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# In-plant real-time manufacturing water content characterisation

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ABSTRACT

To trial the concept of in-plant real-time manufacturing water content characterisation, a commercial optical system for measuring light absorption and backscatter intensity was used with samples of food industry wastewater, and the results compared with conventional laboratory based water analysis. It is shown that the instrumentation is capable of coping with the range of turbidities presented by the wastewater and that there is some correlation between the absorption and backscatter measurements with the conventional parameters COD and TSS. It is suggested that combining backscatter and absorption data may provide an optical fingerprint of effluent that can be used as a management parameter, for example to identify unexpected contamination events. Potential uses of the instrumentation are discussed, including to provide rapid feedback on effects of system changes on effluent production, and in a feedback control loop to allow reuse of water without compromising product safety.

## 1. Introduction

The pressure on fresh water availability for human use is exacerbated by the changing climate and increasing population, particularly where this results in a mismatch between water demand and availability on a local scale. This is because water is not easily transportable in large volume, and, unlike energy, there are no sustainable large-scale new sources. For example the energy cost of distillation of saline water is prohibitively high at 2–4 kWh per cubic metre [23]. In high income countries industry is the majority user of limited water supplies (see Fig. 1) [11]. As the economies of mid to low income countries develop, this pattern is likely to be reproduced. Thus with significant growth in global manufacturing activities, manufacturing water consumption is set to increase by a factor of more than 5 by 2050, over a year 2000 baseline, from 245 to 1552 billion $m^3$ [20], and it has been estimated that global demand for fresh water by 2030 will be 40% above current water supplies [1]. Therefore, the lack of freshwater supply is predicated to act as a restriction on sustainable economic development and improvement in living standards. Simultaneously disposal of industrial wastewater is becoming increasingly costly due to tightened legislation, for example the European Directives on a European Water Policy Framework [9] and on Integrated Pollution Prevention and Control [10].

Against this background use of water has become an important part of commercial sustainability strategies, along with use of energy and materials. Water management measures were classified by Puigjaner et al., (2000) [21] into reactive (which they termed Specific Actions) and proactive (which they termed General Methodologies). Low risk, low cost reactive measures involving little change to process or product are generally the first port of call for manufacturing companies seeking to improve their water profile. Examples include automatic taps in employee facilities, use of grey water hygiene processes such as vehicle washing, and detection and reduction of leaks. However proactive measures involving more fundamental changes are essential for long-term gains, see for example Seneviratne [26]. More generally, a radical approach to resource management in industry and society will be required to

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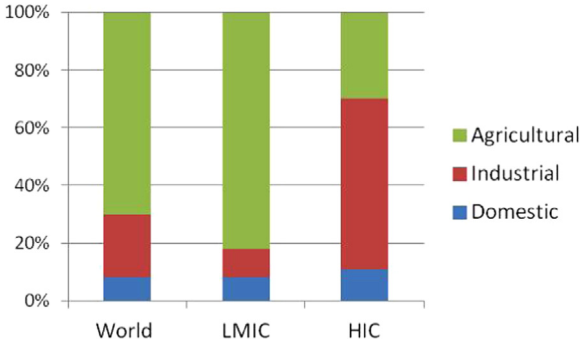


**Fig. 1.** Distribution of water use in low and middle income countries (LMIC) and high income countries (HIC) [11].

achieve long term sustainable economic development [22].

Proactive measures include process redesign, product redesign, and process substitution. Examples include air cleaning instead of water cleaning, re-use of water with single unit processes with and without treatment, or recycling of water effluent from one process as input to another process with and without treatment. Because such proactive measures may have longer payback periods and are more costly, analysis and decision support tools are required to target areas for intervention and manage the investment risk.

In previous work, it was identified that a fundamental barrier to achieving a long term and sustained water reduction and efficiency programme is lack of transparency on water use and waste management within manufacturing plants [24]. This has two aspects: lack of enterprise level engineering understanding of how and why water is used in processes, and lack of numerical data on actual water use and wastewater production. The lack of data is due to the historical view of water as an inexpensive resource to be drawn down and discarded as required.

