

# Development and identification of a control oriented model of $NO_x$ Storage Catalyst for automotive application

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**Abstract:** The  $NO_x$  Storage Catalyst is currently envisaged to be implemented in light-duty passenger cars for nitrogen oxides reduction, in order to comply with strict emission legislation targets. Since robustness and durability of the engine and emission control system is the first priority in automotive application, to satisfy the need of robust on-board real time monitoring, diagnosis and control, computing efficient methods are needed. In this framework, a control oriented model that describes the dynamics of the main physical-chemical processes within the NSC catalyst, while still maintaining affordable computational burden, has been developed and validated. Model calibration has been performed, for light-duty application, along the NEDC test cycle, by using a statistical-based sub-optimal procedure, based on a parametric analysis which allows identifying the more suitable section of NEDC cycle for model identification, without the need to perform cost- and time- expensive experiments on the engine test bench. The procedure also accounts for missing information and sensors inaccuracies. The great potential of this methodology is the possibility to adopt not optimal designed tests for model parameters identification. The proposed methodology is proven to be effective for real time control strategy, directly embedded in ECU, and provide a sub-optimal but effective strategy for complex models calibration.

*Keywords:* Automotive system identification and modeling, NSC, LNT,  $NO_x$  emissions, exhaust gas after-treatment systems.

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## 1. INTRODUCTION

Emissions from compression ignition engines have been recognized as one of the main sources of air pollution, thus increasing development efforts are required to meet the new extremely restrictive legislation (EU Euro 6/VI or U.S. Tier 2) (Williams and Minjares, 2016; Parliament and the Council of the European Union, 2007). After-treatment technologies, such as  $NO_x$  Storage Catalyst (NSC) or urea based selective catalytic reduction (SCR), coupled with ammonia oxidation catalyst (AMOX), may be added alongside technologies already adopted to meet  $CO$ ,  $HC$ , and  $PM$  limits, including Diesel oxidation catalysts (DOC) and particulate filters (DPF). Beside the  $NH_3$ -SCR process, the  $NO_x$  reduction from burned exhaust gas can be achieved using the cycled  $NO_x$  Storage Catalyst - NSC (also referred as Lean  $NO_x$  Trap - LNT). The NSC catalyst operates in fast lean/rich transients. During the lean steps,  $NO$  and  $NO_2$  are trapped on the basic components of the catalyst. The "saturated" trap is then regenerated during short rich period (of approximately 1 min) in order to reduce the stored  $NO_x$  into  $N_2$ . The control oriented model of NSC is intended to be integrated in an optimal control strategy, embeddable on-board. The optimization of regeneration strategy is aimed

at reaching the trade-off between fuel penalty, resulting from the rich conditions generation, and tailpipe  $NO_x$  slip, especially during transient real driving conditions. Such complex emission control systems will require advanced simulation tools for their cost-effective design, development and system integration. In this framework, control oriented models, implementable in Electronic Control Units (ECUs), can be useful to provide accurate knowledge of main state variables, which can be used in control loops for the management of engine and ATs. Guttenke et al. in (Güthenke et al., 2007) used a detailed global kinetic model of reactants. The authors in (Kočí et al., 2007) developed a global kinetic model in which  $NH_3$ , produced by the reduction of stored  $NO_x$  with  $H_2$ , actively participate as intermediate to reduce the remaining stored or released  $NO_x$ , which could explain the delayed ammonia release (Epling et al., 2004). More advanced model was proposed (Kočí et al., 2013) which considers  $N_2O$  as an additional by-product of  $NH_3$  -  $NO_x$  reaction. In order to accomplish the control task and to infer adaptive features vs. system aging and plant-model mismatch, a control oriented mathematical model that accurately describes the dynamics of the main physical-chemical processes within the NSC catalyst, while still maintaining affordable computational burden, has been developed and validated. The paper is

organized as follows: Section 2 describe the main chemical and mechanistic aspects of NSC catalyst, in section 3 the control oriented model derived from a detailed chemical model is described. In the section 4 an accurate description of a sub-optimal calibration model procedure is detailed and finally some validation results along NEDC type-approval test.

