COOP-CT-2004-512912

EXOCAT

Novel Catalyst Converter for Treatment of Engine Exhaust Gases

Co-operative Research Project
Thematic Priority 6: Sustainable Development, Global Change and Ecosystems

Final Activity Report

Period covered**:**	**from 1st November 2004 to 31st January 2007**
Date of preparation:	**17th October 2007**
Start date of project:	**1st November 2004**
Duration:	**27 months**
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Project coordinator organization name:	**EUROPARAMA UAB**

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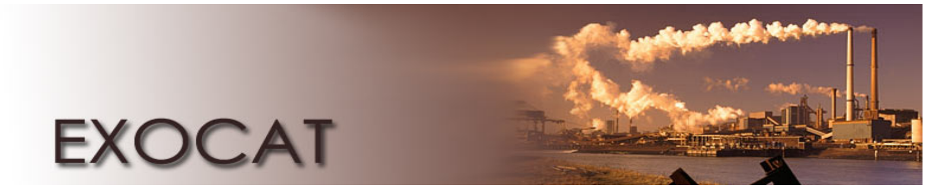
Executive Summary

Novel Catalyst Converter for Treatment of Engine Exhaust Gases



Contract number: COOP-CT-2004-512912

Start date: 01/11/2004

Duration: 27 months

Diesel engines are widely used for on-road (trucks, vans) and non-road applications (agricultural tractors, compressors, bulldozers, off-road trucks, forklift trucks, etc.). Despite a number of advantages, diesel engines have a detrimental environmental downside: relatively high NO_x and particulate matter (soot) emissions, which prompted stringent vehicle emissions regulation, in Europe as well as in Japan and the USA.

Driven by large volumes, current technologies are mainly developed for on-road applications. Indeed, for certain non-road applications they might be technically not feasible or very expensive to use and novel technologies are required to ensure that this diesel non-road mobile machinery are able to comply with environmental legislation. Without exception, the currently available catalyst converters make use of precious (expensive) metals like Pt, Pd and Rh.

The aim of the EXOCAT project was to develop a novel catalyst converter for diesel non-road mobile machinery, based on non-precious metals and by applying Air Spray Coating Technology.

Technical objectives included:

- Reduction of NO_x, and oxidize CO and HC
- Optimisation of the mixture of oxides.
- Improvement of converter performance to Euro III/IV level
- Reduction of the light-off temperature to 100°C.

The work included:

- Selection and testing of prospective cobalt oxides.
- Design of catalyst precursors with alloyed hydroxides, which need to include a mix of metals (Co, Ni, Cu...).
- Selection and optimisation of thermal spraying and its parameters.
- Characterisation of the substrate material and coatings by ageing tests on small lab-scale samples.
- Prototypes of the catalytic block, with a scaled geometry, Furthermore, vehicle roller bench validation of a full-scale converter with new catalytic block prototype will be executed on a forklift truck.

Contractors involved:

	Participant name	Short name	Main contribution
			SME partners
1	Norta UAB	NORTA (LT)	▪ Technical project management ▪ Requirements and specifications for non-road applications ▪ Catalyst precursor development
2	Przedsiębiorstwo Produkcyjno-Handlowo-Usługowe 'ZREMBUD' Sp. z o.o.	ZREMBUD (PL)	▪ Technical specification with focus on installation and service aspects ▪ Field evaluations on logistics and technical feasibility ▪ Dissemination in forklift and other activities such as automotive and generator, and etc. service industries
3	GTV UAB	GTV (LT)	▪ Responsible for corrugation/rolling technology and equipment development ▪ System prototype assembly and pre-testing
4	Metallisation Ltd.	METALLISATION (UK)	▪ Thermal spraying activities ▪ Spraying process monitoring
5	BERSY S.R.L. UNIPERSONALE	BERSY (I)	▪ System specification and manufacturing monitoring.
6	Europarama UAB (Coordinator)	EP (LT)	▪ Non-technical project management ▪ Project and Consortium management
7	Finn Katalyt Ltd.	FINNKAT (FI)	▪ Sales, marketing, exploitation and distribution strategy
			RTD partners
8	Consejo Superior De Investigaciones Cientificas	CSIC (E)	▪ Catalytic composition analysis and evaluation
9	Kauno Technologijos Universitetas	KTU (LT)	▪ Laboratory testing of catalytic activity, selectivity, thermostability and poisoning of catalytic coatings during precursor development.
10	Centro Ricerche Fiat Società Consortile per Azioni	CRF (I)	▪ Emissions after treatment characterization tests ▪ Evaluation of the performance of metal substrates and catalytic layers provided by the partners, accelerated ageing tests in accordance with standard procedures for testing

Results achieved:

- Developed prototype of industrial scale installation for powder precursor production
- Developed method of catalytic coating manufacturing
- Developed industrial scale machine prototype for corrugation of thermally sprayed metal strip
- Developed catalytic elements

The main aim of the Project is not fully achieved – NOx reduction in catalytic elements is about 5-6% instead of 25-30% needed for commercial application in **automotive** industry. However, some applications in the area, where the total volume of catalytic elements is not critical, such as **diesel locomotives** and **ship diesel engines** appear possible.

The most important advantage of developed catalytic elements is their low cost compared to traditional catalytic elements. The cost of catalytic elements strongly depends of cost of materials, where cost of metal substrate with noble metal catalyst come to 80-90% of total cost.

Developed technology gives unique opportunity to decrease not only cost of catalyst (replacing noble metals with cobalt oxides), but also replace very expensive metal substrate – steel strip made from special high alloyed chromium-aluminum steel – on other, more cheap, steel compositions. Developed technology of Soft Plasma Spraying permits to get very good adhesion to any steel substrate.

PROJECT OBJECTIVES AND MAJOR ACHIEVEMENTS

The aim of the EXOCAT project was to develop a novel catalytic block for catalyst converter for diesel non-road mobile machinery, by applying Air Spray Coating Technology and making use of non-precious metals.

Brief description of the manufacturing process (demonstrated on laboratory scale).The substrate is commonly used steel strip with a thickness of ~50 µm, coated by plasma spraying on both sides, with the catalytic layer thickness ~20 µm, on the basis of gamma-alumina. So the total thickness of the material, depending on the steel strip thickness is in the order of 70 µm. The catalytic layer is based on a complex mixture of aluminum oxides, mainly gamma- and theta-alumina, with a thin film of complex oxides of non-precious metals on its surface. The catalytic layer after coating is in a non-activated condition, and its adhesion to the metal substrate is so high, that it permits any mechanical treatment, such as cutting, cold stamping, corrugation, etc. without any deterioration of the catalytic layer. After this basic Air Spray Coating process only thermal activation of the catalytic layer is needed, which requires a temperature range of 400 - 750°C in a common kiln.

The overall objective was to improve the current laboratory process further, and to manufacture an industrial prototype.

Technical objectives included:

- Reduction of NO_x, and oxidize CO and HC
- Optimisation of the mixture of oxides
- Improvement of converter performance to Euro III/IV level
- Reduction of the light-off temperature to 100°C. Light-off temperature (temperature where the catalyst starts converting) is important as to keep the pollution after start of the motor as low as reasonably achievable. Light-off temperature of the laboratory prototype was 175°C. Untreated exhaust emitted at the start of the legislated emissions test and on short journeys in the real world had to be curtailed. Existing catalysts only starts working in the 250°C range.

The work includes:

- Selection and testing of prospective cobalt oxides.
- Design of catalyst precursors with alloyed hydroxides, which need to include a mix of metals (Co, Ni, Cu...).
- Selection and optimisation of thermal spraying and its parameters.
- Characterisation of the substrate material and coatings by ageing tests on small lab-scale samples.
- Prototypes of the catalytic block, with a scaled geometry, will be designed, manufactured and tested.
- Furthermore, vehicle roller bench validation of a full-scale converter with new catalytic block prototype will be executed on a forklift truck.

Startegic project objectives

Objective	Milestone/Deliverable	Month for delivery
▪ To identify the points for improvement of currently available technology and to prepare sample catalytic coatings	Detailed Project Plan prepared.	3
	Functional requirements and specifications complete.	3
	Two most prospective oxide systems selected.	9
	Coating controls established	12
▪ To design catalytic block; ▪ To manufacture and test prototypes of promising concepts with a scaled geometry	Thermal spraying tools and method developed	12
	Coating characterization completed	27
	Prototype designed.	27
	Engine steady state tests performed and evaluated	27
▪ Prepare for dissemination EXOCAT achievements through the targeted potential clients/market segments and preparation of plans for future exploitation.	Draft Plan for use and dissemination of knowledge	27
	Future exploitation plans established.	27
	Website	27

The aim of the project was to develop a novel catalytic block for catalyst converter for diesel non-road machinery by applying thermal spray coating technology and making use of non-precious metals. The work performed during the project was divided into following work packages:

- WP1 – Requirements and specifications for non-road applications
- WP2 – Catalyst properties
- WP3 – Catalyst precursor
- WP4 – Thermal spraying
- WP5 – Coating characterization
- WP6 – Catalytic block design
- WP7 – Catalytic block bench tests
- WP8 – Dissemination and future exploitation
- WP9 – Project and consortium management

Graphical view of listed work packages is listed in a figure below.

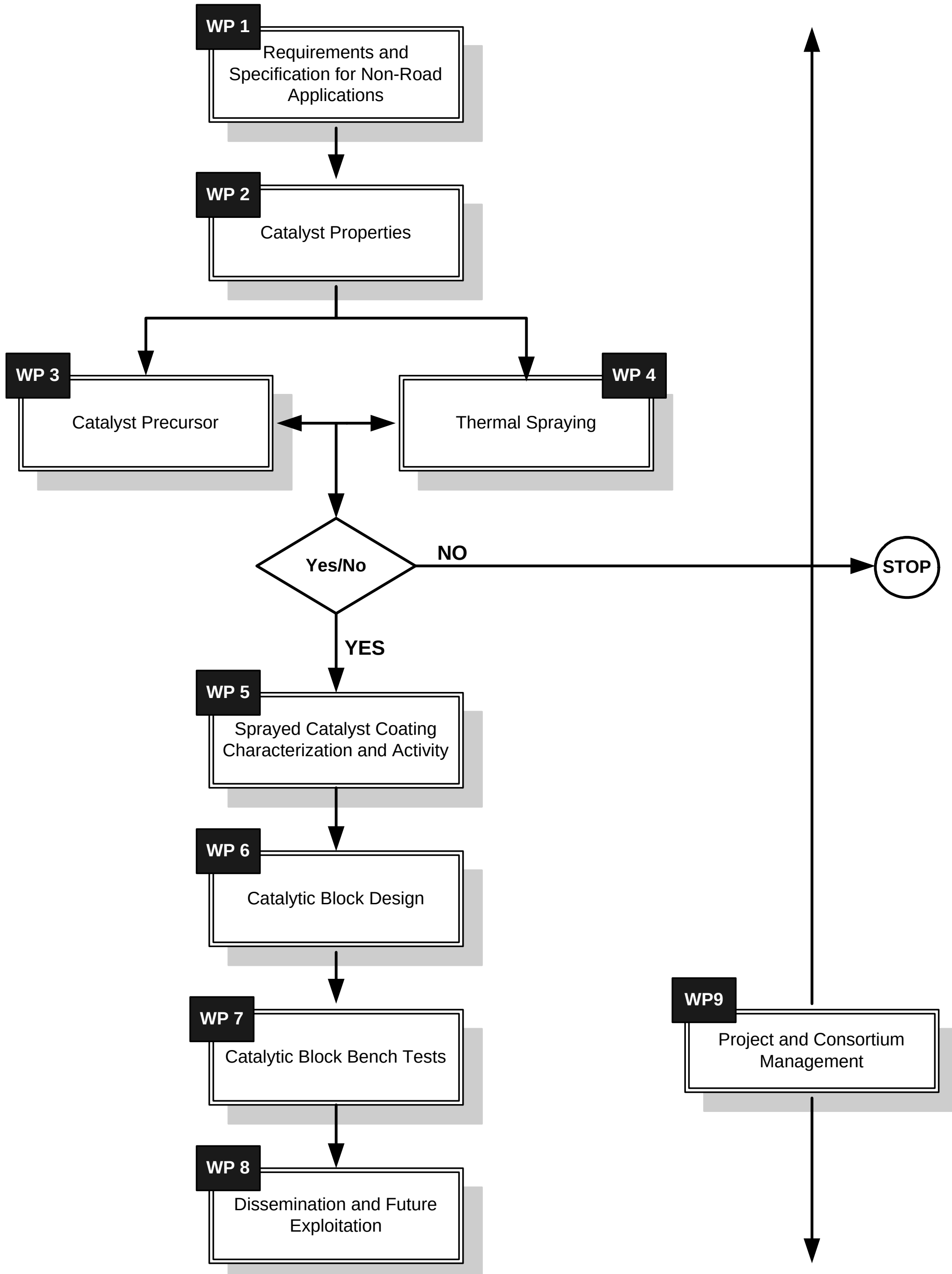


Figure 1: Project flow diagram

1.1 SHORT DESCRIPTION OF WORK PACKAGES IMPLEMENTATION

WP1: (Requirements and specifications for non road applications) was completed successfully.

- Detailed scope of the Project was developed (*Deliverable 1.1:* Project Plan (Detailed))
- The functional requirements and specification for catalytic elements for non-road diesel engine exhaust gases refining were defined (*Deliverable 1.2*: Functional requirements and specifications)

WP2: (Catalyst properties) was completed successfully.

- Evaluation of catalytic activity, selectivity and resistance was done (*Deliverable 2.1*: Report on catalytic activity, selectivity and resistance and *Attachment to deliverable* D2.1)

WP3 (Catalyst precursor) was completed successfully.

- A model of composite powder precursor (three variants) was developed
- Three variants of technology (lab-scale) to produce these precursors (*Deliverable 3.1:* Optimized precursor) were developed
- Prototype of industrial scale installation (*Deliverable 3.3:* Prototype of industrial scale installation for precursor) for powder precursor production was developed and produced
- Precursor properties (*Deliverable 3.2:* Report on precursor properties) after isothermal decomposition were investigated in comparison with hydrotalcite data
- Tests showed that catalytic properties of precursors, developed by NORTA, are at least 30% higher, than catalytic properties of hydrotalcite itself.

WP4 (Thermal spraying) was completed successfully**.**

- Detailed analysis of existing thermal spraying tools and methods was done *(Deliverable 4.1*: Thermal spraying tools and method)
- In WP4 was supposed to use HVOF process (resp. partner METALLISATION) for effective transportation (without damage in gas stream) of particles of precursor, developed in WP3. The plasma spraying process (NORTA) was supposed to use only for comparison, because this process is well known and can be characterized by considerable damage of precursor particles during their transportation
- First tests on precursor (model hydrotalcite composition) HVOF spraying (resp. partner METALLISATION) gave positive results and showed that the mass of particles should be increased
- Next tests on second variant of precursor (developed by NORTA in WP3) HVOF spraying (resp. partner METALLISATION) showed that the increase of mass for precursor particles is not enough for effective penetration into intermediate layer.
- Taking into account received data, NORTA has developed third variant of precursor with aluminum core (to increase the mass of particle), but according to insufficient technical quality of results of the first two variants it was proposed to use spraying technology available to NORTA. METALLISATION has stopped their work in WP4 and their tasks on thermal spraying tools development were overtaken by NORTA
- NORTA has developed a new technology – Soft Plasma Spraying – which was an attempt to replace HVOF spraying in the Project. Soft Spraying Technology includes two steps:
  - Soft Plasma Spraying process
  - Restoration of coating surface, damaged during the spraying

WP5 (Sprayed catalytic coating characterization) was completed successfully.

- The samples, produced in WP4 according Soft Spraying Technology, were characterized by BET, SEM, X-ray and adhesion tests (*Deliverable 5.1:* Report on coating characterization and *Deliverable 5.2:* Report on laboratory reactor experiments)
- Spraying (WP4) and testing the samples (WP5) fulfilled by multi-stage iteration, realized in catalytic coating with needed chemical composition (cobalt hydrotalcite), and well formed macro- and microstructure and good adhesion to substrate

WP6 (Catalytic block design) was completed successfully.

- Design of catalytic block was developed.
- Technology of corrugation and rolling for sprayed (WP4) strip was developed.
- Prototypes of industrial scale installations for corrugation sprayed strip, its rolling and fastening of catalytic block were developed and produced (*Deliverable 6.1*: Prototype design and *Deliverable 6.2*: Prototype)

WP7 (Catalytic block bench tests) was not totally completed.

- About 20 variants of catalytic blocks were produced by NORTA by Soft Plasma Spraying method. These blocks were preliminary tested in KTU on their test bench (to determine the best samples for further testing in CRF).
- After preliminary tests (KTU) with low velocity (2,7 l/min) exhaust gases the most prospective variants of catalytic blocks were selected (Fig. 2 and 3) :
  - Cobalt hydrotalcite (P5-NCT-OG and P5-NCT-BG)
  - Cobalt hydrotalcite with vanadium (B-75-V-55)
  - Block including two different catalytic strips; one with catalyst for CO oxidation, other with catalyst for NOx reduction (P6-PFT-OG)
  - Zirconium based catalyst (P7-1291/1-Cu)

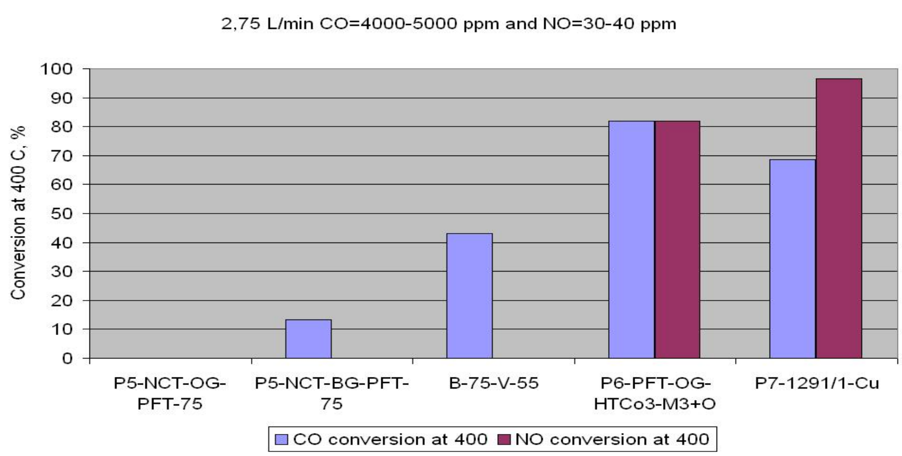


Figure 2: The most prospective variants of catalytic blocks tested at 400 C

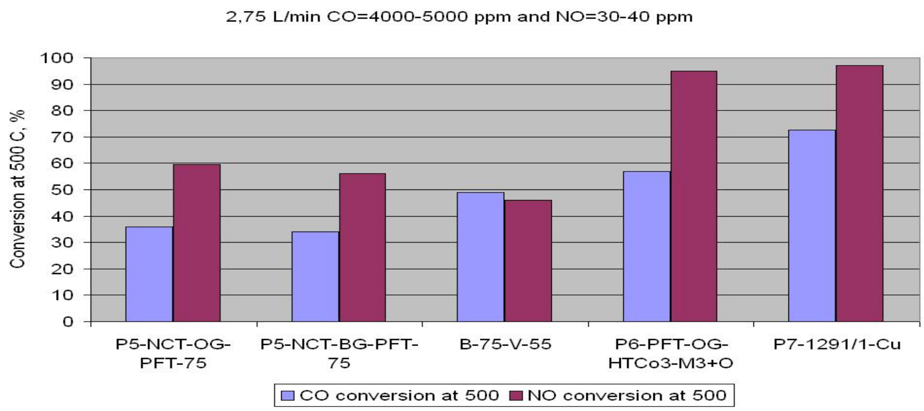


Figure 3: The most prospective variants of catalytic blocks tested at 500 C

- Testing in CRF showed the following (*Deliverable 7.1:* Laboratory test report):
  - Maximal NOx conversion is on the level of 6-8%
  - Temperature of "lighting" in CO oxidation reaction is about 300 C
- According to results received from CRF on catalytic blocks testing it was decided by the consortium to not perform engine steady state tests (*Deliverable 7.2:* Perforemance report on engine steady state tests – was not delivered) because of low NOx conversion rates measured during laboratory testing.

