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Design of Module Type Package Services for Modular Downstream Units and Process Analytic Technology

Modularization of process plants with its standardization activities is one of the current responses to react to dynamic markets, shorter product life cycles, and uncertain supply chains. Standardized solutions for intelligent process equipment assemblies with own automation promise high potential for chemical and pharmaceutical industries. Despite the standardized description of the module type package (MTP) and the corresponding service concept, the implementation of the service logic is left to the manufacturer, which often leads to finding various granular services for different process functions or assemblies. In this contribution, different service design approaches for a generic 'separate' service are investigated on the example of a solvent extraction and a distillation column. Additionally, a Raman spectroscope device for process analysis is implemented via MTP with an 'analyze' service. Pros and cons of the different service design approaches are discussed in the context of a fast and flexible process development in the laboratory.

Supporting Information available online

Keywords: Automation, Modular downstream unit, Module type package, Process analytic technology, Solvent extraction column

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1 Introduction

With the 50 % idea [1] and industrial projects such as the EU-funded F^3 factory project [2], modularization in the process industry gained a momentum and today is often state-of-the-art in environments with short product lifecycles or for process development. Beside the process engineering activities, automation of modules and modular plants were a prominent issue, since each module was integrated with high effort by hand to process control systems. With the concept of decentralized intelligence of modular plants [3] and corresponding concepts, the idea of a manufacturer-independent interface describing the module automation was presented at the NAMUR annual general meeting in 2014 and then followed up by certain research and industry projects. This approach is now standardized in several parts of the guideline VDI/VDE/NAMUR 2658 [4] made by representatives of module manufacturer, owner and operator of modular plants, automation industries, and academia. With the defined module type package (MTP), a manufacturer-independent interface is given that enables fast and flexible modular automation for all relevant players. Now, the MTP technology finds its way into the industry and the plug & produce technology is ready for the market [5], and is now even being used in other domains such as logistics, maritime, and biopharma [6–8].

In the framework of the nationally funded project ENPRO2.0-ORCA, demonstrators and new methodologies for the fast and efficient orchestration of modular plants were developed and evaluated. Among others, results were partly published and presented in [9–15], while demonstrators of the industry and academic partners are further explained in [16]. Beside this, more details about the modular equipment developed at the Laboratory of Equipment Design at TU Dortmund University will be given in this contribution. According to the VDI/VDE/NAMUR 2658 guidelines, only the service interface itself is described, not any method or best practice on how to develop a service or the logic executed by the service. Therefore, certain strategies and approaches for the service design are investigated using the example of a distillation and a solvent extraction column together with the process analytical device of a Raman spectrometer.

2 State of the Art of Modular Automation and Modular Plants

Due to the complex wording and abbreviations in the topic of modular plants, a short introduction for the engineering of modular plants is given and supported by literature for further details. For the general structure of modular plants, the process

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engineering part is explained first, followed by the modular automation.

2.1 Setup of a Modular Plant – VDI 2776

In the guideline VDI 2776 [17], the hierarchical structure of modular plants is defined. A modular plant (MP) consists of one or more process equipment assemblies (PEAs). The modular plant represents an entire process, while each PEA contributes with a procedural step to make this process possible. Furthermore, each PEA is set up with one or more functional equipment assemblies (FEAs). FEAs fulfill a process engineering function and can already have own intelligence. FEAs are assembled with the smallest units, the components (COMPs), which are not anymore separable. By adjusting COMPs and FEAs, the operating range can be varied, e.g., for the use of other chemicals or process conditions such as temperature, pressure, or flow rates. The modularization of process plants and manufacturer-independent use of PEAs can only succeed if there is a common understanding of the structure of these process units and modular plants in general. More detailed explanation can be found in the guideline VDI 2776 itself as well in the publications [18–20] with emphasis on the engineering phase and safety issues.

2.2 Modular Automation – VDI/VDE/NAMUR 2658

A manufacturer-independent interface description of aspects of the PEA automation is required for the integration of the individual PEAs into a process control system. This interface is standardized in VDI/VDE/NAMUR 2658 [4, 21, 22]. An overview of the state of the art of this interface, related concepts and tools are given in the following short description. More information is provided in the Supporting Information.