## 2. MECHANISTIC AND CHEMICAL PROCESSES

The  $NO_x$  Storage Catalyst usually contain a noble metal (Pt) allowing the  $NO$  oxidation into  $NO_2$ , and a basic phase (Ba oxide/carbonate) in order to trap  $NO_2$  as nitrite or nitrate intermediates. Both precious metals and storage phase are usually supported on a modified alumina support (Nova and Tronconi, 2014). The NSC operates in fast lean/rich transients. The NSC processes are presented in the figure 1. During the lean steps,  $NO$  in the exhaust gas is oxidized into  $NO_2$  over the precious metals (Pt) and further trapped on the basic components of the catalyst in the form of nitrates or nitrites through the formation of ionic bonds.  $NO_x$  sorption leads to a near complete removal of  $NO_x$  from the gas stream for appreciable periods of time.

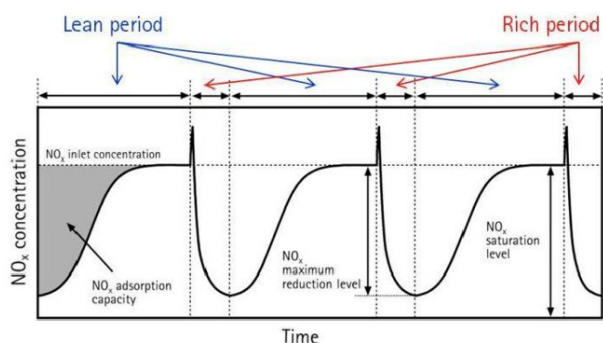


Fig. 1.  $NO_x$  breakthrough and release profile during NSC process. Refer to (Roy and Baiker, 2009)

The trapping ability of the NSC catalyst must be periodically regenerated and the trapped  $NO_x$  reduced to  $N_2$ . To accomplish this, a reducing rich transient (of approximately 1 min) achieved through the introduction of reductant specie is necessary, refer to the short time rich periods represented in the Fig. 1. There are two general methods of introduction of reductants, generally  $H_2$ ,  $CO$  or hydrocarbons (e.g.,  $C_3H_6$  or  $C_3H_8$ ): (i) fuel is directly injected into the exhaust, (ii) fuel is periodically post-processed over an upstream reformer or partial oxidation catalyst.

## 3. NSC MODELING

The trapping efficiency of the NSC Catalyst is a non-linear function of trap temperature, catalyst loading, fuel sulfur content, space velocity, feed gas concentration and trap regeneration frequency. In addition, using a lean A/F ratio for purging allows the trap to be run lean for a longer period of time. But purging at a richer A/F ratio not only has a larger impact on fuel economy, but also increases the likelihood of converting the trapped sulfate to hydrogen sulfide, which would be unacceptable. It is

clear that in order to improve fuel economy and minimize  $NO_x$  emissions, the storage/purge control strategy have to be well designed and optimized. To assist the management strategy development, a control oriented dynamic model of the trap behavior has been developed. The model captures the LNT trapping and purging phenomena and includes the important parameters which affect its behavior. The catalyst devices are basically flow through 3D reactor, with a different conversion rate and temperature for each channel. The suitable modeling should be able to investigate and to represent heat and mass transport in the reactor (e.g. internal and external diffusion, convection and conduction heat transfers) and kinetics of the chemical reactions. The literature widely investigates 1D models, which consider average flow parameters and give characteristic results for the entire catalyst; they are considered successful to calibrate kinetic mechanisms (Depcik and Assanis, 2005). The global reaction model, adopted for the specific application, is derived from the chemical approach proposed by (Rafiq et al., 2017), which considers three main stages: 1-oxygen storage and  $NO$  oxidation, 2- $NO_x$  storage, 3-reduction of stored  $NO_x$ . The global chemical reactions are not reported here for reasons of compactness. In the most general formulation there are three total sites participating in the reaction, modeled in the form of  $BaO$  and differentiated by  $Ba^I$ ,  $Ba^{II}$  and  $Ba^{III}$ , as accurately described by (Rafiq et al., 2017) and (Morandi et al., 2014).

