

THE MECHANICS OF FUEL SULPHUR INFLUENCE ON EXHAUST EMISSIONS FROM DIESEL ENGINES

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Summary. The paper proves that, fuel sulphur influence on exhaust emissions from modern Diesel engines is chiefly connected with sulphur impact on catalytic emissions control systems. The mechanics of impact and negative effects of sulphur contained in fuel on various diesel emission control devices, as well as mechanics of formation of PM (sulphates) in catalytic converters, are described. The paper shows also, that sulphur sensitivity depends considerably on the catalyst formulation and engine operating conditions as well.

Key words: sulphur, diesel fuel, diesel engine, exhaust emissions


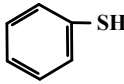
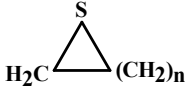
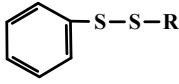
INTRODUCTION

Sulphur is present in petroleum in a form of different compounds (Table 1) of which about 70% are made up by thiophen and its derivatives [Grzechowiak 1987]. The sulphur content in petroleum is in the range of 0.1 to 10% depending on a place of origin. The sulphur content increases as the temperature of boiling point of individual fractions increases so there is less sulphur in the gasoline fraction than in the diesel fuel fraction, and most sulphur is in the heavy ends.

Due to the depletion of the petroleum deposits of a small sulphur content, the oil fields of a higher and higher percentage of sulphur are exploited. In addition to a significant limitation of the admissible sulphur content in fuels, this entails the necessity of using such distillation processes which are aimed at the elimination of sulphur from the individual fractions of petroleum. In today's hydrorefining processes commonly used the sulphur compounds in the presence of hydrogen are decomposed to H_2S which is then subject to a separation from the petroleum fractions.

The most sulphur (about 98%) contained in diesel fuel is oxidized in the combustion process to SO_2 which together with exhaust gas is released to the atmosphere where it can be subject to other reactions contributing to the creation of London-type smog and acid rains.

Table 1. The sulphur compounds present in petroleum and the products of its processing [Grzechowiak 1987]

Compound	Chemical formula
Hydrogen sulphide	H_2S
Thiophen and its derivates	
Aliphatic mercaptans	RSH
Aromatic mercaptans	
Aliphatic sulphides	$R-S-R$
Cyclic sulphides	
Aliphatic disulphides	$R-S-S-R$
Aromatic disulphides	
Polysulphides	$R-S_n-R$

Hydrogen sulphide H_2S is formed only when the rich air-fuel mixture is burned, whereas carbonyl sulphide COS appears at the exhaust gas temperature higher than $700^\circ C$ [Sher 1998]. Certain Part of SO_2 in the presence of oxygen contained in exhaust gas can be oxidized to SO_3 . The high exhaust gas temperature causes that SO_3 remains in its vapour state and easily combines with water formed during the combustion process. In the exothermic reaction the aerosol of sulphuric acid is formed, which with the chemically bonded water makes up one of the components of the particulate emission (PM). Certain amount of SO_3 being formed during the combustion process combines also with hydrocarbons or metals forming sulphates (in the following part of this paper all the chemical compounds containing SO_4^{2-} group, are called sulphates, including sulphuric acid H_2SO_4) i.e. another component of PM. Metals can come from the products of abrasive wear of the engine elements, from lubricating oil, fuel (catalyst residue) or erosion of exhaust system.

In general, forming the sulphates requires SO_2 present in exhaust gas to be oxidized to SO_3 . However, under normal conditions (lack of catalyst) such a process proceeds

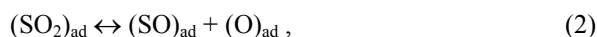
very slowly and as a result of that only approx. 1% of SO₂ is oxidized before leaving the engine exhaust system [Kozak 2000; Voss].

