# The Carbon2Chem® Laboratory in Oberhausen – A Workplace for Lab-Scale Setups within the Cross-Industrial Project

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Within the Carbon2Chem® network, basic research is mandatory for a successful implementation and realization of sustainable technologies for $CO_2$ emission reduction. For this purpose, the exchange of knowledge between the project partners in the individual subareas is as essential as obtaining precise data on the fundamental parameters on a laboratory scale in order to transfer them later to large-scale plants. Therefore, the Carbon2Chem® laboratory offers a platform to gain detailed insights into the individual sub-processes and to then apply these findings at the technical center in Duisburg.

**Keywords:** Cross-industrial network, Gas cleaning, Methanol synthesis, Steel mill exhaust gases

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## 1 Introduction

The past cumulative $CO_2$ emissions and consequently the global rise in temperature require a rethink in the use of economic goods. Thereby, a special focus has to be set to the supply and demand of energy provided as well as the degree of utilization of existing technologies. To accomplish the energy transition sustainably and successfully, numerous aspects have to be addressed involving the economic efficiency of production processes as well as the optimal utilization of all resources provided [1–3]. Carbon2Chem® has created an extensive network between industry and scientific community, which is essential to deal with the inevitable exhaust gases from industrial processes and create an alternative to their thermal utilization for electricity and process heat by converting them economically and in an environmentally friendly way into valuable products, e.g., basic chemicals [4, 5]. Such an approach can effectively support a sustainable economy, since the exhaust gas based production of chemicals enables a more sustainable and careful use of resources compared to the conventional production of chemicals [6, 7]. At the same time, it enables an economic assessment and its feasibility via the classification of the possible and necessary treatment steps.

The basis for any implementation of innovative and sustainable technologies is not only the utilization of systematic scientific investigation, but also cooperation between research institutes and relevant industrial partners to transfer the constantly changing framework conditions directly among the researchers. Owing to these requirements, basic research is essential within the cross-industrial approach of Carbon2Chem® to determine the feasibility of using steel mill exhaust gases as feedstock. The overall project goal of significantly reducing $CO_2$ emissions is only achievable when gas conditioning, gas purification and the influence of impurities on downstream processes are considered [8–10]. While the applied research undertaken by the industrial partners in the technical center in Duisburg investigates sectoral coupling in a central workspace under real conditions [11], laboratory scale experiments are employed to analyze each single parameter in detail. The results from such research always have to be considered in parallel to those gained from setups working under real conditions. Similarly to the technical center in Duisburg, a single location for laboratory scale experiments facilitates the exchange of knowledge between the researchers and avoids unnecessary duplicate investigations by the individual partners, ensuring effective and fast cooperation.

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## 2 Planning and Realization

To achieve the goals of the Carbon2Chem® project and to successfully implement individual measurement tasks, close cooperation between project partners is indispensable. The Carbon2Chem® laboratory in Oberhausen belongs to the core infrastructure of the project and forms the basis for the construction of a wide variety of research apparatus by several project partners. Therefore, the joint laboratory was developed and planned at the beginning of the project in 2016. At first, the existing laboratory facilities and infrastructure in Oberhausen at the location of Fraunhofer Institute for Environmental, Safety, and Energy Technology UMSICHT (Fraunhofer UMSICHT) were used. Within the first years of the project, the existing laboratory was therefore expanded with the infrastructure necessary to quickly establish and adapt the workspace for several of the project partners' setups and to bridge the time until the construction of the new building was completed. In addition to the existing electrical supply, exhaust air and waste gas facilities, a central gas supply was installed for this purpose, which provides several gases in the high-pressure and low-pressure range (Tab. 1) and can be utilized by all partners at the respective points of use. Furthermore, several working areas for wet chemical work such as the preparation or post-treatment of catalysts and reactant samples were installed. During this period, the Max Planck Institute for Chemical Energy Conversion (MPI CEC) operated the existing building on the premises of Fraunhofer UMSICHT as well as supervised and developed the new construction and the implementation of the infrastructure in cooperation with Fraunhofer UMSICHT. Here, the close cooperation between Fraunhofer UMSICHT and MPI CEC ensured the optimal development and lastly the usability of the workspace for all partners involved.