WP8 (Dissemination and future exploitation) was completed successfully.

- All foreseen deliverables were successfully delivered to the Commission (*Deliverable 8.1:* Website; *Deliverable 8.2:* Application notes; *Deliverable 8.3:* Publications; *Deliverable 8.4* Plan for use and dissemination of knowledge)

WP9 (Project & Consortium management) was completed successfully.

Conclusions on implementation of EXOCAT Project:

- Tests in CRF showed, that catalytic elements, developed in the project, can not be applied for automotive applications because of low (6-8%) NOx reduction capacity
- From other side, these catalytic elements possibly can be applied for other non-road application, such as **diesel locomotive** and **trains**, where the requirements to volume of catalytic elements are not so rigid. It can be proved by KTU test results, where NOx conversion reached 90%
- Very important result is that during the project the **complex of industrial scale technologies** were developed, including production of precursor, spraying (Soft Plasma Spraying), technology of mechanical treatment (corrugation, rolling and fastening of catalytic element) and technology of thermal activation.
- The prototypes of **industrial scale installation** (installation for precursor production, installation for corrugation and for rolling and fastening) are developed.

SECTION 2 – WORKPACKAGE IMPLEMENTATION AND DEVIATIONS DURING THE PROJECT

This section covers technical work implemented during entire duration of the project. In separate subsection - Major deviations - cumulative tables with deliverables, their foreseen and actual delivery dates, used person months and explanations on occurred deviations are provided.

WORKPACKAGE 1 - REQUIREMENTS AND SPECIFICATION FOR NON-ROAD APPLICATIONS

Responsible partner: NORTA

Other partners involved: CRF, METALLISATION, CSIC, KTU, FINNKAT, BERSY, EP, ZREMBUD,

Objectives:

- Ensuring correct project direction/definition and efficiency of the project

Main tasks:

Task 1.1. The developing of detailed scope of the Project.

Task 1.2. The functional requirements and specification should determine and formulated.

Implementation of tasks:

Task 1.1. The state-of-art for earlier available technical solutions were analyzed and discussed between all partners (*Attachment to deliverable D1.1:* Project plan (detailed)*)*. New concept of NOx localization during absorption/reduction and simultaneous soot oxidation in small volume has been developed. The design of catalytic element for these processes and new concept of technology for manufacturing of new type catalytic elements has been proposed. For realization these tasks during the Project the Work Plan was developed (*Deliverable D1.1:* Project Plan (detailed).

Task 1.2. The functional requirements and specification for catalytic elements for non-road diesel engine exhaust gases refining was developed (*Deliverable D1.2:* Functional requirements and specifications). The main components of exhaust gases for diesel engine, such as carbon monoxide, hydrocarbons, nitrogen oxides and particulate matter were selected and their influence in spite of current and future legislation was analyzed. The fulfilled analysis showed, that the first aim, that should be realized in the frame of the Project to meet stage IIIA standards should be NO_x content reduction on approximately 25-30%.

Status:

WP# 1 was fulfilled completely.

Deliverables List:

D. No.	Deliverable name	Delivery date	Actual delivery date	Estimated indicative person-months	Used indicative person-months	Lead contractor
D1.1	Project plan (detailed)	3	3	7,49	7,71	NORTA
D1.2	Report on economical evaluation	3	3	7,5	7,72	NORTA

Explanations on major deviations:

During this work package no significant deviations have occurred.

WORKPACKAGE 2 - CATALYST PROPERTIES

Responsible participant: CSIC
Other partners involved: NORTA, METALLISATION, EP, KTU
Objectives:

- To develop optimal catalyst composition on the basis of cobalt content hydrotalcites

Main tasks:

Task 2.1. DeNOx catalytic activity
Task 2.2. Oxidative properties
Task 2.3. Sulphur resistance
Task 2.4. Catalyst characterization
Task 2.5. Optimization

Implementation of tasks:

Task 2.1. In order to test the catalytic activity for the NO removal, Cu(II)Mg(II)Al(III) and Co(II)Mg(II)Al(III) hydrotalcites were prepared by a standard co-precipitating procedure. The hydrotalcites were calcined at 650ºC in air for 3 hours before reaction, obtaining a mixed oxide. Some samples were impregnated with hexachloroplatinic acid and/or palladium nitrate and calcined at 550ºC for 6 hours in order to prepare some catalysts based on hydrotalcite and with a 1% wt platinum and/ or 1% wt palladium (*see Deliverable 2.1*:Report on catalytic activity, selectivity and resistance)

The DeNOx catalytic activity experiments were carried out in a fixed bed, quartz tubular reactor where 1g of catalyst was introduced in the reactor and was heated up to 450ºC under nitrogen flow. At this temperature the flow was maintained for 2 hours. After that, the desired reaction temperature was set and the reaction feed admitted. This consists of 650 ml.min^{-1} of a mixture composed by NO, C_3H_8, oxygen and balanced with nitrogen. The NO_x present in the outlet gases from the reactor were analyzed continuously by means of a chemiluminescence detector. The different samples were tested at different temperatures from 100 to 400ºC and at different reaction cycles.

Initially the activity for the NO removal of the catalyst based on copper hydrotalcite and that of the catalyst based on cobalt hydrotalcite were compared. In all range of the temperatures tested the activity of the catalyst based in Co-hydrotalcite is better than that of the Cu-hydrotalcite, but the activity obtained was no so high. It is known that in order to increase the activity, the presence of Pt or Pd is positive, for this reason some samples with 1% wt. Pt, 1% wt. Pd and 1% wt. Pt-1% wt Pd were prepared and tested in the reaction. The difference is quite significant at 100ºC with the Pt-catalyst, but at higher temperatures it is not so important, obtaining almost the same results with the Pt or Pd catalyst than with the Co-hydrotalcite without noble metals. The combination of Pt and Pd does no result in an improvement of the catalysts activity if compared with the catalyst with only one of these metals.

Some hydrotalcites with different cobalt content were also prepared. The samples with higher cobalt content show the best activity. This activity is even better than that of the catalyst with 5% Co and Pt. The sample with 10% Co shows the best activity at 100ºC, but at higher temperatures the hydrotalcite with 15 % Co is the best.

On the hydrotalcite with 15% cobalt, it was studied the influence of the catalyst activation. The activity of the catalyst increases significantly at any temperatures if the catalyst is activated with hydrogen at 450 ºC instead of nitrogen.

The results obtained from the catalysts tested, suggest that the best activity for the NO removal is achieved with the hydrotalcites with higher cobalt content: 10 % Co-Hydrotalcite and 15 CoHydrotalcite actived with hydrogen.

Task 2.2. In order to test the oxidative properties of the catalysts, two different experiments were made. In the first one a flow of 650 ml/min with a composition of 2% C_3H_8, 8% O_2, 900 ppm of NO and N_2 as gas balance was supplied to the catalyst at 100ºC. The temperature was increased at 5º/min from 100ºC to 700ºC, analysing the CO_2 formed (*see Deliverable 2.1:* Report on catalytic activity, selectivity and resistance). The oxidation capacity of the catalysts was evaluated determining the temperature at which half of the propane introduced in the feed was oxidized (T_{50}). The results obtained shown that the 10 Co-Ht and the 15 Co-Ht catalyse the oxidation of the propane, obtaining a T_{50} 100ºC lower than the blank, being the catalyst with higher cobalt content more active than the other.

In the second type of the oxidative experiments the samples were mixed with black carbon (soot) and the temperature of the samples was increased at 5ºC per minute from room temperature up to 750ºC in presence of air, determining the temperature at which the soot starts to burn (*see Attachment to Deliverable 2.1:* Report on catalytic activity, selectivity and resistance). The hydrotalcite with 10% Co and that with 15% Co catalyse the oxidation of the soot, starting the soot ignition with the Co-hydrotalcites 100ºC lower than with the blank, being the catalyst with higher cobalt content more active. In addition we test some samples supplied by Norta based on the precursor, this is the sample with 15% cobalt deposited on the surface of the core (gibbsite) and we have test it, obtaining with this sample the best results probably because a better dispersion of the metals or due to an additional catalytic effect of the gibbsite.

Task 2.3. The resistance of the cobalt hydrotalcites to water and sulphur was tested adding to the gas flue different quantities of water and SO_2. It was observed that there was no any important change in the catalysts oxidation or reduction activity of the 15 Co-Ht in presence of 15% water and 30 ppm of SO_2. This indicates a high stability of the material.

Task 2.4. In order to characterize the samples, different techniques were used: XRD, diffuse reflectance in the UV-visible region, chemical composition and surface area analysis.

The surface area study shows that all the catalysts tested have areas up to 140 m^2/g. The highest surface areas correspond with the catalysts with the lowest copper or cobalt content, decreasing the surface area when the quantity of the transient metal increases.

The XRD study shows that before calcinations, the samples present the typical X-ray diffractogram of a hydrotalcite. However after calcinations at 650 ºC, it can be observed the formation of magnesium oxide poorly crystalline. The observed peaks are shifted to higher angles if compared with a pure magnesium oxide. This is consequence of the incorporation of aluminium in the framework of the MgO, resulting in the formation of a mixed oxide. No peaks assigned to the transition metal oxide are observed, even in those with higher cobalt content, indicating a better dispersion of the transition metal in the matrix.

The diffuse reflectance spectroscopy measurements of the cobalt hydrotalcites show the presence of different bands that indicates the presence of tetrahedral Co^{2+}.

From the characterization results, it can be concluded that Co-Al-Mg oxide derived from hydrotalcites, are catalysts with high surface area. In these samples, cobalt seems to be present not only as isolated and well dispersed paramagnetic ions, but also as some kind of very small Co-containing particles

Task 2.5 The results obtained from the catalytic tests, suggest that the best activity for the NO removal and for the COVs and CO oxidation is achieved with the hydrotalcites with higher cobalt content: 10 % Co-Hydrotalcite and 15 Co-Hydrotalcite. These are the optimised catalyst formulation that it is passed to the partners.

Status

WP# 2 was fulfilled completely.

Deliverables List:

D. No.	Deliverable name	Delivery date	Actual delivery date	Estimated indicative person-months	Used indicative person-months	Lead contractor
D2.1	Report on catalytic activity, selectivity and resistance	14	14	22,37	27,82	CSIC

Milestones list:

Milestone no.	Milestone name	Workpackage no.	Date due	Actual/Forecast delivery date	Lead contractor
M2.1	Two most prospective oxide systems selected and developed	2	14	14	CSIC

WORKPACKAGE 3 - CATALYST PRECURSOR

Responsible participant: NORTA
Other partners involved: GTV, METALLISATION, EP, FINNKAT, CSIC, KTU
Objectives:

- To investigate specific precursors for different thermal spraying methods

Main tasks:

Task 3.1. Precursor development
Task 3.2. Precursor decomposition research
Task 3.3. Complex precursor development
Task 3.4. Coating deposition and optimization

Implementation of tasks:

Task 3.1. During the work in WP2 (*see Deliverable 2.1:* Report on catalytic activity, selectivity and resistance) copper – Cu(II)Mg(II)Al(III) and cobalt – Co(II)Mg(II)Al(III) hydrotalcites with different content of copper and cobalt were developed by CSIC. The copper content in hydrotalcites was 5%, the cobalt content was 5, 10 and 15%. Synthesized catalysts were tested in reaction of NO reducing and hydrotalcite catalysts with cobalt content of 15% demonstrated the best results. The hydrotalcite with cobalt content of 15% was selected for development of precursor.

Development of the precursor included reproducing and testing the samples of hydrotalcites, selected by CSIC (WP2).

For this purpose NORTA produced a wide range of cobalt hydrotalcites with cobalt content of 10, 15, 25, 50 and 100% (Table 1) and sent them to CSIC to make a comparison with hydrotalcites, developed by CSIC.

Table 1: Specific surface area measurement of cobalt hydrotalcities with different cobalt content

Cobalt concentration in hydrotalcite, %	Marking	Specific surface area in synthesized condition, m^2/g
10	HTCo3-M4	54,8
15	HTCo3-M3	45,4
25	HTCo3-M2	72,2
50	HTCo3-M1	20,6
100	HTCo3	104,6

The test results demonstrated a full coincidence of hydrotalcites, reproduced by NORTA with standard samples (CSIC). According the work plan NORTA has developed three variants of precursor on the basis of CSIC development (cobalt content hydrotalcite). The first variant is hydrotalcite itself with particle dimensions less then 20 microns. Second variant was compositional precursor with gibbsite or boehmite core and hydrotalcite coating and the third variant was compositional precursor with aluminum core, intermediate layer of gibbsite, boehmite and hydrotalcite coating.

The precipitation technology, developed by CSIC, was modified to increase adhesion of the hydrotalcite coating to precursor core. NORTA produced the precursor (on the basis of CSIC development) where the coating was 15% Co hydrotalcite. These precursors were planned to be sprayed by thermal spraying in mutual tests at METALLISATION. Precursors were sent to CSIC for control its chemical and phase composition.

Task 3.2. Produced by NORTA hydrotalcites were investigated by thermal analysis, X-ray and BET method in KTU. Results are presented in the Fig.4 – 12.

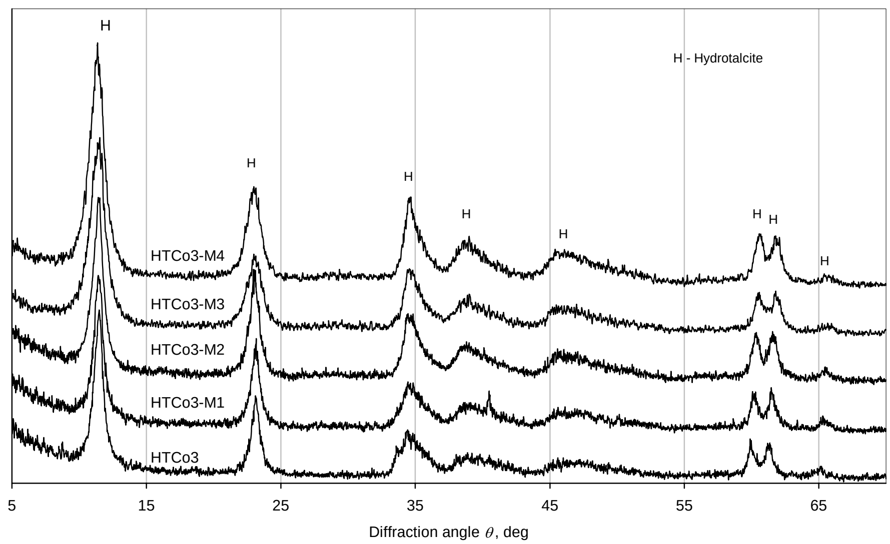


Figure 4: XRD of synthesized hydrotalcites.

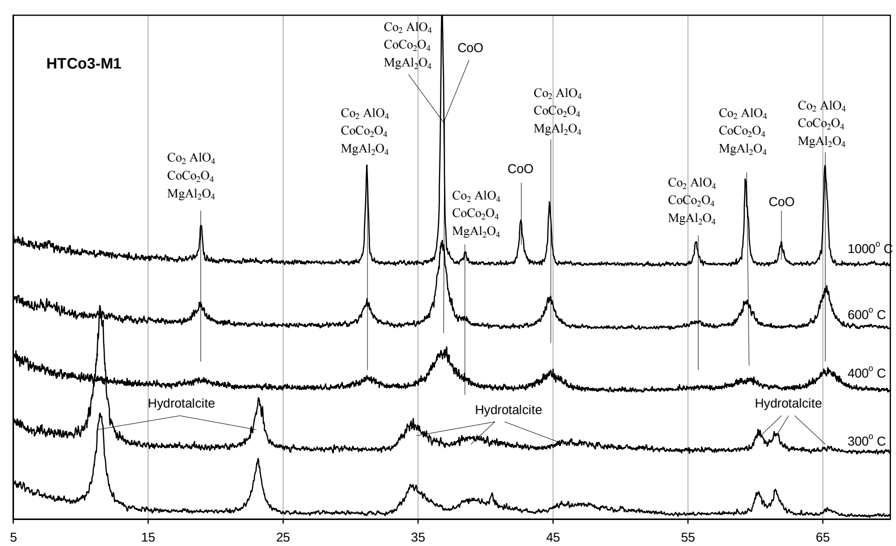


Figure 5: Thermal decomposition of hydrotalcite HTCo3-M1 (50% of cobalt)

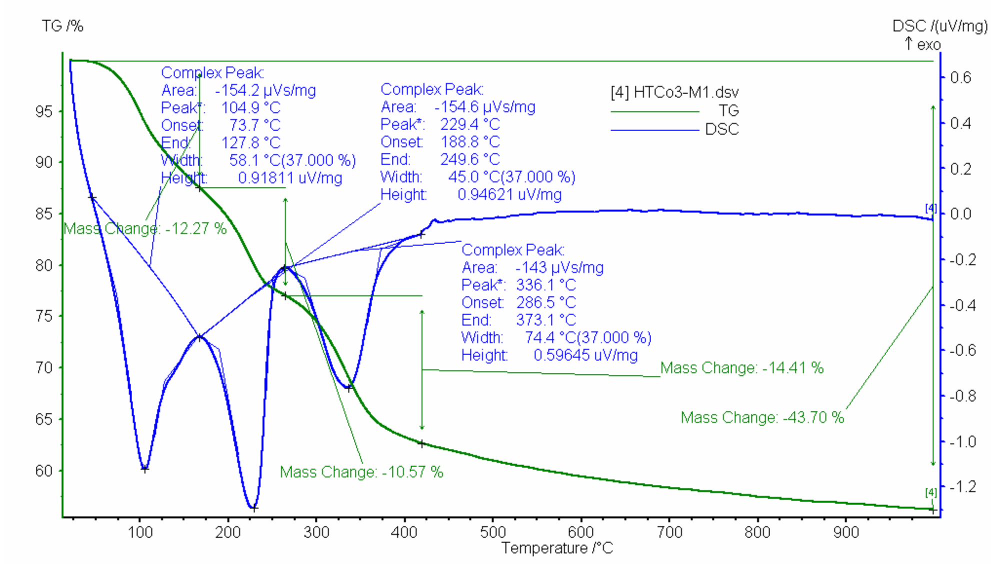


Figure 6: Differential scanning calorimetry–DSC and thermogravimetry–TG of hydrotalcite HTCo3-M1 (50% of cobalt).