The ability to visualize utility usage within manufacturing facilities is at a more advanced stage for energy than for water. This capability for energy has allowed the development of concepts of efficiency of energy use by various researchers [15,27,31,8]. The efficiency concept is used to help prioritise energy using processes, areas of plant and ideas in energy reduction initiatives, and hence promote improvements in industrial sustainability.

To apply a similar approach to water, the added complexity must be dealt with that while energy usage can be described by a single parameter, i.e. joules, water is described not only by volume but by content, i.e. the identity and concentrations of dissolved or suspended species, both at input and at output. Thus the concept of efficiency of use for water must take into account input water quality and contaminants introduced into the water flow by manufacturing process steps [24,25], and measurements of both water flow volumes and water content are required. In this work we have concentrated on process water quality characterisation because this the more significant gap in current capability. By contrast there are a number of off-the-shelf products available for automated volume monitoring [2].

This paper deals with initial steps towards the development of in-line instrumentation for continuous monitoring of water content within a manufacturing plant and hence determination of the effects of individual process steps on individual flows. The requirement for a continuous data stream arises from the need to capture the time variations in water content related to the batch nature of production and varying manufacturing schedules. The requirement to resolve the effects of individual process steps arises from the need to identify opportunities for structural changes to the manufacturing process chain to improve water usage efficiency. Note that the potential of continuous monitoring of wastewater *outside* factories, for example for the management of wastewater treatment plants, has been recognised in the literature [19,5].

A major issue is in defining what the instrumentation should measure. In addressing this question we first briefly outline some of the parameters most commonly measured when talking about industrial water content, and then the sensor principles available for in-line characterisation. We then propose repurposing equipment that is already in use for process monitoring in food for wastewater monitoring. The novelty of this approach lies not only in the application, i.e.; wastewater monitoring, but also in combining simultaneous measurements from different sensors.

Next we present the results of a trial consisting of application of a prototype set of instrumentation to process water samples taken from a food manufacturing plant. Finally we discuss the trial results and the implications for further development of the concept of in-plant real-time manufacturing water content characterisation, including specific applications.

## 2. Factors determining choice of instrumentation

### 2.1. Water content monitoring applications

We briefly review here some water content monitoring applications. For drinking water quality assurance, or environmental monitoring of water courses, wetted sensors are generally used. The most common measurands are driven by legislative or administrative limits on water quality. For example for drinking water, typically measurements are required of free chlorine, total chlorine, temperature, pH, oxidation–reduction potential (ORP), specific conductivity, dissolved oxygen (DO), total organic carbon (TOC), nitrogen (nitrate, nitrite and ammonia), turbidity and particle counts [14].

Effluent content at plant outflow is not generally monitored in–line. To prove compliance with water company discharge consents, a composite sampler may be employed. This machine automatically withdraws samples from the outflow at programmed intervals, with the sample being later analysed in a laboratory. The major parameters of interest in the UK are chemical oxygen demand (COD) and suspended solids (SS), as these are used to determine the disposal costs [32].

Sensors are used for effluent plant control [30], for example to respond to variation in effluent strength and to ensure it falls within the designed operational parameters. In the past lack of online monitoring capability has meant that plants were over specified in order to operate safely with only manual sampling to provide control feedback. The increasingly availability of sensors offers the possibility for such plants to be operated adaptively and hence more efficiently.

Turning to current applications within the process chain, water content sensing is used in specific applications for process control. An example is Cleaning-in-Place (CIP) for food manufacturing where cleansing and sanitation agents and rinse water are flushed through processing vessels. In some automated systems conductivity sensors are used to ensure that the concentrations of cleaning chemistry are correct and that the chemistry has been completely removed by rinsing [3,4]. Sensors in this application also have a role of improving resource efficiency by reducing consumption of water, chemicals and energy and wastage of product [28]. Another application is detection of when product transitions have gone to completion in pipework, for example in fruit juice processors or breweries [18]. In both these cases the direct detection of endpoints, as part of a control feedback loop, improves resource usage efficiency over relying on predetermined protocols established during process setup. A further water efficiency application is use of conductivity sensors for feedback control of blowdown intervals in cooling tower systems, thus reducing the amount of top up water required [26].

