**Physical-Chemical Analysis of Industrial Processes: Advanced Spectroscopy and Chromatography for Real-Time Process Optimization**

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**Abstract.** This research is offering an integrated approach on the physical-chemical characterization of industrial processes including advanced spectroscopic, chromatographic and thermal analysis techniques combined with machine learning methods for real-time tuning of process optimization. Monitoring and Control Engineering / Industrial applications Three industrial sectors will be investigated, petrochemical refining with fluid catalytic cracking unit monitoring being central, pharmaceutical manufacturing including real-time polymorph detection/control and food processing involving contamination detection/quality assurance. The placement of the analytical suite is combination Fourier-transform infrared revealed that they're not spectroscopy and Quantum cascade laser for mid-infrared analysis (resolution 0.5 cm⁻¹, detection limit of 10 ppm for hydrocarbons), Raman with transmission and spatially offset configuration subsurface analysis, Gas chromatography with vacuum ultraviolet (GC-VUV) comprehensive hydrocarbon analysis, mass spectrometry process gas composition monitoring in real time. Machine learning algorithms, such as CNN for spectral pattern recognition and RNN for temporal process prediction demonstrate the classification accuracy of 97.8% in predicting catalyst deactivation states and the prediction error of 2.3% in predicting product yield. Application on a 120,000 barrel per day FCC unit shows that the APPCP technology can provide an early warning for detection of catalyst coking onset by 45 mins ahead of the temperature-derived signals traditionally used in refineries thus informing preventive manipulation to improve yield by 3.2% and save energy consumption by 8.7%. Pharmaceutical manufacturing In pharmaceutical manufacturing, real-time polymorph detection with 99.2% accuracy avoids batch failures valued at $250,000 each occurrence. Food processing applications realize contaminant detection levels of 0.1 ppm with a high specificity of 99.9%, helping to decrease costly product withdrawals. The combined analysis platform diminishes the analytical time from hours to seconds and offers a better accuracy of 42-68% than traditional lab methods enabling process industries to be more well-prepared for being part of Industry 4.0 by integrating digital twin or predictive procedure control.

**Keywords:** process analytical technology, spectroscopy, chromatography, real-time monitoring, process optimization, Industry 4.0, machine learning

**INTRODUCTION**

Physical n2hemical analysis of industrial processes has transformed from off-line laboratory-based testing to on-line integration and monitoring control systems, thus, facilitating proactive process optimisation and control. PAT Market is Worth $8.9 Billion by 2027 – New Industry Data PAT (process analytical technology) for pharmaceutical manufacturing, efficiency in the petrochemical sector and quality assurance for food processing are features driving a $2 billion increase according to Global Industry Analysts. Conventianal analytically based control leads to huge delays between sampling, analysis and adjust the process due to inferior operation or bad product. Contemporary PAT systems combine state-of-the-art analytical instruments with datamining and real-time control technology to deliver close-loop optimization capabilities [1-2].

This project will address long standing challenges in industrial process analysis through the construction of a multifunctional analytical system comprising multiple orthogonal technologies with advanced data analytics. The work is guided by three typical industrial application cases, which are selected to cover various physical-chemical analysis needs (e.g., petrochemical refining - complex hydrocarbon mixture and catalyst monitoring; pharmaceutical manufacturing -polymorph detection and reaction monitoring; and food industry - contaminant detection and quality control). Standardized methodologies for data integration, calibration transfer among instruments and real time analytics are developed , allowing the translation of PAT to multiple industrial areas. An industrial combustion process such as incinerators has been considered in the validations, giving some insight into the practical challenges and economic gain of a full industrial scale implementation.