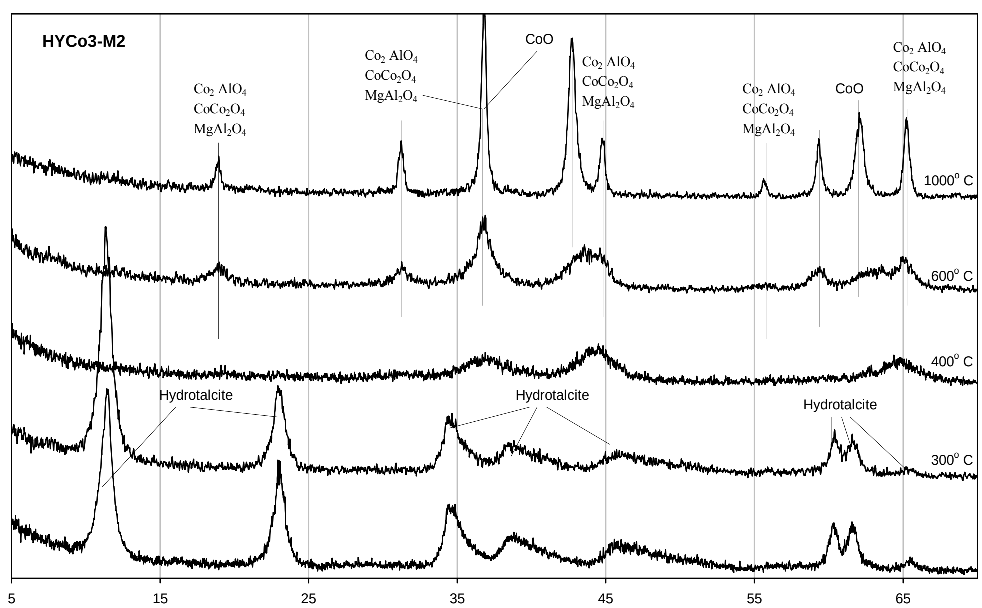


Figure 7: Thermal decomposition of hydrotalcite HTCo3-M2 (25% of cobalt).

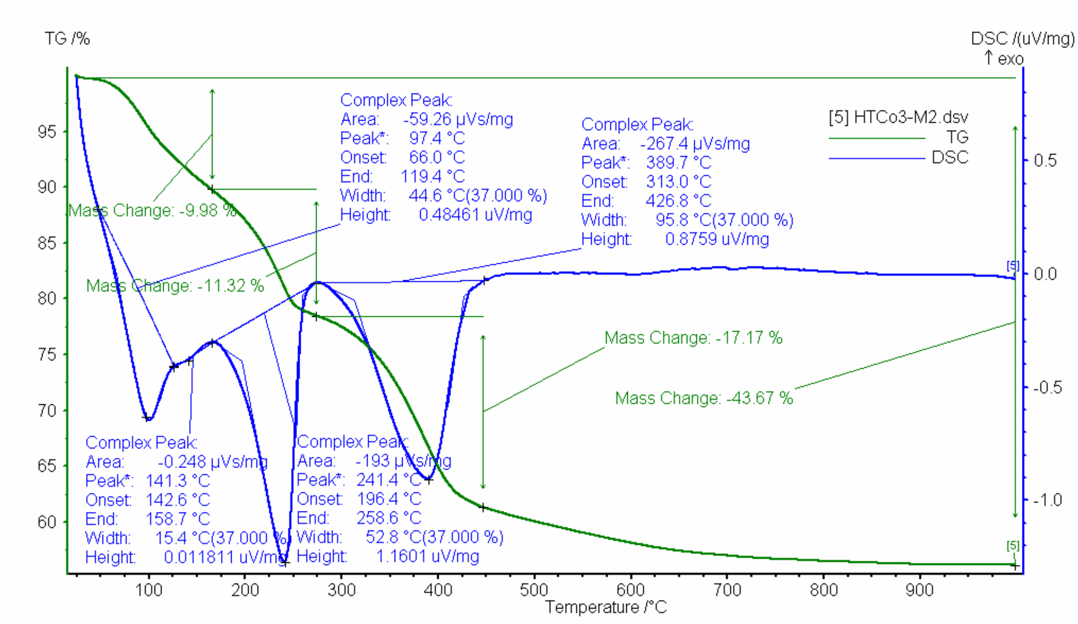


Figure 8: DSC and TG of hydrotalcite HTCo3-M2 (25% of cobalt)

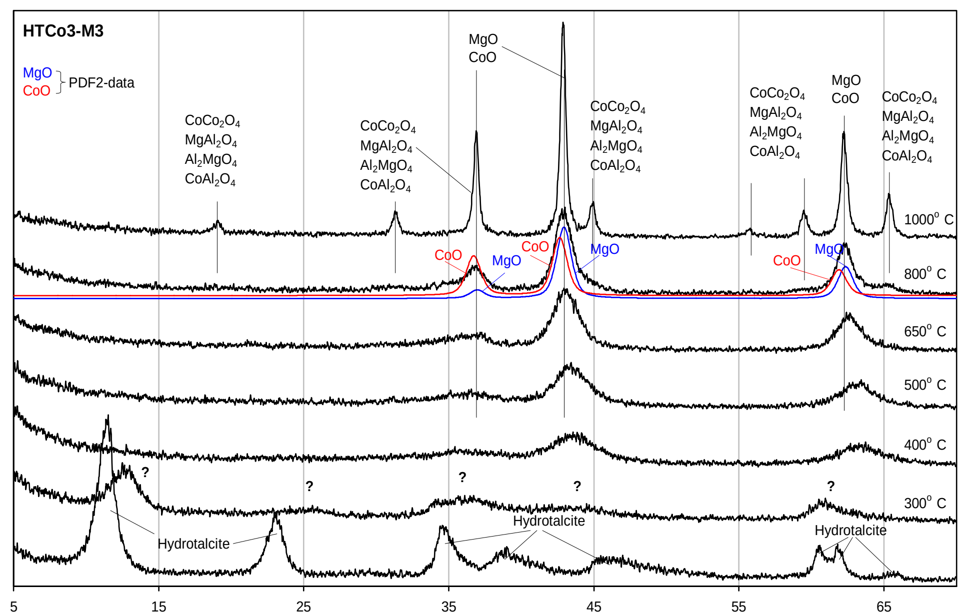


Figure 9: Thermal decomposition of hydrotalcite HTCo3-M3 (15% of cobalt)

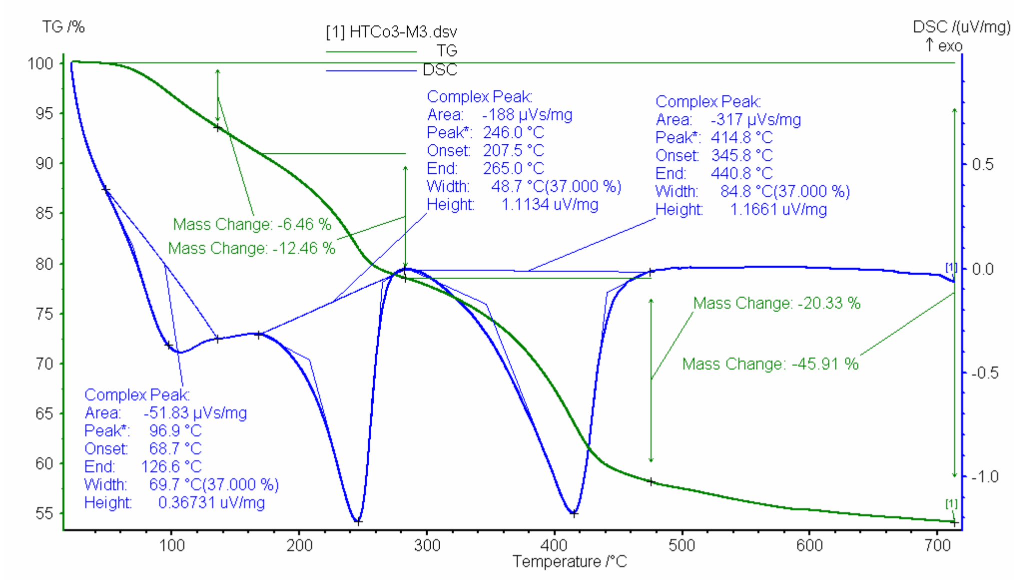


Figure 10: DSC and TG of hydrotalcite HTCo3-M2 (15% of cobalt).

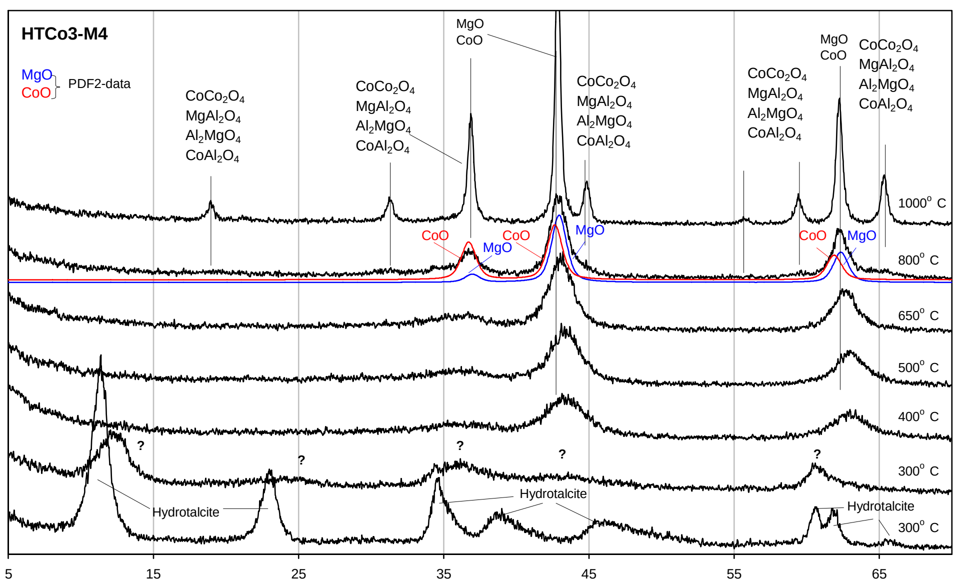


Figure 11: Thermal decomposition of hydrotalcite HTCo3-M4 (10% of cobalt)

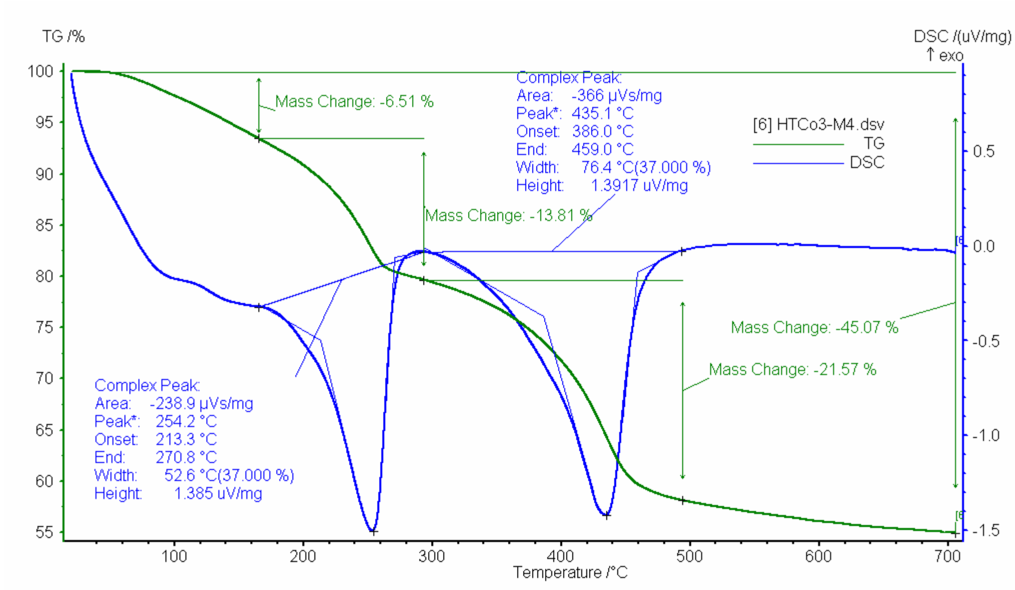


Figure 12: DSC and TG of hydrotalcite HTCo3-M2 (10% of cobalt)

The cobalt hydrotalcites were reproduced well enough in all cases. The thermal decomposition of all hydrotalcites was typical for these materials.

Decomposition of hydrotalcites proceeds between 300 and 400 ºC. Heating over 400 ºC lead to formation microcrystalline cobalt oxides (Co_3O_4 and CoO) and at higher temperatures, in the region of 600 – 1000 ºC, lead to formation of complex oxides (Co_2AlO_4 and $MgAl_2O_4$) with structure of spinal. Described hydrotalcites were sent to CSIC for control their chemical composition and catalytic properties. CSIC confirmed full coincidence received materials with their previous results.

Task 3.3. According to the task of NORTA in the Project the technology of precursor production has been developed. The technology includes next stages:

- Preparation of core material;
- Chemical treatment to activate the surface of the core material;
- Synthesis of needed hydrotalcite on the surface of the core material;
- Drying of received precursor;
- Powdering of the precursor;
- Sieving of the precursor.

This technology was developed at NORTA’s model installation with limited capacity (about 200 grams).

Additional investigations on oxidation soot, fulfilled by CSIC (*see Attachment to Deliverable 2.1*) showed (Table 2) that 10% and 15% catalysts decrease the starting temperature of soot ignition approximately on 100 ºC and the higher content catalyst – 15% - is more active.

The most interesting fact is that complex precursor, produced by NORTA, showed the lowest temperature of ignition (lower than show hydrotalcite, produced by CSIC) namely 425 ºC (see table 2). CSIC partner explains this fact by the better dispersion of the metals or an additional catalytic effect of the gibbsite.

Table 2: Investigations on oxidation soot

Sample	Temperature of ignition, C
Blank	600
10%Co	510
15%Co	485
Precursor (15%Co)	425

NORTA explains this effect by special structure of gibbsite, which was used as a support for precursor. According their point of view, the gibbsite had a sufficiently close to optimal ratio of mezo and micro pores, and it provide the precursor (after hydrotalcite deposition) with high enough catalytic properties. To prove or reject this idea NORTA asked the partner CSIC a more thoroughly investigation of precursors porous structure. The results of this investigation are presented below (Table 3).

Table 3 Specific surface area measurement of precursors

Sample	Specific surface area in synthesized condition, m^2/g
HTC + gibbsite	138,2
HTC + boehmite	204,2
HTC(PFT) + boehmite	291,5

NORTA has developed fundamentally new concept of the catalyst, produced according proposed technology, which permits:

- **improvement of catalyst properties on the stage of precursor production by selection core -** aluminum hydroxides (or other hydroxides with porous structure) by optimization their porous structure
- producing by thermal spraying a composite metal based semiproduct with precursor coating and excellent adhesion to metal substrate, **where chemical and phase composition and porous structure is not damaged during the spraying**
- **creating of needed macro- porous structure** of the catalytic element by mechanical treatment (perforation, corrugation etc)
- **producing of needed structure of the catalyst with controlled phase composition and mezo- and micro- porosity** by thermal treatment (during precursor thermal decomposition)

NORTA has developed an industrial scale reactor (on the basis of work model, mentioned earlier). The scheme of reactor is presented in Fig. 13.

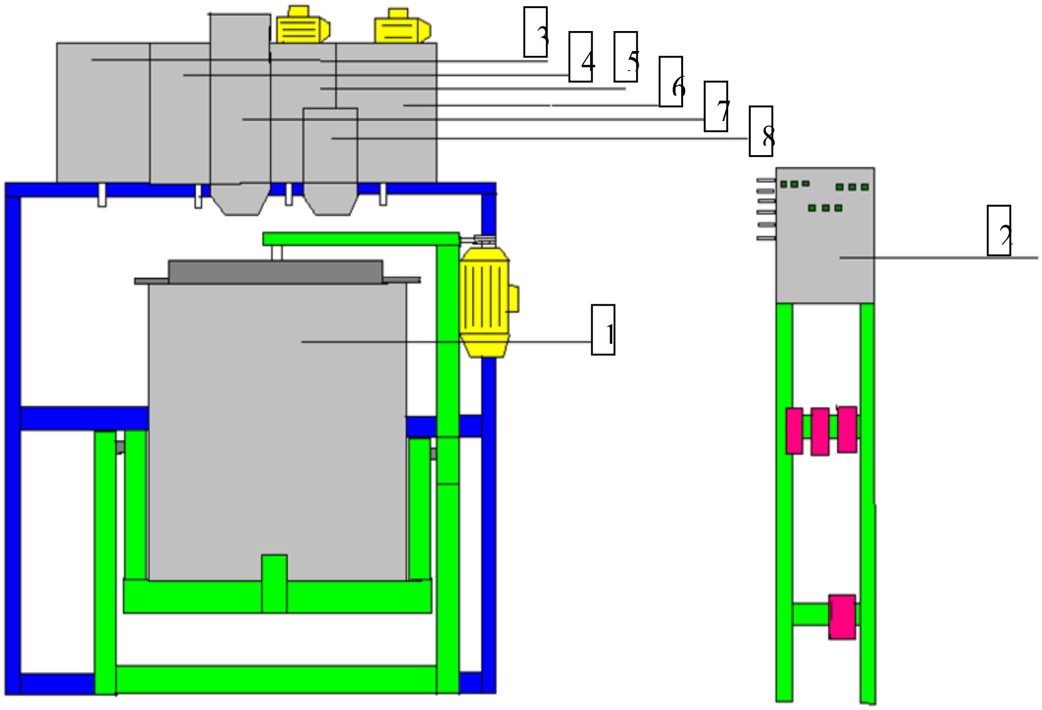


Figure 13: Scheme of industrial scale reactor

The chemical reactor includes:

1. Namely reactor, made from stainless steel (with heating and rotator), tankage: 150 l (39,625 gal). (1)
2. Control unit, including interface for PC-based control support. (2)
3. Batcher for aqueous solutions, made from stainless steel, capacity: 35 l (9,246 gal). (3-6)
4. Powder batcher, made from stainless steel, capacity: 40 kg. (7)
5. Powder batcher, made from stainless steel, capacity: 10 kg. (8)

Principle of operation:

Preparation of solution (solution B): reactor fills of the distilled water. The level of the water can be controlled manually, using automated or PC-based control. Then the outlet valve of powder batcher (8) is opened, and mixed powder enters the reactor. The rotation is activated at the same time, when the outlet valve is opened. The activation of the rotation is automated, so the rotator's motor is protected from being damaged. When preparing the solution, the exothermal reaction is being executed; it takes about 25 minutes to complete.

Preparation of aqueous solutions: Batchers 3 and 4 are filled of distilled water (batcher 4 can be also reserved). Batchers 5 and 6 are filled of solutions. When preparing solution in batcher 6, an exothermal reaction is being executed. It takes about 12 minutes to complete. Because those liquids are preparing just in the batchers, those batchers has an additional rotation. Rotation can be powered on manually, or using PC-based control. It is also protected from being powered, after the AC power is lost, if there is no additional activation. The outlet valves are closed.

Synthesis of the hydrotalcites: When the solution B is prepared and the exothermal reaction is executed, the valve of the batcher 5 is opened. The solution enters the reactor drop-by-drop. The process must take 3 hours, and is controlled manually or using PC-based control. After the process is completed, the additional rotation must be executed. It takes 60 minutes. This part of the process can be controlled using PC

Filtration and aging: After the synthesis and deposition is completed, the powder must be filtered (the valves of the batchers 3 and 4 are opened). After the filtration, the reactor is filled of the distilled water and the heating elements are turned on, the process of the aging is being started. This process takes about 24 hours. The control of the process is automated. The temperature can be adjusted manually or using PC-based control.

End of the process: After 24 hours, the suspension should be cooled, filtered and removed from the reactor.

Task 3.4 To estimate applicability of thermal spraying (HVOF) for precursor spraying on the metal foil substrate preliminary tests at METALLISATION were performed.

For this purpose a model precursor on the basis of traditional hydrotalcite – Mg(II)Al(III) - was synthesized at NORTA. This hydrotalcite was deposited on the core of gibbsite according developed technology. Received precursor was sprayed by HVOF spraying at METALLISATION. A drawing of the spray arrangement is shown in Figure 14.