All these examples have in common that the choice of measurand(s) is driven by the specific application and the system created around the sensor is limited to that application. The aim of the proposed instrumentation is to be more broad, i.e.; to support the management of water use within a factory. This presents a challenge to identify appropriate measurands and associated instrumentation as the possible content of water can vary so widely.

### 2.2. Characteristics required of instrumentation

The ideal set of instrumentation would have the following attributes:

- Be capable of continuous monitoring so as to capture process related changes in water content, e.g.: due to process parameter variation, batch production, and product changeovers;
- Be low-cost to make use in multiple areas of a factory economically viable;
- Be sensitive to a wide range of contaminants to reduce the need to customise for specific applications;
- Be non - or minimally - invasive; meaning that the instrumentation does not interfere with the flow of water or plant operations.
- Have low maintenance and associated staff training requirements. Maintenance factors to be considered are fouling and cleaning requirements, calibration requirement, and the use and replenishment of reagents.

Insight into the required capability can be gained by referring to the categorisation formulated by Callis et al. [6] for process analytical instrumentation used in chemical engineering, i.e.: off-line, at-line, on-line, in-line and non-invasive. Off-line and at-line refers to highly capable laboratory instrumentation, aimed at identifying and quantifying the chemical constituents of a mixture, but requiring manual sampling. With on-line similar instruments are used but the sampling is automated, whereas with in-line no sampling is required as the sensitive element is a probe in direct contact with the process fluid flow. Finally non-invasive refers to measurement techniques requiring no direct access to the fluid.

Off-line and at-line can provide highly detailed data but suffer from the time delay between sampling and analysis, making it difficult to capture variations in water content [5]. On-line automated sampling improves the response time but requires a separate fluid line and potentially complex sample pre-conditioning with associated maintenance requirements. The best matches to the required capability are therefore in-line and non-invasive. The chemical analytical power of techniques fitting these categories is less than for off-line and at-line, but the usefulness of the techniques for process control of production is greater.

Thus we consider that for the present purpose the task of the instrumentation is not primarily to identify species, but to indicate variation of contaminant concentrations with time. It would also be useful to capture and flag-up the presence of unexpected contaminants, even if these are not immediately chemically identified.

### 2.3. Available sensor types

In-line sensing principles have been categorised as optical, electrical, electromagnetic and acoustic. Optical and electrical techniques provide the best fit to the characteristics in Section 2.1 as these require no reagents and little maintenance. Optical sensors are not in direct contact with fluid although optical windows into process pipework are required. Optical path length considerations may require that the window elements protrude into the fluid flow space. Electrodes for conductivity measurement are in contact with fluid but can be made of the similar materials as the pipework, i.e. stainless steel and designed to not protrude into the fluid flow. An alternative is an inductive probe which does not have wetted electrodes, but which requires the sensing element to protrude into the middle of the flow stream.

Potential non-invasive measurement techniques which do not require wetted elements are ultrasonics and electromagnetic and impedance sensing. Ultrasonic transducers have the advantage that they can be attached to the exterior of standard pipework but the interpretation of data is more complex than for other techniques. For example ultrasound studies of colloids rely on theoretical models for relating particle properties such as the size distribution to the propagation of ultrasound through the fluid [13]. Some a priori knowledge of these properties is therefore generally required. Electromagnetic sensing does not yield useful information with all fluids, and requires non-metallic pipework.

