500 ppm inlet H₂S. Satisfying sulfur compounds removal rate of 99% at the transport reactor was obtained at the voidage of 0.98. Solids were circulated continuously and steadily at the rate of 54.6 kg/hr between a transport desulfurizer and a bubbling fluidized reactor at high pressure. The SO₂ concentration produced from a regenerator was

maintained at 2-3 % level that is lower than the value expected by stoichiometry. The sulfur capture availability in the transport reactor depends mainly on the extent of regeneration. Regeneration temperature was varied from 580 °C to 630 °C to keep the effluent H₂S concentration below 40 ppm. We increased regenerator temperature to promote the regeneration of the sorbent as the outlet H₂S concentration was increased. Average sulfur content of a desulfurizer and a regenerator after 160 hour run are 5.51 wt%, and 4.63 wt%, respectively, which means the sorbent is partially sulfided and regenerated.

Acknowledgements

We appreciate the financial support from the R&D center of Korea Energy Management Corporation through the Energy R&D program of Ministry of Commerce, Industry, and Energy of Korean Government for this work.

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Table 1. Physical properties of fresh and reacted sorbents

| | Fresh sorbent | Used sorbent in a desulfurizer | Used sorbent in a regenerator |
|-------------------------------------|---------------|--------------------------------|-------------------------------|
| Bulk density, g/ml | 0.91 | 1.38 | 1.40 |
| Particle density, g/ml | 1.57 | 2.43 | 2.34 |
| Skeletal density, g/ml | 4.10 | 3.60 | 3.74 |
| BET surface area, m ² /g | 16.3 | 11.0 | 8.0 |
| Mercury pore volume, ml/g | 0.395 | 0.135 | 0.161 |
| Average pore diameter (4V/A), nm | 35 | 16.3 | 34.1 |
| Attrition loss, AI, % | 28.9 | 8.4 | 9.2 |

Table 2. Size distribution and sulfur contents at each size of sorbents in two reactors.

| Size class, μm | Desulfurizer | | Regenerator | |
|---------------------------|--------------|-----------|------------------|-----------|
| | Fraction | Sulfur, % | Fraction | Sulfur, % |
| 150 over | - | 5.48 | 0.018 | 4.10 |
| 106 – 150 | - | 5.69 | 0.271 | 4.15 |
| 75 – 106 | - | 5.57 | 0.388 | 4.86 |
| 63 – 75 | - | 5.51 | 0.119 | 4.70 |
| 53 – 63 | - | 5.55 | 0.120 | 4.81 |
| 38 – 53 | - | 5.47 | 0.067 | 4.88 |
| 0 – 38 | - | 5.31 | 0.018 | 4.58 |
| Mean | - | 5.51 | 80 μm | 4.63 |

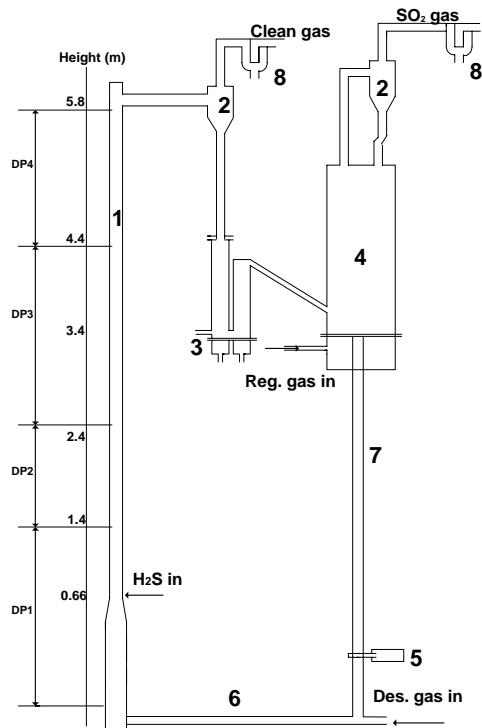


Figure 1. Schematic of the bench scale unit of KIER hot gas desulfurization process.