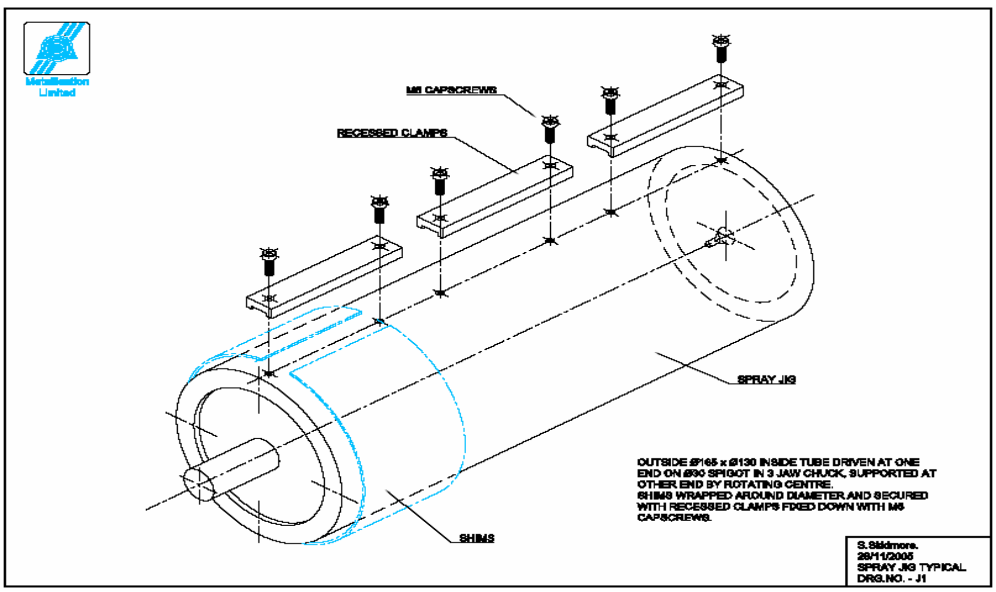


Figure 14: Spray arrangement

The spraying tests showed applicability of method for precursor spraying. The main results of these tests are:

- Adhesion of the sprayed layer is high enough;
- Phase composition of sprayed layer is fully coinciding with phase composition of precursor. It means, that sprayed precursor didn't decompose at all and create the sprayed layer, firmly coupled with metal foil substrate. These results prove the idea to use HVOF spraying for technology development.

At GTV the mechanical properties of catalyst samples, received from NORTA have been analyzed in order to preliminary evaluate the corrugation possibilities. The following main goals were kept in mind while carrying out this research:

1. Evaluation of the precursor surface properties (roughness, profile, hardness and compatibility with the properties of corrugation tool).
2. Estimation of plasticity of metal substrate made of stainless steel foil as an important factor for the right set-up of corrugation technology.
3. Evaluation of adhesion/coupling of catalytic layer with the stainless steel substrate.

Methods used: experimental

Tools and instruments: microscope image computer analysis, special tooth-wheel

Conclusions on goal 1:

- Roughness disbursement lays within the ±10 microns range.
- Corrugation tooth-wheel surface has to be compatible with the obviously abrasive surface of the catalyst.

Conclusions on goals 2&3:

- The samples have demonstrated acceptable plasticity and limited elasticity within wide range of bending radiuses.
- Substrate foil has copied teeth shape of the tooth-wheel with high accuracy, which indicates that tooth-wheels could be considered as one of the applicable corrugation techniques.
- Neither simple bending nor corrugation with the tooth-wheel has caused a visible peeling, which indicates that the adhesion level could be considered as acceptable.
- Should the final catalytic material have similar mechanical properties as the tested samples, the corrugation and probably the manufacture of catalyst converter at this stage could be considered as an affordable task from the point of view of mechanical engineering.

After that partners has dedicated their work on development new types of precursor. The idea of this work was to increase the productivity of HVOF method. Three variants of precursors were developed (see in Fig. 15).

First variant was cobalt hydrotalcite with particles less than 20 microns. The process of spraying this hydrotalcite showed that reliable coating is possible only in case, when we use a thick enough intermediate aluminium layer. But this layer is not good for exploitation.

Second variant was developed taking into account the weakness of the **first variant**. In this case precursor consisted of gibbsite or boehmite core and cobalt hydrotalcite coating. Diameter of particle is about 100 microns. Industrial scale technology to produce big enough quantities of precursor was developed and needed quantity of precursor (about 10 kg) was produced on industrial scale installation. Testing of second variant of precursor in HVOF spraying showed that the mass of particle is not enough for effective penetration into thin intermediate aluminium layer.

Third variant of precursor was developed taking into account the weakness of the second variant. In this case precursor consisted of aluminium core with intermediate layer of boehmite on its surface and coating of cobalt hydrotalcite on its top. The lab scale technology to produce enough quantity of precursor was developed and needed quantity of precursor (3 kg) was produced.

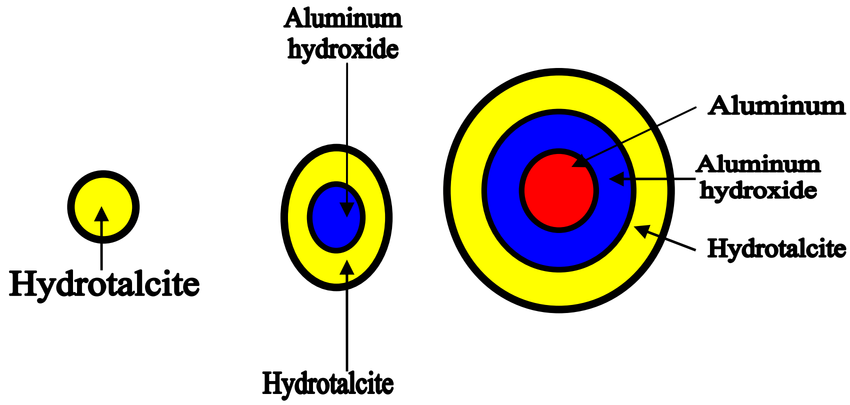


Figure15: Variants of precursor

Testing of third variant of precursor in HVOF spraying was suddenly interrupted in consequence of cessation works of METALLISATION. Nevertheless a new material (**third variant of precursor**) was found so promising that NORTA took a decision on preparing **Patent Application. The PCT Patent Application was prepared and submitted.**

Status

WP# 3 was fulfilled completely.

Deliverables List:

D. No.	Deliverable name	Delivery date	Actual delivery date	Estimated indicative person-months	Used indicative person-months	Lead contractor
D3.1	Optimised precursor	12	12	7,39	12,17	NORTA
D3.2	Report on precursor properties	12	12	7,39	12,04	NORTA
D3.3	Prototype of industrial scale installation for precursor production	22	22	7	9	NORTA
D3.4	Draft plan for using and disseminating knowledge	12	12	5,14	5,14	EP

Milestones list:

Milestone no.	Milestone name	Workpackage no.	Date due	Actual/Forecast delivery date	Lead contractor
M3.1	The technology and installation for precursor production are developed	3	22	22	NORTA

WORKPACKAGE 4 - THERMAL SPRAYING

Responsible partner: METALLISATION
Other partners involved: NORTA, BERSY, FINNKAT, KTU
Objectives:

- To develop the technology for thermal spraying of powder precursors (developed in WP 3).

Main tasks:

Task 4.1. Thermal spraying tools development

- Spraying method selection. Thermal spraying method (air-plasma, HVOF, HVAF) will be investigated for further development according to the results of sub-layer and precursor spraying tests and economic considerations
- Spraying tools design and manufacturing
- Spraying tests and coating control methods

Task 4.2. Sub layer spraying technology development

- Development of the technology of spraying thin (5-10 microns) soft metal layer (e.g. Al) on flexible metal substrate (30-50 microns) to get high adhesion between substrate and catalytic support.

Task 4.3. Catalytic support spraying technology development

- Development of the technology of spraying catalytic support (20-30 microns) using alloyed hydroxides as catalyst precursors. Catalyst support should be sprayed on the surface of sub layer.

Implementation of tasks:

In the beginning of WP4 the feasibility study on various types of thermal spraying technologies was prepared (see *Deliverable 4.1*: Thermal spraying tools and method) and provided to th consortium in order to select the most suitable spraying technologies for thermal spraying of powder precursor**.** It was decided to develop or modify the following technologies:

<u>Plasma spraying (NORTA):</u>

1. *Special tools for air plasma spraying was developed or modified:*
   - air plasma torch for complex hydrotalcite precursor (modified)
   - powder feeders for light powders (developed)
2. *Experimental technology for spraying intermediate layer was developed.* The technology is based on spraying of aluminum powder with particle size less than 5 microns.
3. *Experimental technology for spraying hydrotalcite precursor layer was developed.*
4. *Experimental spraying of model hydrotalcite precursor (developed in WP3 by NORTA) is fulfilled. Experimental samples were sent to KTU for testing.*

<u>HVOF spraying</u>

1. *Special tools for HVOF spraying were developed:*
   - device for steel strip allocation and fastening during spraying (rotating drum)
   - device for spraying gun displacement

2. *Experimental technology for spraying intermediate layer (arc spraying) was modified.* The technology is based on aluminum rod arc spraying.
3. *Experimental technology for hydrotalcite precursor layer spraying was developed.*
4. *Experimental spraying of model hydrotalcite precursor (developed in WP3 by* NORTA*) was fulfilled.* Expeimental samples were sent to KTU for testing.

It was decided to use HVOF process (resp. partner METALLISATION) for effective transportation (without damage in gas stream) of particles of precursor, developed in WP3. The plasma spraying process (NORTA) was supposed to use only for comparison, because this process is well known and can be characterized by considerable damage of precursor particles during their transportation

First tests on precursor (model hydrotalcite composition) HVOF spraying (resp. partner METALLISATION) gave positive results and showed that the mass of particles should be increased. Next tests on second variant of precursor (developed by NORTA in WP3) HVOF spraying (resp. partner METALLISATION) showed that the increase of mass for precursor particles is not enough for effective penetration into intermediate layer. Taking into account received data, NORTA has developed third variant of precursor with aluminum core (to increase the mass of particle), but according to insufficient technical quality of results of the first two variants it was proposed to use spraying technology available to NORTA. METALLISATION has stopped their work in WP4 and their tasks on thermal spraying tools development were overtaken by NORTA.

NORTA has developed a new process for plasma spraying catalytic coatings namely **Soft Plasma Spraying (SPS) process**.

The SPS process consists of two steps:

- First step is a plasma spraying process with using special regimes of spraying to prevent the total decomposition of precursor during its transportation through plasma stream. Special devices were developed to ensure the transportation of precursor into coldest zones of plasma stream. All these actions permit to prevent the total decomposition of the precursor in plasma stream.
- Second step is the special process of reconstitution (should be patented) based on special properties of hydrotalcites (such as “memory effect”). Using this process the structure of hydrotalcites, partly lost during the first step (plasma spraying) can be totally restored.

The only factor, which can not be restored for now, is the structure of porosity, which should include the definite ratio of macro-, mezo- and microspores. Unfortunately, they can not rule the structure of sprayed layer for now, but they plan to develop definite method in future works of NORTA.

According described technology 16 variants of coating were developed and produced for testing in KTU and CRF.

Status

WP#4 was fulfilled completely.

Deliverables List:

D. No.	Deliverable name	Delivery date	Actual delivery date	Estimated indicative person-months	Used indicative person-months	Lead contractor
D4.1	Thermal spraying tools and method	27	27	16,38	25,38	METALLISATION

Milestones list:

Milestone no.	Milestone name	Workpackage no.	Date due	Actual/Forecast delivery date	Lead contractor
M4.1	Spraying method is selected	4	27	27	METALLISATION

WORKPACKAGE 5 - SPRAYED CATALYTIC COATING CHARACTERIZATION AND ACTIVITY

Responsible partner: CRF
Other partners involved: NORTA, GTV, METALLISATION, FINNKAT, CSIC, KTU, CRF
Objectives:

- To characterize the catalytic coatings (small lab-scale samples) by accelerated thermal and chemical ageing tests to evaluate the wash-coat durability.

Main tasks:

Task 5.1. Coating characterization after annealing (500-1200 C)

- Chemical and phase composition (X-ray method)
- Porosity, pore distribution, specific surface area (BET method)
- Microstructure of the surface (SEM method)
- Mechanical properties (cohesive resistance)

Task 5.2. Coating characterization after accelerated thermal and chemical ageing

- Chemical and phase composition
- Porosity, pore distribution, specific surface area (BET method)
- Microstructure of the surface (SEM with semi-quantitative analysis)

Task 5.3. Laboratory test bench development. Laboratory test bench for precursors and coatings Denox properties examination will be developed.

Task 5.4. Laboratory reactor experiments. Six small lab-scaled samples, produced according different regimes of thermal spraying (WP4), will be tested after thermal and chemical ageing for:

- Pressure drop evaluation
- Catalytic activity in simultaneous Nox reduction and soot oxidation in media with SO_2 contamination.

Fulfillment of tasks:

Task 5.1. Coating characterization after annealing (500-1200 C)

Chemical and phase composition (X-ray method). Thermal sprayed samples produced by NORTA have been analyzed before annealing and after.

One of analyzed set of samples was made using complex precursor of hydrotalcite and gibbsite core. The following tests have been made on this sample: X-ray analysis of thermal sprayed and reconstituted Co containing hydrotalcite; X-ray analysis of annealed at 300 ºC thermal sprayed and reconstituted Co containing hydrotalcite; X-ray analysis of annealed at 400 ºC thermal sprayed and reconstituted Co containing hydrotalcite; X-ray analysis of annealed at 600 ºC thermal sprayed and reconstituted Co containing hydrotalcite; X-ray analysis of annealed at 750 ºC thermal sprayed and reconstituted Co containing hydrotalcite; X-ray analysis of annealed at 800 ºC thermal sprayed and reconstituted Co containing hydrotalcite.

Second set of Co containing hydrotalcite coatings made using other complex precursor of hydrotalcite and boehmite core were analysed by the same way, among others the following tests have been made: X-ray analysis of thermal sprayed and reconstituted Co containing hydrotalcite; X-ray analysis of annealed at 650 ºC thermal sprayed and reconstituted Co containing hydrotalcite; X-ray analysis of annealed at 750 ºC thermal sprayed and reconstituted Co containing hydrotalcite.

All samples before catalytic tests have been investigated using X-Ray diffraction analysis. Thermal treatment at 650 ºC for 3 h was not enough for sprayed coatings and it was decided to use higher temperature for pre-treatment.

Porosity, pore distribution, specific surface area (BET method). BET analysis was used to evaluate influence of different spraying variants and usage of different complex of precursors (hydrotalcite, hydrotalcite with gibbsite core, hydrotalcite with boehmite core and other). The results of thermal treatment influence to specific surface area have showed how temperatures affect specific surface area. For example thermally sprayed and reconstituted Co containing hydrotalcite has 92.15 m^2/g specific surface area and after thermal treatment at 650 ºC specific surface area reached 97.8 m^2/g.

Microstructure of the surface (SEM method). The scanning electron microscope (SEM) was used to examine the microstructure of bulk specimens. It is an electron - optical instrument which uses a source of electrons to illuminate the specimen. The information was accessed and displayed in a form of an image. The information from the sample was built up as a two dimensional image. Our samples were analyzed using scanning electron microscope (SEM) EVO 50 from LEO company (Cambrige UK). Resolution was 3nm. To get the image SE (second electron) detector was used. Acceleration strain was 20 kV. Working distance was 10 mm. Analysis was made using EDS detector INCAx-sight. Producer Oxoford instruments (England).

Mechanical properties (cohesive resistance). There is no special equipment to measure these properties of coated materials, so we have used technology of testing, developed by our partners in EXOCAT project CRF.

Testing procedure

1. Retaining of sample at 120 °C for 1 hour
2. Retaining of sample in silica-gel drier at room temperature for 30 min
3. Weighting of the sample
4. Retaining the sample in oven (500°C) for 15 minutes
5. Cooling of the sample in water (at room temperature)
6. Retaining of the sample at 200°C for 1 hour
7. Retaining of the sample in silica-gel drier at room temperature for 30 min
8. Weighting of the sample
9. Repeating of points 4-8 for a total of 3 cycles

The results have showed us which of the coatings have the best mechanical properties.

Task 5.2. Coating characterization after accelerated thermal and chemical ageing

Chemical and phase composition. Chemical and phase composition of catalyst samples after accelerated thermal and chemical ageing have been determined using X-ray analysis.

One of analyzed set of samples was made using complex precursor of hydrotalcite and gibbsite core. The following tests have been made on this sample: X-ray analysis of thermal sprayed and reconstituted Co containing hydrotalcite; X-ray analysis of annealed at 300 ºC thermal sprayed and reconstituted Co containing hydrotalcite; X-ray analysis of annealed at 400 ºC thermal sprayed and reconstituted Co containing hydrotalcite; X-ray analysis of annealed at 600 ºC thermal sprayed and reconstituted Co containing hydrotalcite; X-ray analysis of annealed at 750 ºC thermal sprayed and reconstituted Co containing hydrotalcite; X-ray analysis of annealed at 800 ºC thermal sprayed and reconstituted Co containing hydrotalcite.

Second set of Co containing hydrotalcite coatings made using other complex precursor of hydrotalcite and boehmite core were analysed the same way, among others the following tests have been made: X-ray analysis of thermal sprayed and reconstituted Co containing hydrotalcite; X-ray analysis of annealed at 650 ºC thermal sprayed and reconstituted Co containing hydrotalcite; X-ray analysis of annealed at 750 ºC thermal sprayed and reconstituted Co containing hydrotalcite.

All samples before catalytic tests have been investigated using X-Ray diffraction analysis. Thermal treatment at 650 ºC for 3 h was not enough for sprayed coatings and it was decided to use higher temperature for pre-treatment.

<u>Porosity, pore distribution, specific surface area (BET method).</u> BET analysis was used to evaluate influence of different spraying variants and usage of different complex of precursors (hydrotalcite, hydrotalcite with gibbsite core, hydrotalcite with boehmite core and other) after accelerated thermal and chemical ageing of catalyst samples. The results of thermal treatment influence to specific surface area have showed how temperatures affect specific surface area.

<u>Microstructure of the surface (SEM with semi-quantitative analysis)</u>. The scanning electron microscope (SEM) was used to examine the microstructure of specimens after accelerated thermal and chemical ageing. The information was accessed and displayed in a form of an image. The information from the sample was built up as a two dimensional image. Our samples were analyzed using scanning electron microscope (SEM) EVO 50 from LEO company (Cambrige UK). Resolution was 3nm. To get the image SE (second electron) detector was used. Acceleration strain was 20 kV. Working distance was 10 mm. Analysis was made using EDS detector INCAx-sight. Producer Oxoford instruments (England).

Task 5.3. Laboratory test bench development. Laboratory test bench for precursors and coatings Denox properties examination will be developed

Laboratory test bench for precursors and coatings Denox properties examination has been designed and produced. It allowed investigation of Denox properties of single sample or a set of samples at different temperatures (typically investigations have been performed in a range of 200-600°C, although construction of test bench allows testing at temperatures up to 800°C) and different composition of flue gas. Description of test bench construction and performance is presented in *Deliverable 5.2:* Report on laboratory reactor experiment

Task 5.4. Laboratory reactor experiments. Six small lab-scaled samples, produced according different regimes of thermal spraying (WP4), were tested after thermal and chemical ageing for:

Pressure drop evaluation. Pressure drop of catalyst samples or a set of catalyst samples have been investigated, although presented catalyst samples have been of standard type and significant difference in pressure drop of samples with different coatings has not been observed. Additional investigations have been carried out on influence of the shape of catalyst samples on heat transfer from the surface of the sample to gas flow, passing through such sample as heat transfer is closely related with efficiency of catalyst. It is obvious that the higher heat transfer rate, the better is the contact between surface of the sample and gas flow and, as a result, the better Denox properties of catalyst. Various types of catalyst sample surface and different types of external turbulence influencing devices have been tested. It has been established that external turbulence influencing devices are not very efficient in incensement of heat transfer rate and it is not worth to envisage such devices in working equipment. Contrary, influence of the shape of catalyst sample surface proved to be quite significant and it is advised to use not a flat surface during construction of real life catalyst samples but a surface with disparities. This will significantly increase efficiency of real working catalysts.

