

Experimental Study on LacoO3 on Catalytic Converter

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Abstract: The aim of this research work was to test the catalytic converter with the catalyst LacoO3 on the incinerator of Tabriz Refinery. In this paper first of all, the method of building catalytic converter and the way of placing catalyst in it was investigated. Besides that, testing of converter was done. For testing catalyst, two steps were experimented, at first, the flue gas was changed so that the conversions of CO, Sand could be determined. For the second experiment the effects of CO and on the reduction of Sand were carried out. The results showed there was an optimum flow for the proper function of catalytic converter and if the conditions of process were appropriate, then the conversion of 0.94 for Sand 0.96 for would be obtained. The results also determined that was the suitable substitute for CO in the catalytic converter type LacoO3.

Key words: Catalytic Converter, Incinerator, lanthanum Oxy Sulfide (La₂O₃S), Cobalt Disulfide (CoS₂)

INTRODUCTION

A catalytic converter is a device used to reduce the toxicity of emissions from an internal combustion. The common and generic principles of catalytic combustion and developments have been scrutinized in many major topics. (Hayes R.E., S.T. Kolackzkowski, 1997; Prasad .R, L.A. Kennedy, E. Ruckenstein, 1984; Kolackzkowski *et al.*, 1993; Cybulski and Mouljin, 1994; Weinberg and (Ed.), 1986; Menon *et al.*, 1993; Trimm D.L. and F.R. Hartley (Ed.), 1991) A catalytic converter provides an environment for a chemical reaction where in toxic combustion by products are converted to less-toxic substances. In order to reduce emissions, modern engines have been designed to control the amount of fuel they burn carefully. The main emissions are: Nitrogen gas, Carbon Dioxide (Wright, Matthew, 2009) although carbon dioxide emissions are considered to contribute to global warming (Somerville *et al.*, 2007) and also Water vapor. These emissions are mostly benign, but because the combustion process is never perfect, some smaller amounts of more harmful and hazardous emissions are also produced such as Oxides of nitrogen and sulfur that Contribute to smog and acid rain, Carbon monoxide, Hydrocarbons or volatile organic compounds, that can be broken down to oxidants which react with oxides of nitrogen and cause pollution. There are two usual types of catalytic converter that have been used:

(A) Two-way, this type of converters has two simultaneous tasks:

1: Oxidation of carbon monoxide to carbon dioxide:

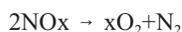


2: Oxidation of unburnt hydrocarbons (unburnt and partially-burnt fuel) to carbon dioxide and water:



(B) Three – way, this kind of catalytic converter has three simultaneous tasks:

1:Reduction of nitrogen oxides to nitrogen and oxygen:



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2: Oxidation of carbon monoxide to carbon dioxide:



3: Oxidation of unburnt hydrocarbons (CH) to carbon dioxide and water:



Usually when the air fuel ratio became less, Three-way catalytic converters could store oxygen from the exhaust gas stream, (Brandt *et al.*, 2000) when oxygen derived from reduction was unavailable, stored oxygen was released and consumed. The Oxygen sensor is the basis of the closed loop control system on a spark ignited rich burn engine, however it is also used for diagnostics. A common cause for catalytic converter failure is a defective oxygen sensor or failure to do a scheduled tune up. A deficient Oxygen sensor causes the engine to run out of tune causing the converter to practically meltdown. The certain sign that shows catalytic converter works badly is the intensive noises emitting from the catalyst which has been broken apart from the converter shell.

There have been lots of ways for reducing Sand N since the previous years, the wet and dry adsorption are the common processes used to eliminate. Also selective catalytic reduction (SCR) often was used to remove N from the gases of combustion operations. The best way of controlling the pollutant gases that are present in the gas flow simultaneously, is designing the composite and en bloc system. Some of these methods were described below:

A) Haldor -Topsoe □ s WSA -SNOX, the German DESONOX, and the Danish SNOX (Haldor Topsoe; June 1999; Blumrich *et al.*, 1990; Jelsbak *et al.*, 1990). These have been the same technologies conclude the various processes of elimination. First of all, the output gases of incinerator entered filters and the ash was eliminated. After heating, the filtered gases entered the SCR part and N was reduced by ammonia. Then the gas flow entered the second reactor and Swas oxidized to S. Consequently S was hydrolyzed and sulfuric acid produced. These are the multi steps processes and completely were not dry.