In case of exhaust gas from engines which are not equipped with catalytic exhaust emissions control systems, the sulphur present in the fuel is released with exhaust gas almost only in the SO₂ form and the sulphur compounds are only a small part of the PM emission even at the high sulphur content in fuel and no effect of sulphur on the emission of the gaseous exhaust emissions can be also observed. There is a quite different situation in case of the catalytic converters located in the exhaust system, where SO₂ can be subject to various reactions affecting the qualitative and quantitative changes of the exhaust emissions [Bielaczyc and others 2002 and 2003; Kozak 2001 and 2003; Merkisz 2001 and 2002; Summers and others].

THE EFFECT OF SULPHUR ON THE DIESEL OXIDATION CATALYST (DOC)

For the vehicles equipped with the diesel engines, the diesel oxidation catalyst (DOC) is a fundamental device of exhaust after-treatment, i.e. designed to be used for the treatment of the engine exhaust gas. A lack of an effective and cheap catalyst eliminating NO₂ under conditions of the oxidizing atmosphere of exhaust gas causes that the reduction of the NO_x emission from the diesel engines is today achieved mainly by a significant delay of the fuel injection and using the high rates of the exhaust gas recirculation. Both these actions limit the amount of formed NO_x efficiently, however, they cause a significant increase in the emission of products of the non-complete combustion process i.e. CO, HC and PM. In order to keep the emission at the currently required level EURO III/IV it is necessary to eliminate such compounds by using the catalyst DOC which is able to oxidize CO and HC and the soluble organic fraction SOF of the particulate matter. The oxidation catalyst does not affect the soot (the ignition point of soot is in the range of 480 to 540°C [Rao] significantly exceeding the exhaust gas temperature of a diesel vehicle under its normal operation conditions) and NO_x emission levels.

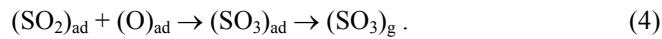
Sulphur dioxide SO₂ present in exhaust gas can interact with the oxidation catalyst in many ways. Its interaction with the converter element decisive for the conversion of the toxic compounds, i.e. with the catalytic layer, is of special importance. Sulphur dioxide can be adsorbed at the surface of a noble metal in accordance with the following reactions (index „g” means gaseous state, index „ad” means adsorption) [Truex 1999]:



Adsorbing the sulphur compounds at the catalytic layer surface blocks an access of exhaust gas to the layer itself (so called *sulphur poisoning*) what makes the course of the conversion reactions of the exhaust toxic compounds difficult. Besides the physical and chemical blockage the adsorbed compounds change also the electric and steric properties of the catalyst [Truex 1999].

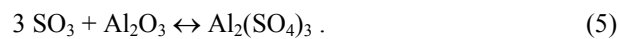
The reactions (1) and (2) are very fast whereas the course of reactions (2) and (3) is inhibited by the presence of oxygen. Thus it can be concluded that in case of the diesel engines, at partial loads especially, sulphur will be adsorbed chiefly in a form of SO₂. According to [Mador 1984] SO₃ does not cause any catalyst poisoning. Adsorption of the sulphur compounds is mainly dependent on the composition of the catalytic converter and exhaust gas, whereas the effect of temperature up to about 500°C is insignificant. The noble metals can be ranked according to their intensity of adsorption of the sulphur compounds under the excess of oxygen conditions in the following way: Pt > Pd >> Rh.

Under some conditions the removing of the adsorbed sulphur compounds can occur. However, under the oxidizing atmosphere conditions (diesel engine exhaust gas) the desorption of the sulphur compounds requires the high temperature conditions which in general are not met under the normal operation conditions: i.e. the temperatures higher than 650°C for the palladium catalyst and more than 700°C for the platinum one [Truex 1999]. The removal of the sulphur compounds consists in the conversion of a course of the reactions (1-3) and the occurrence of the reaction [Truex 1999]:



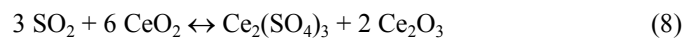
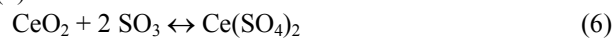
Many papers show [Henk and other, Mogi and other 1999, Truex 1999] that the poisoning proceeds the most quickly in case of the platinum catalyst and its course is the slowest for the palladium catalyst. The same happens in case of the *de-poisoning* procedure of the catalyst. The palladium catalyst recovers its primary activity with difficulty as the sulphur compounds are not only adsorbed but can also migrate deeply into the structure.

