Autonomous Processes in Particle Technology

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Dedicated to Prof. Dr. Thomas Hirth on the occasion of his 60th birthday

Battery materials, pharmaceuticals, solar cells, coffee powder, 3D printed components, etc., all these products have in common that they are predominantly made of particles. Ensuring high product quality with optimal raw material and energy utilization is only possible with extensive and many years of experience in the operation of such processes. This unsatisfactory situation is due to the complexity of particulate products, which still hinders extensive automation and autonomous process control. The challenge is to couple the respective basic operations with characterization devices, process dynamics and modern control algorithms to form a closed loop for process control. As a result, some day it should be possible to set the desired property profiles of particulate products with the most energy- and raw material-efficient operation possible with a “push of a button”.

Keywords: Autonomous processes, Machine learning, Particle characterization, Particle technology

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1 Background

Particulate systems are the basis for many products from the chemical, energy conversion and storage, materials technology, pharmaceutical and food industries, among others. Due to their importance, the fundamentals of particle process technology have also been the subject of extensive research projects to date. Among other things, extensive models have been developed for the processes, detailed measurement techniques for characterizing the specific properties are now available, and process strategies for the most efficient operations of particle-producing and particle-processing plants have been explored. A particular challenge is that the properties of particulate systems can only be described by distributed functions according to the material behavior. As a result, the product properties as well as the process behavior are difficult to predict when process conditions change.

The measurement of the previously mentioned particle properties is usually carried out in a time-consuming and offline manner using special characterization methods, which only allow a determination with a certain statistical uncertainty. In addition, the properties are always distributed, i.e., they cannot be described with a single measurement value. However, with the help of soft sensors, which are methods for reconstructing property distributions from easily accessible measurement information, the particle properties can be reconstructed and correlated with the product properties. With a view to flexible and robust process control, online or, even better, in situ characterization methods are therefore necessary, where the measurement signals permit the fastest possible process response. ‘In situ’ here means that the measurement directly takes place in the process, without any sampling. In the meantime, there are methods that can be used as an in situ characterization technique, but these are rarely used to interfere directly in the process in the sense of an autonomous process control.

Another important development for particle production and processing in recent years has been the extensive development of simulation tools on different time and length scales. Extensive progress has been made as a result of the rapid development in computer technology. These have only made it possible to understand the often very complex processes in machines and plants and thus form the basis for optimized and more efficient processes.

However, the results of simulations are not used to exert a direct influence on the process. It seems to be conceivable to derive so-called short-cut or surrogate models from the simulations for process control and to use these for a permanent comparison with the measured values. If this can be done in real time or even predictively, it would ensure robust and safe process control while maintaining the process and product properties. However, short-cut models are not only obtained by means of complex simulations. Also, on the basis of physical balance equations and, under certain circumstances, empirical relationships, many proven process models are available in relatively simple, analytical

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relationships (so-called parametric models). Due to the extraordinary development in learning algorithms and GPU (Graphics Processing Unit) hardware in recent years, machine learning or artificial intelligence methods have led to breakthroughs in various technological areas. It is even conceivable that machine learning methods could be used to map product or process properties from data (non-parametric models) that cannot be described directly with physical relationships. Combining methods in the form of semi-parametric models, e.g., analytically existing, physically based correlations in conjunction with data-driven methods (e.g., using an artificial neural network or multivariate data analysis) appears attractive.

Ultimately, the determined correlation between the measured particle or product properties and the parametrically or non-parametrically existing process model can be used via the control or automation technology to directly influence the process. The basis for the development of autonomous, particle-based processes and thus for far-reaching digitization in the process industry is the feedback of the measured variables to the model-based process control.

Finally, the overall objective is to develop the basis for setting defined property spectra of particulate products by autonomous process control with optimum utilization of resources. In this context, autonomous process control is understood to mean processes that, ideally, only require observation by a human being and are capable of setting defined property distributions themselves without external intervention. This requires basic developments with respect to process models capable of being controlled, in situ characterization techniques for determining the particle properties in the process and new process control strategies.

The vision is that in the future a digital image of the process will interfere in process control in such a way that compliance with the quality of the product, process and product safety and resource efficiency is achieved independently. The proposed approach therefore provides the basis for autonomous processes by stringently combining modern control technology with particle technology, incorporating information technology, and thus lays the foundation for far-reaching digitization of the particle manufacturing and processing industry.

