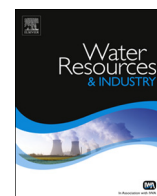


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## Assessment of the capability of an optical sensor for in-line real-time wastewater quality analysis in food manufacturing



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### ABSTRACT

This work investigates the use of a commercial optical product monitor to achieve in-line real-time water content analysis. Test fluids were used and optical measurements of attenuation of light intensity at four colours were made. These measurements were used to identify any relationship between these and the water quality parameters of turbidity and colour. Variation in light attenuation for turbidities up to 1700 NTU was successfully resolved by the instrument, with optical data for turbidities  $\geq 20$  NTU fitting well the Beer-Lambert model. The sensor was also able to clearly identify the effect of filtering out suspended solids with unfiltered samples (apparent colour) exhibiting significantly higher attenuation coefficients than filtered samples (true colour). Further studies will concentrate on whether the instrument can analyse samples with turbidities higher than 1700 NTU, together with further investigating the variation in the attenuation coefficient seen with turbidity and colour of light.

### 1. Introduction

Water, as a resource, has often been neglected when it comes to considering its consumption, with many manufacturing companies failing to explore less obvious measures for optimizing their water operations. This is usually shown by ad-hoc water efficiency efforts based on information that is only monitored on a factory level and assigned to respective process chains via allocation rules [10]. However, fresh water is a limited resource and increasing pressure on its supplies, continued supply uncertainties and costs linked to legislative compliance, such as costs relating to wastewater treatment, have nowadays led manufacturers to start adding water use reduction in their agendas [14,22]. As such, water, as an industrial commodity, must start being increasingly considered as a valuable material that requires a rational and systematic approach for its careful use. This is more critical to the food-industry where water is widely-used as it is involved in many processing methods and unit operations, e.g. for soaking, washing, rinsing, fluming, blanching, scalding, heating, pasteurising, chilling, cooling, steam production, as an ingredient, for general cleaning, and sanitation and disinfection purposes [3,18].

In fresh water management, water quality monitoring is a key tool with water quality being a generic term used to define the general suitability of the water for a specific purpose [9,11]. However, off-line assessment based on application of standard laboratory-based methods is typically laborious (requiring extensive samples preparation with potential for sample corruption), time-consuming requiring one to three days to return results as well as involving hazardous reagents [2,4,8]. To overcome this, in-line monitoring (no sampling and immediate analysis) by means of a rapid and reliable technique that can provide instant feedback,

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would be most beneficial [4]. One of the most promising advances in the recent years for in-line monitoring is the availability of commercial optical product monitoring equipment. The sensors used in this equipment rely on the absorbance, fluorescence, or scattering properties of materials that are either dissolved or suspended in water [19]. A non-exclusive list of such sensors is as follows: optical-density probes, in-situ microscopes, optical biosensors, fibre-optic sensors, surface plasmon sensors, refractometers, near infrared sensors, fluorescence sensors, and ultra-violet sensors [9,12,24].

Monitoring technologies within the food industry mainly concentrate on process control, pH and conductivity measurements, sugar content determination, food freshness and detection of microbes and toxins, ingredient freshness and food quality including taste. Focusing especially on quality management during food processing operations, this management appears to be one of most critical processes to prevent contamination and improve product safety, quality and production hygiene [1]. In addition, it becomes an even more challenging field when minor components such as flavours, vitamins, antioxidants, natural toxins, more or less unwanted toxic heavy metals from soil, herbicide or pesticide residues, etc. have to be detected, or, in the case of some special clinical problems, e.g. allergies, detection of even traces of cross contaminants is required [20]. Optical sensors, e.g. optical chemical sensors, optical biosensors, optodes, have already been widely used in food industries for food quality measurements, e.g. for detection of glucose and urea (bio-optodes), heavy metal ions (optodes utilising immobilised chelating ligands), humidity (optical humidity sensors), multi-analysis, e.g. simultaneous determination of humidity and NH<sub>3</sub> using single optodes, food-safety-related analytes like chemical contaminants, pesticides and drug residues, foodborne pathogens and toxins (optical fluoro-immunosensors, surface plasmon resonance (SPR) biosensors, optical fibre biosensors) [16,17]. In such applications, optical sensors and bio-sensors are suitable, as highly selective devices have already been constructed [20]. As such, optical sensors can indeed be useful in food factories, however, it may be worth mentioning that some in-situ optical sensors may require further characterisation for interferences and correction schemes prior to their widespread use [19].