**ANALYTICAL PLATFORM DEVELOPMENT**

Advance thinking A full portfolio of advanced analytical instrumentation was combined for the various needs in petrochemical, pharmaceutical and food processing analyse. The spectroscopic core comprised a Fourier-Transform Infrared Quantum Cascade Laser (FTIR-QCL) system in the mid-infrared range (700-4000 cm⁻¹)) with spectral resolution of 0.5 cm⁻¹ and fast scan rate of 100 spectra/second. This setup was connected to a home-made, high-pressure and high temperature flow cell that can be used at up to 400 °C and 200 bar. Raman systems (Ex 785 nm, range 100-3200 cm⁻¹, resold. 4 cm⁻¹) were used for surface anal.; Spatially Offset Raman Spectroscopy (SORS), Non-invasive sub-surface probing to a depth of up to 5 mm; and Transmission Raman Spectroscopy (TRS) Bulk comp-analysis of pharma-tablets. The real-time analysis of gasses was accomplished with a Process Mass Spectrometer, which employed electron impact ionization for continuous monitoring (to 1-500 amu/mass range) at 100 amu/second scan frequency through multiplexed sampling from as many as 16 different process points [3].

Chromatographic capabilities were equally robust. Gas Chromatography with Vacuum Ultraviolet (GC-VUV) detection provided a universal hydrocarbon detectability with compound specific spectral characteristics in the 115-240 nm window. For the most complicated mixtures, comprehensive (GCxGC) was used up to a peak capacity of over 10,000. Drug applications were facilitated by Ultra High-Performance Liquid Chromatography (HPLC) systems connected to photodiode array and mass detection. The samples were analyzed by DSC to determine the Tg (using a modified with flow-thru cells for process stream analysis) and in the TGA-MS for evolved gas analysis [4].

Durable, application-specific sampling interfaces were also necessary for the preservation of valid sample data. For petrochemical applications, filters were integrated which can resist high temperatures and pressure levels (ceramic filing grade 0.5 μm) for the specific - particulate separation and multiple cooling stages. For pharmaceutical applications, the sampling had to be aseptic with CIP and SIP possibility which guarantees sterility. In food industry, preference was given to non-contact optical sampling (e.g., for NIR and Raman if applicable) where possible; and automatic cleaning cycles were included in the contact probes in order to avoid cross-contamination and biofilm formation.

Data lifecycle was coordinated by one software architecture. Real-time data collection employed communication via the secure and standardized OPC UA protocol, with dynamic sampling rates ranging between 1-100 Hz according to the analytical technique. The acquired data, and specifically large spectral and chromatographic files, were stored within a custom-built time series database using efficient compression techniques. The calibration of the instruments was automated and transferred between instruments of the same model during the analysis.

Sophisticated machine learning software served as the analysis engine. Spectral analysis employed a 5-layer CNN (kernel sizes ranging from 3×3 to 7×7), including one max-pooling and three fully connected layers, for the pattern recognition and quantification. Process prediction and trend analysis was also conducted with the use of LSTMs, which had 128 memory cells to encode temporal dependencies. For quantitative modelling, PLS (Partial Least Squares) regression combined with genetic algorithms for best set selection was used. One-Class SVM and deep learning autoencoders were used for anomaly detection, to detect process disturbances and quality issues in real time [5-7].

**PETROCHEMICAL REFINING APPLICATIONS**

To satisfy these needs, a 120,000-B/D FCCU was significantly instrumented. The FTIR-QCL system was located at the outlet of the reactor with six representative sampling points for on-line HC speciation. A process MS was allocated for on-line definition of the regenerator flue gas to monitor emission and combustion. A high-temperature Raman probe was placed into the catalyst recycle loop to directly track coke formation and catalyst integrity, while a GC-VUV apparatus allowed detailed liquid product analysis every 15 min.

The built-in analysis system provided accurate monitoring control. With respect to catalyst monitoring, the coke content estimation error was 0.12 wt% ([Formula: see text] = 0.96), metal poisoning was detected consistently at thresholds >50 ppm for Ni and >30 ppm for V, and an excellent correlation (R² = 0.94) with offline microactivity test (MAT) results was observed. For product quality control, the system estimated gasoline octane number (mean absolute error 0.3) and diesel cetane number (0.4), determined light gas (C1 to C4) content within ±1.5%.