**Table 1.** Key facts of the Carbon2Chem® laboratory.

| | |
|---|---|
| Location | Fraunhofer UMSICHT, Oberhausen |
| Start of operation | December 01, 2017 |
| Date of opening the new building | March 19, 2019 |
| Space of research (existing + new) | $120\,m^2 + 500\,m^2$ |
| Office workplaces / meeting rooms | 30 / 2 |
| Provided gases | $N_2$, $H_2$, CO, $CO_2$, Ar, $CH_4$, 20 % $O_2$/Ar at 70 bar and $N_2$, $H_2$, Ar, He, 2 % $H_2$/Ar at 14 bar |

When completing the extension of the laboratory in 2019, the main part of the infrastructure was again planned and constructed by MPI CEC and Fraunhofer UMSICHT and combined with the existing laboratory. At the end of the first project period, the entire central gas supply with the respective specific gas supply stations for the individual plants, the electrical distribution for the specific equipment as well as the compressed air and exhaust gas lines were extended to the new building, so that the move-in of individual plants and the initiation of measurement campaigns could occur towards the end of 2019. The general workspace of the whole laboratory including existing and new constructions is shown in Fig. 1b. Some key facts about the laboratory are summarized in Tab. 1. An overview of the general workspace of the laboratory is shown in Fig. 2.

## 3 Activities and Setups within the Subprojects

### 3.1 Gas Analysis

First of all, one key question the project has to deal with is the nature of unknown trace components in steel mill exhaust gases as well as their concentrations and possible fluctuation behavior. Although they occur on an only small scale of ppb/ppt, the trace compounds can cause significant deactivation of the catalysts and thus, have an enormous impact on the efficiency of the catalytic processes. Therefore, a detailed analysis of the exhaust gases is one central task in Carbon2Chem®. The analysis of gases is separated into several tasks. Firstly, the investigation of the real gas compositions before and after gas treatment are performed on-site in Duisburg in the technical center of Carbon2Chem® [12, 13], where we rely on conventional gas chromatography and a proton transfer reaction quadrupole interface time-of-flight mass spectromer (PTR-QiToF-MS). To establish a solid basis for the latter analytical method, a detailed time-resolved long-term analysis method of the different gas compositions was developed [12]. Due to the complex distribution of gas traces within the working gas, the analytical method and instrumentation has to be validated, optimized and further developed. For this propose, the simulation of exhaust gases is mandatory to obtain a better insight into the gas composition under controlled conditions and hence, a gas mixing system to evaluate requirements of gas cleaning technologies was constructed in the joint laboratory (Fig. 3).

This facility allows for the generation of gases with controlled compositions to evaluate requirements of gas cleaning technologies. To analyze trace components individually, i.e., without the influence of the numerous other trace compounds present in real exhaust gases, this setup can generate off-gas-like gas compositions from simple mixtures with selected trace impurities to complex compositions with up to 200 compounds. Either gaseous compounds (e.g., hydrocarbons, N-, S-containing gases) or the transference of liquids into the gas phase (e.g., moisture, alcohols, higher hydrocarbons, aromatics) can be added into the feed gas stream in the ppm range. The main focus of this setup is to provide these complex gas mixtures for screening the activ-

  