### 3.1 Control oriented model development

The catalyst is described as a series of continuously stirred tank reactors (CSTR) and the model equations are derived by assuming lumped approach for the main thermal fluid dynamics phenomena and chemical kinetics in each section (Arsie et al., 2017, 2018). The kinetic modeling has been accomplished by considering the global reactions rates  $R_i$ , expressed in  $mol/m^3s$ . The reaction rates are assumed to be first order with respect to each reactant concentration and they are modeled by means Arrhenius-like exponential decay formulation 1.

$$Rate_j = A_j \exp \left( \frac{-E_{a,j}}{RT} \right) [C_j]^\alpha [C_y]^\beta \quad (1)$$

No energy balance is solved due to the small temperature gradients when switching from lean to rich conditions. The reason for the small temperature differences is heat losses in the reactor system. Further, the inlet gas temperature is assumed to be adopted for simulating all the catalyst. These assumptions, in addition to the radially symmetric monolith channel, keep the equations of the model 0-dimensional. However, due to the transient nature of the real process, the differential equations describing the process are time dependent. Based on the overall mechanisms occurring in the NSR catalyst, the control oriented model accounts for the following processes: (i)  $NO$  oxidation over Pt and Water gas shift reaction, (ii)  $NO_x$  storage on Ba active site in the form of nitrite or nitrate specie, (iii)  $NO_x$  reduction over Pt and (iv)  $NO_x$  regeneration over Ba. To describe the reduction processes, it is supposed to consider negligible contribution of  $HC$  specie due to the low concentration in the feed stream along the investigated operating conditions and because of the presence of a DOC (devoted to  $HC$  and  $CO$  oxidation) upstream the NSC.

On the Pt catalytic sites, the  $NO$  oxidation and  $NO_x$  reduction processes occur; the oxidation is modeled by one-step reaction; while the  $deNO_x$  process over Pt is modeled by assuming  $NH_3$  and  $CO$  as reductants for  $NO$  and  $NO_2$ . On the Ba catalytic sites, two-site approach is adopted for  $NO_x$  storage and reduction mechanisms, thus the chemical equations related to the third Ba-site are neglected:

- $Ba^I$  mainly devoted to the  $NO_2$  storage in the form of nitrates by means the single-step disproportionation law.
- $Ba^{II}$  which refer to the  $NO_x$  storage in the form of both nitrites and nitrates. Particularly, the second site consider the  $NO_2$  storage as nitrates ( $Ba^{II,a}$ ) and the  $NO$  storage on the catalytic substrate in the form of nitrites ( $Ba^{II,i}$ ).

The interaction between the BaO active sites and the  $NO_x$  in the feed stream is represented by the coverage factor  $\theta_i$ , defined for the two sites as the ratio between adsorbed  $NO$  and/or  $NO_2$  and total adsorption capacities respectively of sites 1 and 2. The resulting set of equations derived from global mass balances for the relevant specie concentrations and storage fractions (refer to Eq. 2).

$$\begin{bmatrix} \dot{C}_j \\ \theta_i \end{bmatrix} = \begin{bmatrix} \dot{Q}(C_{j,out} - C_{j,in}) - \sum_{i=1}^k R_{i,j} n_{i,j} \\ \frac{R_{ads,i} - R_{des,i} - \sum_{i=1}^k R_{i,j} n_{i,j}}{\Omega_i} \end{bmatrix} \quad (2)$$

$R_j^i$  is generic formula for the  $i$ -th chemical reaction rate based on Arrhenius formulation 1, where the  $NO_x$  regeneration process occurring on Ba catalytic sites, involve  $CO$ ,  $H_2$  and  $NH_3$  as reductant for nitrite or nitrate specie adsorbed on first or second Ba sites.

The set of equation defined in 2 takes into account  $NO$  and  $NO_2$  reactions in the form of nitrates and nitrites specie on the site I and II for  $NO$  oxidation,  $NO_x$  adsorption and  $deNO_x$  reactions; it also consider  $H_2$  produced from Water gas shift (WGS) and  $NH_3$  as additional reductants and  $N_2O$  as undesired by-product. The derived NSC twelve-state model exhibits a computational burden that is still not suitable for on-board control application (Schar et al., 2006), thus a further model reduction should be carried out. It can be assumed that the dynamics relating to the storage fractions  $\theta^I$ ,  $\theta^{II,a}$  and  $\theta^{II,i}$  are predominant in the process, then chemical equilibrium can be assumed for the other formation processes. The resulting state equations can be derived by assuming negligible the accumulation terms in the balances for each chemical specie ( $\dot{C}_j = 0$ ). In the three-state model, only the storage dynamic process has to be modeled over time. A total of 41 parameters relating to kinetics constant of reaction rates (pre-exponential terms and Activation Energies) and storage capacity on two Ba sites need to be identified.