Catalytic activity in simultaneous Nox reduction and soot oxidation in media with SO_2 contamination. Catalytic activity in simultaneous NOx reduction and soot (CO) oxidation of catalyst samples has been tested using developed test bench. Typically investigations have been performed in a range of 200-600°C. Quantity of test samples reached approximately 25, which is well over 6, forecast during initial planning of the project. Results of experimental investigations are presented in *Deliverable 5.2:* Report on laboratory reactor experiment

Status

WP#5 was fulfilled completely.

Deliverables List:

D. No.	Deliverable name	Delivery date	Actual delivery date	Estimated indicative person-months	Used indicative person-months	Lead contractor
D5.1	Report coating characterization	27	27	8,5	23,75	CRF
D5.2	Report on laboratory reactor experiments	27	27	8,41	23	CRF

WORKPACKAGE 6 - CATALYTIC BLOCK DESIGN

Responsible partner: GTV

Other partners involved: NORTA, ZREMBUD, BERSY, FINNKAT, CRF

Objectives:

- To develop and manufacture prototype of catalytic block for the engine bench tests. Variables such as exhaust flow rate, exhaust gas composition of a particular engine will have to be taken into account.

Main tasks:

Task 6.1. Catalytic block design

Task 6.2. Catalytic block manufacturing

Fulfillment of tasks:

Task 6.1. The aim of this task was to develop a prototype of industrial scale machine for corrugation of thermally sprayed metal strip. Corrugation is a very important stage in new technological process of catalytic unit production.

The process include following stages:

- **materials preparation**, where there are two processes – metal stripcleaning and synthesis of complex powder precursor,
- **thermal spraying** of steel strip,
- **corrugation** of sprayed strip,
- **mechanical assembling** of catalytic unit
- **thermal activation** of catalytic unit.

A technological process, proposed by NORTA, is totally new, because in all existing technologies, devoted to production of metal based catalytic unit, stage of corrugation **always preceded** to stage of catalyst deposition. In proposed technology the stage of corrugation is **after** the stage of catalyst deposition, in this case stage of thermal spraying.

This chain is possible only because of very high adhesion of sprayed layer to metal substrate, which is the feature of mentioned technology.

The main conclusions of this investigation are:

- thermally sprayed layer has sufficient adhesion to steel strip for corrugation process;
- ceramic surface of the sprayed layer can course intensive abrasion of corrugation device during prolonged exploitation;
- sprayed layer has an inequality in transversal direction, which can cause the transversal displacement of sprayed strip during the process of corrugation.

Taking into account the peculiarities of the spraying strip corrugation, determined in preliminary investigations (see below) the corrugation machine and machine for mechanical assembling of catalytic unit were constructed by GTV.

The complete of design drawings for both machines was produced by GTV.

Because lack of time and according the modified Work Plan the works on corrugation machine and machine for mechanical assembling were performed by NORTA under author's control of GTV.

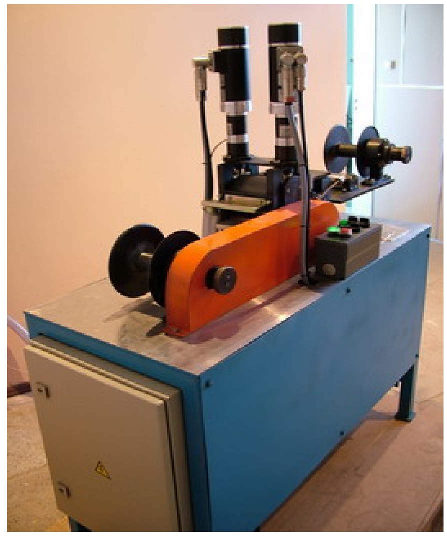


Figure 16: Corrugation machine

Figure 17: Machine for catalytic unit mechanical assembling

Produced machines were successfully tested according special program, developed by GTV and NORTA, and placed in operation.

Task 6.2. According air plasma spraying technology, developed by NORTA, metal strip with thickness 30-50 μm and width of 45, 90, 100 and 120 mm, produced by Sandwik, Sweden was selected as substrate (see Fig. 18)

Figure 18: Metal strip

Named above metal strip should be coated both sides with precursor layer with thickness about 15 – 30 μm (see Fig. 19).

Figure 19: Coated metal strip

It was decided, that the best way for catalytic element manufacturing is to use flat strip as first component and corrugated strip as the second by fixing them together, winding both till needed diameter and then fixing finally. Catalytic element, produced using this technology, will have a cylindrical shape, so it will be easily fit to tube-based measuring equipment.

Main concept feature of new technology, developed by NORTA, is the fact, that flexible metal substrate is sprayed before the corrugation of it. This is fundamental fact which distinguishes this new technology from many other variants of catalytic elements preparing, where the process of corrugation always forego to catalyst deposition process.

Development of this kind of process is possible only because of unique adhesion of sprayed catalytic layer to metal substrate. This is one of the basic requirements for sprayed substrate and this problem is solved using plasma spraying technology, developed by NORTA.

The corrugation is a process of passing flexible metal substrate **perpendicularly** to direction of rolling in toothed rolls. With respect to high adhesion of sprayed layer, it should be affected mechanically in minimum under corrugation. From here, a tooth of the roll must be designed in appropriate form (having touch-contacts as less as possible) and size, to prevent losing of coating or losing it in minimal scale. On the other hand, profile of corrugation should provide the maximal rigidity of corrugation cell and corrugation step should be adequate to unforced gas flow throw the catalytic block, still keeping enough quantity of the catalyst (bigger step – less quantity of the metal strip in the same diameter). It was decided, that the best area of transversal cross section of the single channel after corrugation should be in the range 1-1,5mm^2. According this, the prototype of the sprayed strip corrugation machine was designed and the corrugation tests were produced (Fig. 20).

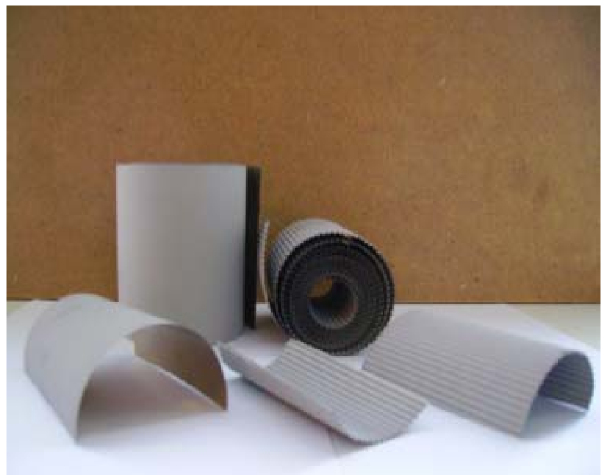


Figure 20: Corrugated strips

The corrugation of sprayed metal strip is big problem because of inequality of sprayed layer. Small deviations, about 3-5% course a uniform displacement of the metal strip during its processing. That is why the continual control of the distance between rolls and also the borders of the strip during its processing is needed.

According the Work Plan, the prototypes of industrial scale machines for sprayed strip corrugation and wounding were designed. The technical requirements for these machines are represented lower.

The installations should include the next components:

- corrugation device
- wounding device
- fixing device

Requirements to catalytic element:

- using of not corrugated strip as second component during wounding
- cylindrical shape of catalytic element
- diameter of catalytic element in the range 50-150 mm
- mechanical fixing of the element

Requirements to corrugation conditions:

- corrugation direction – perpendicularly to direction of rolling
- profile of corrugation should provide the maximal rigidity of corrugation cell and minimal damage of coating layer surface during wounding

- area of transversal cross section of the single channel after corrugation should be in the range 1-1,5 mm^2
- Productivity of corrugation and wounding machine:
- Productivity should be in the range 70-100 meters/hour

To start rolling process, two elements (sheet material and corrugated one) must be fixed. It can be done mechanically, using thin metal stick or small tube with a higher length than a width of the strip and scarf, or using arc welding. However, using of tube will result in hole in the center of the block, so some quantity of gas will flow throw non-catalytic surface (inner surface of the tube). The welding use also requires coating to be removed in the place of welding, because ceramics can not be welded. According these specifications, it was decided to use metal stick with a scarf as a fixing element.

Because of the fact that metal strip is **sprayed before rolling,** it is necessary to perform rolling process without mechanical affect to the surface of it but keeping enough tension to reach stable construction. It could be done by turning stick-form fixer, feeding sheet material and corrugated material from separate reels under appropriate tension. Should be noted, that increasing of the diameter of the roll causes lose of the tension and it also depends on the width of the strip.

To keep the tension of the rolled element, it should be fixed finally. As at the fixing at the beginning of the rolling, welding is not a good idea, because of the necessity to remove coating from the strip.

Alternative solution is to use a tube of the same length as the width of the strip with wall thickness to keep the element stable enough and inner diameter the same as the outer diameter of the rolled element.

According technical decisions, tests of the catalytic element formation in final shape (Fig. 34) were produced successfully:

Fig. 34 Catalytic element

Described technology of development of the catalytic element has very promising feature. Because of use of two elements (first – sheet material, second – corrugated material), two separate catalyst can be placed into the same element (for example, one – oxidative, and the second one – reductive). This feature is protected by Patent Application of NORTA (PCT PCT/LT2006/000010)

A prototype of catalytic element (catalytic block) for diesel off-road machinery presents a cylinder element with honeycomb structure inside and:

- length of 90, 100, 120 mm
- triangular shape of channel cross-section
- area of channel cross-section is 1,5 mm^2
- diameter from 20 mm up to 120 mm

Conclusions on this task:

1. The new technological process of catalytic element production was analyzed from the position of corrugation process. Main features of the corrugation **after spraying** are determined. Solutions for different stages of corrugation and winding are developed.
2. The prototype of catalytic element (catalytic block) is developed.

Status

WP#6 was fulfilled completely.

Deliverables List:

D. No.	Deliverable name	Delivery date	Actual delivery date	Estimated indicative person-months	Used indicative person-months	Lead contractor
D6.1	Prototype design	24	24	6.51	5	GTV
D6.2	Prototype	24	24	6.51	5	GTV

WORKPACKAGE 7 - CATALYTIC BLOCK BENCH TESTS

Responsible partner: CRF

Other partners involved: NORTA, ZREMBUD, GTV, METALLISATION, BERSY, EP, FINNKAT, KTU, CRF

Objectives:

- To develop and manufacture prototype of catalytic block for the engine bench tests. Variables such as exhaust flow rate, exhaust gas composition of a particular engine will have to be taken into account.

Main tasks:

Task 7.1. Laboratory tests

Task 7.2. Engine steady state tests

Fulfillment of tasks:

Task 7.1. Laboratory tests. *Prototype manufacturing*. The developing and experimental activity performed in NORTA, CSIC, KTU and CRF led to the choice of the catalytic formulation used for the P6 lot as the one used for the manufacturing of the final prototype: the prototype (P8-PFT-OG-HTCo3-M3+O) has the characteristic reported in table 4:

Table 4: Final prototype characteristics

Sample code	Catalyst	Cell density	Notes	Dimension
P8-PFT-OG-HTCo3-M3+O (3)	HT on gibbsite + HT Co	Normal	One part coated with new soft plasma spraying technology. The other part with axial plasma spraying technology	Diameter: 38 mm Length: 150 mm Volume: 0.170 dm^3

The prototype has been tested on the CRF test bench described in *Deliverable 5.2:* Report on laboratory reactor experiments. It is able to reproduce different engine conditions in terms of temperature, flow rate and exhaust gas composition. Since the results reported in D5.2 were not optimal, the aims of the tests carried out on the prototype were mainly the followings:

- To evaluate the catalytic performance with test conditions of a typical diesel engine at steady state engine points;
- To analyse the catalytic performance at low flow-rate, in order to confirm the hypothesis that unsatisfactory results obtained on lab-scaled samples were due to the very high flow rate (compared to reactor experiments).

Pressure drop evaluation

The pressure drop evaluation was carried out on the prototype. Similar investigation was performed also on lab-scaled samples but not reported in the D5.2 since the values were not significant (below 2 mbar at flow-rate of about 40-45 dm^3/minute). Pressure drop results on the P8 prototype are reported in fig. 21.

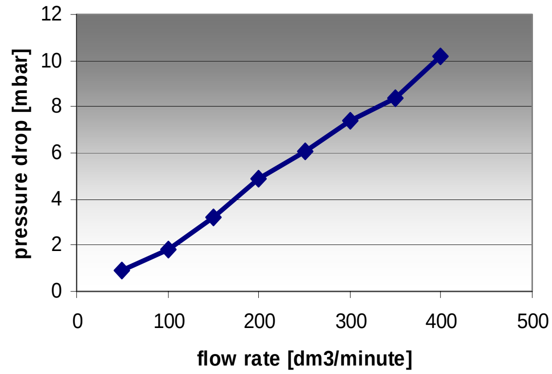


Figure 21: Pressure drop analysis

As 400 dm^3/minute corresponds to a space velocity of 140000 h^{-1}, that pressure drop is not a critical parameter for the investigated sample.

CO conversion

In order to investigate the oxidation property of the P8 prototype, it has been decided to carry out tests at the conditions reported on table 5, while the results are reported in fig. 22.

Table 5: Experimental conditions for CO conversion test

Gas concentration	SV = 100000 h^{-1}	SV = 45000 h^{-1}	SV = 20000 h^{-1}
CO	200 ppm	2000 ppm	1000 ppm
		1000 ppm	500 ppm
O_2	10%		
N_2	balance		

A space velocity of 45000 h^{-1} (coupled with a CO concentration of 1000/2000 ppm) is the commonly CRF test condition used to rate most of the catalysts supplied for automotive applications. As seen in the D5.2 deliverable, those conditions lead to results far from target. Probably the main reason for that behaviour is the high space velocity adopted. Thus, some test modifications were undertaken to verify the catalyst behaviour: first it was decided to reduce the SV down to 20000 h^{-1} (that is in any case a unusual condition for car passenger and for heavy duty application) and CO concentration down to 1000/500 ppm. Finally, a more realistic condition (100000 h^{-1}) was applied to the sample, the CO concentration chosen was based on data supplied by FINNKAT (fig. 23 – Euro 2 heavy duty diesel engine that would be labelled as a Stage II off-road engine), i.e. a maximum CO concentration of 200 ppm. According to shown cycle data, the average exhaust gas temperature is about 350°C (exhaust gas temperature in real driving conditions would be much lower, in the range of 220-320°C).

The following considerations can be done:

- The CO conversion increase when the SV decrease (at equal CO concentration);

- The CO conversion at higher SV seems to be weakly affected by the CO inlet concentration;
- The CO conversion at the more realistic point (100000 h^{-1} – 200 ppm) has a light off (T50) of about 400°C. We can estimate that a CO conversion of about 30-40% is expected at 350°C.

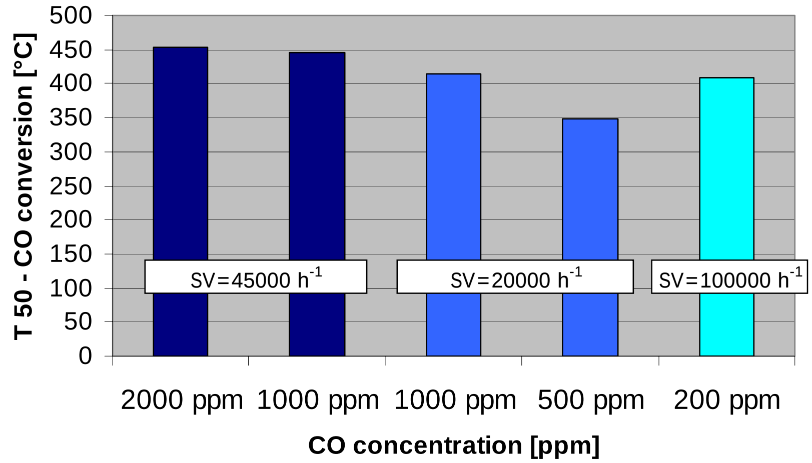


Figure 22: CO conversion results

ESC Results:			
	CO	0.42	g/kWh
	HC	0.75	g/kWh
	NOx	5.99	g/kWh
	PM	0.183	g/kWh

Test details:

SPEED	TORQUE	POWER	B_COL	B_CO2	B_THC	B_NOx	Humidity	Testcell T	Receiver T	Exh_1 T	Exh_2 T	Estimated O2
rpm	Nm	kW	ppm	%	ppm	ppm	%	°C	°C	°C	°C	%
702	3	0	105	1.5	50	161	38.5	24.5	25.5	173	173	18.5
1948	390	80	93	8.9	33	574	42.2	24.5	34.7	437	419	11.1
2404	190	48	93	5.2	35	319	35.9	25.1	37.4	312	307	14.8
2395	285	71	80	7.0	37	448	34.9	25.0	37.6	395	385	13.0
1946	200	41	96	5.5	41	361	34.2	25.1	34.7	312	307	14.5
1948	300	61	70	7.1	31	497	33.3	25.2	35.7	368	358	12.9
1948	100	20	154	3.8	59	214	33.1	25.2	30.9	246	245	16.2
2397	363	91	114	8.8	29	478	33.5	24.9	36.6	493	477	11.2
2401	95	24	127	3.7	46	214	32.6	25.2	36.0	265	269	16.3
2847	351	105	131	9.3	26	535	32.6	25.4	43.7	542	524	10.7
2855	91	27	67	3.8	24	491	31.2	26.2	40.6	271	271	16.2
2848	274	82	59	7.2	39	877	31.6	26.3	41.9	397	387	12.8
2849	183	55	41	5.4	11	704	31.0	26.3	42.8	319	313	14.6

Figure 23: Engine emissions for a Euro 2 heavy duty diesel engine

NOx conversion

NO_x conversion up to 30% is the main target of EXOCAT project. Samples manufactured during the optimisation/improving phase (D5.2) did not evidence a NO_x conversion higher than about 5-6%. As told before the main reason (shared by the partners) is probably the high space velocity. It was therefore decided to reduce the SV down to 20000 h^{-1} while maintaining all the other testing parameters unchanged (see D5.2). The conversion results are reported in fig. 24.

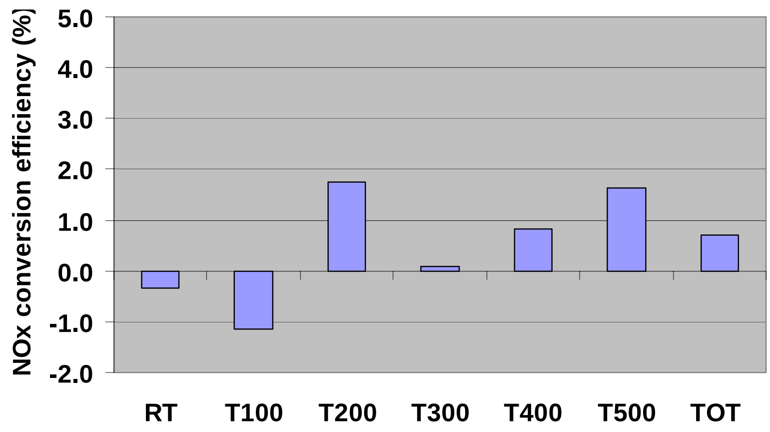


Figure 24: NO_x conversion results on P8 prototype vs. temperature

The reduced SV has not improved the conversion that remains always above the 5% threshold.