B) Babcock & Wilcox company □ s SO_x-NO_x-RO_x box (SNRB) (Doyle *et al.*, 200): Ash and tiny particles were eliminated by filters made of ceramic at high temperatures. The elimination of Swas done by injecting calcium or sodium adsorbents to the gas flow. N was reduced to nitrogen and water by injecting ammonia.

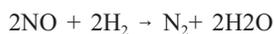
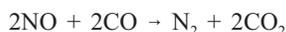
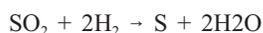
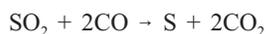
C) The Technology of NOXSO (Haslbeck *et al.*, 1984): The alkali alumina was used to adsorb Nand S. First of all the sorbent of NOXSO was reduced by heating and N was synthesized and ejected from the sorbent. Then the sorbent entered next reduction process and methane gas was used to emit the sulfur compounds from the adsorbent. The output gas of this part entered the Claus reactor and sulfur compounds were changed to sulfur. The reduced sorbent then chilled and recycled to the tower. These were also the multi steps processes that were consisted of one adsorption step, two reduction processes and the Claus operation to produce sulfur (Goar *et al.*, 1986):



The Claus operation has been used in refineries or the plants which has accessibility to hydrogen sulfide but there are some problems also about this operation such as having the precise Stoichiometric proportions for the reactants through the reaction. Another problem is presence of oxygen. This operation is two steps.

There are also the ways that eliminate Sand N simultaneously such as J.M Longo and N.L. cull (Longo J.M. and N.L. cull, 1979). There are some important points that we should review them before testing the catalyst doubtlessly. As we know, LaCoO₃ has displayed the significant role because of its special catalytic properties and it can be prepared via several methods (Bini *et al.*, 1997; Fierro J.L.G, M. A. Peña, 2001; Valencia *et al.*, 2009; Keshavaraja A., and Ramaswami, 1994; Lin huang 2005; Susumu Nakayama 2002; Goodenough and Senaris-Rodrigues, 1995; Kakihana and Popa, 2002; Popa .M, J. Franti and M. Kakihana, 2002; Yamada *et al.*, 2002; Zhu 2000; Zhecheva, 2006; Hideki Taguchi 2001; Nakayama *et al.*, 2007). This material usually has been synthesized by ceramic method (Ivanova *et al.*, 2009; Ngai Ting Lau, 1997). The catalyst LaCoO₃ should be reduced before normalization operation. The product of reduction operation was the mixture of S and Co (Ngai Ting Lau, 2005; Ngai Ting Lau *et al.*, 2003). These works showed that lanthanum oxysulfide - based catalysts are active in the reduction of sulfur dioxide to elemental sulfur (Ngai Ting Lau, 2005; Ngai Ting Lau *et al.*, 2003; Ma *et al.*, 1999; Fang *et al.*, 1996) and obviously would be

efficient way in industry and of course catalytic converters to purify the pollutant gases such as sulfur dioxide. At first, the active catalyst materials for converter were prepared in the laboratory and after building the converter, the reduction operation and also testing the catalyst with the output combustion gases of the incinerator of Tabriz Refinery was performed. Any way, we can say that the reactions that happened in the catalytic converter would be supposed as below (Ngai Ting Lau, 1997; Ngai Ting Lau, 2005; Ngai Ting Lau *et al.*, 2003; Ma *et al.*, 1999; Fang *et al.*, 1996):



The temperature of operation for these converters usually was between 450 -650 Celsius degrees and approximately in many cases it was assumed 550 °c (Ngai Ting Lau, 1997; Ngai Ting Lau, 2005; Ngai Ting Lau *et al.*, 2003) and we put the atmospheric pressure in our calculations too.

2. Experimental

2.1. Building the catalytic converter:

Calculating catalyst value and flue gas:

The relation between flue gas and catalyst value was given in reference (Ngai Ting Lau, 2005) so the required reduced catalyst value for flue gas conversion with these properties:

Table 1: The properties of flue gas

CO	1.1 %
NO	1000 ppm
SO ₂	5000 ppm

Was $21600 \frac{\text{ml}}{\text{g.h}}$, which in these situations the conversions of 0.99 for NO and 0.92 for Sat temperature 550°c were reported.

In many cases the output flue gas □ s situations from incinerator fitted the value of S in the base gas (5000 ppm) but the values of CO and NO did not, hence, this was the reason using reductive gases such as NH₃.