The MTP is the automation interface description of a PEA. For control of a modular plant, the PEAs are integrated into a process orchestration layer (POL). Via the MTP, the POL gets necessary information about the automation structure of the PEA. The PEAs offer services with encapsulated process functions that can be executed by an automation system according to a service-oriented architecture (SOA) [23]. The POL is an automation tool that supports the entire engineering process from PEA selection to modular plant operation. A detailed description of requirements for a POL has already been published by Klose et al. [9].

For the communication between the modules, the concept provides that the PEA describes defined process values (ProcessValueIn or ProcessValueOut) as defined in the guideline VDI/VDE/NAMUR 2658 part 4 [24]. During engineering, these process values are either graphically linked directly to each other or interconnected via additional logic blocks in the POL. At runtime, each ProcessValueOut of the PEAs are continuously read by the POL, and each ProcessValueIn is continuously provided by the POL to the PEA to ensure communication between multiple PEAs.

For a detailed overview of the standardized interface description MTP and corresponding topics like the service-oriented architecture, process orchestration layer, and module-to-module communication, further references and short summaries are given in the Supporting Information.

3 PEA Pool at Laboratory of Equipment Design at TU Dortmund University

At the Laboratory for Equipment Design several micro- to mini-scale, continuously operated devices are investigated within the framework of various public-funded research projects. Although not all of the investigated PEAs can currently be integrated to a POL via MTP, they are equipped with automation systems that generally can be extended with MTP functionality. Some of them already use service-like architectures in order to completely execute desired tasks automatically. With the addition of MTP interfaces to these units, fast and flexible interoperability can be easy applicable. However, for the investigation of service design the focus is here on two downstream units, a distillation and a solvent extraction column, as well as a Raman spectrometer.

3.1 Solvent Extraction Column Setup

In prior research projects, stirred-pulsed extraction columns were developed in DN 15, DN 32, and DN 50 size. Here, the investigated column is a DN 32 stirred-pulsed extraction column with ten stirred cells [25]. Both feed streams, organic (light phase) and aqueous (heavy phase), are fed via dosing pumps in the top and bottom of the column as indicated by the arrows in Fig. 1. The heavy phase is fed at top streams to the column bottom and is pumped out for constant level control. The light phase fed at the bottom is rising to the top and leaves the column via an overflow. With applied level sensors (CleverLevel-LBFS, Baumer, Switzerland) in the settling zone

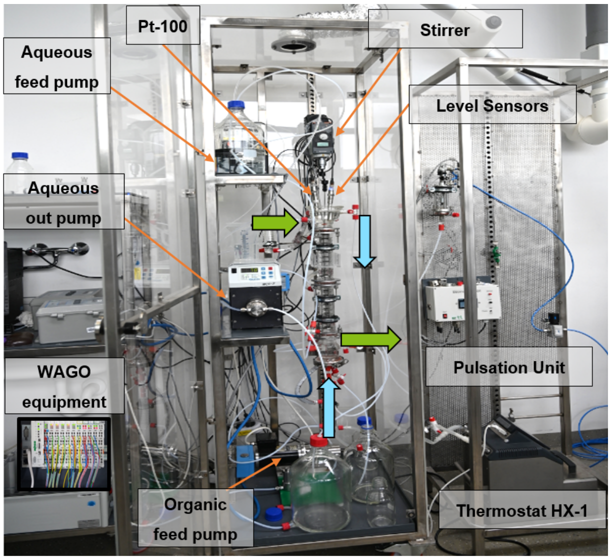

Figure 1. Monolithic setup of laboratory DN 32 stirred pulsed extraction column.

in the top, the phase interface can be continuously observed for continuous operation, hence, operator intervention is not necessary in normal operation mode [25, 26].