Performance display were: The benefits of implementation were significant in terms of economics and operations; a 3.2% increase in yield (4,000 BPD), saving – reduced energy consumption by 8.7 % in the reactor/regenerator system due to better heat balance; catalyst savings improved from 12%, while emissions dropped by --15% for SOx and –-8% for NOx.

In addition, hydroprocessing unit optimization was also carried out using supplemental sensing techniques: UV fluorescence for real-time sulfur measurement (LOD 0.1 ppm), chemiluminescence for on-line nitrogen analysis and near-IR (NIR) spectroscopy for determining aromatic content.

This method provided a very good precision with the sulfur prediction error at 0.5 ppm for the level of 10 ppm. The system had a built-in 1-minute delay time to monitor the desulfurization efficiency and take appropriate actions. In addition, the use of real-time data allowed optimization of hydrogen consumption, which saved 8.2% by matching hydrogen feed to sulfur load and product specifications on a minute-to-minute basis.

**PHARMACEUTICAL MANUFACTURING APPLICATIONS**

One of the most challenging aspects in pharmaceutical production is crystal form control, as 65% to 90% of drugs exist as more than one polymorph and each possesses different physicochemical properties influencing bioavailability, stability and manufacturability. For this purpose, an advanced analytical strategy encompassing the use of Raman spectroscopy in combination with multivariate analysis for primary recognition while being backed-up by established reference measurements using X-ray diffraction (XRD) and high-throughput screening methods employing Near-infrared (NIR) spectroscopy was conducted.

**TABLE 1.** Polymorph Detection Performance

|  |  |  |  |  |
| --- | --- | --- | --- | --- |
| **Compound** | **Polymorphs** | **Raman Accuracy (%)** | **NIR Accuracy (%)** | **Detection Limit (% minor form)** |
| **Carbamazepine** | Forms I, II, III | 99.2 | 97.8 | 0.5 |
| **Ritonavir** | Forms I, II | 98.7 | 96.3 | 1.0 |
| **Indomethacin** | α, β, γ | 97.5 | 95.2 | 1.2 |

These enabled the monitoring of polymorphic transformations during crystallisation processes in real time as well as providing on-line control of the final form. This function was responsible for helping avoid expensive batch failures, saving $250,000 per incident, so it became a particularly useful feature. The implementation of real-time IPC monitoring also enabled a 75% decrease in off-line laboratory tests, resulting in significant reduction in the analytical workload and faster release times.

For both chemical synthesis and unit operations, a combined multi-technique approach was used; in-situ Raman measured reactant and product concentrations, FTIR tracked functional group transformations and reaction calorimetry provided real-time heat flow for kinetic analysis and safety monitoring.

This tight coupling resulted in very precise process control. The reaction endpoint determination was accurate to 99.5% and over-processing was minimized by the system. It was instrumental for the early detection of impurities formation at 0.1% level, and corrective measures were taken prior to specification breach. Furthermore, the models offered accurate yield prediction with a granary mean absolute error less than 1.5%, and hence will help in improved production planning and resources apportioning.

**FOOD PROCESSING APPLICATIONS**

Monitoring of beat safety for chemical and biological hazards should be rigorously conducted. Important targets are pesticide resi-dues (organophosphates, carbamates and pyrethroids), mycotoxins (aflatoxins, ochratoxin, fumonisins) and allergens (peanut, gluten, milk proteins). A two tiered analytical approach was selected based on a trade-off between speed, specificity and sensitivity. The above-mentioned confirmative, quantitative analysis for investigation of regulatory limits was carried out by liquid chromatography-tandem mass spectrometry (LC-MS/MS). For fast off-line or on-line screening, Raman spectroscopy combined with surface-enhanced substrates (SERS) yielded sensitive detection of several classes of contaminants without the need for elaborate sample preparation. Such immunoassays were also used for initial high-throughput screenings of certain allergens and toxins.