Having these conditions and also the increasing of catalyst through the reduction process (with respect to transforming of catalyst LaCoO3 to the catalyst mixture of CoS₂ and S through the reduction process), the value of output flue gas versus one gram of catalyst was:

$$21600 \frac{\text{ml}}{\text{g.h}} * 1.2\text{g} * \left(\frac{1}{60}\right) \frac{\text{h}}{\text{min}} = 432 \frac{\text{ml}}{\text{min}}$$

Because the maximum value for SO₂ in flue gas was under 2000 ppm through the experiment, so the value of flue gas passing through the reactor for 2000 ppm of SO₂ was:

$$432 \frac{\text{ml}}{\text{g.h}} * \frac{2000}{5000} = 173 \frac{\text{ml}}{\text{min}}$$

Briefly we can say to convert 2000 ppm S, the pollutant flue gas passed through the reactor was 173 versus one gram of catalyst.

2.2. The reactor □ s dimensions and structure:

Because the prepared catalyst was not too much then we used ceramic yarn as catalyst carrier and one gram of catalyst was dispersed on adequate ceramic yarn precisely and also monotonously. With respect to yarn volume, one stainless steel tube with dimensions 50cm* 1 in was chosen, and then the yarn included catalyst □ s particles were placed inside the tube. To prevent carrying the catalyst particles by flue gas, some pieces of yarn without catalyst were placed in the beginning and the end of tube. We used the stainless steel because of the intensive corrosion property of flue gas and the high temperature of experiment. The leaky plate

of stainless steel for keeping yarn inside the tube was used with the size of tube diameter. The number and dimension of holes were determined to decrease the pressure drop of flue gas till minimum and also for proper keeping of yarn inside the reactor. The inside leaky plate was fixed inside the tube by hammering method. Two entrances were placed on the tube for flue gas. These entrances were linked to the side of tube by one stainless steel junction for entering the main flue gas and reductive gas NH_3 (for compensating lack of CO gas in the flue gas flow). There was exit way for inside gas flow by linking the joint made of stainless steel to the other side of tube. To analyze the input gas flow to the reactor, one sample point was placed after the entrance part. There were some other junctions such as:

- (A) The entrance junctions to the chimney of furnace and the injection junction of
- (B) The exit way of reactor to the condenser.
- (C) The sample point \square s junction to the analyzer system by copper tubes.

These categories were all linked together and the schematic sketch was given below:

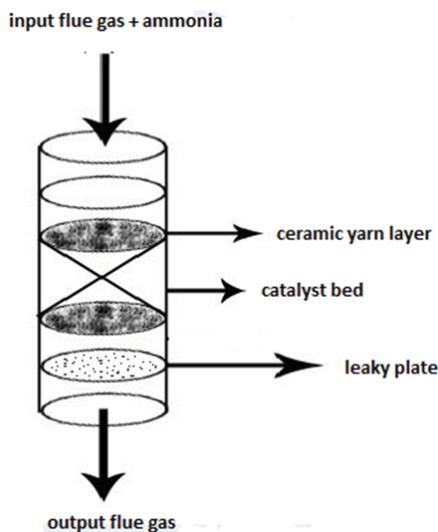


Fig. 1: The inside schematic sketch of the catalytic converter

2.3. Testing of catalyst:

The experiment was arranged for testing the catalytic converter and the required equipments were shown by figure 2:

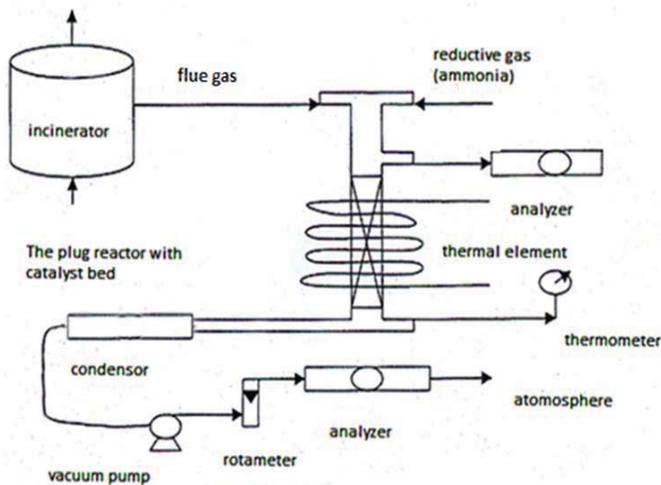


Fig. 2: Testing catalytic converter on the chimney of incinerator

It was necessary to obtain the optimum temperature for the reaction. The optimum temperature of the catalytic reactions type LaCoO_3 was in the reference (Ngai Ting Lau, 1997; Ngai Ting Lau, 2005) so the optimum temperature showed the conversion of SO_2 and NO was obtained and given in the table 2. (The results were calculated for the gas with these properties: 1.1% CO, 1000 ppm NO and 5000 ppm SO_2)