3.2 Spinning Band Distillation Column Setup

The distillation column (Normag, Hofheim) is set up very flexible with several additional PEAs necessary for the complete distillation process. This PEA arrangement ensures high modularity for exchanging the existent PEAs with those of other vendors with similar services. Additionally, the utility PEAs such as thermostats (Ministat 230cc, Huber Kältemaschinenbau AG, Offenburg) or feed pumps (Ismatec Reglo-Z, Ismatec, Wertheim) can be easily reused in other laboratory setups. However, this requires services designed in a way for compensating fluctuations of the process and that can be used in versatile setups.

The laboratory spinning band distillation column is intended to conduct distillation experiments and to provide information about the separability of mixtures with small amount of material and in a short time. For further literature regarding the process engineering and general capabilities of the distillation column it is referred to [27, 28]. In the following, the assembled PEAs and FEAs and the distillation process are described shortly. The labortory setup is depicted in Fig. 2.

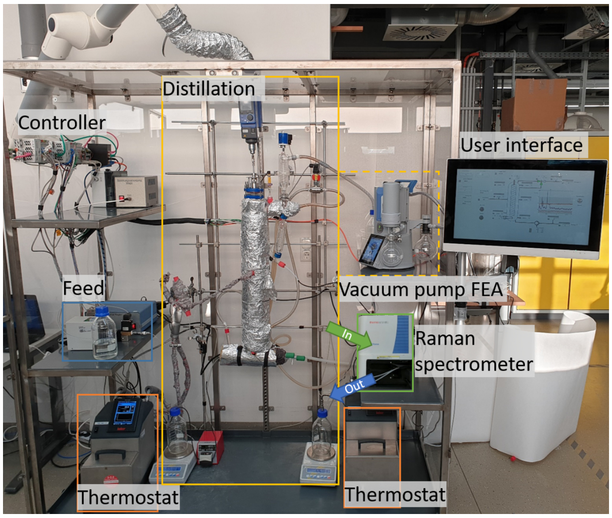

Figure 2. Modular setup of laboratory DN 25 spinning band distillation column and periphery units.

The feed PEA feeds a mixture of components into the distillation PEA with a defined mass flow rate between generally possible 1 and 50 g min^{-1}. Via a glass preheater located in the distillation PEA, the feed stream is preheated to a mixture-dependent temperature. A thermostat PEA tempers the glass preheater. Within the column, a heating rod in the bottom of the column boils up the liquid enabling the distillation process. The motor that drives the spinning band adjusts the speed of the spinning band. A compromise must be made here between high speed for better separation efficiency and flooding of the column, which occurs at high band speed. Flooding can be recognized by monitoring the pressure drop. As in conventional distillation columns, the purity of the distillate can be influenced by the reflux ratio. The distillation stream can be observed via the Raman analysis PEA described in Sect. 3.4.

Additionally to normal operation of the distillation column, a vacuum FEA can be added. Carrying out a vacuum service enables the vacuum distillation down to 50 mbar which is beneficial, e.g., for temperature-sensitive components, high-boiling components, and energy-efficient operation. The vapor in the head of the column is separated into reflux and distillate streams via a vapor divider and totally condensed in glass condensers. Similar to the feed preheater a thermostat PEA tempers the condensers. Bottom product and distillate are collected in glass vessels placed on scales to determine the overall mass balance.

In Sect. 4.2 the services of the distillation column are described more closely, the different procedures are also explained in the Supporting Information. The encapsulated services refer exclusively to the distillation PEA and could be performed in the same way even if the other PEAs (periphery and analytic) are replaced by functionally identical other PEAs, e.g., of another manufacturer.

3.3 Periphery Units (PEAs and FEAs)

For investigation of the mentioned service design of the downstream units distillation and extraction, some periphery units, also provided with MTP, were set up. The Huber thermostats, which are already equipped with an integrated Pilot-One controller, are available in different sizes, i.e., cooling or heating capacity. The thermostats can already be quickly and easily integrated into the POL via MTP.