Finally, it was decided to modify the testing procedure in order to be, as much as possible, representative of the real engine emissions. Based on the Finnkat emissions results (fig. 22) a CO concentration of 200 ppm was added both in the lean and the rich phase. The results are reported in fig. 25, while in fig. 26 the NO_x trend on the entire cycle is shown.

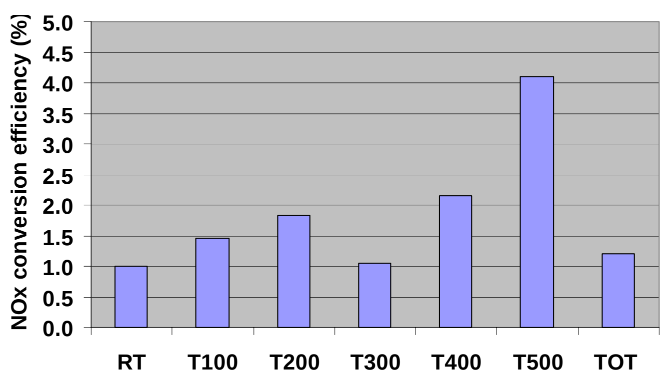


Figure 25: NO_x conversion results on P8 prototype vs. temperature (CO addition)

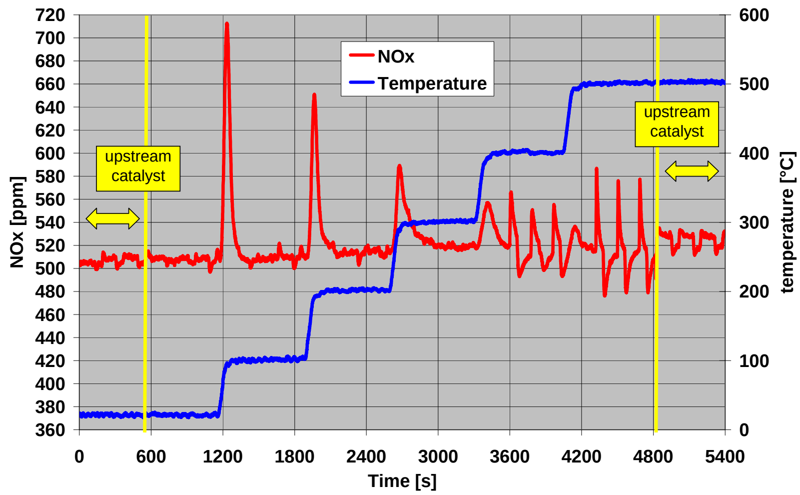


Figure 26: NO_x trend during the entire cycle

Some considerations were stated as follows:

- The addition of a reductant should be an advantage for the NOx conversion: but this aspect is noted only if compared with results shown in fig. 4 at constant temperatures;
- The overall conversion value (TOT) is in any case quite similar between the 2 testing conditions;
- The overall conversion value of P8 is even lower than P6 one;
- The reduced SV did not generate improvements on the NO_x conversion;

HC and CO conversion at constant temperature

NO_x conversion test results could be used to quantify HC and CO oxidation . It is not a typical light–off evaluation, but it is made at constant temperature (only the rich phase HC content has been considered); the results are reported in fig. 27 and fig. 28 respectively for HC and CO. The catalytic formulation adopted for P8 prototype hardly oxidise the hydrocarbons (i.e. a mixture of butane, pentane and hexane): at testing condition we reach a conversion of 50% at about 500°C degrees. CO, as expected, is oxidised more easily and we reach a 50% conversion at a temperature just above 300°C.

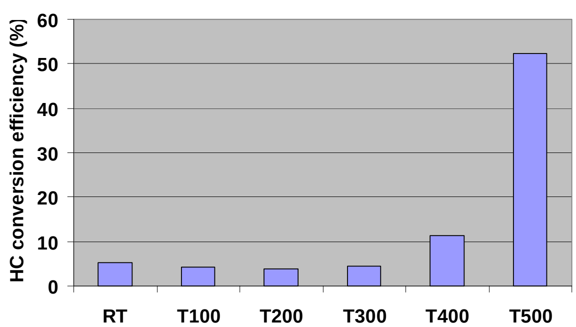


Figure 27: HC conversion results vs. temperature

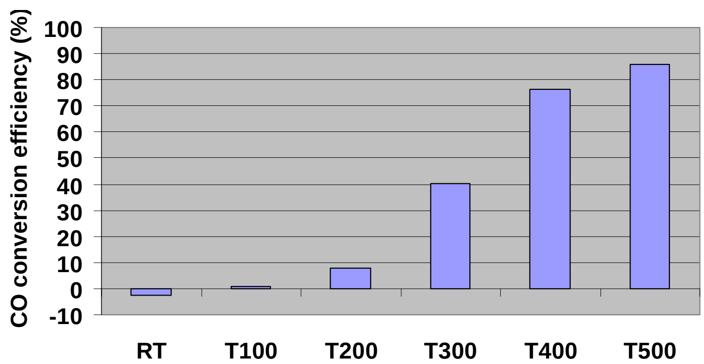


Figure 28: CO conversion results vs. temperature

Ageing considerations

During Exocat project a deep research has been done in order to optimise/improve the basic catalytic properties. The major CRF efforts were focused therefore on a deep bench characterization in order to select the best formulation as well as the selection of the testing conditions that guarantee the best CO, HC and NO_x conversion performances. This effort was bigger than expected, but in any case it was required in order to reach some of the goals of the project. The ageing characterisation was therefore considered an experimental aspect that could follow only satisfactory catalytic results on fresh samples, nevertheless the durability of a catalytic converter, as well as its robustness and reliability at the severe conditions applied on it , represent one of the main topic in the selection of converters for automotive applications. Therefore, no specific ageing tests were performed on P8 prototype, and some preliminary test were performed only on lab-scale samples.

Conclusions

P8 prototype manufacturing has been based on the P6 catalytic formulation. P8 prototype has been used as demonstrator and specific tests that reproduce as much as possible the real engine conditions have been carried out on CRF test bench, with the following results:

- The pressure drop does not represent a critical parameter.
- The CO conversion is quite far from state of the art catalytic converter properties;
- The CO conversion at the more realistic point (100000 h^{-1} – 200 ppm) has a light off (T50) of about 400°C. The CO conversion expected at 350°C is about 30-40% (i.e. the average exhaust gas temperature found in the data supplied by FINNKAT and used as reference).
- NO_x conversion remain the most critical point with very low values that have not been improved at low space velocity (20000 h^{-1})
- The addition of a reductant (CO – 200 ppm) does not affect the overall NO_x conversion;
- The estimated T50 (HC conversion) evaluated at constant temperatures is about 500°C;

From the CRF point of view and as a final conclusion, the scale-up of the catalytic formulation selected and tested in CSIC/NORTA with very good results, should be improved.

Task 7.3. *Engine steady state tests*

According internal decision of the consortium, engine steady state tests were not performed as catalytic converter properties should be further improved.

Status

WP#7 was not fulfilled completely. According internal decision of the consortium, engine steady state tests were not performed as catalytic converter properties should be further improved thus *Deliverable 7.2:* Performance report on engine steady state tests was not delivered.

Deliverables List:

D. No.	Deliverable name	Delivery date	Actual delivery date	Estimated indicative person-months	Used indicative person-months	Lead contractor
D7.1	Laboratory tet report	27	27	11,1	17,58	CRF
D7.2	Performance report on engine steady state tests	27	27	11,03	0	CRF

WORKPACKAGE 8 - DISSEMINATION AND FUTURE EXPLOITATION

Responsible partner: ZREMBUD

Other partners involved: NORTA, GTV, METALLISATION, BERSY, EP, FINNKAT, CRF

Objectives:

- To ensure that the achievements are made known to the targeted potential clients / market segments.
- To prepare plans for future exploitation to ensure that the results are implemented in real-world applications.

Main tasks:

Task 8.1. Preparing publications

Task 8.2. Designing website

Task 8.3. Making application notes

Task 8.4. Plan for using and disseminating the knowledge

Fulfillment of tasks:

Task 8.1 Preparing publications

The results obtained in the project have been protected with a PCT Application PCT/LT2006/000010 with priority from 23.11.2006.

The most interesting and innovative scientific results are going to be published for its dissemination in the scientific community in some of the most important journals related with Chemical and Mechanical Engineering. In this way from the results obtained in the study of the catalysts properties, at least two articles are going to be sent this year to the Journal of Catalysis and to Applied Catalysis B: Environmental, both with a high impact factor. In these articles, the catalytic properties of the tested catalysts are going to be shown and discussed; the catalytic properties will be related with the chemical and textural properties of the materials prepared.

In addition, it is planned to present a contribution in the VII European Congress on Catalysis (EUROPACAT) that is going to be held this year in Turku (Finland). This congress is the most important European congress on Catalysis. Another contribution will be sent to the next International Congress on Catalysis that will be held in Seul next year. Last year a contribution based in the results obtained in the preliminary tests of the catalysts was presented in the XX Iberoamearican Symposia of catalysis.

On the other hand, the results obtained in this project are the basis for two PhD theses, one that is being developed in Spain and another in Lithuania. Finally, a video showing the scheme of the sprayed catalyst preparation system was produced and the web site http//:exocat.europarama.lt created to provide various information related to the project has been opened for public access since January 2005

Task 8.2 Designing website

The first Dissemination deliverable was the development of a dedicated Web page. EXOCAT web site (http://exocat.europarama.lt) has been developed to provide wide range of information related to the project. It includes a restricted area for the consortium, mainly used as document repository, and a public section.

The site has been opened for public access in January 2005. All the relevant information regarding the EXOCAT web site is to be found in the corresponding

Deliverable 8.1: Website*)*. The server is run by staff from EP and is regularly updated.

The EXOCAT web site (http://exocat.europarama.lt) has been developed as a main dissemination tool. Not only does it present the goals and objectives of the project in itself, but it also provides information on its on going activities aiming at the creation of an open discussion within partners.

Task 8.3 Making application notes

According to Project Work Plan the Consortium should develop the catalytic elements for exhaust gas refining for non-road diesel engine.

Also the main aim of the Project is not fully achieved – NOx reduction in catalytic elements is about 5-6% instead of 25-30% needed for commercial application in automotive industry, some applications in the area, where the total volume of catalytic elements is not critical, such as diesel locomotives and ship diesel engines appears possible. More detailed information is available in *Deliverable 8.2:* Application notes.

Task 8.4 Plan for using and disseminating the knowledge

Final plan for using and disseminating the knowledge could be found in respective *Deliverable 8.4:* Plan for using and disseminating the knowledge. There are summarized all dissemination activities including past and future activities.

Status

WP#8 was fulfilled completely.

Deliverables List:

D. No.	Deliverable name	Delivery date	Actual delivery date	Estimated indicative person-months	Used indicative person-months	Lead contractor
D8.1	Website	12	12	5,15	5,15	EP
D8.2	Application notes	27	27	2	1,5	ZREMBUD
D8.3	Publications	27	27	2	1,5	ZREMBUD
D8.4	Plan for use and dissemination of knowledge	27	27	2	1,12	EP

MAJOR DEVIATIONS

All deliverables and milestones foreseen in EXOCAT project are listed in tables 6 and 7.

Table 6: Deliverables List

Deliverable No	Deliverable Title	Work package no.	Date due	Actual/Forecast delivery date	Estimated indicative person-months	Used indicative person -months	Lead contractor
D1	Project Plan (detailed)	1	3	3	7,49	7,71	NORTA
D2	Functional requirements and specifications	1	3	3	7,5	7,72	NORTA
D3	Report on catalytic activity, selectivity and resistance	2	14	14	22,37	27,83	CSIC
D4	Optimized precursor	3	12	12	7,39	12,17	NORTA
D5	Report on precursor properties	3	12	12	7,39	12,04	NORTA
D6	Prototypeof industrial scale installation for precursor	3	22	22	7	9	NORTA
D7	Draft of Plan for using and disseminating knowledge	3	12	12	5,14	5,14	EP
D8	Thermal spraying tools and method	4	27	27	16,38	25,38	METALLISATION
D9	Report coating chracterization	5	27	27	8,5	23,75	CRF
D10	Report on laboratory reactor experiments	5	27	27	8,41	23	CRF
D11	Prototype design	6	24	24	6,51	5	GTV
D12	Prototype	6	24	24	6,51	5	GTV
D13	Laboratory test report	7	27	27	11,1	17,58	CRF
D14	Performance report on engine steady state tests	7	27	27	11,03	0	CRF
D15	Website	8	12	12	5,15	5,16	EP
D16	Application Notes	8	27	27	2	1,5	ZREMBUD
D17	Publications	8	27	27	2	1,5	ZREMBUD
D18	Plan for use and dissemination of knowledge	8	27	27	2	1,12	EP
D19	Management Reports	9	27	27	3,44	3,44	EP
D20	Final Report	9	27	27	3,44	3,44	EP
Total:					**150,72**	**197,47**	

Table 7: Milestones List

Milestone no.	Milestone name	Work package no.	Date due	Actual/Forecast delivery date	Lead contractor
M2.1	Two most prospective oxide systems selected and developed.	2	14	14	CSIC
M3.1	The technology and installation for precursor production are developed	3	27	27	NORTA
M4.1	Spraying method is selected	4	24	24	METALLISATION
M9.1	Implementation of the project assured	9	27	27	EP

A tabular overview of budgeted person-months and actual person months is presented in table 8.

Table 8: A tabular overview of budgeted person-months and actual person months

CONTRACT N°:	512912												
ACRONYM:	EXOCAT												
PERIOD:	01/11/2004	31/01/2007											
			TOTALS	NORTA	ZREMBUD	GTV	METALLISATION	BERSY	**EP**	FINNKAT	CSIC	KTU	CRF
WP 1) Requirements and specification for non-road applications	**Actual WP total:**		**15,43**	7,79	0,35		1,1	2,2	0,61	1,56	0,6	0,9	0,32
WP-leader - NORTA	**Planned WP total:**		**14,99**	7,31	0,35		0,66	2,2	0,61	1,56	0,6	0,9	0,8
WP 2) Catalyst Properties	**Actual WP total:**		**27,82**	9,03			0,2		0,25		5	13,34	
WP-leader - CSIC	**Planned WP total:**		**22,37**	9,03			1,33		0,31		7,8	3,9	
WP 3) Catalyst Precursor	**Actual WP total:**		**38,35**	9,08		0,26	0,26		1,55	0,19	5,4	21,61	
WP-leader-NORTA	**Planned WP total:**		**26,92**	8,60		0,5	0,33		1,53	0,36	3	12,6	
WP 4) Thermal Spraying	**Actual WP total:**		**25,38**	5,01		0	1,6	2,2		0		16,57	
WP-leader -METALLISATION	**Planned WP total:**		**16,38**	4,30		0	2,32	2,2		0,36		7,2	
WP 5) Sprayed Catalytic Coating Characterization and Activity	**Actual WP total:**		**46,75**	6,45		1,5	0	0		0	1	33,8	4
WP-leader - CRF	**Planned WP total:**		**16,91**	6,45		1,5	1	0		0,36	0,6	3	4
WP 6) Catalytic Block Design	**Actual WP total:**		**10,00**	2,15	0	2	0	3,85		0			2
WP-leader - GTV	**Planned WP total:**		**13,02**	2,15	1,75	2,25	0	3,85		1,42			1,6
WP 7) Catalytic Block Bench Tests	**Actual WP total:**		**17,58**	3,01	0	0,25	0	0,55	0,30	0		5,67	7,8
WP-leader - CRF	**Planned WP total:**		**22,13**	3,01	4,2	0,25	0,33	0,55	0,31	2,28		2,4	8,8
WP 8) Dissemination and Future Exploitation	**Actual WP total:**		**9,28**	0,48	0,33	0,49	0,51	2,2	3,39	0			1,88
WP-leader - ZREMBUD	**Planned WP total:**		**11,15**	2,15	0,7	0,5	0,66	2,2	3,36	0,78			0,8
WP 9) Management	**Actual WP total:**		**6,88**	0,22					6,66				
WP-leader and project Coordinator - EP	**Planned WP total:**		**6,88**	0,22					6,66				
	Actual total:		**197,47**	**43,22**	**0,68**	**4,5**	**3,67**	**11**	**12,76**	**1,75**	**12**	**91,89**	**16**
Total Project Person-month	**Planned total:**		**150,72**	**43,22**	**7**	**5**	**6,64**	**11**	**12,76**	**7,11**	**12**	**30**	**16**

There are some major deviations:

Project parner	Expanatory notes
ZREMBUD FINNKAT	<u>FINNKAT used only about 25% and ZREMBUD only about 10% of their planned person months.</u> Actual person months used by FINNKAT and ZREMBUD is reduced comparing to planned because of the shift of the scope of the project to applications where the total volume of catalytic elements is not critical allowing cost effective solutions without requirement for low volume of catalytic elements namely to diesel locomotives and ship diesel engines. These applications were not in line with mainstream of FINNKAT and ZREMBUD as end users of project results activities therefore they used less than planned person months for project activities.
KTU	<u>Exceeded total person months used.</u> This deviation has occurred due to project partner KTU. KTU has stated that they have exceeded PM due to restrictions, imposed by laws and regulations of the Republic of Lithuania on salaries of research personnel of scientific and educational institutions; they have not been able to enlist into project team the researchers with the required qualification and versatility. Due to that KTU had to employ larger quantity of researchers' staff of low salary. As tasks and deliverables within responsibility of KTU were implemented with relevant quality consortium accepted this explanation as sufficient. Detailed explanation is provided in KTU's letter to project coordinator (see page 71);
METALLISATION	During the project, NORTA has developed three variants of precursors. First two variants were experimented in METALLISATION, but according to insufficient technical quality of results of these two variants it was proposed to use spraying technology available to NORTA. METALLISATION has stopped their work in WP4 and their tasks on thermal spraying tool development were overtaken by NORTA Consequently, METALLISATION used 3,62 PM during 1st period and only 0,05 during 2nd reporting period. Due to overtaken tasks the total planned person-months for METALLISATION was reduced from 11 to 6,64. In the latest approved DoW (01/06//2007), the total planned person-months are indicated wrongly 155.09 – because of a technical mistake in updating relevant tables. Total planned person-months **should be 150.7 instead of 155.09** (see tables 9 and 10)

Table 9: Project effort form from DoW (01/06/2007) with technical mistake indicated.