Using outputs from two or more sensors simultaneously is known as sensor fusion. Sensor fusion is not just about calculating the value of a desired property of the liquid from calibrated relationships, but by including more sensors than the minimum number required non-standard conditions such as sensor failure can be detected. In the application under discussion the non-standard condition of interest is a fluid with properties falling outside the expected range [12].

## 3. Prototype set of instrumentation

The food industry was chosen as a first application for continuous monitoring of water content within a manufacturing plant. Food manufacturing is a major consumer of water for many different purposes including raw ingredient washing, the flushing through, cleaning and sterilisation of processing equipment, cleaning and sterilisation of processing facilities, cooking, heating, cooling and fluming [16]. Improving the efficiency of water use (along with energy use) was nominated by the UK food industry as one of the top ten pre-competitive areas for research [29]. In 2008 a large number of UK food manufacturing companies signed the Federation House commitment, committing them to reduce water consumption over a 2007 baseline by 20% by 2020 [33].

A prototype set of instrumentation consisting of commercial sensors already used in the food industry for process control was selected. The optical sensor was a Mettler Toledo Inpro 8300 RAMS unit, capable of measuring transmitted and back-scattered light (180°) intensities for light emission from four differently coloured LED sources (red, green, blue and infra-red). Turbidity values can be calculated if necessary by calibration with a formazin suspension. For conductivity a four point probe with SS316L electrodes designed to fit flush to the pipe wall was included. The probe includes a Pt1000 thermometer. Finally a liquid electrolyte pH sensor was included. This last requires regular calibration and so violates the low maintenance requirement, but was included for its high temperature robustness. A gel electrolyte version pH sensor could be substituted instead. The sensors are capable of withstanding typical food industry corrosive cleaning in place conditions such as 1.4 wt% NaOH at 137 °C for 50 min. The capability of the instruments according to the manufacturers' data sheets is summarised in Table 1.

Although the instruments are already used for process control in the food industry, the novelty of the current work is to combine the sensors and apply them for in-plant effluent content characterisation and monitoring.

## 4. Trial of prototype instrumentation set

### 4.1. Methodology

In order to trial the concept of in-plant real-time manufacturing water content characterisation, the prototype instrumentation set was tested with samples of food industry wastewater. It was specifically not the purpose of the trial to take the approach of calibrating the sensor outputs to enable calculation of wastewater quality parameters, but rather to identify whether different water types can be distinguished.

A test rig for the prototype set was constructed from food standard hygienic pipework, connectors and a valve and takes the form of a column, as shown in Fig. 2. Wastewater samples were obtained from a manufacturing plant making frozen, pre-cooked, filled and unfilled pastry products. Individual samples were taken from different areas of the plant during the weekly deep clean as listed in Table 2. The deep clean takes place at the weekend after all production is stopped and involves partial disassembly and mostly manual washing of equipment and pipework using a sequence of fluids, including caustic cleaners, acid cleaners, disinfectant and rinse steps. Floors are washed manually using hoses with automated dosing of chemical into the flow.

The samples were processed within 48 h of being obtained. Each sample was allowed to settle and then decanted into the test rig. Readings were taken and the equipment rinsed with de-ionised water before the next sample was processed. Readings of de-ionised water were used as a control and to ensure there was no measurement drift due to residue build-up within the equipment. Simultaneously portions of the samples were passed to a commercial effluent analysis service to carry out conventional laboratory based analyses. The protocols used by the service are described in method statements available from their website (turbidity: WAS066, colour WAS065) [17]. The parameters measured were COD and TSS, chosen because they determine the cost of wastewater disposal in the UK [32].