The feeding PEA was designed and built in the laboratory of equipment design. Equipped with a storage tank, level sensor, pressure and flow sensor, pump and valves and automated with a WAGO PFC200 controller, the PEA offers a dosing service and an MTP according to the VDI/VDE/NAMUR 2658 guidelines. The vacuum pump was subsequently integrated into the distillation as an FEA. It can be operated via the vacuum service of the distillation PEA.

3.4 Process Analytical Units with MTP

Similar to periphery units such as the Huber thermostats, which are already provided with an MTP by the manufacturer, same is conceivable for online analytical units. Especially for modular plants operated in a continuous mode, online process analytical technology (PAT) is essential for observation and control of the process [29]. In modular plants, where the process, the product, and the setup change regularly, the PAT must also be correspondingly flexible with the possibility to be integrated manufacturer-independent to a POL. In addition, many PAT applications are associated with high investment costs, which makes it profitable to reuse them for process development. By providing the manufacturer-independent interface description MTP for a PAT device, the integration can be done automatically.

For publishing the data on an OPC UA server and making the data available for a POL, a python application has been developed for this analysis PEA. Within this application, the correct execution of the Raman software is observed and the recent concentration value including the timestamp are written to a local OPC UA server. Additionally, the correct service behavior for the 'AnalyzeQuant' service is executed. For example, when the Raman software or hardware has an issue and is not providing new concentration values, the service state changes is triggered from execute to hold by the logic within the Python application. By providing the requested data structure on the OPC UA server according to the MTP guidelines, an MTP is written for the analysis PEA including the three DataAssemblies AnaView for the three concentration values and a single service with a single procedure 'AnalyzeQuant'. The concentration values are additionally provided as ProcessValueOut for the module-to-module communication within the prototypical ABB POL. The architecture is illustrated in Fig. 3.

This method can be transferred to several applications for analytic units in a laboratory, e.g., for process development. The obtained flexibility allows rearranging modular process development plants very fast and efficient. In the Laboratory of Equipment Design one can imagine such analytic PEAs or FEAs for further spectroscopic methods (such as UV-Vis, refractometer and more), for slug recognition (like in two-phase flow microreactors) or even for image analysis tools extended by AI methods (exemplary for recognition of flooding behavior in extraction columns). These modular sensors can be applied iterative at different locations in order to experimentally find the best position and the best sensor for the task or the process with less integration effort for automation interfaces. The data availability is always given due to the OPC UA server and the fast integration time due to the MTP. Additionally, when applying PAT in real industrial environments, this approach can be very well extended by NAMUR Open Architecture (NOA) for safe integration of the device, e.g., into cloud architectures.

4 Service Design for Downstream Units and Analytical Devices

Generally, downstream processing units refer to all processes used to separate and purify multicomponent systems by mechanical, thermal, electrical or physicochemical processes. In this context of the proposed separate service, we will focus on continuously operated thermal and physical processes with liquid and vapor phase without chemical reactions or solids formation taking place. For these continuous downstream processes, typically a start-up procedure needs to take place before going into the steady state of the continuous process. Normally, this steady state is maintained as long as the feed flow does not change significantly in mass and concentration conditions and the outgoing streams meet the purification requirements. When the process is taken out of operation, also the downstream unit needs to be shut down in a controlled manner with a suitable shutdown process.

In the context of modular plants and the service design of the considered downstream PEAs, two possibilities for the general service 'separate' were identified. Option A and Option B are depicted in Fig. 4. First (Option A), the mentioned start-up, continuous operation, and shut-down processes are all carried out in a single procedure within the states starting, execute and completing of the state machine of the service. Another possibility (Option B) is to execute every step in an own procedure of a service 'separate'. Both options are generally possible with regard to the VDI 2658 guideline and the defined service state machine, but both have pros and cons regarding flexibility and usability.

Option A is more to integrate and easy to operate, as there is only one service which has to be started. However, for Option B, complex start-ups of a PEA can be carried out with more states which might be beneficial for the internal control logic and for handling exceptions or product specific start-up rules. In addition, if there are different procedures that can be used after the start-up, this option of a start-up procedure might be helpful.