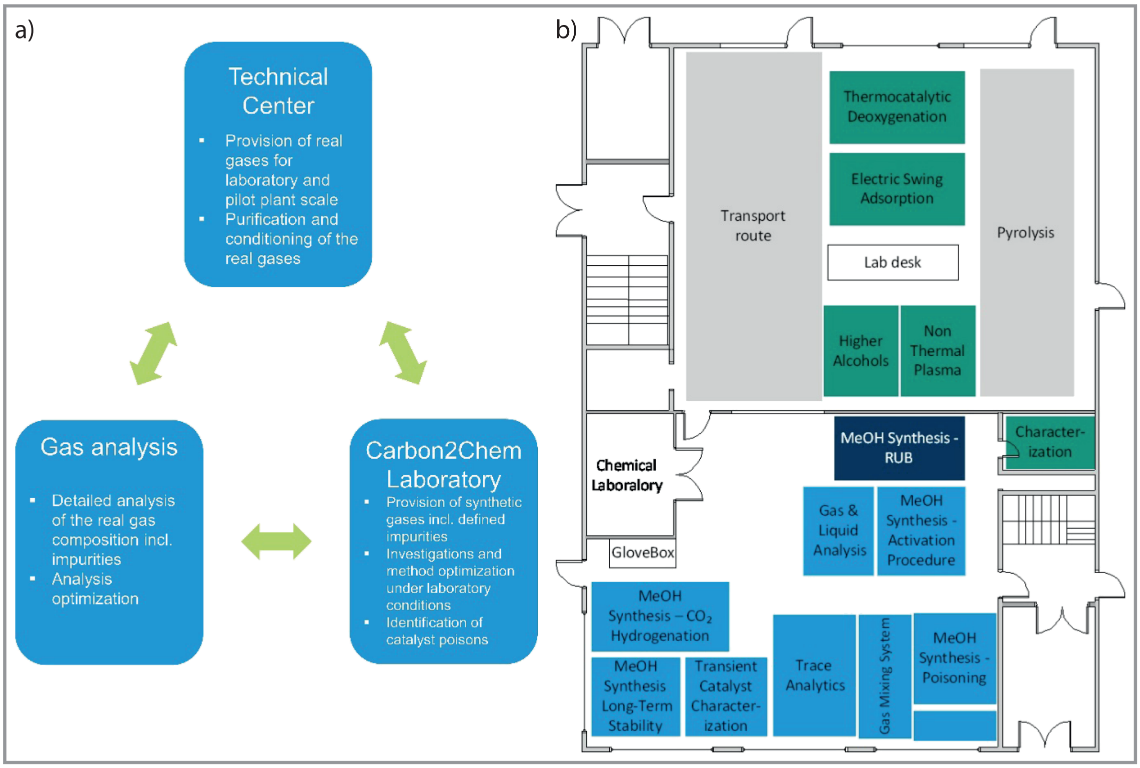


**Figure 1.** a) Role of the Carbon2Chem® laboratory within the Carbon2Chem® network. b) Floor plan of the Carbon2Chem® laboratory showing the workspace in the existing (top) and newly constructed (bottom) building for all project partners including Fraunhofer UMSICHT, Ruhr-University Bochum and MPI CEC.

**Figure 2.** Overview of the newly constructed laboratory of Carbon2Chem®.

ity in methanol synthesis, testing of gas purification systems, and validation of the previously named analytical equipment [14, 15].

Oxygen separation emerged as a particularly interesting challenge in gas cleaning, but at the same time it is also relevant for methanol synthesis. Using the gas mixing system, it was possible to work in close cooperation with other subprojects in Oberhausen as well as the technical center in Duisburg to clarify structure-activity relationships in methanol synthesis under the influence of trace oxygen. Simultaneously to the analysis of the simulated gases, the validation of the analytical equipment can be conducted offline using gas samples taken at different locations of the piping system with respect to the fragmentation pattern of individual components as well as the influence of the different treatment steps. In order to support the characterization of trace components in the exhaust gases and its purified form as well as for a trace analysis in the experimental plants operated in other subprojects, the bandwidth of analytical equipment is extended with a thermal desorption gas chromatograph coupled with mass spectrometry (TD-GCMS). The focus of the TD-GCMS features the validation of real exhaust gases, ensuring the functioning of the gas purification in the Carbon2Chem® technical center as well as a detailed analysis of liquid samples produced within the project.