**Table 1**
Manufacturer data on components of the prototype instrumentation set.

| Sensor type | Measurement range | Units | Notes |
| --- | --- | --- | --- |
| Turbidity | 160–2400 | NTU | |
| Colour | Red, Green, Blue, Infra-red | | |
| Conductivity | 0.02–650 | mS/cm | + / − 5% |
| pH | 0–12 | pH | Temperature compensated |
| Temperature (of product) | 0–106 | °C | |

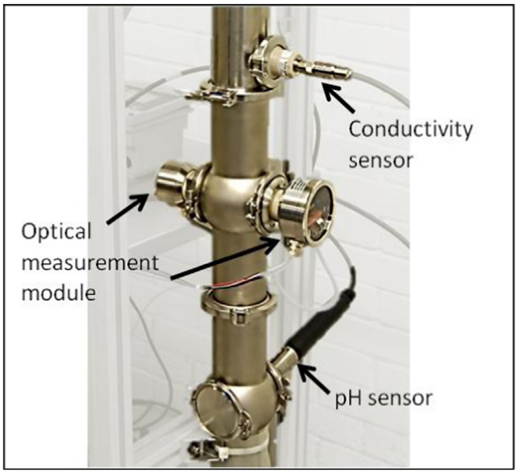


**Fig. 2.** Test rig used for trial incorporating instrumentation as indicated.

### *4.2. Results of trial and discussion*

The values of each measurement made on the samples are listed in Table 2. Scat. NIR, Scat. R, Scat. G and Scat. B are backscatter values, and Abs. NIR, Abs. R, Abs. G and Abs. B are absorption values, all measured using the optical sensor on the test rig. The values for turbidity, COD, TSS, conductivity (cond.) and pH were measured by the commercial effluent analysis service.

The optical (absorption and backscatter) measurements are in arbitrary units such that a measurement on de-ionised water corresponds to a value of zero and a value of one corresponds to the maximum signal output from the equipment. The entries in the table are arranged in order of increasing turbidity. It can be seen that sample 13 is anomalous as it exhibits a very high COD value but a low turbidity. The COD content is probably the cleaning agent, consistent with the highly alkaline pH value of 13.2 pH units compared to the range of 4.0–7.8 pH units exhibited by the other samples, and high conductivity, at $9.12 \times 10^4$ S/cm, compared to values on the order of $10^3$ S/cm for the other samples.

The data in the table is plotted in various ways in Fig. 3. In Fig. 3(a), the back scattered light intensity for the four LED colours are plotted on a logarithmic scale against turbidity measured in nephelometric turbidity units (NTU). In general the backscatter signal increases roughly linearly with the log of turbidity and then saturates. The saturation occurs at different turbidity values for the different LED colours (blue – around 10 NTU, green and red – around 200 NTU, infra-red – not saturated at highest turbidity seen). The variation in behaviour with colour allows the equipment to probe fluids with turbidity ranging over three orders of magnitude.

The absorption signal variation with turbidity, Fig. 3(b), shows a different functional dependence to that of backscatter with no saturation behaviour, although there is still a general rise in signal with increasing turbidity. The difference between the graphs is an indication that there is information to be gleaned from comparison of the backscatter and absorption signals. This point is further illustrated by comparing graphs of backscatter and absorption versus TSS, Figs. 3(c) and 3(d). The backscatter plot again shows saturation behaviour while the absorption trend is more complex. Plots against COD show similar behaviour (not shown). The difference in behaviour between plots is probably because the backscatter signal is more sensitive to suspended matter than the absorption signal.

In the light of these results we discuss how such general purpose instrumentation could be used for both reduction and active management of water and effluent, with reference to Fig. 4 illustrating three potential system configurations. Fig. 4(a) shows characterisation of variation in water effluent production of an individual manufacturing process step for different operational conditions, for example product mix and sequencing, batch sizes and machine operation parameters. The diagram shows two process steps, numbered $i$ and $i + 1$. Water quality is monitored using sensors at both the inlets and outlets, allowing the contribution of each process step to effluent water content to be characterised. The data obtained can then be used for process or system redesign to improve water sustainability, as illustrated in the diagram by connection of the sensors to an industrial network and a computer running system analysis and optimisation software. The inline nature of the instrumentation would allow taking a trial and error approach, with instant feedback on the effects of changes. The instrumentation could be permanently installed or used only for optimisation of a facility and then removed.