Besides this, it is always possible to realize the start-up, normal operation, and shut down completely in a recipe conducted by the POL by triggering highly granular services without any procedural logic (Option C). In this case, the plant operator who configures the recipe in the POL needs the process knowhow for each PEA in order to conduct the start-up correctly. In addition, the integration effort for these recipes is already high for small PEAs. As mentioned before, it needs a lot of process knowhow for the operation of the PEA itself, which is not always given for the owner/operator, who prefers a plug & produce unit.

In cases where safety issues occur or an emergency shutdown must be carried out, this is always handled by the held or the stop and abort loop of the current procedure.

As verification and for better investigation of pros and cons for the suggested architectures for a 'separate' service, both were implemented to real PEAs. First, an extraction-PEA with the service 'extract' and second a distillation-PEA with the service 'distill'. Both PEAs are small-scale apparatus and operated continuously.

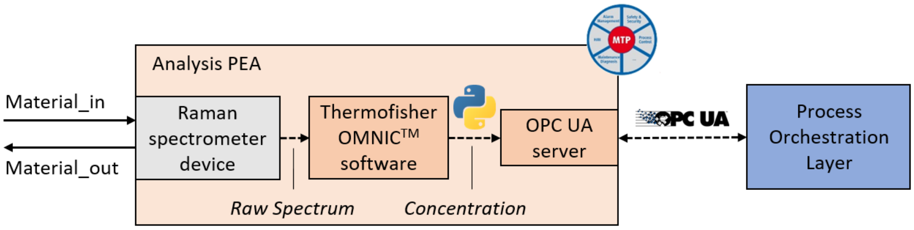

Figure 3. Architecture for the analysis PEA with a Raman device measuring a material flow, the OMNIC software processing the raw spectrum data, the python application writing the concentration data to an OPC UA server described via the MTP for integration to a POL.

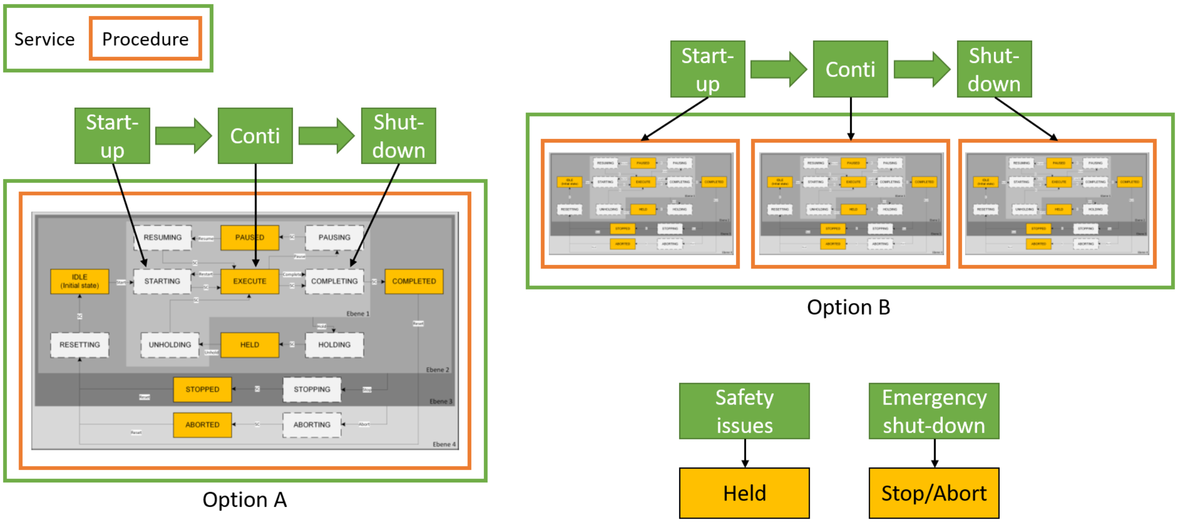

Figure 4. Options A and B for location of recipes for start-up, continuous operation, and shut-down with regard to services (green box) and procedures (orange box).

4.1 Liquid-Liquid Solvent Extraction Column

For the extraction column, the decision has been made for Option A because of the monolithic setup and yet no interaction with further PEAs are investigated, as shown in Sect. 3.1.