#### 4. IDENTIFICATION PROCEDURE

The aim of this section is to present model identification procedure by exploiting experimental tests collected on a commercial NSC catalyst, at the engine test bench, during the typical NEDC type-approval test cycle and therefore, not optimally designed for calibration step. The model calibration consists in a iterative sub-optimal procedure,

able to perform the model parameters tuning by exploiting a generic test transient. The identification procedure is formulated as a constrained optimization problem and it is aimed at evaluating the reaction rate parameters (pre-exponential factors  $A_i$ , the activation energies  $E_i$  and Ba-sites capacities) which minimize the error function expressed by the following equation:

$$J = \frac{1}{N} \sum_{i=1}^N (C_{NO_x,s} - C_{NO_x,r})^2; \quad (3)$$

where  $C_{NO_x,s}$  and  $C_{NO_x,r}$  are respectively predicted and reference  $NO_x$  concentrations of downstream the NSC converter.

The experimental data exploited for model tuning were collected on a commercial  $Pt - Ba/Al_2O_3$  catalyst with honeycomb monolith (400 cps) washcoated, whose geometrical data are listed in Table 1. The catalyst was placed at the exhaust of a light-duty Euro 6 Diesel engine equipped with DOC and NSC systems, along the type-approval NEDC. The global  $NO_x$  concentrations are measured upstream and downstream of the converter by using  $NO_x$  sensors. Nevertheless, in order to exploit the three-state model, the  $NO$  and  $NO_2$  inlet/outlet concentrations are needed; for sake of simplicity and due to the lack of further details, an arbitrary split has been adopted (it is assumed 80% of  $NO$  and 20% of  $NO_2$  in the feed stream). To overcome the drawback deriving from the missing informations, the error function has been evaluated on the total  $NO_x$  concentration predicted by the model (by summing the predicted amount of  $NO$  and  $NO_2$ ). The reductants specie concentration are measured upstream the converter such as temperature profile and volumetric flow rate during the test.

Table 1. Geometrical data of the NSR catalyst.

Diameter [m]	Length [m]	Volume [l]
0.14	0.147	2.3

The proposed control oriented model require the calibration of 41 parameters related to the chemical kinetics of each reaction and total adsorption capacity of each Ba site. It is well-known that, the best practice for model calibration would be to perform TPR test transient and to tune each reaction until the entire process is calibrated (Rafiq et al., 2017; Arsie et al., 2017). The computational burden required to control each inlet specie concentrations and the impossibility to achieve this detailed control on the engine test bench during real-world operation, made this procedure time and cost expensive. Thus, an iterative sub-optimal method for model parameters identification has been developed. The main idea behind the proposed method derives from the alternative operation of the system, thus it is assumed that different macro-phenomena (i.e. Adsorption, Desorption, regeneration etc) can be tuned along selected part of the test transient in which such phenomena prevail compared to the others. To detect the more representative part of the test cycle for such phenomena compared to other, first a sensitivity analysis, is performed over pre-defined split of the test transient; second, the actual identification is implemented. The main tools jointly exploited for this procedure are: box plot statistical distribution and correlation factor. The following main steps describe the proposed procedure:

- Definition of a-priori splitting of driving cycle;
- Definition of a-priori coupling criteria;
- Identification of j-th parameter along i-th transient;
- Box-plot distribution and correlation index;
- Definition of priority criteria;
- Model parameters identification.

The NEDC driving cycle has been arbitrary divided in five different sections on the basis of required load.