By contrast an active management use requiring permanent installation of the instrumentation is shown in Fig. 4(b). In this case the water quality is assessed only on the outlet side of a process. Software then decides whether the water can be reused as an input to another process or should be rejected, and subsequently operates a valve to direct the wastewater stream appropriately. Data and control signals are passed over an industrial network. The software could run on a computer connected to the industrial network, or as embedded software in an industrial controller (neither shown on the diagram).

In Fig. 4(c) the instrumentation takes a similar role on the input side of a process to ensure that water input from an alternative source to potable town-water is of acceptable quality. Again, software processes the data and if water quality falls below a required threshold, operates a valve to cut off the supply and illuminates a warning light to alert production staff to the issue. In this case the

**Table 2**
Samples and measurements on samples from the trial (A.U. = arbitrary units).

| Sample ID | Source | Appearance | Turbidity (NTU) | Scat. NIR (A.U.) | Scat. R (A.U.) | Scat. G (A.U.) | Scat. B (A.U.) | Abs. NIR (A.U.) | Abs. R (A.U.) | Abs. G (A.U.) | Abs B (A.U.) | COD (mg/l) | TSS (mg/l) | Cond (uS/cm) | pH |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| 1 | Belt oven area floor | Clear | 1.4 | −0.02 | −0.09 | −0.02 | 0.07 | 0.00 | 0.00 | 0.00 | 0.01 | 1.70E + 01 | 6.00E + 00 | 8.03E + 02 | 7.8 |
| 2 | Pastry cutting area drain during deep clean | Strong yellow | 10.1 | 0.08 | 0.20 | 0.51 | 0.97 | 0.00 | 0.00 | 0.07 | 0.33 | 2.17E + 05 | 8.78E + 02 | 9.12E + 04 | 13.2 |
| 3 | Tray washer drain (not in operation) | Cloudy | 36.4 | 0.37 | 0.46 | 0.67 | 0.87 | 0.01 | 0.02 | 0.14 | 0.16 | 2.44E + 02 | 3.20E + 01 | 9.20E + 02 | 7.5 |
| 4 | Mixing vessel water and detergent mix | Cloudy | 56.5 | 0.59 | 0.68 | 0.88 | 0.99 | 0.03 | 0.05 | 0.39 | 0.40 | 3.48E + 03 | 1.08E + 02 | 9.66E + 02 | 4 |
| 5 | Main effluent outflow water after traps | Cloudy | 113 | 0.57 | 0.67 | 0.88 | 0.99 | 0.03 | 0.04 | 0.37 | 0.37 | 8.27E + 02 | 1.64E + 02 | 1.05E + 03 | 6.5 |
| 6 | Gyro freezer external drain prior to cleaning | Cloudy | 174 | 0.65 | 0.77 | 0.96 | 1.00 | 0.04 | 0.09 | 0.64 | 0.74 | 1.71E + 03 | 8.20E + 01 | 1.12E + 03 | 5.4 |
| 7 | Mixing area drain during deep clean | Strong yellow | 1180 | 0.77 | 0.79 | 0.96 | 1.00 | 0.53 | 0.74 | 0.80 | 0.80 | 5.13E + 03 | 2.15E + 0.3 | 1.68E + 0.3 | 7.4 |