### 3.2 Methanol Synthesis

One promising way to benefit from waste gas streams is methanol synthesis. This well-known reaction converts $CO_2$ into methanol and the industrial process has been investigated in detail in the recent decades [16–18]. Methanol is

  

Figure 3. Overview of the structure of the gas generator subdivided into individual plant parts from left to right: the two gas supplies, the liquid dosing station and the analysis section.

an important intermediate for the production of basic chemicals and fuels as well as a hydrogen storage molecule [19] and can be easily integrated into the current petroleum industry value chain [20]. The well-known $Cu/ZnO/Al_2O_3$ catalyst is used for this purpose and, especially because so much research has already been conducted on this catalyst and reaction, it is also the only alternative to enable a fast implementation [21]. Despite the known fundamental literature data, there are some big challenges to overcome for the industrial methanol synthesis using exhaust gases from a steel mill. In the Carbon2Chem® laboratory, several setups address this reaction and test the influence of critical reaction parameters, which can reduce or even prevent an economically sufficient productivity. The results obtained here can act as an interface between the different subprojects as well as the technical center in Duisburg.

In order to ensure comparability of the various investigations in methanol synthesis running in the differently constructed plants, a benchmark test as described in detail in [9] was performed (Fig. 4). All setups access the same central gas supply but differ in terms of design and manufacturer of the individual components. Nevertheless, the results showed an only small deviation of < 3 %, which highlights the comparability of the plants despite deviations in components and individual analytical methods.

Methanol synthesis is studied by MPI-CEC researchers at the various setups in Oberhausen, with a focus on the activation procedure of the catalyst by variation of absolute temperature, $\Delta T/\Delta t$ or reactant concentration. The activation procedure is indeed well studied, but the framework conditions differ greatly due to the changed process gas. For industrial applications, mostly purified high purity gases are used as synthesis gas, so that the aspect of catalyst poisons normally plays a minor role. However, the utilization of steel mill exhaust gases in methanol synthesis makes it essential to determine the catalyst's limiting threshold of selected trace substances that are present in the gas stream as well as the required purification steps of the process gas for a successful implementation. To achieve this, the undesirable components of the gas stream are investigated directly within the methanol productivity featuring their poisoning potential by coupling of the gas mixing system with a methanol synthesis setup [15]. In addition, the determination of the limiting load of the catalysts makes it possible to reliably dimension the industrial plants or the necessary purification steps. Furthermore, since the steel mill exhaust gases largely consists of $CO_2$, studies focusing on pure $CO_2$ hydrogenation and addressing the optimization of the corresponding reaction parameters are carried out in the laboratory. Here, the effects of the dynamic changes of the feed gas compositions, as a result of the different processes in the steel production, as well as the reuse of utilized process gases are investigated to study the interaction of methanol synthesis and reverse water gas shift reaction, and its impact on productivity.

Further methanol synthesis setups are operated by Fraunhofer UMSICHT at the location in Oberhausen with methanol production capacities between a few milliliters and up to 50 L a day in the demonstration plant shown in Fig. 4f. While the smaller setup serves to optimize the process operation under different conditions and to develop new control concepts, the demonstration plant is used to obtain first information about the catalyst behavior on a technical scale. This plant will be transferred to the technical center in Duisburg and operated with purified gases from the steel mill during the project. However, as a first step, several measurement campaigns are carried out in Oberhausen using artificial steel mill gases, leading to a better understanding of the process [22]. Moreover, setup configuration is adopted and optimized according to the first results as a prerequisite for smooth operation at the technical center.