- Overall NEDC;
- UDC;
- EUDC;
- Single UDC;
- Intermediate part of EUDC;

The statistical analysis (described in the following paragraphs) allow identifying for each macro-phenomena, the part of the cycle in which the aforementioned phenomenon has a dominant effect and thus the optimal dataset for the official identification procedure (refer to the next section). Different reactions attributable to similar macro-phenomena are grouped, with the aim to simplify the identification procedure (14 macro-processes are defined). Particularly the following processes are grouped:

- $NO_2$  Desorption on Site I and Site II;
- $NO_2$  Adsorption on Site I and Site II;
- $H_2$  based Reduction on Site I and Site II;
- $CO$  based Reduction on Site II,i and Site II,a;
- $NH_3$  based Reductions on Site II,i and Platinum;

The key tools adopted for selecting the optimal dataset along the driving cycle for macro-phenomena identification are: the box plot and correlation index. The box plot is a method for graphically depicting groups of numerical data through their quartiles and it allows to visualize various L-estimators, notably the interquartile range, mid hinge, range. The correlation index defined as in the Eq. 4 is a numerical measure of their linear dependence.

$$R^2(A, B) = -\frac{1}{n-1} \sum_{i=1}^n \left( \frac{A_i - \mu_A}{\sigma_A} \right) \left( \frac{B_i - \mu_B}{\sigma_B} \right) \quad (4)$$

where  $\mu_A$ ,  $\sigma_A$  and  $\mu_B$ ,  $\sigma_B$  are respectively the mean and standard deviation of generic variables A and B.

Each macro-phenomena has been fictitious calibrated along each part of the cycle (originated from the driving cycle splitting). From this identification, a pre-exponential parameter and an activation energy has been achieved for each cycle part, by comparing the outlet concentrations estimated by the control model with those experimentally collected along the test cycle, refer to Fig. 2. The box-plot refers to single reaction or macro-phenomena under investigation and shows statistical distribution for both identified parameters along splitted cycle (i.e. pre-exponential factor -A- and Activation energy -Ea-). Six markers are depicted, they are relating to each cycle part recognized during cycle splitting and a benchmark parameter value provided from literature results (Rafiq et al., 2017). Each marker, is obtained from this preventive identification, which in turn corresponds to a correlation index  $R_i^2$ , Eq. 4. For each chemical phenomena to be calibrated, a table of correlation indices Tab. 2 for both outlet specie  $NO$  and  $NO_2$  and, for each cycle part, can be evaluated.

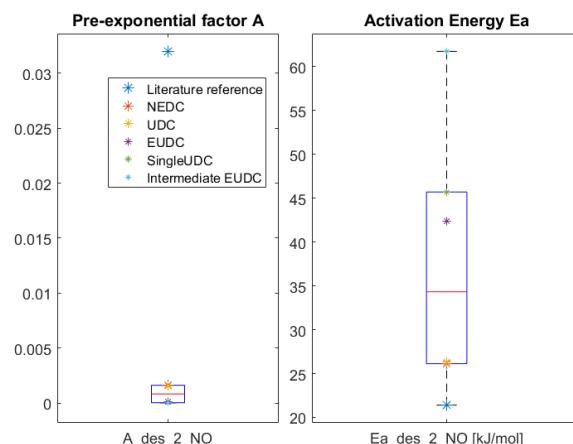


Fig. 2. Box-plot representation for control oriented model calibration. Identification of Arrhenius parameters for  $NO$  desorption on Site II, along each part of the splitted cycle.

Table 2. Correlation coefficients for  $NO_x$  desorption process calibration along different NEDC cycle sections.

Cycle split	$R_{NO_x}^2$
Overall NEDC	0.46
Overall UDC	0.51
Overall EUDC	0.43
Single UDC	0.36
Intermediate EUDC	0.18

The box plot represent the inter-quartile distribution of calibrated parameters and their dispersion for different operating conditions with relating minimum/maximum values. Thus, the most stable and robust processes, show a narrow variability of the parameter, because the optimal values for different dataset are quite similar.

The mid-hinge represents the average behavior of the chemical process along the test transient and it represents a good compromise to optimize calibration on the overall test cycle, thus the NEDC cycle part whose parameter corresponds to the marker nearest the mid-hinge, has been selected as the preferred data-set for the model parameters identification. In addition, jointly to the box plot, the correlation index has been evaluated in this process to average the parameters distribution. Particularly, among parameters contained in the quartile area and close to the median, the section of the NEDC cycle which corresponds to the highest correlation, has been definitively selected. To summarize, to perform the actual parameters identification, the optimal experimental dataset along NEDC cycle has been selected considering the section in which the relating parameter is nearest to the mid-hinge and corresponding to the highest correlation index.