Table 2: The conversion of NO and S at various temperatures

T °C	NO	SO_2
400	30%	46%
450	84%	79%
500	95%	89%
550	99%	92%
600	100%	90%

The table 2 showed that the optimum temperature was 550 °c and for the temperature more or less than it, the conversion of SO_2 decreased. At this temperature the conversion of NO was almost complete therefore the test operation was scheduled at temperature 550 Celsius degree so that the conversion of SO_2 stayed at its optimum value.

With respect to the catalyst □ s oxidation state, the first step of testing was the reduction operation and indeed in this operation the main catalyst □ s structure formed and being ready for its performance. To reduce the catalyst, the flue gas included Swas used through the catalyst bed. Through reduction operation no sulfur was obtained, therefore we used this property to recognize the final point for the reduction operation and this operation continued till appearing the sulfur particles at the end of reactor. In this experiment, the catalyst □ s particles appeared after two hours and consequently the reduction operation ended.

After the reduction operation, the testing began. The important problem that we had in this process was the lack of CO in the flue gas in order to reduce all SO_2 and NO_x . Therefore to solve this problem and with respect to CO as a main source for reducing the catalyst LaCoO_3 (Ngai Ting Lau, 1997; Ngai Ting Lau *et al.*, 2003; Fang *et al.*, 1996) we tried using another source. The ammonia gas was abundantly present around the incinerator and it was distinguished the proper case for this situation. It was used for rescuing gas and reducing SO_2 and NO_x . The ammonia gas also vent into the reactor with the main flow.

For investigating the effects of changing gas flow □ s conditions on the conversion of pollutants, some experiments were done. While the incinerator was under use, we could not change any of its conditions but because the conditions of flue gas were changed by manipulating the other unit operation □ s conditions, then the possibility of investigation about the changing of flue gas was accomplished. At first step the gas flow changed so that we could investigate the conversion of CO, SO_2 and NO_x . Therefore the catalytic converter with two flows was tested. The first experiment was with about one gram of catalyst and the second with more value of catalyst. To confirm the sufficiency of reductive gas, we entered more ammonia gas to the reactor up to appearing its surplus at the end of reactor. The results of these experiments were given in the tables 3, 4, 5 and 6.

For the next experiment, the competitive influences of CO and H_2 over the reduction of SO_2 and NO_x were investigated. So we injected ammonia with its minimum value so that the CO gas was utilized for the reduction operation. The results of this experiment were shown in the tables 7 and 8. There were some points we should say about these tables. Firstly, in these tables the flue gas was given in the standard of pump flow and with respect to placing the equipments, this number showed the value of flue gas passing through the reactor for the exit flow but for the entrance flow, it only showed value of entering gas to the analyzer. Secondly, in these tables the analysis of input gas showed the mixing of flue gas and ammonia gas and because it was impossible to analyze ammonia gas in the analyzer, its value was not given in input gas. Thirdly, in these tables, the gas temperature was given smaller with the standard of flue temp and it was because of cooling the flue gas before entering the analyzer system. Attending to this point was so important because the analyzer system was such a sensitive device. The last point, since there was one analyzer, there was a difference between the analyzing time of input and output gases because of moving the analyzer between input and output parts but it would not influence over the results and can be neglected.

RESULTS AND DISCUSSION

By doing the experiments on the catalytic converter, these results were obtained:

According to the results which were given in the tables we realized that the catalytic converter did its job well about decreasing SO_2 and NO_x , and it obviously satisfied us about reducing pollutants to the value less than its allowable limit (800 PPM for SO_2 and 350 PPM for NO_x). Our calculations showed that the

conversions for SO₂ and also NO_x were 0.94 in the first experiment and for the second one, even though the conversion decreased because of the conditions of experiment and reached 0.71 for SO₂ and 0.81 for NO_x, but the concentration of pollutants was less than their maximum allowable limit. For the third experiment, we obtained the conversion of 0.91 for SO₂ and 0.96 for NO_x.