The raw materials, the final product, and the individual products after each process step are characterized using appropriate measurement techniques, if possible in situ. Controllable process models for robust process control are to be provided based on physical relationships and data-driven methods (see Fig. 1). In process control, these models are used to exert a direct intervention on the manipulated variables of the particle technology process via control algorithms or under knowledge of the process dynamics to be studied. The modeling, the measurement technology and the process control interact directly with each other on the physical and information technology level and thus form the basis for an autonomous process.

2 Mathematical Modeling

Mathematical modeling is a powerful method to quantify the relationships between process variables and particle properties, as well as between these and the product properties. The resulting models are an important basis for process modeling, optimization, and process control. Basically, they

Figure 1. Interaction between modeling, measurement technology and process control for the autonomous operation of a particle technological process.
can be obtained by theoretical and empirical modeling approaches and classified according to various criteria. A particularly important criterion is whether the parameters of a mathematical model can be associated with a physical or empirical meaning in the real world. In this context, we often speak of white, black and gray box models. However, these terms are also used to describe the transparency of models to an end user. Due to this, the classification proposed by von Stoch [1] will be used in the following as follows:
- parametric models (white box),
- non-parametric models (black box),
- semi-parametric models (gray box).

Parametric models (white box models) are developed based on prior knowledge and have a fixed number of parameters with physical or also empirical significance. Essentially, these are based on empirical correlations or on ordinary or partial differential equations. The main advantage of this type of modeling is the profound understanding of the process, which results in a high transparency of the model. Nevertheless, such models often tend to be very complex. The consequence is high costs in model creation and calculation, which sometimes makes them unattractive for process control tasks. A large number of parametric models already exist for predicting particle size distributions from process parameters [2, 3]. However, it should be noted that although parametric models are sometimes very complex and computationally expensive, their predictive power is limited. While, e.g., the particle size distribution can often still be predicted with corresponding approaches, product properties with rather imprecise causality, such as the stickiness, electrochemical properties or taste, cannot be determined directly in the process.

An alternative is offered by non-parametric models (black box models), which are developed based explicitly on data. In this context, ‘non-parametric’ does not mean that these models have no parameters at all; rather, the type and number of these parameters is flexible [1]. The advantage of data-driven models is the low computational effort, but they are mainly only applicable in the trained domain and can only extrapolate beyond this to a limited extent. Forms of such models that establish a connection between the relevant process variables in a purely data-driven manner are, e.g., multivariate data analyses, hidden Markov models (HMM) and machine learning (ML). A particularly prominent example of machine learning models are artificial neural networks (ANN), which undergo an iterative model discrimination and parameter identification process [4–12].

Semi-parametric models are a combination of parametric and non-parametric models. These hybrid models are frequently called gray box models. This approach allows process knowledge, such as the material and energy balances, to be combined with process data. Polynomial approaches, ANNs, support vector machines (SVM), etc. find their application as non-parametric models [13–15]. The modeling effort is reduced if the different information spaces are efficiently linked. In principle, the parametric and non-parametric sub-models can be interconnected in series or in parallel. This results in different application possibilities for the non-parametric models. In a serial structure, they can reproduce unknown reaction kinetics or mass transfer coefficients, while in a parallel structure, e.g., the deviation of a simple model from the measured data can be corrected additively. In current research, work is being done on stationary models for the design of processes, on dynamic models for process control and on the life-cycle-spanning use of the different models [16].

As an example, the stationary gray box modeling for the optimization of processes using dewatering of calcium carbonate water slurries in decanter centrifuges is described here. The mineral typically occurs during processing as finely dispersed calcium carbonate water slurry, which is often dewatered using decanter centrifuges to increase the solids content. The suspension is fed into the continuously operating centrifuge, pre-accelerated and directed into the rotating liquid pool within the machine. Due to centrifugal forces, the particles settle towards the wall of the drum, where sediment builds up. The clarifying slurry flows towards the weir, where it is drawn-off as centrate. Inside the centrifuge, a so-called screw rotates with a slightly different rotational speed compared to the actual rotational speed of the drum. This causes shear forces on the consolidating sediment and transports it out of the centrifuge via the conical section. Settling, sediment consolidation and sediment transport occur parallel. Additionally, the geometry parameters, process variables and material parameters influence each other and partly with contrary effects on the separation results. Both makes modeling of this apparatus complex.