1. Transport desulfurizer
2. Cyclone
3. Loopseal
4. Fluidized Regenerator
5. Slidevalve
6. Horizontal pipe
7. Standpipe
8. Filter

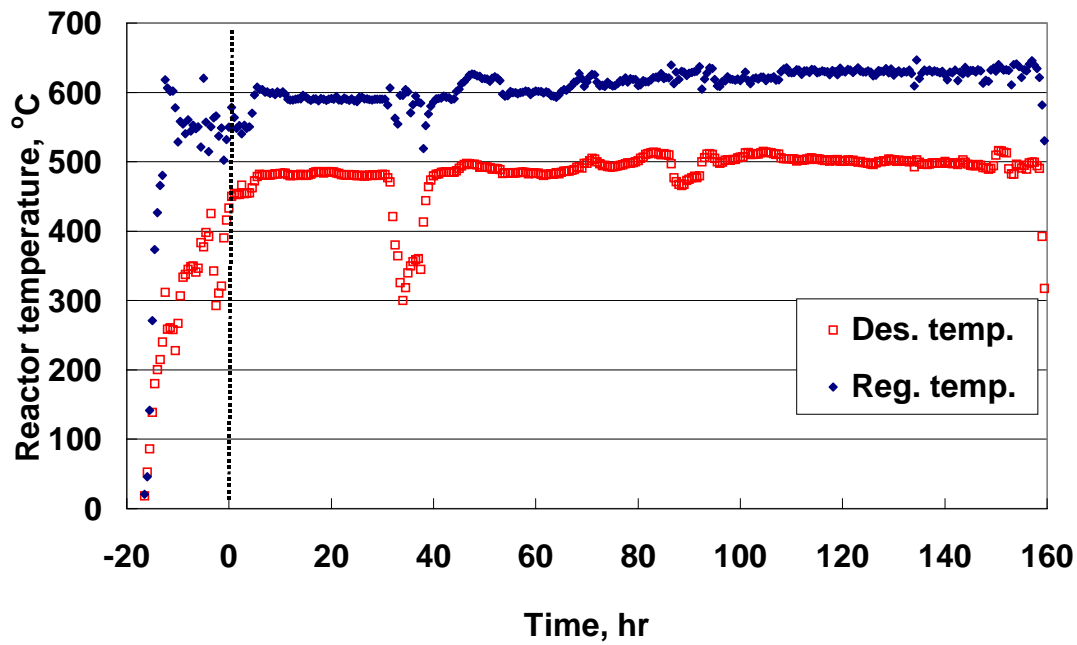
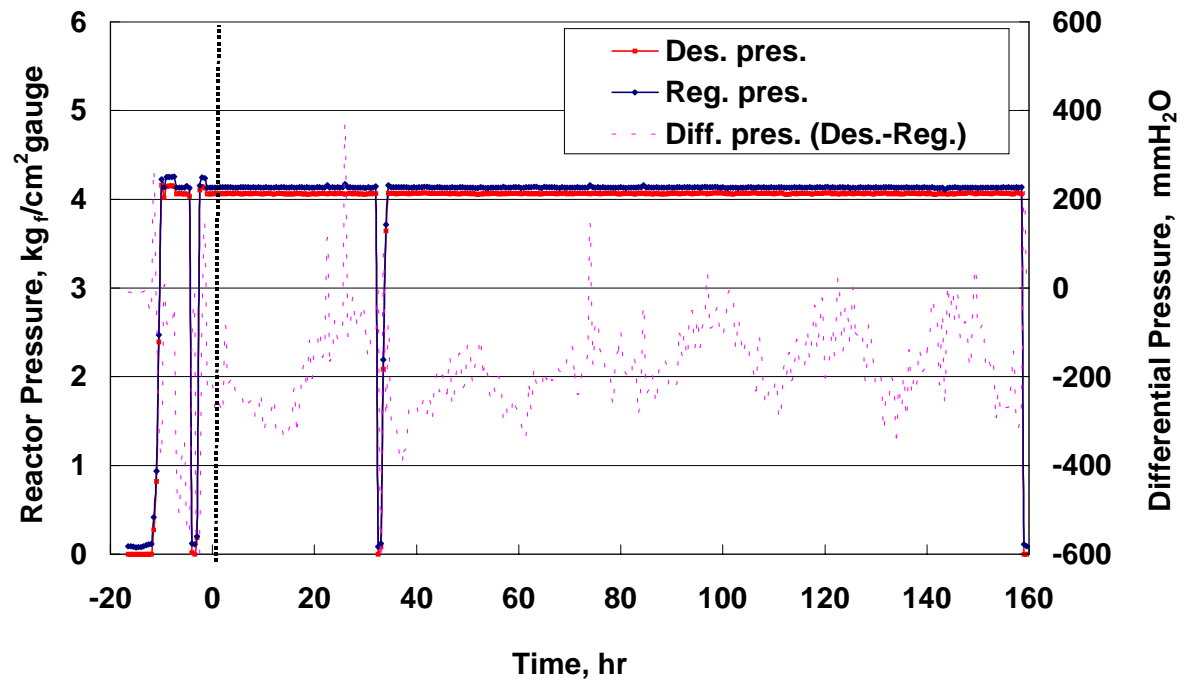