Before model identification has been carried out, a priority criteria based on experience has been established. The main difference between actual identification procedure and the previous sensitivity analysis is the recursive and iterative usage of parameters. During the sensitivity analysis, to avoid contaminating the sensibility process with the previous steps, no iterative parameters update, based on the previous calibration, has been carried out.

Conversely, during the identification procedure, a recursive and iterative process optimization is required, thus each identification is updated with the previous parameters. For the 14 macro-phenomena, deriving from the grouping procedure, a priority criteria has been established to execute the iterative calibration procedure, which performs a recursive optimization of model identification. Firstly Adsorption and Desorption phenomena and total adsorption capacity are tuned because they are not constrained from other processes, furthermore they are process controlling. Then reduction phenomena (by using respectively  $H_2$ ,  $CO$  and  $NH_3$ ) are calibrated. The Water Gas Shift reaction is calibrated one the reduction kinetics from  $CO$  has been evaluated; the WGS reaction is only affected by the  $CO$  inlet concentration. Finally, the Oxidation phenomena are tuned and Reduction processes over Platinum.

Once optimal dataset has been identified from previous sensitivity analysis, model identification can be performed by comparing the outlet concentration predicted by the model with collected data on the experimental engine test bench. To perform the identification procedure, the inlet concentration of the relevant specie measured on the engine test bench, have to be provided as model inputs. Thus the model processes the experimental concentrations measured upstream the catalyst to provide outlet concentrations prediction. The typical  $NO_x$  outlet concentration trends during identification procedure are shown in Fig. 3. The model identification described in this section is a

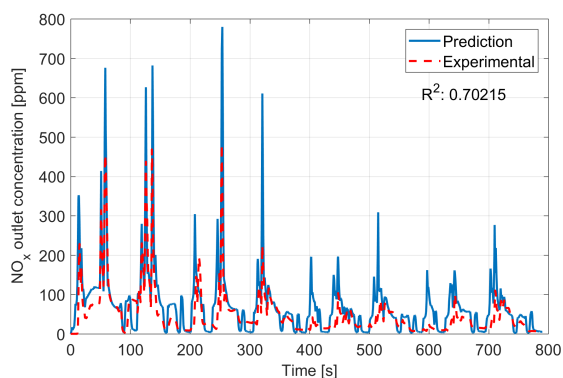


Fig. 3. Identification results.

sub-optimal procedure, able to perform model parameters tuning with a generic test transient, without the need to perform cost- and time- expensive experiments on the engine test bench. Although sensors uncertainty and missing informations, the procedure shows robustness in tuning a quite complex control oriented model, which originally required identification of 41 parameters. Despite having a generic test transient, instead of programmed reactions tests, optimally designed for model tuning, the statistical approach allow achieving good fitting and accuracy in predicting experimental results.

### 5. NSC MODEL RESULTS AND VALIDATION

The NSC model validation has been performed by using experimental data collected along the NEDC type-approval driving cycle. The same driving cycle is adopted

for both model identification and validation, but it is worth remarking that the identification considers limited well-selected sections of the whole test. Several assumptions affect the validation results:

- The inlet and outlet  $NO_x$  concentrations of relevant specie are measured by  $NO_x$  sensors located upstream and downstream the catalyst; the model requires inlet concentration of  $NO$  and  $NO_2$  species to solve the chemical modeling. To figure out this issue an arbitrary split has been assumed, considering 80% of  $NO$  and 20% of  $NO_2$  at the inlet feed stream, while outlet  $NO_x$  concentrations are the result of sum of predicted  $NO$  and  $NO_2$ .
- The temperature trend along NEDC test cycle has been predicted by using the forward vehicle-powertrain model (Arsie et al., 2013).