### 3.3 Gas Cleaning and Conditioning of Steel Mill Process Gases

For a practical further use of still mill process gases, cleaning and conditioning is a key for success. Trace components harmful for the subsequent process steps have to be removed, and reaction components have to be adjusted in the feed gas. In addition to the application of well-known gas cleaning technologies that are already adapted and tested in the Carbon2Chem® technical center in Duisburg, the applicability of new approaches is initially verified in the Carbon2Chem® laboratory in Oberhausen under controlled conditions. After successful testing with artificial steel mill gases, the functionality of new processes has to be proven under real gas conditions. One example is the application of the electric swing adsorption (ESA) technology to

  


**Figure 4.** a) Normalized degree of conversion of $CO_x$ ($X_{COx}$) of the standardized benchmark test for methanol synthesis over an industrial $Cu/ZnO/Al_2O_3$ catalyst in the applied setups at 523 K and 50 bar. b–e) Setups used in the Carbon2Chem® laboratory of MPI CEC and RUB for methanol synthesis as well as f) Fraunhofer UMSICHT demonstration plant for methanol synthesis at the location in Oberhausen.

separate high-boiling hydrocarbons from coke oven gas (COG). In a small setup in Oberhausen (Fig. 5a) Fraunhofer UMSICHT identified and tested suitable adsorbents to study real effects in multicomponent adsorption at volume flows up to 100 NL $h^{-1}$. Moreover, different electrode and adsorber designs were investigated to realize efficient heating. Based on these results a basic pilot plant was designed, constructed, and put into operation in the technical center in Duisburg (Fig. 5b), which allows for the treatment of 9 $Nm^3h^{-1}$ COG. While gathering the first results under real gas conditions, investigations in Oberhausen are continued to optimize adsorption and desorption process, e.g., by varying the electrode setup. Furthermore, the separation of $CO_2$ and CO from nitrogenous gases by the ESA technology will be investigated in the laboratory. Therefore, specially doped adsorbents are synthesized, tested, and optimized regarding their potential to separate the target molecules. Here, the separation of CO from nitrogen appears to be a

  

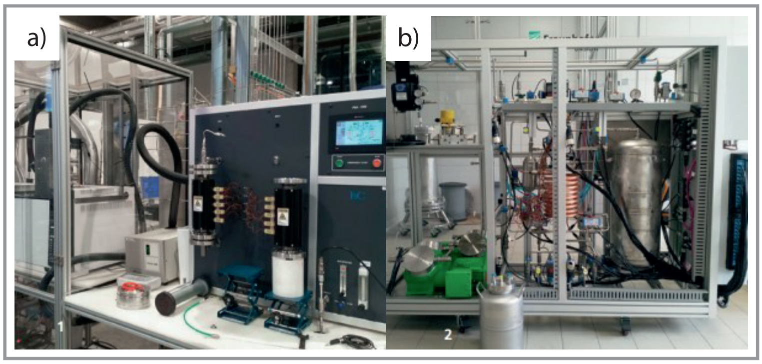


**Figure 5.** a) ESA setup with GC system for the measurement of minor components in the Carbon2Chem® laboratory. b) Pilot plant ESA for the analysis of steel mill gases in the Carbon2Chem® technical center in Duisburg.

particular challenge, since typical adsorbents show low $CO/N_2$ selectivity in pressure swing applications [23]. Finally, promising adsorbent materials will be applied in the pilot plant in Duisburg and their functionality will be tested using blast furnace gas.

Another topic of interest is the removal of oxygen traces from COG. Within the frame of the Carbon2Chem® project two different technologies for this are tested by Fraunhofer UMSICHT. One approach is thermocatalytic deoxygenation, which is investigated in a high-temperature fixed-bed reactor (so-called "TomCat", Fig. 6) enabling a time-resolved measurement of the main and trace components of artificial steel mill gases in the range of seconds. An electrochemical oxygen sensor is used to detect oxygen concentrations up to 1 vol % and a quadrupole mass spectrometer is used for the analysis of all other components. This investigation aims to identify suitable low-cost catalysts and to determine promising process parameters. To date, various catalytic systems (based on precious and non-precious metals) have been tested and showed encouraging oxygen removal behavior [24, 25]. Therefore, the results are used to plan and construct a pilot plant that will be connected and operated with COG upstream of the pressure swing adsorption system at the technical center in Duisburg. Accompanying tests with artificial COG in the laboratory will be continued.