Figure 2. Pressure and temperature profiles during 160 hour run

(a) each pressure at a desulfurizer and a regenerator, and the pressure difference between two reactors (b) temperature of desulfurizer and regenerator

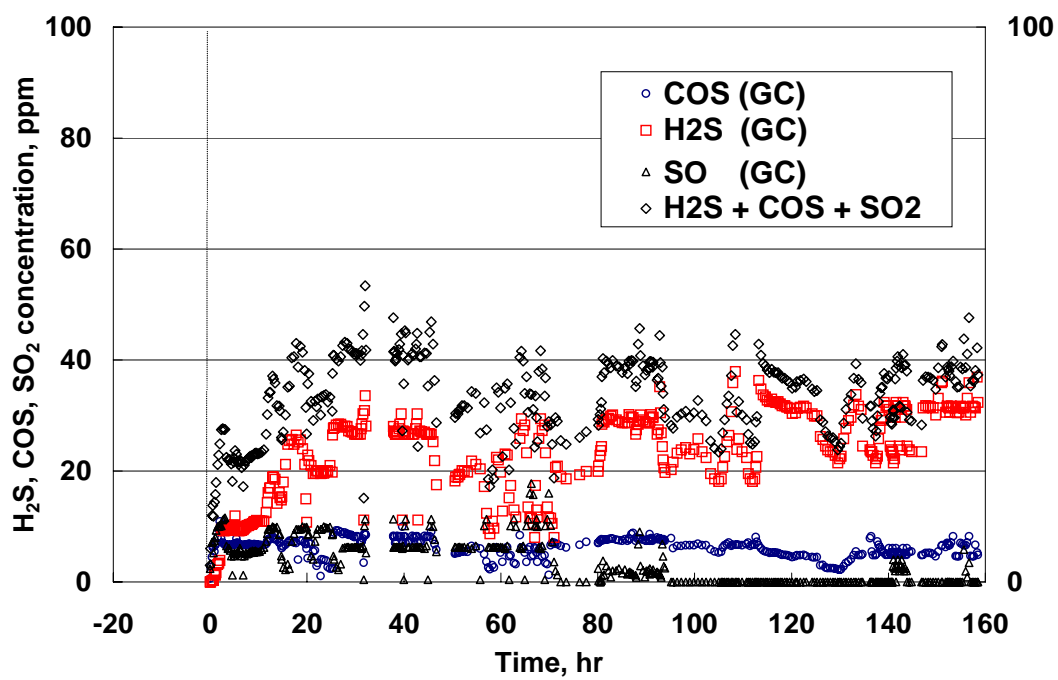


Figure 3. H₂S, COS, SO₂ concentration in the exit of the transport desulfurizer for 160 hour run.