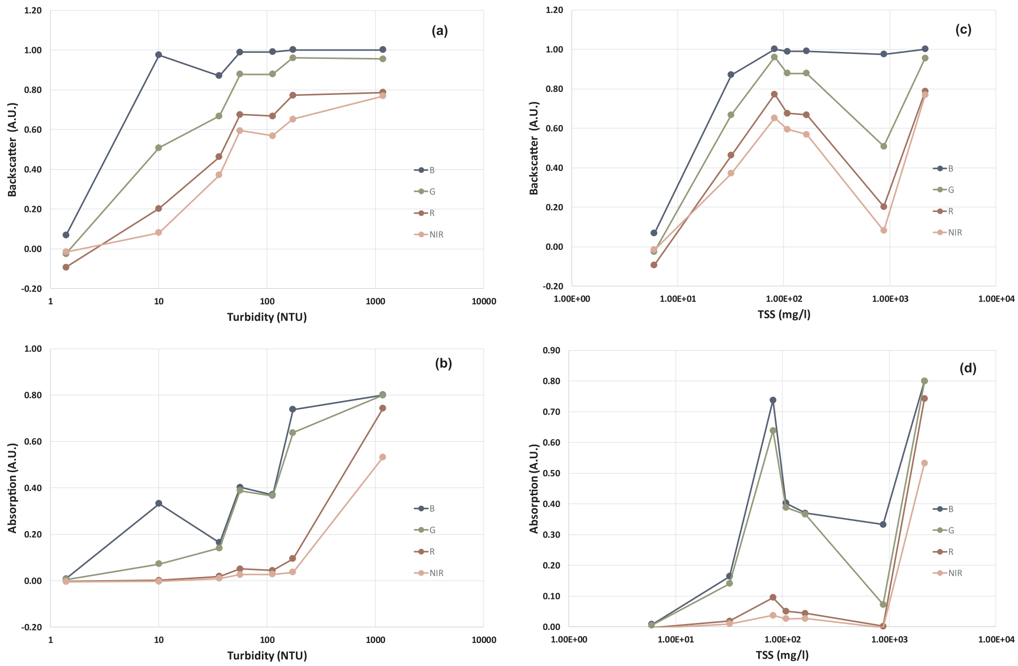


**Fig. 3.** Optical measurements on wastewater samples at different illumination colours plotted against standard wastewater parameters; (a) Backscatter signal versus sample turbidity, (b) absorption signal versus turbidity, (c) backscatter signal versus total suspended solids (TSS), (d) absorption signal versus TSS.

instrumentation acts as a safeguard to ameliorate the risk to production of using a variable quality water source.

In fact variability in quality is a barrier to wastewater re-use, particularly in food manufacturing [16,7]. Such variability can arise from variation in raw material streams to manufacturing, for example fresh produce, or from process factors such as personnel non-compliance with procedures. Active management of water as in Figs. 4(b) and (c) can therefore promote both water sustainability and food safety.

For operation in the applications in Fig. 4(b) and (c) detection of a change in the character of the water stream is the desired capability. A possible approach to this is presented in Fig. 5. This shows how an optical fingerprint for each fluid can be defined by plotting the scatter signal against the absorption signal at each colour. In this plot sample 2 is not obviously anomalous. However further work may show that the combination of a large range in scatter signal over the colour spectrum combined with a small range in absorption is characteristic of, for example, the middle part of a cleaning regime.

A possible limitation to this formulation of an optical fingerprint is saturation in the backscatter signal. As can be seen in Fig. 3(a) the backscatter signal appears to be saturated, i.e. there is no variation with turbidity, for turbidity over most of the range sampled (i.e. ≥ 10 NTU). This means that the fingerprints for all the samples in the saturation regime would be distorted, at least in the blue. To investigate this effect follow up work to that reported here is concentrating on exploring the signal state space through controlled variation of the content of samples of industrial effluents using dilution and filtering.

## 5. Conclusions

Lack of data on water flows and water quality within factories is a major barrier to radical progress in improving industrial water sustainability. While water volume data can be obtained by installing sub-metering, water quality data is much more difficult to obtain and instrumentation dedicated to this application is not available. We have argued that instrumentation to provide such data should be of in-line type, i.e; where no sampling is required, in order to provide continuous monitoring capability while presenting minimum interference with factory water flows. We have also identified that optical and electrical sensor principles are the most suited to providing the required capability. While the proposed instrumentation would not provide chemical specificity, we consider that the actual task of the instrumentation is to be sensitive to variation of contaminant concentrations in real-time.