**Figure 6.** Front view of the high temperature fixed bed reactor "TomCat".

An alternative approach to remove oxygen traces from COG is the treatment with non-thermal plasma (NTP). The corresponding setup of Fraunhofer UMSICHT allows for investigation of artificial coke oven gases and other complex gas mixtures with volume rates up to $1\,Nm^3h^{-1}$ and trace oxygen concentrations up to 1 vol %. The generation of NTP in the laboratory in Oberhausen is based on the volume dielectric barrier discharge (DBD) reaction system, whereas a surface DBD reactor system is the subject of investigations at the Ruhr-University Bochum. Fundamentals about DBD reactor design are shown elsewhere [26]. The investigation in Oberhausen started with a small volume DBD reactor applicable for volume flows of $0.1\,Nm^3h^{-1}$, which contains a quartz glass tube enabling the optical assessment of plasma formation (Fig. 7a). A thermal conductivity detector is used to detect $H_2$, while infrared cells are applied for the detection of CO, $CO_2$ and $CH_4$ and an electrochemical sensor is used for $O_2$ detection. With this experimental setup the technical feasibility for the deoxygenation of an artificial COG has been successfully proven [27, 28]. However, scale-up of the glass reactor is limited with respect to its maximum volume flow as well as operation at increased pressure. Therefore, the reactor design has been modified by substituting the quartz glass jacket by a stainless-steel jacket, whereas the quartz glass tube now covers the inner electrode (Fig. 7b). The new reactor design enables an easy scale-up and has been successfully tested in the laboratory for maximum volume flows of $1\,Nm^3h^{-1}$. Building on the promising results in Oberhausen and Bochum, a pilot plant with a capacity of $10\,Nm^3h^{-1}$ COG using both reactor designs is planned to verify the suitability of NTP for the removal of oxygen traces from real COG at the technical center in Duisburg. This plant is planned to start operation in 2022. Accompanying laboratory measurements in Oberhausen and Bochum will be continued to study the influence of increased pressure as well as selected trace components on deoxygenation.

### 3.4 Production of Higher Alcohols

In addition to the methanol synthesis from steel mill gases, the production of short-chain alcohols and olefins is investigated, which can be used as fuels and are starting materials for other important chemical building blocks. In contrast to methanol synthesis, this production route is not based upon the application of an already well-known and industrial applied catalyst. Within the frame of the Carbon2Chem® project, Evonik Resource Efficiency GmbH is developing a new specialized catalyst whose suitability has yet to be tested. During the first years of the project Fraunhofer UMSICHT used the so-called "Spider" test system in the Carbon2Chem® laboratory (Fig. 8) to screen the perfor-