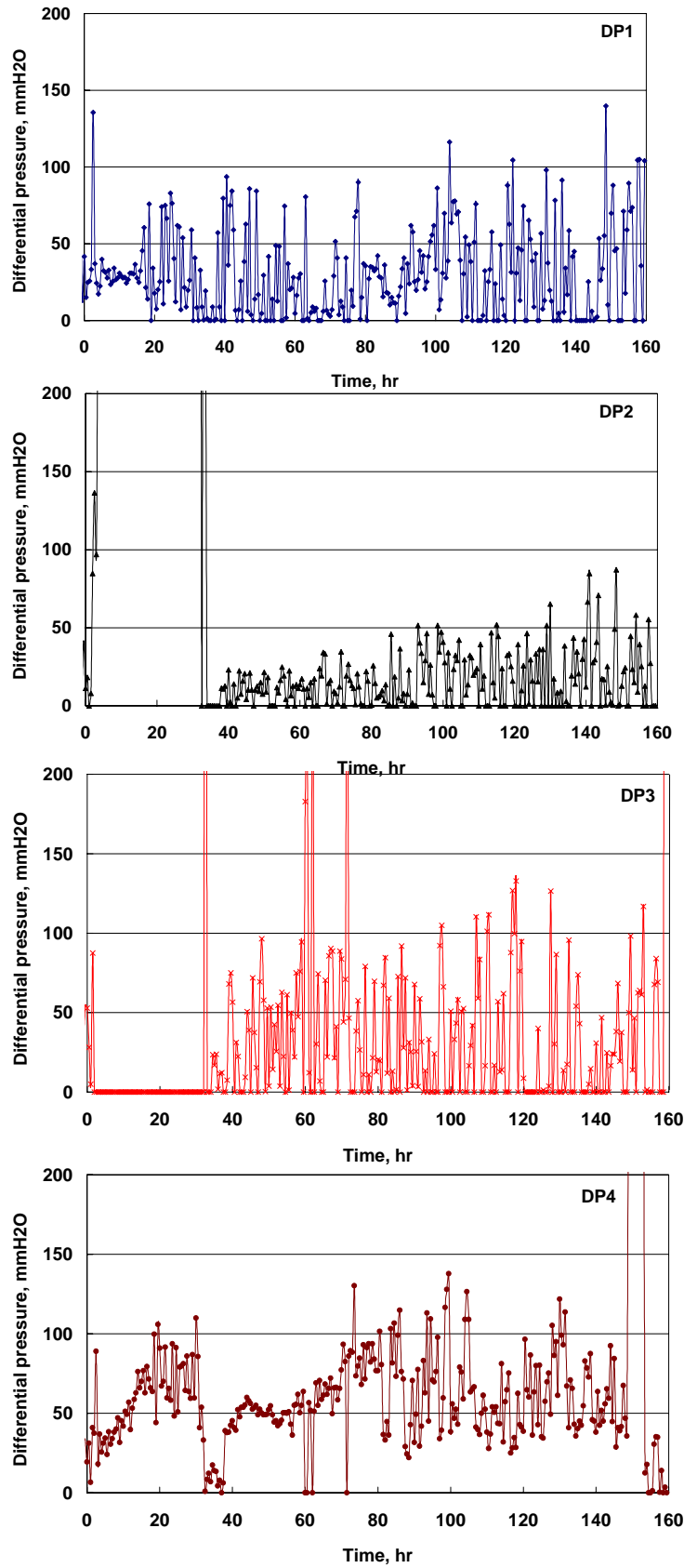


Figure 4. Differential pressure fluctuation profiles measured by four differential pressure transducers with the heights in the transport desulfurizer.