The sulphur compounds present in exhaust gas can also combine with the washcoat layer of the catalyst. In the oxidizing atmosphere and in the presence of platinum Pt (intensive oxidation of SO₂) large amounts of sulphur are deposited on the Al₂O₃ layer following the reaction:



This reaction is very intensive at the low temperatures and it proceeds inversely at the temperatures above 700°C only.

Sulphur can also react with CeO₂ which is added to the washcoat layer as a promoting and stabilizing agent. The reaction (6) takes place at the temperature below 200°C and the reaction (7) above this temperature. Sulphur dioxide SO₂ can also reduce CeO₂ according to the reaction (8).



In the oxidizing atmosphere CeOSO₄ and Ce₂(SO₄)₃ are stable up to the temperatures above 650–700°C. Other compounds added to the washcoat layer such as La₂O₃, BaO and ZrO₂ also form sulphates which are thermally stable.

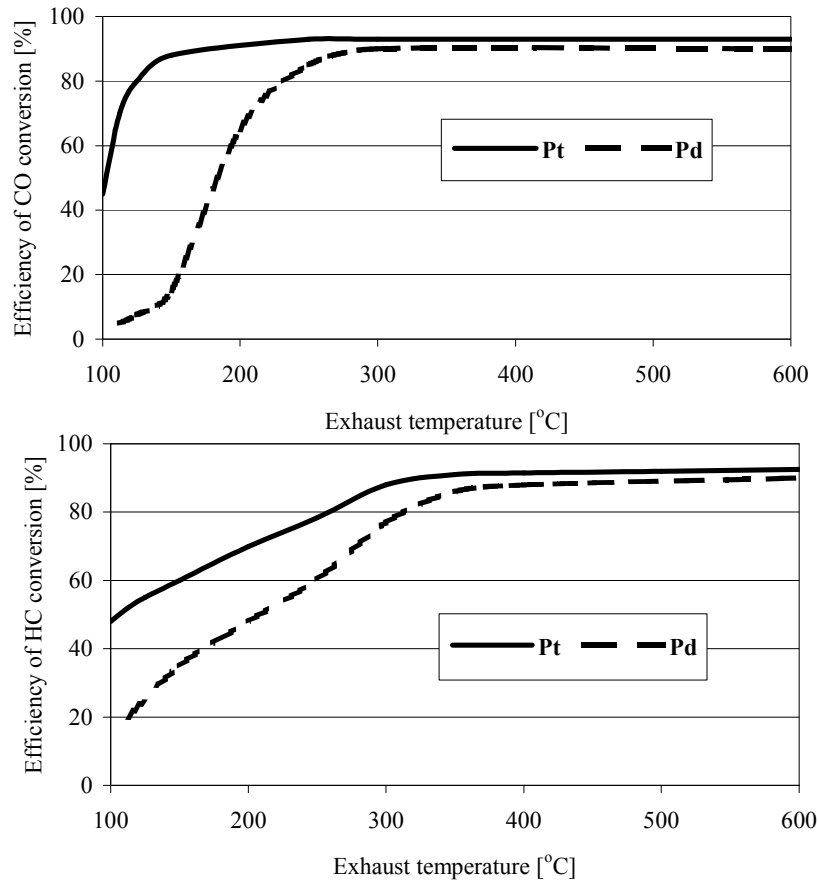


Fig.1. The CO and HC conversion efficiency for the diesel oxidation catalyst with use of the platinum (Pt) and palladium (Pd) catalytic layer as a function of the exhaust gas temperature [Engler]