The exhaust gas temperature range 200-700 degC, outside this range, the chemical kinetics can significantly change and NSC conversion efficiency can decrease due to the slower reaction rate. Model validation results are reported in the Figs. 4.A and 4.B. The first one shows the trend on both predicted and measured outlet  $NO_x$  concentration vs. time and the exhaust gas temperature profile upstream the NSC. The figure in the bottom 4.B shows the cumulative  $NO_x$  emissions vs. time and the vehicle speed profile (refer to the right axis). The results reported in the figure 4

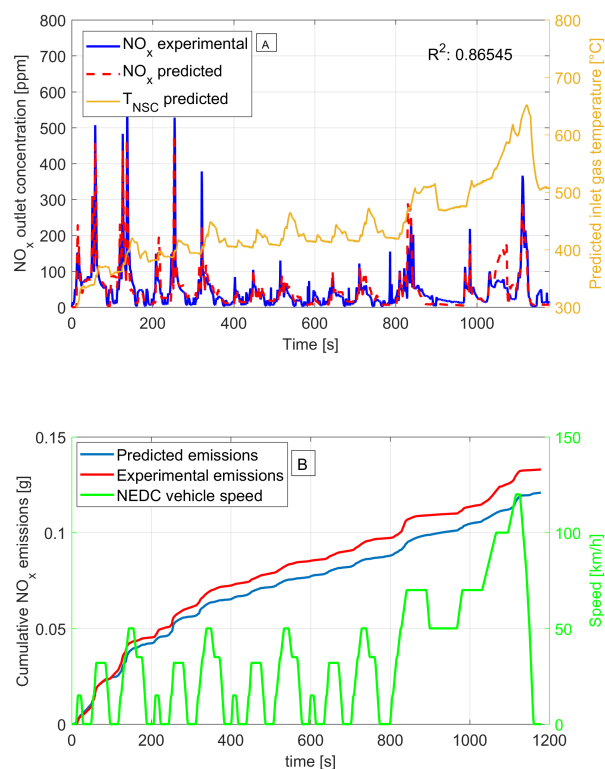


Fig. 4. Validation results along NEDC test cycle. A) predicted and measured outlet  $NO_x$  concentration vs. time and the exhaust gas temperature profile upstream the NSC; B) cumulative  $NO_x$  emissions vs. time and the vehicle speed profile.

show a good prediction accuracy of relevant specie concen-



tration both on instantaneous emissions and cumulative along the test transient ( $RMSE = 30$  ppm). The instantaneous outlet concentration (refer to upper Figure 4) shows a good reactivity of the model during transient conditions, as well. Mismatching conditions are visible especially at time 100 s and 1100 s, both of them are due to suboptimal identification procedure of adsorption/desorption phenomena, which are reaction controller. Particularly, at time 100s the catalyst is filling up, the slight underestimation of adsorption rate affect the outlet  $NO_x$  prediction; while at time 1100 s the temperature increase, the main effects on  $NO_x$  prediction are due to desorption rate. The cumulative predicted and measured mass along the cycle are shown in the lower part of the graph 4.B. Accordingly with the load request, the rapid growth of  $NO_x$  emissions can be seen during the cold start conditions time ranging [0 - 150 s] and at the beginning of EUDC section time ranging [800 - 900 s]. The model slightly under-estimate the emissions. Finally, the emissions in terms of [g/km] can be computed by considering the test distance 11.3 km, thus resulting in a global emissions: 0.0115 [g/km], less than the actual emission limitation.

## 6. CONCLUSIONS AND MAJOR FINDINGS

The present research activity proposes a lumped control oriented model of NSC catalyst suitable to be integrated in a real-time control strategy for emissions reduction. Model calibration has been performed on a light-duty application, along the NEDC test cycle, by exploiting a novel sub-optimal procedure based on box-plot analysis and correlation factors. The potential of this methodology is the possibility to adopt a generic test transient, not optimally designed for model parameters identification, thus avoiding expensive specifically-designed experiments on the engine test bench. The validation results show robustness again plant-model mismatching conditions, missing information and sensor inaccuracies. A percentage variation of 7% has been achieved during validation, while good prediction of dynamic response is proven. The computational burden required from the model is 10.3 s to simulate a complete NEDC test cycle, by using an Intel Core i7-6700HQ processors and a not optimized algorithm; thus it is suitable for control oriented applications if embedded on-board in an Electronic Control Unit.

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