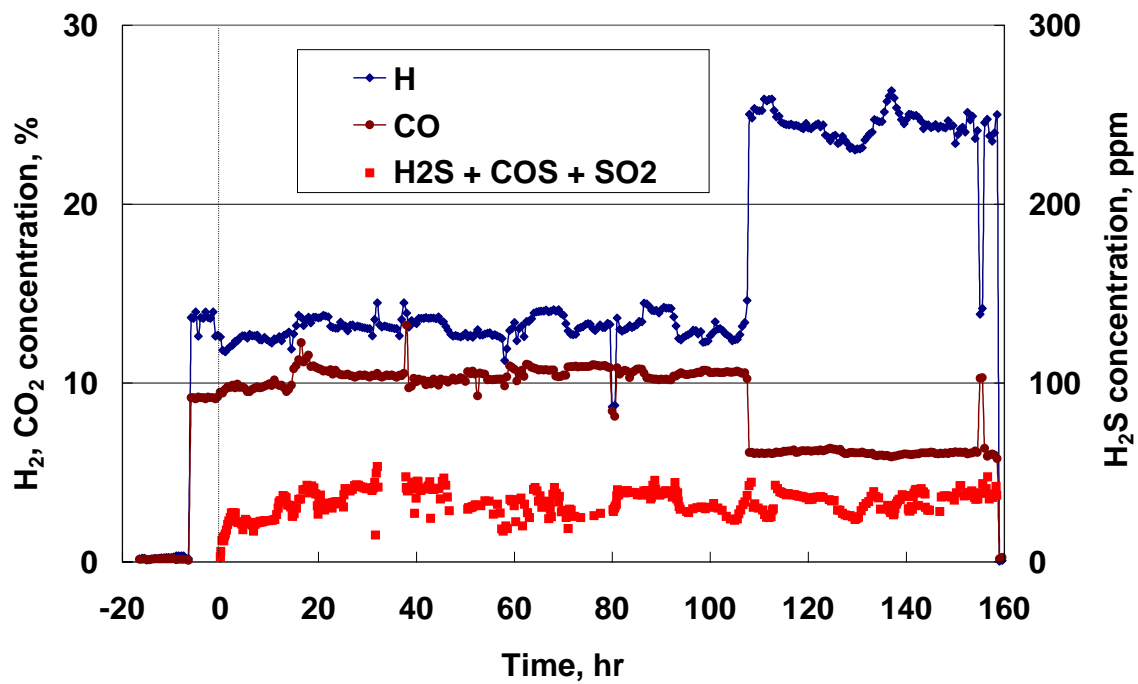


Figure 5. Effect of inlet gas composition on desulfurization reaction.