The effect of the exhaust gas sulphur compounds on the catalyst and washcoat layer, as described above, disturbs the operation of the catalytic converter. The oxidation of SO_2 on the catalyst is a reaction competitive to the oxidation of CO, HC and PM present in exhaust gas what additionally reduces the efficiency of their conversion. The main effects of the sulphur presence in exhaust gas are: decrease of efficiency of the toxic components conversion by the oxidation catalyst and an increase in the light-off temperature – by 40 to 100°C are observed for the palladium catalysts) temperature of the catalyst. According to [Engler and others] and [Zelenka and others] at the higher temperatures an ability to oxidize HC is mainly reduced, and at the lower temperatures (approx. 200°C) the oxidation of CO is also significantly worse. At generally low temperature of exhaust gas during the transient test cycles, an increase in the oxidation catalyst light-off temperature caused by the sulphur even by over a dozen degrees can sig-

nificantly intensify the emission because in the range of temperatures which are reached by exhaust gas during those cycles the changes of the catalytic converter efficiency as a function of temperature are very high (Fig. 1).

As already mentioned the catalysts with the platinum active layer are the most sensitive to the sulphur present in exhaust gas. However, due to their low light-off temperatures and very high CO and HC oxidation efficiency they are used most often. More common application of the sulphur-resistant rhodium is still limited due to its very high price (price ratio for Pd/Pt/Rh is like 1:4:60) and significantly lower efficiency of the HC conversion.

THE EFFECT OF SULPHUR ON THE PARTICULATE MATTER EMISSION

Sulphur can indirectly or directly affect the particulate matter emission. The indirect action of sulphur consists in the reduction of an ability of the catalytic exhaust emissions control systems to remove the particulate matter formed during the combustion process. Above all it applies to the lowered ability of the oxidation catalyst, as described in chapter 2, to remove the SOF fraction composed mainly of hydrocarbons and to the negative effect of sulphur on the efficiency of the catalytic particulate matter filters.

The direct effect of sulphur consists in its participation in the formation of new particulates in exhaust gas – the sulphates, what generally occurs after the cylinder leaving by exhaust gas. Forming the sulphates is determined by the presence of SO_3 (sulphuric acid anhydride). As said above, SO_2 is dominant in the cylinder exhaust gas and its oxidation proceeds with difficulty despite the presence of oxygen. However, the SO_2 oxidation process can be significantly intensified in the presence of a catalyst, i.e. the catalytic converter which vehicle is equipped with. In the vehicle's catalytic converter the SO_2 oxidation process is strongly dependent on the exhaust gas temperature and on the composition of the catalytic active layer. Under normal operation conditions of the diesel engine, when the exhaust gas temperatures are in the range of 100–500°C [Rao and others], the formation of SO_3 in the presence of catalyst is controlled by the chemical kinetics laws what causes that the intensity of forming increases very significantly as the temperature increases (Fig. 2). Only for the highest exhaust gas temperatures which are hardly ever reached under normal operation conditions, the SO_3 formation is to some extent limited by the thermodynamics reasons. The presence of platinum (platinum is used as a catalyst for the SO_2 to SO_3 oxidation in the sulphuric acid production contact method) in the catalytic layer of the catalyst significantly promotes the oxidation of SO_2 to SO_3 . Much less SO_2 oxidizes in the palladium catalyst, and only a few percent of SO_2 can be oxidized in the rhodium catalyst which is seldom used [Kim and others 1998].

Sulphur trioxide present in exhaust gas is subject to further reactions tending to the formation of sulphates. Firstly, it combines with water forming the sulphuric acid and, in general, it leaves the exhaust system in such a form. Part of the sulphuric acid (perhaps even part of SO_3 itself) can react with hydrocarbons and metals, if any, present in exhaust gas forming the sulphates of higher molecule masses than the sulphuric acid one

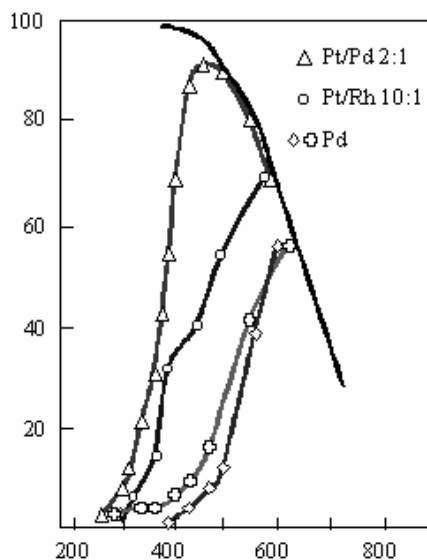
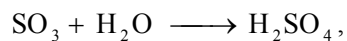
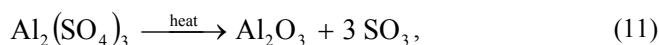