	Partners	**Research/innovation activities**	Requirements and specification for non-road applications	Catalyst Properties	Catalyst Precursor	Thermal Spraying	Sprayed Catalytic Coating Characterization and Activity	Catalytic Block Design	Catalytic Block Bench Tests	Dissemination and Future Exploitation	Total research/innovation	**Management activities**	Project & Consortium Management	Total management	TOTAL ACTIVITIES
1	NORTA		7,31	9,03	8,60	4,30	6,45	2,15	3,01	2,15	43,00		0,22	0,22	**43,22**
2	ZREMBUD		0,35					1,75	4,20	0,70	7,00				**7,00**
3	GTV				0,50		1,50	2,25	0,25	0,50	5,00				**5,00**
4	METALLISATION		1,10	2,20	0,55	3,85	1,65		0,55	1,10	11,00				**11,00**
5	BERSY		2,20			2,20		3,85	0,55	2,20	11,00				**11,00**
6	EP		0,61	0,31	1,53				0,31	3,36	6,10		6,66	6,66	**12,76**
7	FINNKAT		1,56		0,36	0,36	0,36	1,42	2,28	0,78	7,11				**7,11**
8	CSIC		0,60	7,80	3,00		0,60				12,00				**12,00**
9	KTU		0,90	3,90	12,60	7,20	3,00		2,40		30,00				**30,00**
10	CRF		0,80				4,00	1,60	8,80	0,80	16,00				**16,00**
TOTAL PARTNERS			**15,43**	**23,24**	**27,13**	**17,91**	**17,56**	**13,02**	**22,34**	**11,59**	**148,21**		**6,88**	**6,88**	**155,09**

Table 10: Corrected Project effort form from DoW (01/06/2007)

	Partners	Research/innovation activities	Requirements and specification for non-road applications	Catalyst Properties	Catalyst Precursor	Thermal Spraying	Sprayed Catalytic Coating Characterization and Activity	Catalytic Block Design	Catalytic Block Bench Tests	Dissemination and Future Exploitation	Total research/innovation	Management activities	Project & Consortium Management	Total management	TOTAL ACTIVITIES
1	NORTA		7,31	9,03	8,60	4,30	6,45	2,15	3,01	2,15	43,00		0,22	0,22	**43,22**
2	ZREMBUD		0,35					1,75	4,20	0,70	7,00				**7,00**
3	GTV				0,50		1,50	2,25	0,25	0,50	5,00				**5,00**
4	METALLISATION		0,66	1,33	0,33	2,32	1,00		0,33	0,66	6,64				**6,64**
5	BERSY		2,20			2,20		3,85	0,55	2,20	11,00				**11,00**
6	EP		0,61	0,31	1,53				0,31	3,36	6,10		6,66	6,66	**12,76**
7	FINNKAT		1,56		0,36	0,36	0,36	1,42	2,28	0,78	7,11				**7,11**
8	CSIC		0,60	7,80	3,00		0,60				12,00				**12,00**
9	KTU		0,90	3,90	12,60	7,20	3,00		2,40		30,00				**30,00**
10	CRF		0,80				4,00	1,60	8,80	0,80	16,00				**16,00**
TOTAL PARTNERS			**15,00**	**22,36**	**26,91**	**16,38**	**16,90**	**13,02**	**22,12**	**11,15**	**143,85**		**6,88**	**6,88**	**150,72**

SECTION 3 – CONSORTIUM MANAGEMENT

Responsible partner: EP, NORTA
Other partners involved: -
Objectives:

- To ensure a smooth project management and communication.

Meetings

The management and co-ordination of the EXOCAT project activities have been implemented within the plan. During the project meetings with participation of Consortium representatives were organized:

- Kick-off Meeting, held in Kaunas, Lithuania
- 6M Progress meeting held in Turin, Italy
- 12M Progress meeting held in Valencia, Spain
- 18M Progress meeting held in Oulunsalo, Finland;
- Final project meeting held Orbassano, Torino, Italy

For topics discussed reference is made to the Minutes of Meeting as distributed in EXOCAT web site.

Other meetings (no formal minutes taken) include discussions between partners regarding technical progress. Discussion at the meetings has been open and constructive, enabling the direction and content of the technical work to be defined and agreed. Outside of these meetings, communication within the consortium has been good, mainly by e-mail/phone, enabling work programmes to progress smoothly.

<u>Kick-off Meeting, held in Kaunas, Lithuania</u>

Participants

Name	Organization
Alexander Khinsky	Norta
Paulius Lucinskas	Norta
Juras Ulbikas	Europarama
Dmitrij Mamajev	Europarama
Terry Lester	Metallisation
Eugenijus Milcius	GTV
Massimo Asti	CRF
Eduardo Palomaves	C.S.I.C.
Arno Amberla	Finn Katalyt
Kestutis Buinevicius	KTU

Key issues:

- During the kick-off meeting each participant gave a brief presentation about his organisation, relevant achievements in the near past and about their role in the project;
- *Dr. Juras Ulbikas* (EUROPARAMA) (non-technical project coordinator) presented situation with PIPECRAFT: no response during more than two months, no signed documents provided after communicating several time about deadlines. According preliminary information owner of PIPECRAFT cancelled its business activities and new owners of facilities foreseen to use in the project were not interested in the projects.

After discussion Consortium decided that PIPECRAFT behavior could be qualified as serious breach of consortium agreement and on the basis of paragraph 8.1.2 was requested termination of defaulting party reallocating amounts and tasks foreseen to this party. Coordinator was obliged to inform Commission about this decision and prepare updated version of Description of Work and other relevant documents. Amended documents were sent to the Commission on December 7, 2004.

Consortium agreed that NORTA will be responsible for Scientific and Technological project flow and EUROPARAMA will take care of Project Management;

It was agreed that Role of EUROPARAMA will be general assistance with administrative procedures. EUROPARMA will chase the participants for a timely deliverable of cost statements, hourly records and technical reports, but for all clarity:

- Participants do their own bookkeeping
- EUROPARAMA will not be involved in any technical issues
- EUROPARAMA will edit technical reports to the Commission (formal deliverables such as reports and the Plan for dissemination and use of knowledge)
- Decisions regarding project scope and major deviations from the plan (see annex to the contract) will be formally made during consortium meetings, which are scheduled per 6 months.

▪ It was agreed that the 40% advance payment will be paid as soon as the necessary admin details (IBAN number, etc) have been received.
▪ It was agreed that audited cost statements are necessary annually.
▪ It was agreed that all time spent on the project is eligible that would not be spent when there would be no project. This accounts for traveling time, time spent on admin, reading reports, discussions on the phone, etc.
▪ It was agreed that RTD Performers will (have to) spend time on the project in line with efforts put in by the individual SMEs. In any case, overspending by the RTD Performers need to be avoided to ensure that SMEs will not be confronted by bills that cannot be matched by efforts.

6M Progress meeting held in Turin, Italy

Name	Organization
Alexander Khinsky	Norta
Paulius Lucinskas	Representing GTV and KTU
Juras Ulbikas	Europarama
Lorenzo Garro	Bersy
Terry Lester	Metallisation
Eduardo Palomaves	C.S.I.C.
Massimo Asti	CRF
Flavio Parussa	CRF

Key issues:

▪ *Dr. Juras Ulbikas* (EUROPARAMA) gave a short review of activities regarding WP.

- *Alexander Khinsky* (NORTA) and *Massimo Asti* (CRF) presented deliverables they were responsible for. After short discussion deliverables were adopted as final.
- According to CSIC's financial department, CSIC was not able to participate in AC model – they prefered FC. Dr. Juras Ulbikas informed that CSIC partner was invited to the project not only taking into account their expertise but also as R&D organization having possibility to work under AC model. During long proposal preparation time (preparation started in the very beginning of FP6 and proposal was submitted only to second CRAFT call) no information about CSIC decision on cost model reached any of the consortium partners. All contract documents and Consortium agreement was signed without any comments from CSIC. First reaction of CSIC reached Coordinator only more than month after kick-off meeting. Despite all efforts of Coordinator to discuss the matter with responsible persons of CSIC during 6M period no solution were reached. Budget was already totally planned and transfer of CSIC to FC model would force SMEs to bear additional costs.

  Dr. Juras Ulbikas proposed that CSIC should find the possibility to participate in AC model. Otherwise CSIC should be replaced by another organisation working on AC model – new partner was found in Turin, Italy by efforts of NORTA.

  CSIC partner was given 2 weeks (short time was due to already lost 6M solving this problem without any result) to discuss possibility to work on AC model under EXOCAT project with the Commission. *Dr. Juras Ulbikas* was obliged to inform CSIC about Consortium decision immediately (letter to Prof. Corma was send on June 13, 2005).

  All agreed, by a solid vote, to this position. Decision of the Consortium was attached and is integral part of 6 M project progress minutes.

<u>Project suspension and resumption (Meeting in Brussels, Belgium)</u>

According to CSIC problem EXOCAT project was suspended by the Commission in accordance with Article II.7.3. on August 1, 2005. To solve this problem Commission convened meeting in Brussels with participation of EXOCAT project partners. Since the agreement has been reached during the meeting in Brussels – CSIC budget was modified due to their switch to FC model – by the letter send from the Commission on September 29, 2005 project was resumed.

12M Progress meeting held in Valencia, Spain

Participants:

Name	Organization
Alexander Khinsky	Norta
Kristina Klemkaite	Norta
Paulius Lucinskas	Representing KTU
Juras Ulbikas	Europarama
Valdas Jokubavicius	Europarama
Avelino Corma	C.S.I.C
Eduardo Palomaves	C.S.I.C.
Eugenijus Milcius	GTV
Arno Amberla	Finn Katalyt
Massimo Asti	CRF

Key issues:

- Technical project co-coordinator - *Alexander Khinsky* (NORTA) presented status of project deliverables and technical issues, all partners presented their work done during 12 months individually as well (presentations are published on EXOCAT web site);
- Prof. *Avelino Corna* (CSIC) concluded that work done during last months gave wonderful results. The chemical composition of catalyst is determined. It should be alloyed hydrotalcite (composition is confidential). The technology of precursor production developed by Norta is very promising; it could be applied for trucks. **Technology is almost ready for patent.** All partners discussed on possibility for patent. Norta and CSIC agreed to start preparation of necessary documents for patent application.
- *Dr. Juras Ulbikas* (EUROPARAMA) and *Alexander Khinsky* (NORTA) presented the main changes made in EXOCAT DoW (amended EXOCAT DoW was sent to the Commission by e-mail and by currier, it is also available on EXOCAT web site), in particular:
    - Modified Work Plan:
        - *WP2 extended until December of 2005* (Further participation of CSIC is foreseen in WP3 and WP5 – end in October of 2006)
        - *WP3 extended until August of 2006* with further works on precursor optimisation in the frame of WP5 – end in October of 2006)
        - *WP4 extended until August of 2006*
        - *Additional tasks included in WP3* **:**
            - development of model composite precursor on the basis of boehmite (core) and Al-Mg hydrotalcite (covering) – responsible Norta
            - development of industrial scale installation (prototype) for precursor production – responsible Norta
        - *Additional task included in WP5* : Laboratory test bench development – responsible KTU

    Modified budget for CSIC due to their switch to FC model
- *Alexander Khinsky* (NORTA) presented the main scientific work to be done during next reporting period;
- Requirements for the first reporting period were presented and discussed.

Consortium status

There were some changes in responsibilities performed during first project reporting period:

- Due to internal decision in KTU, responsible person – Kestutis Buinevicius was replaced to Mindaugas Jakubcionis;
- Due to internal decision in FINNKAT, to Arno Ambero was given power to sign all documents regarding EXOCAT project.

Consortium contact persons

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Deliverables and results

- Kick-off meeting
- 6M Project Steering Committee meeting
- 12 M Project Steering Committee meeting
- EXOCAT webpage (http://exocat.europarama.lt)

Conclusions:

The EXOCAT project activities for the first reporting period (from 2004-11-01 to 2005-10-30) of the project were implemented completely despite of:

- Modification in membership – PIPECRAFT was removed from the consortium as they were no responding for more than two months and no signed documents were sent to the coordinator after communicating several time about deadlines;
- Problems with CSIC partner – their cost model was switched from AC to FC.

18M Progress meeting

Name	Company
Juras Ulbikas	EUROPARAMA
Valdas Jokubavicius	EUROPARAMA
Arno Amberla	FINNKAT
Jari Lotvonen	FINNKAT
Alexander Khinsky	NORTA

Lorenzo Garro	BERSY
Paulius Lucinskas	Representing KTU
Massimo Asti	CRF

Key issues:

- Technical project co-coordinator - *Alexander Khinsky* (NORTA) presented status of project deliverables and technical issues, presentation covered following points:
  - Overview of the main tasks of NORTA in WP# 3;
  - Presentation of lab scale and prototype of industrial scale installations for compositional precursor production;
  - Presentation of developed prototype of industrial scale corrugation machine (developed by GTV);
  - Presentation of developed precursors (three types of precursor were developed);
  - Presentation of new soft plasma spraying technology for hydrotalcite precursors.

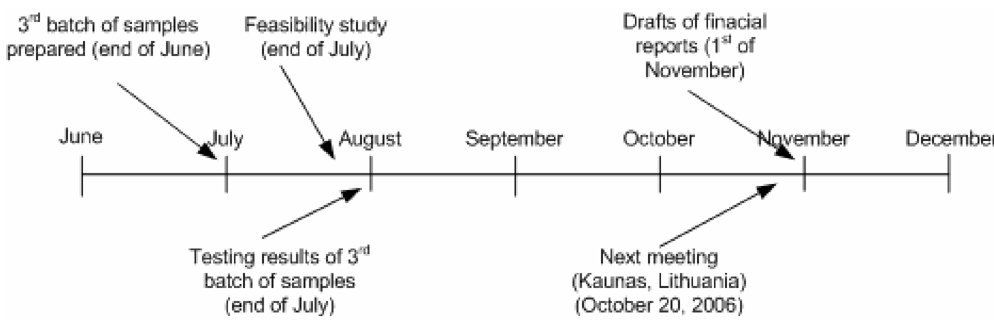
- NORTA simultaneously with CSIC has fulfilled preliminary works on preparation of PCT patent application. The patent search was fulfilled, more than 300 patents were looked through, analogs and prototype were selected. According their mutual position, the patent should include:
  - The chemical and phase composition of hydrotalcite (part of CSIC)
  - Deposition technology (part of NORTA).

  Preliminary description of technological part (NORTA) and the first version for claims is prepared and preliminary description of material part (CSIC) is partly prepared. The first version for claims addition (CSIC) is under work now.

  Ownership of the patent is not decided yet. *Alexander Khinsky* suggested to join all SMEs to patent application.
- *Valdas Jokubavicius (representative of EUROPARAMA)* presented the main requirements for Final Activity and Management reports, in particular discussed issues:
  - Activity report:
    - Partners responsible for workpackages are requested to contribute *reports on activities carried out* until the 1st of November.
    - *Activity report* should include: overview of the activities carried out; publishable executive summary; updated plan for using and disseminating the knowledge.
  - *Management report* should include: detailed justification of the costs (each partner); financial statements (Form C) (each partner), audit certificates.

- Partners concluded:
  - Feasibility study for economical evaluation of precursor production on industrial scale will be prepared by NORTA till the end of July, 2006.
  - 3rd batch of samples should be prepared by the end of June, 2006 and will be sent to CRF for laboratory testing;
  - Testing results of 3rd batch of samples will be presented by CRF by the end of July, 2006;
  - As soon as testing results will be available technical meeting will be organized. All partners will make decision on further activities of the project;

- *Juras Ulbikas* will check possibility for 3 months extension of the project immediately after the meeting;
- Drafts of financial reports should be sent to EUROPARAMA till 1st of November, 2006
- Next meeting will be organized in Kaunas, Lithuania in October 20, 2006. If the project will be extended meeting timing and place will be reconsidered.



Final project meeting

Name	Company
Juras Ulbikas	EUROPARAMA
Arno Amberla (through SKYPE)	FINNKAT
Eduardo Palomares	CSIC
Alexander Khinsky	NORTA
Lorenzo Garro	BERSY
Paulius Lucinskas	Representing KTU
Massimo Asti	CRF
Marco Federico Pidria	CRF
Flavio Parussa	CRF

Key issues

- Dr. Alexander Khinsky’s presentation covered following points:
  1. Overview of main tasks of NORTA in WP3 (NORTA leader)
  2. Participation of NORTA in WP4 (Thermal spraying)
  3. Participation of NORTA in WP6 (Catalytic block design)
- Dr. Massimo Asti presented testing results provided in CRF facilities
- NORTA has demonstrated virtual project of all technology steps: powder preparation, thermal spraying, strip corrugation, block making, thermal activation.
- NORTA demonstrated movie about equipment of powder preparation and catalytic block making;
- NORTA should work on developing of it’s Soft Plasma Spraying technology to increase specific area. After pre-selection in KTU samples will be sent to CRF. If tests will be positive, the sense of the discussion for possibility of new project will appear. NORTA will prepare sample P6 and send to CRF for testing. If results will be good, then NORTA will

produce samples of P6 and will send to FINNKAT. NORTA will prepare and send to all project press-realise. All partners will prepare some input for it. All partners will work on the report in parallel. If there will be not enough time for preparing report, the draft will be prepared.

Consortium contact persons

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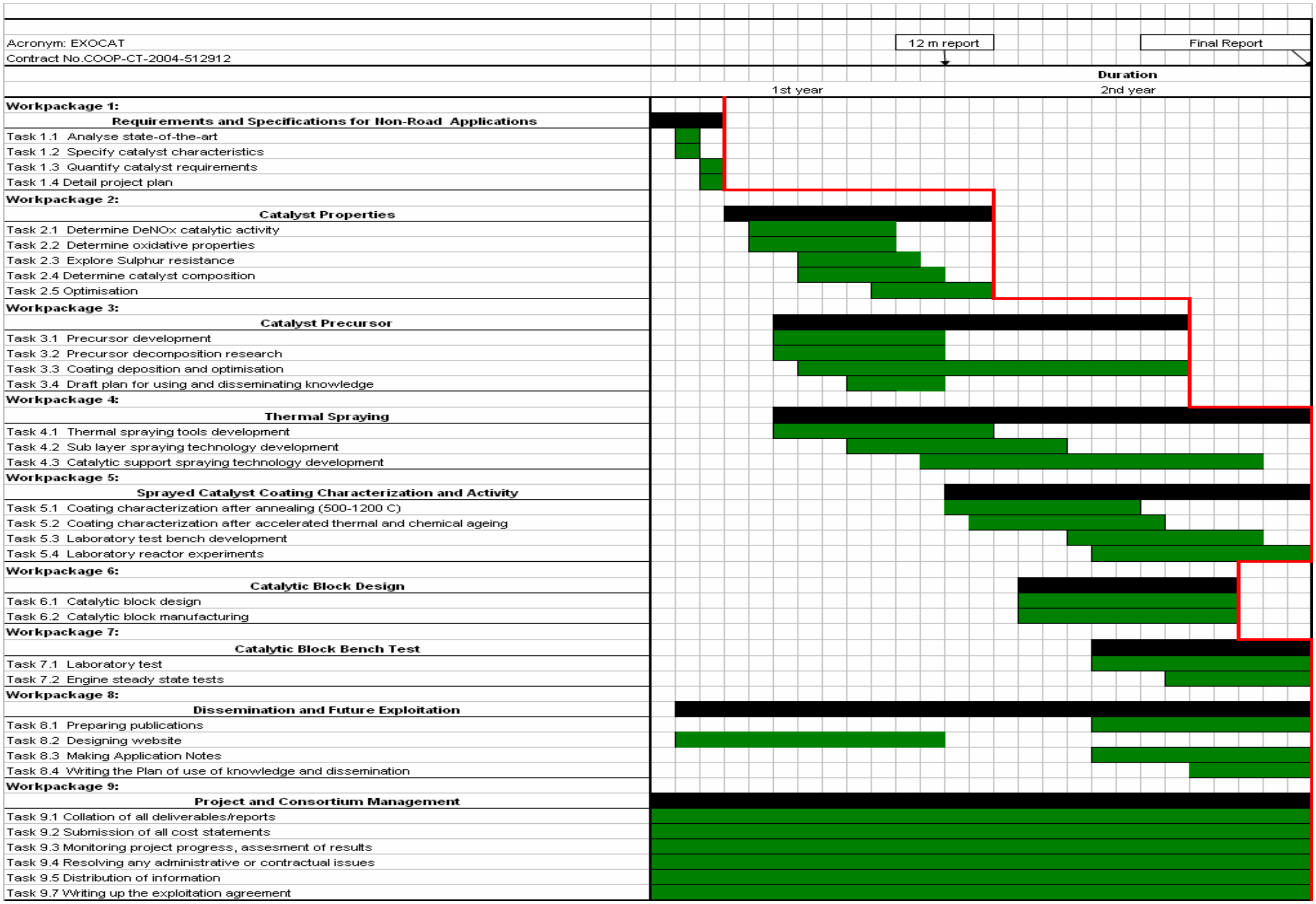
Deliverables and results

- 18 M Project Steering Committee meeting
- Final project meeting
- Updated EXOCAT webpage (http://exocat.europarama.lt)
- Management reports
- Final report

Conclusions:

- The EXOCAT project management activities for the second reporting period (from 2005-11-01 to 2007-01-31) of the project were implemented completely.