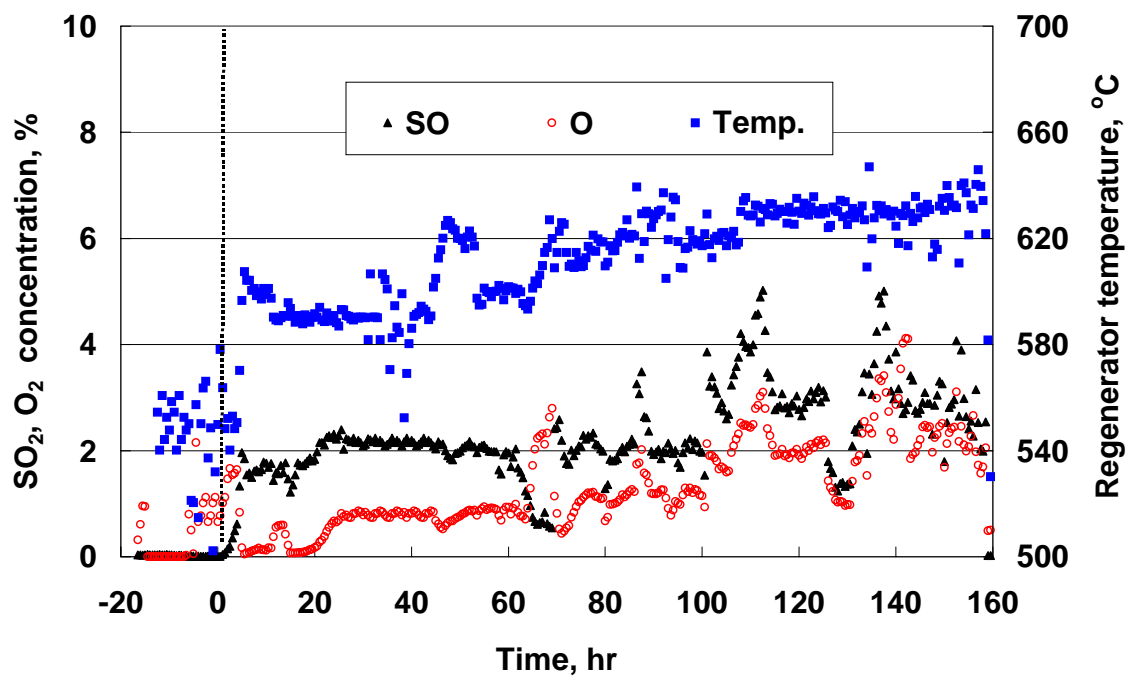


Figure 6. SO_2 and O_2 concentrations in efflux of regenerator and the temperature in the center of a regenerator bed.

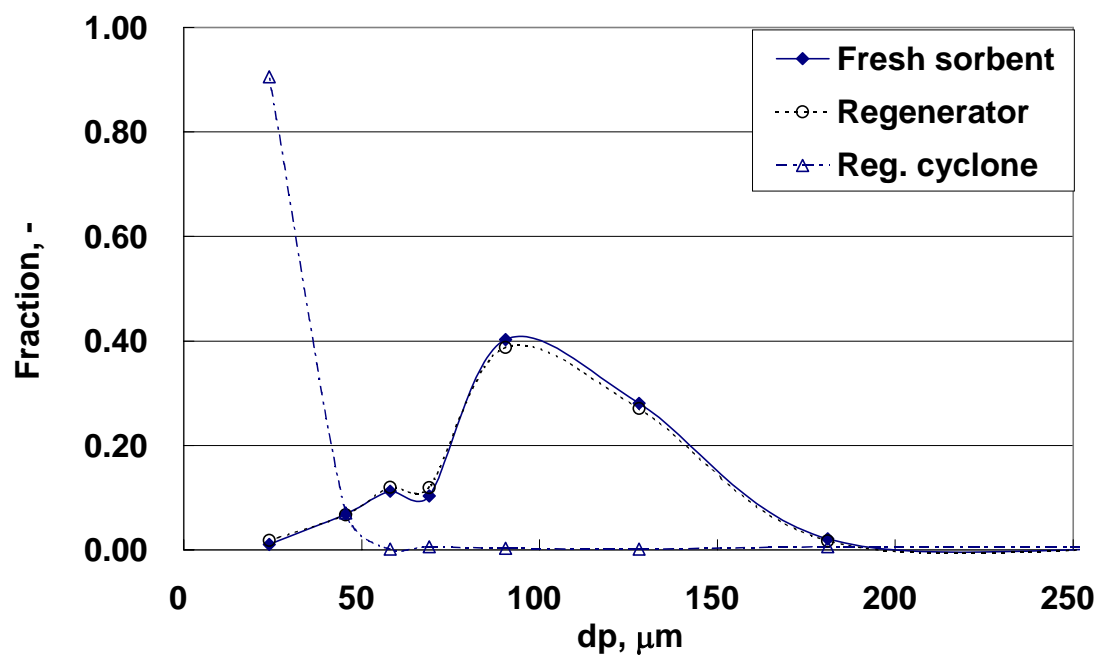


Figure 7. Particle size distributions of fresh sorbent, sorbent remained at the regenerator and captured sorbent in a regenerator cyclone after 160 hour ru