Menesklou et al. [17] described a dynamic process model to simulate a decanter centrifuge considering settling, sediment consolidation and sediment transport. The simulation tool links the material with the machine behavior and reduces the complexity by reasonable assumptions, which enables real time simulations. The authors have shown that the model is indeed valid and usable for scale-up if the material characterization is successful [18]. However, this is coupled with some limitations. For example, the shortcut model assumes a plug flow through the entire pool and therefore does not calculate the flow via momentum equations. This would require detailed CFD simulations, which would increase the computational effort enormously. Especially for relative deep pools, deviations from this ideal behavior may occur due to local flow effects, which is not included in the mechanistic model currently. Often the precision of the white box model is sufficient, e.g., in process design, and it is very useful to have a simulation tool like that. However, in some industrial applications, a high accuracy specifically is required. The aim is to increase the accuracy of the actual model without losing the advantages of the dynamic simulation tool. Therefore, a stationary, parallel gray box model has been developed, whose structure is illustrated in Fig. 2.
The input vector $\mathbf{x}_m$ contains the relevant process variables: the rotational speed, the differential speed, the volumetric flow rate of the feed, the solids mass fraction of the feed and the pool depth. These variables serve as input for the white and black box model. On the one side, the mechanistic model calculates (among other output variables) the solids mass fraction of the cake and centrate. On the other side, the artificial neural network corrects the results from the mechanistic model, if it is necessary. This is possible because the ANN was trained with representative experiments in this range to model the deviations between the white box model and the experimental data due to effects that are not considered in the white box model. However, this does not mean that the white box model is not valid over a wide range. The point here is to combine experimental experience with a mechanistic model to increase the accuracy for specific practical applications. Fig. 3 demonstrates the validity of the presented gray box model for one exemplary case. The solids mass fraction of the sediment is plotted against the rotational speed at a constant volumetric flow rate and pool depth $h_p$.

Here, the experimental results, the simulation results of the pure white box model and the simulation results of the gray box model for different confidence intervals are compared. The confidence interval is used to avoid over-modeling. Both, simulation data and experimental data that are assumed in this range lead to a better statement about the reliability of the sampling, a confidence interval of ±2 wt % is recommended here. However, it is difficult to determine this mathematically, because it depends on the required modeling accuracy and the accuracy with which the samples can be measured. Generally, more measurement data in this range lead to a better statement about the reliability of the experimental results, so that the confidence interval can be determined more precisely and ideally becomes smaller. Furthermore, the black box model assists only in parameter ranges within the range of training data of the neural network. Thus, the neural network is just used to interpolate, because extrapolation of a neural network may lead to incorrect results. For these cases, the white box model is used as it is described in Menesklou et al. [17].

Thus, the neural network has learned to model additional effects and the gray box model can reflect the trend correctly. This shows that a gray box model is indeed a useful extension of existing mechanistic models. Of course, this single case represents only one example from the process industry. However, the training data set can be extended as
required and the neural network retrained. So, gray box modeling is a very useful tool to couple process data with existing physical models to receive a better estimation for the reality.

3 Particle Characterization

An essential prerequisite for effective process control and thus process management is the characterization of particle properties. The measurement of particle properties (e.g., mean particle size, particle size distribution, specific surface area, structure of agglomerates etc.) should be carried out directly at the process, either online or, if possible, in situ.

Extensive research has been conducted in this area in recent years, and a number of landmark publications can be found in the literature [19–25]. Crystallization, as an exemplary process involving phase transfer, often lies in the center of online image analysis. Ferreira [19] uses image analysis to understand crystallization phenomena of different sugar crystals in the presence of different impurity levels. Wang [20] relies on ultrasound emission and correlates it not only to crystallization phenomena but can also relate it to physicochemical crystallization conditions. Much can be expected from deep learning in the future, especially for fast and reliable image analysis [26]. Deep learning is a modern variant of a neural network that involves a large number of hidden layers, which allows practical application and optimized implementation. For example, the article by Scherr [27] shows how deep learning can be used in a high-throughput analysis to detect marker molecules on bacterial cells via rapid image analysis.