Fig 2. The oxidation of SO_2 to SO_3 in the 5% O_2 presence as a function of temperature for different compositions of the catalyst

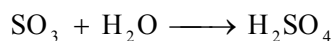
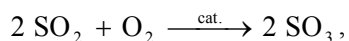
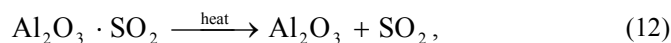
Owing to fact that in some conditions SO_2 or SO_3 can be adsorbed in the catalytic and washcoat layers of the catalytic converter the formation of the sulphates can be of either permanent or periodic nature. In the first case the course of reactions tends immediately towards the formation of the sulphuric acid in the following way:



In the second case sulphur can be stored in a form of SO_2 or sulphates by the washcoat layer of the catalytic converter in accordance to the reactions (1–3) and (5–8). At the specified temperature the stored sulphur can be released from the washcoat layer and form the sulphates according to the following reactions [Henk and others]:



and



The Pt/Al₂O₃ catalytic converters are the most susceptible to the accumulation of the sulphur compounds, whereas considerably smaller amounts of the sulphur compounds are accumulated in the Pt/SiO₂, Pd/Al₂O₃ and Pd/SiO₂ catalysts [Henk and others]. The accumulation of the sulphur compounds in the washcoat layer is the most intensive at the temperatures below 250°C [Zelenka and others]. The release of SO₂ and SO₃ occurs only at high temperatures where the thermodynamic equilibrium is shifted already towards the SO₂ side. Both these facts allow to conclude that the mode of the permanent creation of the sulphates performed according to the reactions (9–10) has the most decisive importance for the sulphates formation in the catalytic converter.

As already stated in this chapter the sulphur compounds can also adsorb onto the noble metal surface. However, due to a small amount of the noble metals in the vehicle catalytic converters, the accumulation and release of the sulphur sulphates from the noble metals is negligible for their total emission [Henk and others].

Owing to a strong temperature dependence of the SO₂ to SO₃ oxidation process and the fact that the sulphates are accumulated in and released from the catalytic converters the serious fluctuations of concentration of the sulphates in exhaust gas can occur. At low temperatures the sulphates emission can be low due to lower efficiency of the SO₂ oxidation and the adsorption in the washcoat layer, whereas at high temperatures the sulphates emission will be significantly increased due to the intensive formation of SO₃ and the release of the sulphates from the washcoat layer. Considering that the deterioration of the catalyst ability to perform the HC/SOF conversion is the most intensive at low temperatures (the most SO₂, strong temperature dependence of the conversion) and the sulphates are created at high temperatures, it can be stated that the indirect effect of sulphur on the PM emission is dominant at low temperatures, whereas the indirect one dominates at high temperatures.

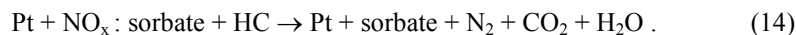
THE EFFECT OF SULPHUR ON OTHER EXHAUST GAS AFTERTREATMENT SYSTEMS

DeNO_x catalytic converter

is designed to reduce NO_x in the oxidizing atmosphere of exhaust gas. It is built on a base of zeolites with platinum as an active layer and is the most effective in a narrow range of temperatures of about 200–300°C [Kandylas and others 1999, Johnson 2000]. To improve the catalytic converter efficiency additional amount of hydrocarbons, as the NO_x reducer, is provided. The NO_x conversion efficiency is dependent on the amount of platinum. In order to reduce the SO₂ oxidation in the presence of platinum, the composition of the active layer is selected in a compromise way and the efficiency of NO_x conversion is reduced. The SO₂ adsorption onto the catalytic layer surface reduces additionally the efficiency of the DeNO_x converter.