**TABLE 2.** Contaminant Detection Performance

|  |  |  |  |  |
| --- | --- | --- | --- | --- |
| **Contaminant Class** | **Analytical Technique** | **Detection Limit** | **Analysis Time** | **Accuracy (%)** |
| **Pesticides** | LC-MS/MS | 0.01-0.1 ppm | 15 minutes | 99.9 |
| **Mycotoxins** | HPLC-FLD | 0.1-1 ppb | 20 minutes | 99.7 |
| **Allergens** | ELISA | 1 ppm | 45 minutes | 99.5 |
| **Broad Screening** | SERS-Raman | 0.1-10 ppm | 2 minutes | 95.2 |

Quality and authenticity are also equally important, if not more than safety. Routine composition analysis – that is, measurement of fat, moisture and protein content – was effectively carried out by Near-infrared (NIR) spectroscopy. Fatty acid profiling: Detailed fatty acid composition was determined using a gas chromatograph (GC) an d vitamin content was assessed by high performance liquid chromatography (HPLC). Authentication methods were used to prevent food fraud and validate claims. The stable isotope analysis showed high accuracy in assignment of geographical origin of the samples (measured δ¹³C, δ¹⁵N and δ¹⁸O ratios). Moreover, Sophisticated spectroscopic fingerprinting (e.g., FTIR-, NIR- or Raman-spectroscopy) and the corresponding chemometric analysis made it possible to identify adulteration (dilution with cheaper oils, addition of synthetic sweeteners), by comparing the complex spectral signature observed on a sample with that registered for known references.

**VALIDATION AND PERFORMANCE ASSESSMENT**

The methods were thoroughly validated to be suitable for the process control. The precision of the FTIR-QCL system was very satisfactory, represented by a repeatability and reproducibility of 0.5% and 1.2%, respectively. Raman spectroscopy demonstrated good performance (day-to-day variation in key peak intensity ratios was less than 2%). This chromatographic performance was also reflected in the statistical evaluation of retention times, as the GC-VUV system showed a retention time precision of 0.01% and a peak area precision of 0.5%, ensuring precise identification and quantification.

**TABLE 3.** Analytical Method Performance Summary

|  |  |  |  |  |
| --- | --- | --- | --- | --- |
| **Technique** | **Application** | **Detection Limit** | **Linear Range** | **Analysis Time** |
| **FTIR-QCL** | Hydrocarbons | 10 ppm | 10 ppm - 100% | 1 second |
| **Raman** | Polymorphs | 0.5% minor form | 0.5-100% | 30 seconds |
| **GC-VUV** | C1-C12 hydrocarbons | 1 ppm | 1 ppm - 100% | 15 minutes |
| **Process MS** | Permanent gases | 10 ppm | 10 ppm - 100% | 10 seconds |
| **LC-MS/MS** | Pesticides | 0.01 ppm | 0.01-100 ppm | 15 minutes |

Economic analysis: Specific industry. A separate economic assessment was performed for each of the industries:

Petrochemical: Capital cost of $2.1million. The annual savings obtained from the system were estimated to be $4.8 million through better yield, reduced energy consumption and catalyst savings with a very attractive payback period of 5.3 months.

Pharmaceutical: Costing $850,000 to implement, the solution delivered annual savings of $1.2 million from higher yields and reduced testing (excluding the substantial added value of avoiding batch failures). This led to a payback period of 8.5 months off the infrastructure investment.

Food Processing: At an installed price of $420,000 the system saved over $680,000 per year that would have been lost as a result of waste plus recalls and inefficient processes for a payback in 7.4 months.

In addition to the tangible financial indicators, significant soft benefits were obtained by this implementation, such as solid assurance for regulatory compliance, strengthened brand protection (by product quality and consistency) and lesser environmental impact through reduced energy consumption and waste..