For the startup process, it is assumed that initially the column is empty, the feed vessels of both phases are filled, and the filling tanks of the column outlets for both phases are empty. These initial conditions are also partially checked within the service logic via level sensors in the column and the tanks. For the startup process the column is first filled with the heavy phase, here water at maximum possible volume flow rate until the lower top-level sensor recognizes this phase. Afterwards, the volume flow rate can be adjusted to the operating point that was set via a procedure parameter of the service and the light phase volume flow is subsequently introduced. At this point, the two countercurrent flowing phases are contacting, making the energy input via stirring and pulsating necessary for not running into flooding condition. Depending on the set value of the volume flow rate, the column is operated at this operating point. The time is determined by the geometry of the column (total internal volume) divided by the volume flow rate of the light phase. The resulting residence time is doubled and waited for until the service is switching from the transient state 'starting' to the state 'execute'.

The continuous operation is now running and the column is in a steady state requiring no manual interaction. Should it be necessary to set new values for the volume flow rates for the heavy and light phases, the procedure parameter can be overwritten and the service must be 'restart'. Like this, the 'starting' step is again conducted, new values are written to the PEA and after waiting the mentioned two residence times of the light phase, the service automatically switches to 'execute' again. Once the operator or the recipe within the POL is about to shut down the process, the shutdown can be initiated via the transient 'resetting' state.

For shutting down the process, the organic feed is shut down and the remaining organic component in the column is expelled via the overflow until the top-level alarm sensor detects the aqueous phase. This initiates to shut down the aqueous feed as well and the column is emptied completely through the bottom outlet. Additionally, further washing cycles could be carried out, e.g., with pure water but has not yet been implemented in the service.

4.2 Distillation Column

For the distillation column setup (described in Sect. 3.2) two services are implemented, the distilling service with four procedures, and the vacuum service with only one procedure.

4.2.1 Distilling Service

The distilling service offers four procedures for running the distillation process. With encapsulated logic for the start-up and shutdown processes, the operator of the column does not need detailed process knowledge, since all heuristics and necessary steps for start-up are already provided by the internal logic of the service. A manual procedure without internal control loops is offered which is sometimes necessary for specific experiments in the laboratory environment. Contrary to this, an automatic procedure was developed with control loops for several procedure parameters such as setpoints for spinning band speed, bottom liquid level, and temperature or concentration specification in the distillate. The description of start-up and shutdown procedure as well as manual and automatic operation is given in detail in the Supporting Information.

4.2.2 Vacuum Service

The vacuum service only has a single procedure for continuously maintaining the setpoint of the column pressure. The pressure is set via a procedure parameter generally allowing values between 50 and 1050 mbar. The low value limitation is additionally limited by the processed substance, detailed information follows. Since the supplier of the vacuum pump already offers internal modes of operation with own logic for, e.g., holding or releasing the pressure, the service was designed in such a way that these functionalities are addressed in the respective service states. Hence, the vacuum pump acts as an FEA with own intelligence, but is encapsulated within the distillation PEA via the vacuum service.

When interacting with the vacuum service, which maintains the pressure in the distillation column, it is necessary to monitor the highest actual column temperature in combination with the processed components and their vapor pressures. At all time, it must be ensured that the operator is not allowed to set a column pressure in a way where flash evaporation of the liquid occurs. This can be ensured, e.g., by setting configuration parameters that reflect the material properties for boiling temperature and vapor pressure of the substances. For the here investigated use-case in the laboratory, the question-answer functionality of the MTP was used. In the logic of the service, various substances and mixtures were stored with corresponding vapor pressure curves. The service inquires which substance is currently present in the column and proposes a list of available selectable substances to the operator. Additionally, the vacuum service is not allowed to start when the distilling service is not in the “Idle” state.