The overall aim of this study was to characterise the capability of optical instrumentation, intended to be used together with other sensors in the final implementation, for in-line characterisation of factory wastewater streams. The instrumentation consists of a commercial optical product monitor. Optical measurements of the attenuation of luminous intensity were carried out, and the relationship between this bulk optical property and two fundamental water quality parameters relating to food-manufacturing, that is: turbidity and colour (e.g. olive oil classification by [13], or determination of sunflower oil colour by [15]), was investigated. To this end, the following questions were addressed:

**Question 1.** What is the range of turbidities the instrument can handle?

**Question 2.** Can it distinguish between true (turbidity-free samples) and apparent (turbid samples) colour?

The study was carried out on two test fluids, synthetic high-turbidity water prepared in the laboratory, and used process water collected in different places within a brickyard.

## 2. Materials and methods

### 2.1. Beer-Lambert law

When a beam of light falls on a material, incident light can be absorbed by its volume, scattered (light emerging in a different direction from the incident light) or transmitted (light emerging in the same direction as the incident light). The attenuation of light intensity due to the properties of the material through which the light is travelling is summarised as follows:

$$\frac{I}{I_0} = e^{-\alpha l} \Rightarrow \ln\left(\frac{I}{I_0}\right) = -\alpha l \quad (1)$$

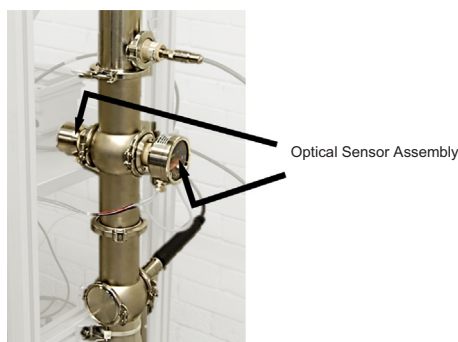
where:  $I_0$ : initial light intensity and  $I$ : light intensity after travelling through the material (both expressed as radiant intensity or power per unit area),  $l$ : the optical path length (depth), in mm, and  $\alpha$ : the attenuation coefficient, in  $\text{mm}^{-1}$

Eq. (1) is one of the ways of expressing what is known as Beer-Lambert Law. In the case of water samples attenuation may be due to both scattering and absorption by suspended matter and absorption by dissolved matter.

### 2.2. Optical sensor and test rig

The commercial optical product monitor, in use in the food industry, is a Mettler Toledo Inpro 8300 RAMS unit that is capable of measuring transmitted and back-scattered light (180°) intensities at four different wavelengths (visible-red, visible-green, visible-blue, and near-infrared) using light emission from eight light-emitting diodes (LED) sources and a detector. The instrument manual does not identify the exact wavelengths, but these are comparable with the wavelengths specified for standard measurement of turbidity and colour. For example, turbidity measurement standards require a broad spectrum source such as a tungsten lamp emitting in the visible range 400–680 nm (violet to red), or a narrowband source emitting in the near-infrared at 860 nm [23]. Colour measurement wavelength ranges depend on application, such as 465 nm (blue) for pulp and paper effluent [7].

The commercial optical product monitor is directly integrated into a test rig that is constructed from food-standard hygienic pipework in the form of a column - see Fig. 1. For the tests the column was filled with static test fluid - because of the current structure of the instrument, only static fluids can be tested for now. However, the fact that measurements take place immediately after the sample preparation or collection, the samples are shaken well before being emptied into the pipework, and the results are taken



Optical sensor incorporated into the test rig

**Fig. 1.** Optical sensor incorporated into the test rig.

within seconds, the samples do not undertake any alteration. Eight values in total, four relating to back-scatter, one per wavelength, and four relating to transmission at the same four wavelengths, were able to be recorded. However, this work will concentrate only on measurements relating to transmitted light.