The monitoring of a time-sensitive separation process is studied here as an example for an efficient evaluation of the product properties. Concentration data of individual species and their particle size distribution can be defined as target variables representing the quality of the product. In a best-case scenario, this valuable information is obtained in situ from easily measurable process data enabling operators to avoid frequent, expensive, and time-consuming laboratory analyses.

The advantageous integration of soft sensors in separation technology was already demonstrated by Konrath [28], who determined the product loss of nanosized particles at the overflow of a tube centrifuge by in situ light scattering measurements. The tracked increase in product concentration was linked to the progressive sediment buildup inside the centrifuge. This led to a negative impact on the desired particle size distribution during prolonged nanoparticle classification. With in situ monitoring and signal processing, the grade efficiency of the isolated fine fraction was kept constant via a controlled and responsive increase of the rotor speed during the semi-continuous separation process.

However, the determination of multidimensional product properties, such as the composition of a suspension containing several species, requires a more adequate measuring principle, which can also function as a hardware component of the soft sensor. Here, the literature lists numerous evidence that UV/vis multi-wavelength spectroscopy is suitable for offline [29–31] and in situ [32, 33] monitoring of multiple physical properties in finely dispersed suspensions. Furthermore, in [34] UV/vis spectroscopy is applied for in situ monitoring of zinc oxide (ZnO) quantum dots processed continuously. Fig. 4 shows a schematic flow sheet of density fractionation in tubular centrifuges, in which a mixture of light polymethylmethacrylate (PMMA) and heavy ZnO nanoparticles are sorted according to their particle size and material density.

The separation outcome is set by the operating parameters of the centrifuge, the volumetric flow rate and the rotor speed.
speed. After sampling and time-consuming, invasive laboratory analyses by means of inductively coupled plasma optical emission spectrometry (ICP-OES), a specific product loss

$$P_n = \frac{\phi_{n,\text{overflow}}}{\phi_{n,\text{feed}}}$$

(1)
of the \(n\)-th component can be determined. Its value describes the ratio of the specific solids volume fraction \(\phi_n\) of one component at the overflow to the initial particle concentration in the feed.

Alternatively, a soft sensor allows the translation of in situ recorded UV/vis spectra into a prediction of the specific solids volume fraction of both dispersed substances. Multiple linear regression (MLR), as one of many applicable machine learning model structures, evaluates the relationship between multi-wavelength extinction data and the bivariate information of relative solid volume fractions. The resulting *Black Box Model* is then used to evaluate process data in real time as outlined in Fig. 4.

Exemplary results of this setup are shown in Fig. 5 where the sensor output (solid lines) is compared to the real product loss of PMMA and ZnO determined by the offline ICP-OES analysis (markers). In this example, the sensor reacts to changes in rotor speed. During a gradual increase, the nanoparticles are separated more effectively resulting in a decrease of product loss \(P\). Simultaneously, the fine fraction composition can be monitored in real time, giving access to an important aspect of product quality. More information on the experimental setup, modeling and effectiveness of this UV/vis soft sensor can be found in Winkler et al. [35].

Regarding measurement methods for the characterization of particulate materials, it can be summarized that in some cases adequate methods are available for in situ use. However, for the measurement of specific particle or product properties, the setup is very complex or not possible, so that model-based measurement methodologies must be incorporated. This is a necessity when no measurement information is available for direct intervention on the process. In addition, the methods also explicitly serve to determine the relationship between particle and product properties. For the process control envisaged in this application, measurement methods are to be used in which the time for measurement acquisition, evaluation and, if necessary, modeling is considerably shorter than the characteristic process time.

### 4 Model-Based Control

Examinations concerning model-based control for processes with particles mainly concentrate on a limited selection of few examples. One of them which is to be mentioned here is the mechanical separation of Li-ion battery materials for their direct recycling.

Without doubt, battery recycling is imperative and urgent. Sensible and sustainable recycling processes are still rather lacking, though, since batteries are complex compounds of a variety of materials challenging to actually recycle, especially the electrode composites. The most efficient, but also most complex approach is direct recycling, which makes use of different physical properties of the materials in order to separate them, like density, allowing for a specific recycling treatment afterwards. After opening the case and sorting, the remaining black mass (basically a slurry of electrode material) must be subdivided into its components, i.e., active materials, additives enhancing the electrical conductivity, as well as binders [36, 37].