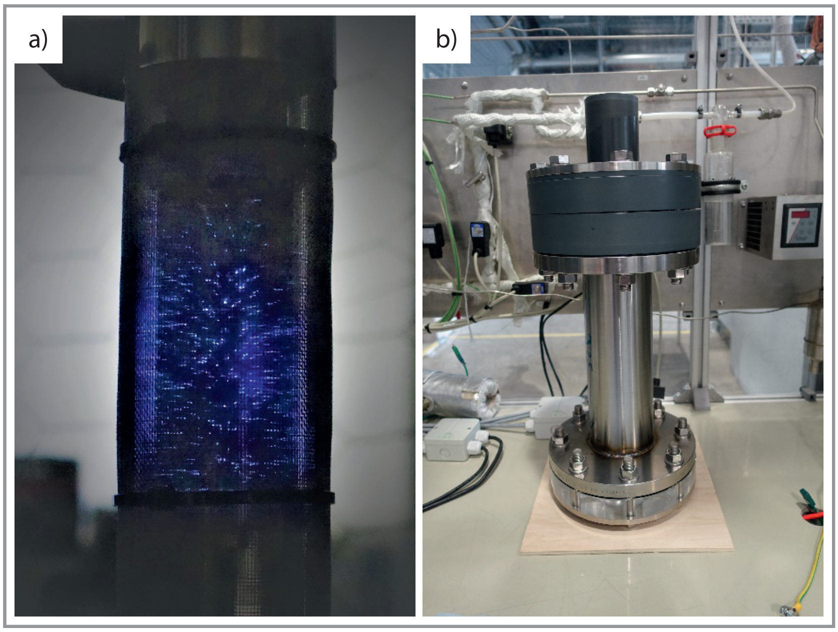


**Figure 7.** a) Non-thermal plasma in a quartz glass volume DBD reactor; b) scalable volume DBD reactor with stainless-steel jacket.

mance of over 70 newly developed catalyst samples. This setup enables the parallel investigation of eight powder catalyst samples with defined synthesis gas compositions under identical reaction conditions in an electrically heated 8-fold parallel reactor system with a subsequent GC-FID/TCD for analysis. Screening results are used to iteratively develop the new catalysts. The most promising samples are selected and modified by Evonik for a new iteration step. In this way, a first generation of promising catalyst materials was designed, which will be optimized further during the upcoming project period [29].

**Figure 8.** "Spider" setup for catalyst screening.

In addition to the optimization of catalyst productivity and selectivity regarding alcohols and olefins, a special emphasis is given to a catalyst design well suited for an industrial process. Investigations with shaped technical catalysts are necessary to gather information about mass and heat transport in the shaped catalyst as well as to answer important questions regarding optimal process conditions and long-term stability. These measurements will in part also be conducted in the "TomCat" setup, but a miniplant from thyssenkrupp Industrial Solutions at the technical center in Duisburg, another miniplant at the Fraunhofer UMSICHT institute branch in Sulzbach-Rosenberg, and the demonstration plant for methanol synthesis (Fig. 4f) will also be used for this purpose. Each setup delivers complementary information, which will finally help to demonstrate the scalability of the production of higher alcohols and olefins to the technical scale.

## 4 Summary and Outlook

"The goals of Carbon2Chem® can only be achieved in close cooperation between industry and science …." notes Prof. Görge Deerberg, Deputy Director of Fraunhofer UMSICHT [30]. The realization of a close cooperation in daily work within the individual projects and between all project partners is a great challenge. In order to improve coordination and cooperation, the establishment of a central workplace as a cooperation site for collaborative research is essential. There, the consortium partners are jointly researching different processes for gas treatment as well as the production of methanol, ammonia, and higher alcohols. The results obtained on laboratory scale with the constantly changing parameters of simulated steel mill exhaust gases or modes of operation resulting in different impurity concentrations and, consequently, the degree of purification required, provide the scientific basis for the work with real exhaust gases and should always remain part of the scientific research. The influence of dynamic behavior in a running process must be continuously monitored and then tested again on a smaller scale to avoid productivity losses or major failures. Based on these results, the design of a Carbon2Chem® pilot plant on-site at the steel mill in Duisburg can be further optimized and prepared for tests under industrial conditions. By this, the real steel mill gases can be accessed and tests under industrial conditions can be carried out.

  

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## Symbols used

| | | |
|---|---|---|
| *m*/*z* | [–] | mass-to-charge ratio |

## Abbreviations

| | |
|---|---|
| COG | coke oven gas |
| DBD | dielectric barrier discharge |
| ESA | electric swing adsorption |
| NTP | non-thermal plasma |
| PTR-QiTOF-MS | proton transfer reaction quadrupole interface time-of-flight mass spectrometer |

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