Table 11: Project Barchart and Status



SECTION 4 – OTHER ISSUES

Ethical aspects

Table 11: **Ethical Aspects Assessment Table**

Sensitive ethical issues related to:	**YES**	**NO**	**Appropriate Measures**
Human beings		√	
Persons not able to consent		√	N/A
Children		√	N/A
Adult healthy volunteers		√	N/A
Human biological samples		√	
Human embryonic stem cells in culture		√	N/A
Human foetal tissue/human foetuses		√	N/A
Personal data (privacy aspects)	√		Statistical data acquired from interviewing individuals will be processed according European Directive 95/46/EC
Genetic information		√	N/A
Animals		√	
Transgenic animals		√	N/A
Non- human primates		√	N/A
Dogs, pigs, cats		√	N/A

The partners confirm that the proposed RTD project does not involve:

- Research activity aiming at human cloning for reproductive purposes;
- Research activity intended to modify the genetic heritage of human beings which could make such changes heritable;
- Research activities intended to create human embryos solely for the purpose of research or for the purpose of stem cell procurement, including by means of somatic cell nuclear transfer;
- Research involving the use of human embryos or embryonic stem cells with the exception of banked or isolated human embryonic stem cells in culture.

Gender issues

The Treaty of Amsterdam formalises the mainstreaming commitment at the European level, as it explicitly mentions the elimination of inequalities and the promotion of equality between women and men among the tasks and objectives of the Community (Articles 2 and 3).

To take this important principle into account, a Gender Impact Assessment (GIA) has been carried out to avoid unintended negative consequences of neglected or disregarded gender issues and to improve the quality and efficiency of the project. The GIA addressed two main stages:

- Existing differences between women and men, both in the research community and the applicable industry.
- Proposal contribution to the elimination of existing inequalities and promote equality between women and men, both during the project and the impact it may have after the project, again both for the research community and the industry.

The GIA has been made against following criteria.

Participation addresses aspects such as:

- sex-composition of the target/population groups
- representation of women and men in relevant research / industry
- representation of women and men in decision-making positions.

Resources addresses issues related to distribution of resp. access to crucial resources such as:

- time and space
- information and money
- political and economic power
- education and training
- job and professional career opportunities
- new technologies
- health care services
- housing, means of transport
- leisure.

Suitability of applied or generated Technologies deals with inequalities caused by technologies to men and women or to masculine and feminine characteristics, in particular to ergonomical aspects (necessary force, human factor aspects; complexity of technology; user-interface, etc)

Norms and Values reviews topics, which influence:

- gender roles
- division of labour by gender
- the attitudes and behaviour of women and men respectively
- inequalities in the value attached to men and women or to masculine and feminine characteristics.

Rights pertaining to:

- direct or indirect sex-discrimination
- human rights (including freedom from sexual violence and degradation)
- access to justice, in the legal, political or socio-economic environment.

EXOCAT contributed to make this work sustainable for both women and men. The partners ensured equal opportunities between women and men in the proposed project. Women scientists was involved in research and dissemination of the results to potentially interesting parties.

ANNEX 1 – Plan for using and disseminating the knowledge

1.2 Exploitable knowledge and its Use

1.2.1 Patent application

During EXOCAT project PCT application was submitted at The State Patent Bureau of the Republic of Lithuania (SPBRL) in November of 2006. Information regarding PCT application is provided in the table below.

Table 12. PCT application

Exploitable Knowledge	Exploitable product(s) or measure(s)	Sector(s) of Application	Timetable for commercial use	Patents or other IPR protection	Owner & Other Partner(s) involved
The method of catalytic coating manufacturing	*Method*	*Catalysis sector, Automotive sector*	*2008*	*Patent*	*Owner: NORTA*

Application number: PCT/LT2006/000010.
Date of submitting to SPBRL: 23.11.2006.
Authors: Khinsky Alexander, Klemkaite Kristina, Laurinaitis Nerijus, Corma Avelino, Palomares Eduardo.
Owner: NORTA UAB. Proposal to participate in process of submitting of application, patenting and owning was sent to all SME's of consortium of the project before submitting. None of other SME partners expressed intention for participation in patenting process.
Annotation of the patent application: The method of catalytic coating manufacturing.
The invention concerns to methods of manufacturing catalytic coatings by thermal spraying of powder material and can be applied in various branches of chemistry, power and automobile industry. The aim of the given invention was necessity of obtaining a double sided catalytic coating on a metal strip which, besides a high level of catalytic properties in reactions of nitrogen oxides reduction and hydrocarbons oxidation, would possess a high level mechanical and operational properties, namely, high adhesion to a metal substrate and high resistance to a poisoning in an atmosphere of sulphurous gases and water steam.
For achievement of this aim a special composite powder, consisting aluminium metal core, of is used an intermediate layer on the basis of aluminium hydroxide and covering on a surface of a particle on the basis of cobalt hydrotalcite which is put on an intermediate layer is offered during thermal spraying of a metal strip. The structure of a composite powder - qualitative and quantitative is offered.
Catalytic covering of the specified type is sprayed by the method of thermal spraying on metal strip, then mechanical treatment and forming of a product is made.

1.2.2 Exploitable results

Scientific results achieved during EXOCAT project are/will be used in following FP6 projects:

- Taking into account excellent adhesion and quite good catalytic properties of cobalt oxide catalyst coating, developed and patented in EXOCAT project, it was tested (in concordance with Partners) as catalyst in other FP6 project, named BURNERCAT (Catalytic, environmental-friendly, fuel flexible and cost effective burner for domestic boiler, contract number 016937). This Project devoted to development of catalytic burner for domestic boilers. The catalyst passed the preliminary tests and now included into the BURNERCAT project for investigation.
- Also, now other question under discussion is application of cobalt oxide catalytic elements, produced by NORTA in EXOCAT project, for stationary diesel exhaust gases refining in other FP6 project, named BIG POWER (Advanced Biomass Gasification for High-Efficiency Power, contract number 500311) and devoted to biofuel gasification.

During the project the *corrugation machine and machine for mechanical assembling of catalytic unit were constructed by GTV*, due to modified Work Plan the works on corrugation machine and machine for mechanical assembling were performed by NORTA under author's control of GTV. Produced machines were successfully tested according special program, developed by GTV and NORTA and placed in operation. Corrugation machine and Machine for catalytic unit mechanical assembling (Fig.29 and Fig.30) will be used for further scientific work in NORTA.

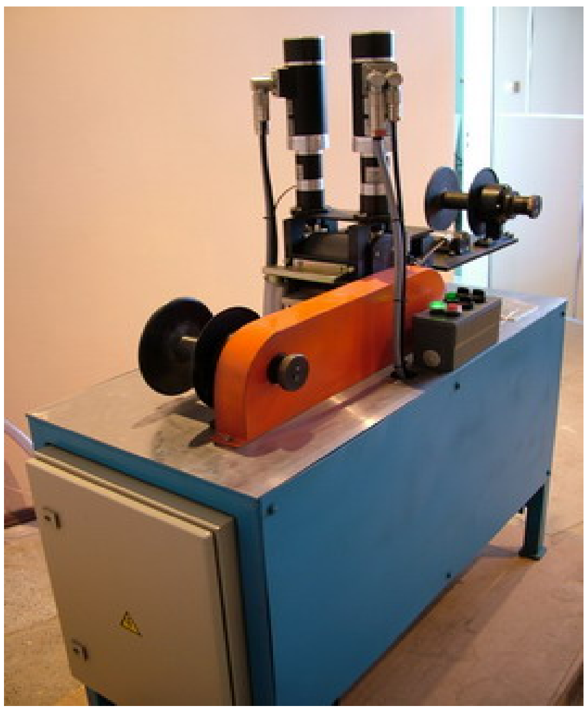


Figure 29: Corrugation machine

Figure 30: Machine for catalytic unit mechanical assembling

Despite the fact that not all EXOCAT project objectives were fulfilled (low NOx reduction), the new technology for catalytic elements production and needed installations are developed and produced on industrial scale level. NORTA is ready for small-batch production (about 2000 units/month).

NORTA is considering that proposed solution (using non-precious metals as catalysts and their plasma spraying application) is right and undertakes obligations to continue the development at its own expense with cooperation with CSIC (Consejo Superior De Investigaciones Cientificas) and CRF (Centro Ricerche Fiat Societa Consortile per Azioni) until the moment, when these organizations will be ready to organize a new European Project, which should be the continuation of EXOCAT.

1.3 Dissemination of knowledge

Wide range of dissemination activities were active during entire duration of the project and there is foreseen dissemination policy after accomplishing EXOCAT project.
Dissemination activities will include:

- Participation in leading conferences related with automotive and catalysis research such as *International Conference on Environmental Catalysis, International Congress on Catalysis, EUROPACAT (European Congress on Catalysis)* ;
- Publications in relevant magazines such as *International Journal of Automotive Technology*, obviously being careful not to reveal sensitive proprietary information. The results related with the catalyst studies will be published for its dissemination in the scientific community in journals related with catalysis with a high impact factor as *Journal of Catalysis, Applied Catalysis B: Environmental*, or *Journal of Molecular Catalysis A: Chemical*. They will be also published in journals related with *Physical Chemistry* as *Journal of. Physical Chemistry B* or related with materials as *Journal of Materials Chemistry*. Finally it is planned to publish in journals related with Chemical Engineering as *Industrial & Engineering Chemistry Research.*
  Note: CRF has a "double role" given that they are 100% daughter of FIAT who also has a division in non-road mobile machinery. CRF plans to disseminate the results within the automotive world via publications in leading automotive and autocatalyst magazines such as Journal of Automotive Engineers, as well as within their own organisation.
- Seminars: EXOCAT project was presented in several seminars listed in Table 11.
- Set-up and maintenance of a project website which is already available on http://exocat.europarama.lt. EXOCAT web site has been created to provide a wide range of information related to the project. It includes a restricted area for the Consortium, mainly used as document repository, and a public section. The site has been opened for public access in January 2005. All the relevant information regarding the EXOCAT web site is to be found in the corresponding Deliverable D8.1 Website The server is run by staff from EUROPARAMA and is regularly updated.
  The EXOCAT web site has been created as a main dissemination tool. Not only it presents the goals and objectives of the project in itself, but it also provides information on its on going activities aiming at the creation of an open discussion within partners.
- Dissemination of information to the networks and established distribution channels of the individual partners. Every partner involved in EXOCAT project is already driven towards promoting the project in its institution. In that perspective, means of dissemination vary: annual review meetings and internal presentations.

Table 12: Overview table

Planned/ actual Dates	**Type**	**Type of audience**	**Countries addressed**	**Size of audien ce**	**Partner responsible /involved**	**Status**
	Conferences:					
In September 2008	International Conference on Environmental Catalysis	Researchers	EU countries	200-300	CSIC	Will be doe
In July 2008	International Congress on Catalysis	Researchers	EU countries	200-300	CSIC	Will be done
In August 2007	EUROPACAT (European Congress on Catalysis)	Researchers	EU countries	150-250	CSIC	Will be done
	Publications:					
In 2007 May	International Journal of Automotive Technology	Researchers, general public	To all	-	CSIC, CRF, NORTA	Will be done
In 2007 May - October	Applied Catalysis B: Environmental	Researchers, general public	To all	-	CSIC, CRF, NORTA	Will be done
In 2007 May - October	Journal of Automotive Engineers	Researchers, general public	To all	-	CRF	Will be done
In 2007 May	Journal of Molecular Catalysis A: Chemical	Researchers, general public	To all	-	CSIC, CRF, NORTA	Will be done
In 2007 May - October	Journal of Physical Chemistry B	Researchers, general public	To all	-	CSIC, CRF, NORTA	Will be done
In 2007 May	Materials Chemistry	Researchers, general public	To all	-	CSIC, CRF, NORTA	Will be done
In 2007 May	Industrial & Engineering Chemistry Research	Researchers, general public	To all	-	CSIC, CRF, NORTA	Will be done
	Seminars:					
In 2006 November	R&D results transfer to SMEs – stories of success (Kaunas)	Researchers, general public	Lithuania	20	EP, NORTA	Done
In 2006 November	Event of Agency for International Science and Technology Development Programmes in Lithuania – main representative and organiser for FP programs in Lithuania	Researchers, general public	Lithuania	80	NORTA	Done
In 2006 October	R&D results transfer to SMEs – stories of success (Vilnius)	Researchers, general public	Lithuania	37	EP	Done
In 2006 November	R&D results transfer to SMEs – stories of success (Klaipeda)	Researchers, general public	Lithuania	16	EP	Done
	Project web-site					
In January 2005	http://exocat.europarama.lt	Restricted and public access	To all	-	EP	Done

1.4 Publishable results

The results achieved by project EXOCAT will be documented in project deliverables. A list of deliverables will be published during the project is given below (Table 12). Deliverables that will be published have a dissemination level of "public" (PU). It is expected, that these deliverables contain a significant part of the project achievements and therefore dissemination activities will largely base on the results reported in these documents.

Table 14. Publishable results

Deliverable No	ID	Deliverable Title	Delivery Date	Dissemination level
D9	D5.1	Report coating characterization	27	PU
D13	D7.1	Laboratory test report	27	PU
D15	D8.1	Website	12	PU
D16	D8.2	Application Notes	27	PU
D17	D8.3	Publications	27	PU

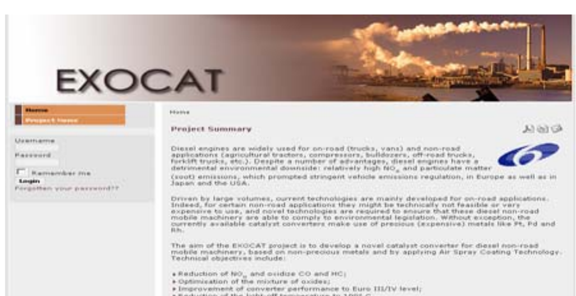
1.4.1 Project LOGO

In order to immediately improve the Project visibility, a logo was designed and will be used in all the dissemination tools, ranking from the web site to fact sheet and brochures:

1.4.2 EXOCAT Web Site

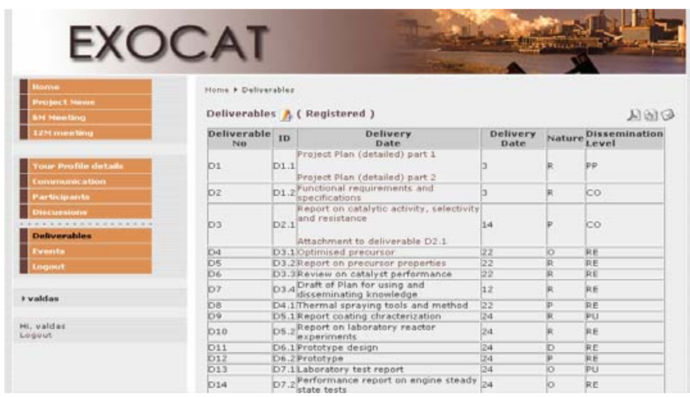
The first Dissemination task was the creation of a dedicated Web page. EXOCAT web site (http://exocat.europarama.lt) has been created in January 2005 to provide various information related to the project. It includes a restricted area for the Consortium, mainly used as document repository, and a public section. Information available in public section:

- Project summary, which includes short project description and the main project objectives;
- Project news, in this section visitor can find all available project news for external users.



Information available in the restricted access area:

- Your profile details. Every partner can check and update his own profile to be available for other project partners.
- Communication. It is available to send a message directly to chosen partner through EXOCAT website.
- Participants. All information about project partners is distributed in this section, including: name of the contact person, telephone number, e-mail address;
- Discussions. This section was created for better communication and dissemination of results between partners;
- Deliverables. This section provides direct access to all project’s deliverables with due dates. As the project progresses, links to deliverable documents in a variety of formats will be provided;
- Events. Information concerning past (Kick-off meeting, 6M meeting, 12M meeting and final meeting) and future events is available in this section. Partners can download all documents from the meetings including: minutes, power point presentations, photos and oth;



1.4.3 Virtual project and movie

A virtual project and movie about NORTA’s equipment developed during EXOCAT project was made for more clearly understanding and presentation of catalytic unit manufacturing route.

Annex 2 – KTU explanation on exceeded costs

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For:

Project EXOCAT
Contract No.512912

Director Dr. Juras Ulbikas
Project coordinator "Europarama" Ltd.

From:
Team leader Dr. Mindaugas Jakubčionis
Kaunas University of Technology (KTU)

In the budget of EXOCAT project 30 person months have been allocated for completion of Kaunas University of Technology (KTU) activities, foreseen in Annex 1 of consortium agreement. Due to restrictions, imposed by laws and regulations of Republic of Lithuania on salaries of research personnel of scientific and education institutions, we have not been able to enlist into Project team required researchers of required qualification and versatility. Due to such reason we decided to enlist larger quantity of cheaper but more specialized researchers and auxiliary personnel. Due to enlistment of larger quantity of cheaper researchers into EXOCAT project team, KTU exceeded quantity of person months, allocated for its activities in the budget of the Project but we can state, that our team carried all the work foreseen in Annex I and remained within the estimated costs.

Attached hereto link to Frequently asked question on Project management in FP6 ftp://ftp.cordis.europa.eu/pub/ist/docs/projects/faq_en.pdf. Page 16 of this document is attached to this letter. Question 63 of this document is related to exceeding of person months.

Team leader Dr. Mindaugas Jakubčionis

informative or binding? (total eligible costs and EC contribution)	(with the exception of the Community contribution), and some deviations from them are allowed, as long as the work is carried out as foreseen in Annex I and deviations can be justified in a subsequent review or audit.
UPDATED 61. What is the procedure to reallocate person months and costs in general between partners, if the consortium decides that this is for the best of the project? Is it possible to request budget shifts between Types of Activities? Are there limitations in making budget adjustments during the years or between different partners, given that the total project budget remains unchanged?	Budget transfers **between partners** or **between activities** are possible, as long as the work is carried out as foreseen in Annex I and transfers can be justified in a subsequent review or audit. No approval is needed by the Commission for such transfers, but the Project Officer should be informed. Important: *In the case of important budget transfers between partners not formalised via a contract amendment, in case the Commission is obliged to apply collective financial responsibility, it will use the share of budget as indicated in the contract or the last amendment (where pre-financing is to be recovered) or the share of budget based on accepted certified costs (where a settled payment is to be recovered).*
62. Can a partner reallocate part of his budget, by moving money from travel, consumables, equipment to personnel? May he reallocate money between personnel and the other costs?	The concept of budget transfers **between cost categories** does not exist in FP6, as the only distinction that is made in the contract is between direct and indirect costs (and the separation of the cost of subcontracting from direct costs). Contractors may reallocate money between their own defined cost categories as long as the work is carried out as foreseen in Annex I.
63. If we have two cheap researchers can we charge for more person months than allocated in the budget so long as we remain within the estimated costs?	Yes, as long as the work is carried out as foreseen in Annex I and deviations can be justified in a subsequent review or audit.
64. What happens if some project partners have spent more on the first year of the project than initially planned in the first 18 month plan of the project on a given activity (in an IP) ?	**Deviations** from the plan are allowed, as long as the work is carried out as foreseen in Annex I and deviations can be justified in a subsequent review or audit. This overspending will then have to be taken into account in the planning of the remainder