Table 3: Input results for the first experiment:

Oxygen	19.90%
CO	5 ppm
NO ₂	0.0 ppm
Flue Temperature	16.0 °C
NO _x	16 ppm
SO ₂	1929 ppm
H ₂	3 ppm
Amb.temperature	11.9 °C
Device.temperature	21.6 °C
Pump flow	770.34 $\frac{ml}{min}$
O ₂ ref	3.0%
CO ₂ max	11.9%

Table 4: Output results for the first experiment:

Oxygen	21.43 %
CO	46 ppm
NO ₂	0.0 ppm
Flue Temperature	14.5 °C
NO _x	1 ppm
SO ₂	108 ppm
H ₂	14 ppm
Amb.temperature	12.3 °C
Device.temperature	21.2 °C
Pump flow	186.33 $\frac{ml}{min}$
O ₂ ref	3.0 %
CO ₂ max	11.9 %

Table 5: Input results for the second experiment:

Oxygen	19.86 %
CO	7 ppm
NO	21 ppm
Flue Temperature	17.6 °C
NO _x	21 ppm
SO ₂	1449 ppm
H ₂	2 ppm
Amb.temperature	16.7 °C
Device.temperature	20.6 °C
Pump flow	527.06 $\frac{ml}{min}$
O ₂ ref	3.0 %
CO ₂ max	11.9 %

Table 6: Output results for the second experiment:

Oxygen	20.79 %
CO	194 ppm
NO	4 PPM
Flue Temperature	16.5 °C
NO _x	4 ppm
SO ₂	422 ppm
H ₂	57 ppm
Amb.temperature	16.3 °C
Device.temperature	21.1 °C
Pump flow	229.37 $\frac{ml}{min}$
O ₂ ref	3.0 %
CO ₂ max	11.9 %

Table 7: Input properties for the third experiment

Oxygen	19.18 %
CO	10 ppm
NO	25 ppm
Flue Temperature	13.3 °C
NO _x	26 ppm
SO ₂	207 ppm
H ₂	4 ppm
Amb.temperature	8.9 °C
Device.temperature	17.2 °C
Pump flow	594.76
O ₂ ref	3.0 %
CO, max	11.9 %

Table 8: Output results for the third experiment

Oxygen	21.25 %
CO	2 ppm
NO	1 ppm
Flue Temperature	12.5 °C
NO _x	1 ppm
SO ₂	19 ppm
H ₂	4 ppm
Amb.temperature	6.8 °C
Device.temperature	16.8 °C
Pump flow	51.32
O ₂ ref	3.0 %
CO, max	11.9 %

For describing the results obtained because of changing the flue gas, these points should be said. In the first experiment with the flow of 186, 33 $\frac{ml}{min}$ next to base flue gas, for one gram of catalyst 173 $\frac{ml}{min}$ and the maximum of conversions for SO₂ and NO_x was obtained but in the second experiment, by increasing the flow to 229, 37 the conversion decreased extremely. These results admitted the calculations about the value of catalyst and flow and totally we could say that the optimum value of flue gas for one gram of catalyst and the gas with the properties 1.1% CO, 1000 PPM NO and 5000 PPM SO₂ was 173 $\frac{ml}{min}$.

Saying some points was necessary about the tables 7 and 8. In the first and second experiments, the injection of ammonia and consequently producing H₂ were more than the third one and therefore the gas CO had the less role for reducing the SO₂ and NO_x and had important role. But for the third experiment CO could do its role for reducing and decreasing of CO at the end of the reactor proved this.

Conclusions:

This project was tested on output gases of the incinerator as a catalytic converter and satisfactory results were obtained. One of the positive points was replacing CO with as a reductive agent. Through the operative conditions of incinerator, value of CO was negligible so using substitute was remedial and corrective. Results showed NH₃ was a proper substitute for CO and could be synthesized through the process to produce required hydrogen for reduction operation. There was an optimum flow for the proper function of catalytic converter and the conversion of 0.94 for SO₂ and 0.96 for NO_x would be obtained by proper operating conditions. Although the theories about the design of catalytic converters were investigated but using these discussions to design catalytic converters, has required more values and data such as the physical and chemical properties of catalyst and also kinetics of reactions. Unfortunately these properties about LaCoO₃ were found scarcely and obtaining them needed precise investigative experiments that required modern equipments. Fortunately the experimental values about the relationship between the flue gas □ s conditions and catalyst □ s value and also the conditions of catalytic converter such as temperature have been available. Finally with respect to success of this project in the laboratory, it will be such a hope to industrialize this method.

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