4.2.3 Distilling Test Service

Besides the separate services, a further service with intention to automate test recipes of new chemical systems is developed. As mentioned above, the distillation column is used as a small-scale continuously operated column for gaining first insights of the separation efficiency of new component systems. Therefore, a new product is often to be expected, for which the operation boundaries in terms of heating power and spinning band speed or gas factor and liquid load must first be determined again. This is of course possible by recipes and orchestration of previously presented services, but requires deeper process knowledge, while the experiments usually always run the same way. For this reason, as service with two procedures has been provided which already run through the process for the experiments internally, and only certain boundary conditions of the component system are set via service parameters initially.

In the ‘TestMaxQ’ procedure, a maximum of the heating power is experimentally determined. As this parameter is depending on several physical parameters of the component system as heat of evaporation, heating capacity, density of liquid and vapor, surface tension and further, simulation models fail due to little data of new component systems. By conducting the experiment with this self-completing procedure, a fast determination within 1 h and a material input of approximately 100–300 mL of the mixture is possible.

The second procedure ‘FloodTest’ is intended to gain the operating limits of the mixture for this column. As mentioned above, the spinning band speed forces the column to flood at a certain band speed. This critical band speed must be determined for further experiments regarding separation efficiency. By giving the range of allowed heating power according to the previous procedure ‘TestMaxQ’ and a step size for heating power and spinning band speed, the operating range is systematically determined by the self-completing procedure. Depending on maximum heating power and chosen step sizes for heating power and band speed, the procedure is taking between 1 and 5 h for determining the whole operation range of the column.

By completely encapsulating the logic within the service, the recipes for these tests are stored internally in the PEA logic. This eliminates the need to draw up detailed experiment plans, which saves a lot of time and material in the laboratory.

5 Automated Distillation Experiments for Characterization of Solvent Mixtures

As stated before, the start-up of the distillation column is carried out completely automatically by executing the service distilling and the procedure start-up. The behavior of the column in the service procedure start-up is exemplarily shown in Fig. 5 for the binary mixture of methanol and ethanol at ambient pressure.

Until minute 9 after start-up, the service is in executing state. When reaching a desired head temperature according to the

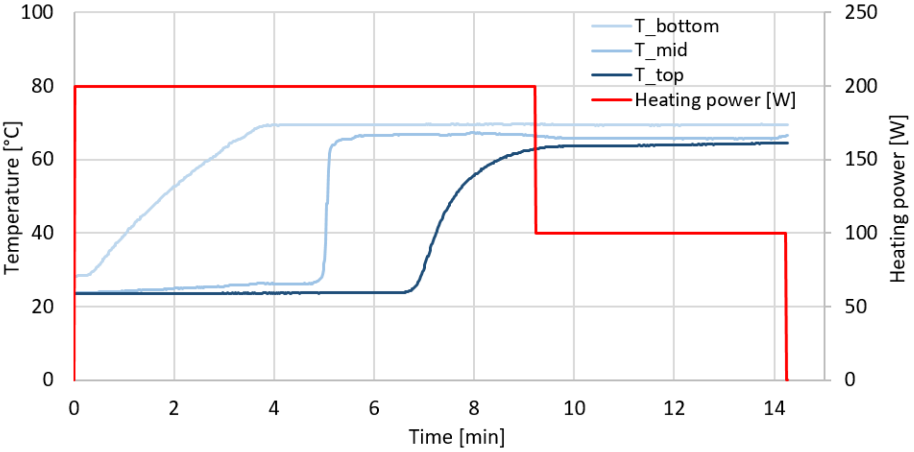

Figure 5. Exemplary behavior of the distillation column by executing the procedure start-up of the distilling service with the binary mixture methanol/ethanol.

current processed light boiler, the service is self-completing to the transient state completing. In the completing state, the heating power decreases and the state is resting for 5 min to allow the process to stabilize and ensure that a steady-state operating point is reached. The service is then completed, reduces the heating power down to zero and expects the start of a new procedure from the operator.