In the exemplary process presented here, the cathode active material lithium iron phosphate (LiFePO₄, LFP) is to be separated from conductive carbon black (CB). The also present binders carboxymethylcellulose (CMC) and styrene butadiene rubber (SBR) may be neglected in the following. The separation mechanism is based on their unequal densities, whereby LFP has the higher one, and carried out in a tubular centrifuge. In this specific case, the centrifugal process shall result in the separation of LFP particles exclusively (obtained as sediment at the end of the process) and the centrate containing all CB particles, i.e., complete fractionation. A tubular centrifuge can only be run semi-continuously but has been chosen due to the small particle sizes partly down to 300 nm, which require very high centrifugal forces to be separated. During the separation process, sediment accumulates inside the rotor, reducing the free cross-section and consequently separation efficiency over time if no countermeasures are taken. Therefore, tubular centrifuge processes always show a strong time dependency. If a certain outcome like complete fractionation of LFP and CB is desired, such countermeasures are inevitable. Generally, adjustments can be made via the operational parameters, i.e., rotational speed and feed flow rate, whereby the model-based increase of rotational speed has been
examined in the experiments elucidated below. More detailed information can be found in [38]. It is worth noting that the properties of LFP particles require strong centrifugal forces and a tubular centrifuge was the first choice, justified under research conditions. Other centrifuge types for continuous processing and large throughput of active materials are subject of actual research. In order to separate CB from the process water in a second (cleaning) stage, the very high forces in a tubular centrifuge are necessary yet.

The separation process inside the centrifuge rotor depends on the actual operational settings, materials used, and previous settings in the specific process run, i.e., time and the spatial distribution of particles in the rotor. All these influencing factors are also interlinked in their effects. It is evident that with this number of concatenated factors, a valuable description of the process with one small invertible set of equations is not possible. Therefore, a short-cut model has been developed according to [39], closely following the principles mentioned in Sect. 3 describing the behavior of LFP and CB in the tubular centrifuge. Evaluative faster than in real-time, it is appropriate to serve in a nonlinear model-based predictive control (NMPC) approach [40] in order to set sensible rotational speed values yielding the desired fractionation outcome.

The MPC (model predictive control) concept generally relies on a process model depicting well the state and development of the process variables. NMPC is a variant of MPC with the feature to use a nonlinear process model, which is the case for the herein used tubular centrifuge model. The fundamental idea behind MPC is to use such a model to predict prospective values of the regarded process variables, given time and actual manipulated variable values, and utilize it to find the optimal settings for the manipulated variable (one or several operational parameters) to proceed close to the desired set point. For this purpose, the model calculations run short time intervals into the future while the outcome is evaluated in a dynamic optimization loop. Of course, the process variables have to be measured, too, in order to give feedback to the controller about the actual state of the process and thus the control quality. Deviations between model predictions and measured reality are taken into account as bias, so that model imperfections and unforeseen disturbances are detected and compensated [41]. Thus, MPC is a convenient option to control processes that are generally complex but can be modeled with adequate correctness. A principal scheme of model predictive control loop is outlined in Fig. 6. Compared to classic control approaches, mainly the controller is simply replaced by an MPC controller (or generally any model-based controller for another model-based control concept). It has to be mentioned that the MPC controller only gives recommendations, but still requires the process control system as an interlayer with simpler, cheaper controllers, e.g., PID, actually putting the setting into practice.

The mentioned model for fractionation of LFP and CB in a tubular centrifuge was correspondingly applied to design a model-based feed forward control for first examinations within a feasibility study for the intended adjustment of rotational speed. The entire study and more background information is accessible in [38]. The aim of the study was to prove that the model-based rotational speed increase results in the desired output over the entire operational time, namely a constant particle size distribution in centrate. Some results are shown in Fig. 7. Three operational cases are compared, one setting with constantly weak forces (20 000 rpm, i.e., a centrifugal acceleration about 94 000 m s\(^{-2}\)), one with constantly strong forces (40 000 rpm, equivalent to an acceleration of approximately 377 000 m s\(^{-2}\)) and the model-based increase. The overall separation efficiency indicated in Fig. 7a is defined as the ratio of solid mass in centrate to solid mass in feed and runs horizontally if the desired constant output is achieved. The same applies to the particle size distribution characteristics shown in Fig. 7b. This is obviously not the case for the scenario with weak forces (blue), which shows rather the undesirable impact of the rotor filling up over time. The scenario with strong forces (red) performs better, but still not constant over time as desired. Especially the characteristic particle sizes indicate that too many particles are separated most of the time, including CB particles entering sediment, which is only slightly acceptable in the posterior LFP regeneration process. Finally, the rotational speed adaption based on the real-time model yields good results as desired, the characteristic particle sizes remain particularly constant.