A certain type of the DeNO_x catalytic converters are the so called NO_x absorbers (NO_x trap, NO_x sorbate catalyst) i.e. the catalytic converters which are able to accumulate NO_x and then to reduce them to N₂. The complete process proceeds [Parks and others 2000] in accordance with the reactions as follows:





Barium oxide BaO as the NO_x absorber which is used most often, and alternatively other metal oxides are used as well. SO₂ after being oxidized to SO₃ in the presence of Pt reacts with BaO forming barium sulphate which is stable up to the temperature of about 700°C:



and reducing in this way the converter ability to accumulate NO_x and perform their conversion. [Ingram and Surnilla 2002, Izuika and others 2002]. On the other way the oxidation of SO₂ in the presence of platinum Pt results in the increase in the sulphate emission.

Selective Catalytic Reduction (SCR)

is a method of reducing the NO_x emission from the stationary and marine sources. The SCR technology used in the present applications is comparatively strongly sensitive to sulphur contained in fuel. However, to ensure the high conversion efficiency for a wide range of temperatures of exhaust gas from the vehicle diesel engines, it is necessary to equip the SCR system with a catalyst effective at the temperatures below 250°C. The systems of that type are more sensitive to sulphur, especially if there is a platinum-based catalyst ensuring the high NO_x conversion ratio at low temperatures. In this case the mechanics of the adverse effect of sulphur is same as in case of the catalytic converters. Another adverse feature of sulphur contained in fuel in the context of using the SCR system is a risk of formation of ammonium sulphate which when accumulating on the catalyst surface additionally lowers its efficiency [McKinnon 1999, Gieshoff and others 2001].

Catalytic Diesel Particulate Filter (CDPF)

is an effective device for removing both the soluble organic fraction and soot and other particulate as well. The sulphur contained in fuel inhibits the operation of the CDPF in a similar way like the one observed in case of the oxidation catalysts. Additionally, SO₂ increases the temperature at which the burning of accumulated particulates and the regeneration of filter starts [Merkisz and others 2000].

Continuously Regenerating Diesel Particulate Filter (CRDPF)

enables the particulate emission to be reduced by more than 85% with more than 80% efficiency in removing PAH [McKinnon 1999]. The CRDPF filter operates on the following principle: there is a catalyst with the platinum component of its active layer oxidizing NO to NO₂, placed in the exhaust system before the particulate filter. NO₂ obtained in such a way (a strong oxidizer, at low temperatures stronger than oxygen) is used then for burning the particulates accumulated in the filter. The sulphur contained in fuel inhibits the oxidation of NO what results in the reduced efficiency of the exhaust gas treatment by the CRDPF. Moreover, SO₂ contained in the CRDPF is subject to the oxidation and the formation of sulphates takes place, what results in the increase in the PM emission [Hawker and others, Merkisz and others 2000, Zelenka and others 2000].

CONCLUSIONS

As the catalytic exhaust gas aftertreatment systems are commonly used in the compression-ignition engines, the sulphur content is at present the most important normalized „emission” parameter of the diesel fuel. It is also such a parameter which has been recently subject to the most significant changes (Table 2). In the end of the present decade reaching the maximum fuel sulphur contents at a level of some, or over a dozen at the most ppm are expected, i.e. of 10 and 15 ppm in the European Union and the United States respectively. At the moment it is difficult to state explicitly whether the sulphur content will continue to be reduced. Presumably the answer is positive, as even such a small amount of sulphur as 10 ppm still noticeably affects the efficiency of the advanced catalytic exhaust emissions control systems. Taking into account the guidelines of the Worldwide Fuel Charter, the shift of the sulphur content limit downwards to the value of 5 ppm can be expected in the foreseeable future.

Table 2. The evolution of Polish regulations determining the admissible sulphur content in vehicle diesel fuels

Year of implementation	1967	1999	2000	2002	2005	2009
Maximum sulphur content	0.2–1,0 %	0.2 %	500 ppm	350 ppm	10–50 ppm	10 ppm

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