As possible follow-up of the start-up procedure, the service DistillingTest with procedure FloodTest is exemplarily displayed in Fig. 6 for evaluation of the flooding behavior with the binary mixture *n*-heptane/methylcyclohexane at 600 mbar pressure. This test system is a common and well-known ideal mixture with a boiling point difference of 2.6 K at ambient pressure. The algorithm executed within the PEAs control logic and encapsulated behind the service is carried out completely automatically within the service 'Execute' state. After completion of all necessary operating points, the service self-completes and the operator can evaluate the experimental data. The testing algorithm was validated with three different binary component mixtures at various column pressures. Hence, this service allows a fast and automatic evaluation of flooding points in this distillation column. Similar flooding services are conceivable for other downstream PEAs, as long as appropriate sensor technology is available to detect flooding behavior online.

6 Conclusion and Outlook

Increasingly dynamic markets, uncertain supply chains, and high energy prices demand for flexible and adaptable solutions. Modularization with MTP and flexible, small-scale, and continuous operated apparatus is one promising solution for a fast process development and modular production. With the presented approach of certain service concepts for 'separate' services and the integration of PAT devices via MTP, practical examples were used to show how process development can benefit from the MTP approach and become faster.

Depending on the complexity and requirement for flexibility of the equipment, it is advantageous to either provide one service and procedure that fulfills the whole task of the PEA and completely automatically, or otherwise to provide several procedures or services for higher flexibility. In both cases, however, the PEAs can be extended with versatile PAT applications to more accurately monitor and control the process. This was shown by integrating a Raman PAT device via MTP measuring the concentration of a product stream of the distillation PEA. For the distillation process and its automation, it does not matter, where the concentration value for the product stream comes from, which allows the use of the best measuring method for a particular substance system.

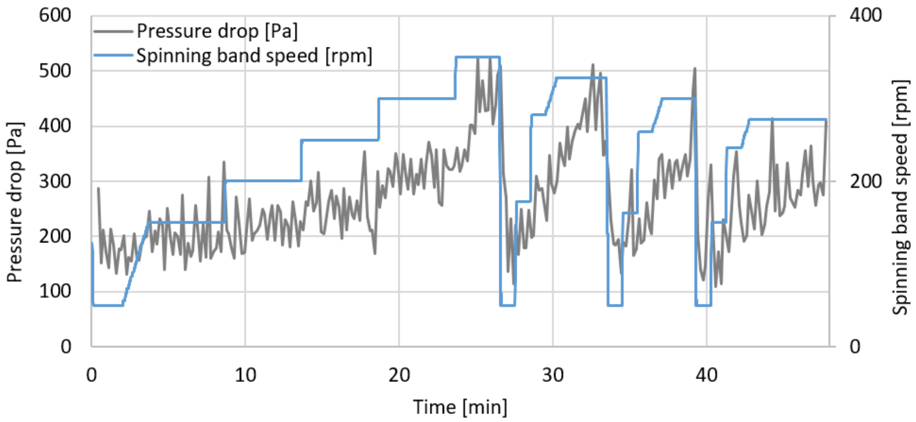

Figure 6. Exemplary behavior of the distillation column for the service DistillingTest service with the procedure FloodTest with the binary mixture *n*-heptane/methylcyclohexane at 600 mbar in the top.

Finally, appropriate sensor technology must be available for automatically recognizing certain operating states. Such recognition of operating states is not always possible by single sensors, therefore advanced smart sensors must be considered. Thus, machine learning (ML) algorithms can also help to reliably detect such conditions. To merge such ML methods within the service concept of the MTP, further investigations with demonstrator plants have to take place. First approaches have already been described by Oeing et al. [30] with the services and experimental setup presented here.

Supporting Information

Supporting Information for this article can be found under DOI: https://doi.org/10.1002/ceat.202200390. This section includes additional references to primary literature relevant for this research [31–48].

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Symbols used

P	[bar]	pressure
T	[°C]	temperature

Abbreviations

COMP	component
FEA	functional equipment assembly
ML	machine learning

MP	modular plant
MTP	module type package
NOA	NAMUR open architecture
OPC UA	open platform communication unified architecture
PAT	process analytical technology
PEA	process equipment assembly
POL	process orchestration layer
SOA	service oriented architecture

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