Overall, these experiments show that model-based prediction yields the desired permanent output, which is necessary in order to allow for proper treatment of the distinct electrode materials before they are used in new batteries. NMPC here provides the opportunity to treat materials with complex behavior in an interlinked process adequately, paving the way to direct battery recycling utilizing con-
trolled centrifuges for the key step, which is clean fractionation. In the exemplary process shown, naturally online measurement is necessary in order to provide feedback signals. Besides, e.g., UV/vis spectroscopy for the composition (cf. Sect. 4), particle size distribution measurement is required to completely monitor the fractionation of the mixture. Both will ultimately be combined in a soft sensor, finalizing the fractionation setup to run fully automatically.

The potential of model-based optimization and process control is already visible today in rare process examples but is far from being exhausted. An indispensable prerequisite for the development of future autonomous particle processes is a well-coordinated management of entire process chains, which represents absolutely new territory in particle technology. This requires targeted investigations on the dynamics of coupled particle processes as well as the development of suitable modularization and/or hierarchization concepts to control complex multi-stage systems.

5 Outlook

To achieve these goals, control-capable process models, in situ measurement techniques and suitable methods of process control are essential. The described challenges are part of the newly started DFG Priority Program 2364 “Autonomous processes in particle technology – Research and testing of concepts for model-based control of particulate processes”. In close cooperation between scientists from the fields of mechanical process engineering and particle technology, control and systems process engineering, as well as computer science and mathematics, new strategies for autonomous processing of particles will be developed. Existing approaches are to be taken up and further developed in a targeted manner. However, these must be supplemented by the development of completely new methods. In addition to individual process types, which have hardly been investigated so far, the focus is now on entire process chains and their targeted interaction with regard to the above-mentioned objectives.

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Marvin Winkler studied chemical process engineering at the Karlsruhe Institute of Technology and is working as a research associate in the group process machines under the supervision of Prof. Nirschl since 2017. His research investigates the semi-continuous centrifugation of nanoparticles in high-speed tube centrifuges. This includes a multidimensional examination of the separation process and real-time monitoring of essential particle properties.

Tabea Sinn studied chemical process engineering at the Karlsruhe Institute of Technology and is working as a research associate in the group process machines under supervision of Prof. Nirschl since 2018. Her research addresses the automation of processes for particle separation and classification, mainly focusing on Li-ion battery recycling applying centrifuges. This includes the development of real-time models, investigation and implementation of model-based control strategies as well as the design of appropriate soft sensors.

Philipp Meneskou studied chemical process engineering at the Karlsruhe Institute of Technology and is working as a research associate in the group process machines under the supervision of Prof. Nirschl since 2018. The focus of his research is on the development of simulation tools for the optimization of decanter centrifuges. This includes modeling, experimental material characterization and application of AI in process engineering.

**Symbols used**

- $h_p$ [m] pool depth
- $n$ [min$^{-1}$] rotational speed
- $P_n$ [--] product loss
- $t$ [min] time
- $\phi$ [--] solids volume fraction

**Abbreviations**

- ANN Artificial neural network
- CB Carbon Black
- CMC Carboxymethylcellulose
- ICP-OES Inductively coupled plasma – optical emission spectrometry
- LFP LiFePO$_4$, lithium iron phosphate
- MLR Multiple linear regression
- MPC Model(-based) predictive control
- NMPC Nonlinear model(-based) predictive control
- PMMA Polymethylmethacrylate
- SBR Styrene butadiene rubber
- UV/vis Ultraviolet visible

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Review Article: The extensive developments in modeling, characterization devices, computing and storage technology and data communication in recent years now enable extensive research and testing of methods for autonomous process control in particle technology.