As a trial of this concept we have carried out measurements using a commercial optical system with samples of food industry wastewater. Absorbance and backscatter intensity results were compared with conventional laboratory based water analysis. The instrumentation was shown to be capable of coping with the range of turbidities presented by the wastewater and also that there is some correlation between the absorption and backscatter measurements with the conventional parameters COD and TSS. We have suggested that combining backscatter and absorption data may provide an optical fingerprint of effluent that can be used as a management parameter, for example to identify unexpected contamination events. This idea is the subject of follow up work exploring the signal state space with further industrial samples.

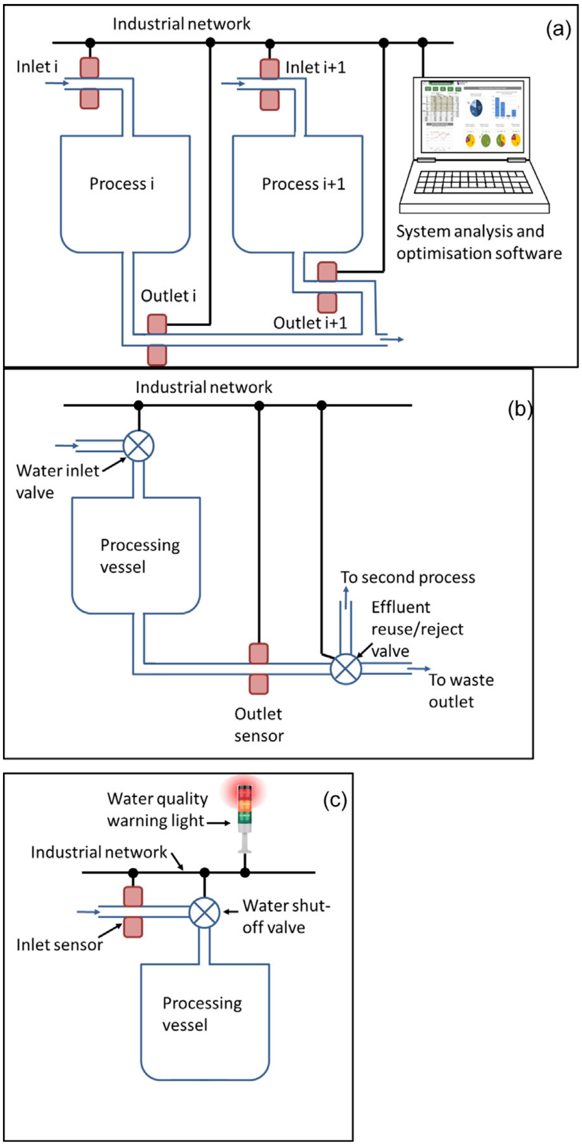


**Fig. 4.** Conceptual applications for in-plant water quality instrumentation: (a) system optimisation, (b) automated routing of process stream to waste or reuse, (c) quality assurance of inlet water.

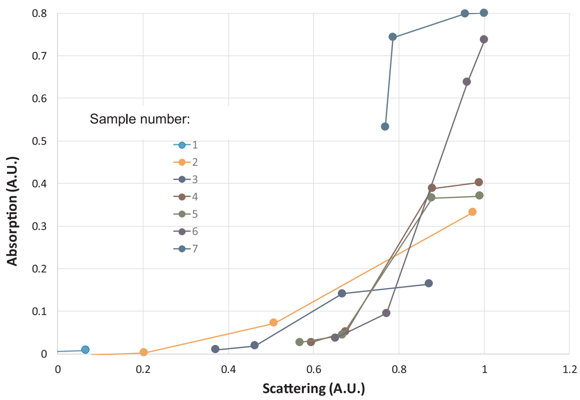


**Fig. 5.** Absorption signal versus scattering signal at four colours for each sample.

Potential uses of the instrumentation has been discussed, including providing rapid feedback on effectiveness of system changes to reduce effluent production, and in a feedback control loop to allow reuse of effluent water without compromising product safety.

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