**IMPLEMENTATION CHALLENGES AND SOLUTIONS**

There were substantial technical challenges for implementing the analysis platform into challenging industrial settings that needed to be engineered around. Specialized ceramic and alloy probe materials were employed allowing high-temperature, high-pressure sampling along with continuous purging to avoid clogging. Powder in the process lines was treated with multi-stage filters that would backflush themselves automatically to ensure proper performance. In order to get the sample isokinetically, sampling probes were designed and qualified individually for all applications so extracted samples are a true reflection of the main process flow.

Analytical interferences were systematically managed. Spectral interferences in FTIR, Raman and similar techniques were solved using complex algorithms such as multivariate calibration that mathematically resolves overlapping signals. Matrix effects were compensated for through standard addition techniques and the use of the same internal standards. And environmental temperature and pressure variations were then real-time compensated by embedded sensor data with a complex proprietary compensation algorithm built into the analytical software.

Successful deployment required attention both to hardware and to the complexities of organization and data management. Change management was supported by extensive training programs for operations and maintenance staff, a staged approach to implementation with clearly defined success metrics at each milestone, and active sponsorship from key levels of senior management who provided resources. Data integration was another significant challenge that we overcame by utilizing standardized data formats (e.g. AnIML, AIA) for vendor-independent data exchange, establishing secure interfaces to connect with existing DCS systems and ensuring strong IT security measures including network segmentation and encrypted communications with endpoints on the process floor in order to safeguard integrity and confidentiality of process data.

**FUTURE DIRECTIONS**

PAT will be driven by several essential technological trends in its development. Microfluidics and lab-on-chip technology also offers miniaturization in analysis and sophisticated instruments closer to the point of need, with associated foot print and reagent savings. Able to deploy distributed wireless sensor networks will allow high spatial resolution monitoring of processes and escape from point measurements that miss gradients and inhomogeneities. The platform’s capacity to identify unknown compounds, predict the behaviors of complex systems, and autonomously make analytical decisions will be expanded by advances in AI—specifically deep learning architectures.

The PAT framework developed here is easily extendable to emerging and new industrial sectors. In biotechnology, metabolomics and multi-parameter sensors enable real-time monitoring of fermentation and cell culture processes for yield optimization and product quality control. Not only can water treatment plants use the same to continuously test for such emerging contaminants, and thereby maintain a safe and effective water system. In advanced materials manufacturing, such as polymerization processes and composite formation, in-line spectroscopy and in -line rheology can be employed to offer an accurate determination of reaction kinetics and final material characteristics.

The path to scale is a sightline through standardization. Next steps should aim at the joint definition of industry-wide standards for PAT methods, data formats and communication protocols. This will be conditional on the generation of robust reference materials and methods that enable calibration transfer between instruments and sites, enabling comparable outcomes to be achieved across the globe. Ultimately, spreading one common set of best practices throughout each sector would act to hasten adoption, reduce risk and facilitate a faster realization of the economic and operational advantages from intelligent process analytics integration in organizations.

**CONCLUSION**

This work illustrates the successful design and application of a combined physical-chemical analysis platform towards process optimization at an industrial scale. Key achievements include:

* Establishment of reliable analytical methods involving several complementary approaches;
* Advanced data analytics and machine learning integration for real time decision support;
* Validation through industrial-scale deployments in three sectors;
* Showing of material economic benefits - yield gains, energy savings, and quality assurance.

The service has the capacity to shift from reactive to proactive process control with analytics latency decreasing from a few hours up to several seconds and accuracy increasing by at least 42 or almost 68% using standard methods. Interface to process control systems enable closed-loop, automatic tuning of constantly evolving conditions.

The study constructs a model for PAT adaptation on technical and organizational level. The proposed solutions for sampling, data integration and change management offer concrete advice on implement- ation in industry. Future work will include downsizing, extension to other industry sectors, and the development of standardised methodologies for an accelerated implementation.

The output results further form the basis for a successful implementation of Industry 4.0 in process industries, allowing them to digitalize and optimize their processes based on data already stored in the control system. The combination of physical-chemical analysis with process control fundamentally changes how industrial operations are run: from only monitoring quality once in a while to continuously assuring and optimizing quality.

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