### 2.3. Test fluids and measurements

Test Fluid 1 was a milk/water solution made of commercial full-fat milk ( $0.036 \text{ gr}_{\text{fat}} \text{ mL}^{-1}$ ) and de-ionised water (stock solution). The final concentration was equal to  $16 \text{ mL}_{\text{milk}} \text{ L}^{-1}$ . This sample had a turbidity value almost equal to 1700 NTU, which was selected to be the highest value to be tested during this experiment. Based on this stock solution, a range of dilutions (using de-ionised water) was made, such as: 0 (de-ionised water only),  $\approx 0.5 \text{ mL}_{\text{milk}} \text{ L}^{-1}$ ,  $1 \text{ mL}_{\text{milk}} \text{ L}^{-1}$ ,  $2 \text{ mL}_{\text{milk}} \text{ L}^{-1}$ ,  $4 \text{ mL}_{\text{milk}} \text{ L}^{-1}$ ,  $8 \text{ mL}_{\text{milk}} \text{ L}^{-1}$ ,  $16 \text{ mL}_{\text{milk}} \text{ L}^{-1}$  (stock solution only). This fluid was prepared to characterise the dynamic range and sensitivity of the equipment with respect to turbidity and consequently to address [Question 1](#). Milk/Water solutions are quite turbid solutions that are very common in food factories, in particular, in the dairy industry. More dilute milk solutions than  $0.5 \text{ mL}_{\text{milk}} \text{ L}^{-1}$  were prepared but RAMS unit measurements on these are not reported here as the signal to noise ratio was too low, due to the low turbidity ( $< 20 \text{ NTU}$ ).

The second test fluid was surface water that had already been used for cleaning purposes within a brick yard i.e.: used process water. Even though this clay-based sample is not directly related to the food industry, it is characterised by a large number of suspended solids that can easily be filtered out, together with little dissolved organic contamination. It was then ideal to deal with [Question 2](#), in particular during these experiments which aim to assess the capability of the instrument. Samples of this kind of water were collected from different areas within the brickyard. Prior to their analysis, the samples had been left to stabilise for at least one hour, and then, the supernatant was collected - through this procedure, the settleable fraction of the suspended solids was removed. This fluid was then used to conclude whether the instrument can identify the effect of filtering out suspended solids, or whether it can distinguish between true colour (turbidity-free filtered sample) and apparent colour (turbid unfiltered sample) and consequently to address [Question 2](#). Measurements had first been made on unfiltered samples, then the samples were filtered using a glass microfiber filter, a standard one for removal of suspended solids, and the tests were repeated.

The optical measurements were performed at a range of optical path lengths, achieved by inserting different window holders into the optical sensor assembly. Different combinations of the window holders were used to create path lengths of 12, 23, 31, 46, 54, and 64 mm. Each window holder combination could be inserted in two orientations, both giving the same path length. Measurements for each path length reported in this paper were carried out using both orientations, as described in the results section. Regarding off-line measurements, turbidity was measured using a HACH 2100 N Turbidimeter and colour was measured using a Hazen Lovibond PCCheckit Colorimeter.

Regarding the turbidity values presented in this paper, measurements were made and the confidence intervals for 95% confidence level were calculated as follows. After allowing readings to settle the nominal reading was taken. The confidence interval was calculated assuming standard deviation in the measurement equal to the instrumental accuracy and one degree of freedom (i.e. worst case). The instrumental accuracy used was that quoted by the manufacturer ( $\pm 2\%$  of reading plus 0.01 NTU from 0 to 1000 NTU with Ratio ON). Regarding colour values presented in this paper, only one reading was shown by the instrument and that reading was taken. The confidence interval was calculated based on what we did for turbidity values. Once again, the confidence interval used was that quoted by the manufacturer (2% from 0 to 500 Hazen).

## 3. Results and discussion

The Inpro 8300 RAMS unit outputs a current proportional to the light intensity measured by the incorporated optical sensor. In the study, this current was determined by measuring the voltage across a  $500 \Omega$  current sensing resistor. To calculate the quotient  $\frac{1}{I_0}$  for a given fluid under test measurements of current for the fluid under test and deionised water were made, and Eq. (2) applied.

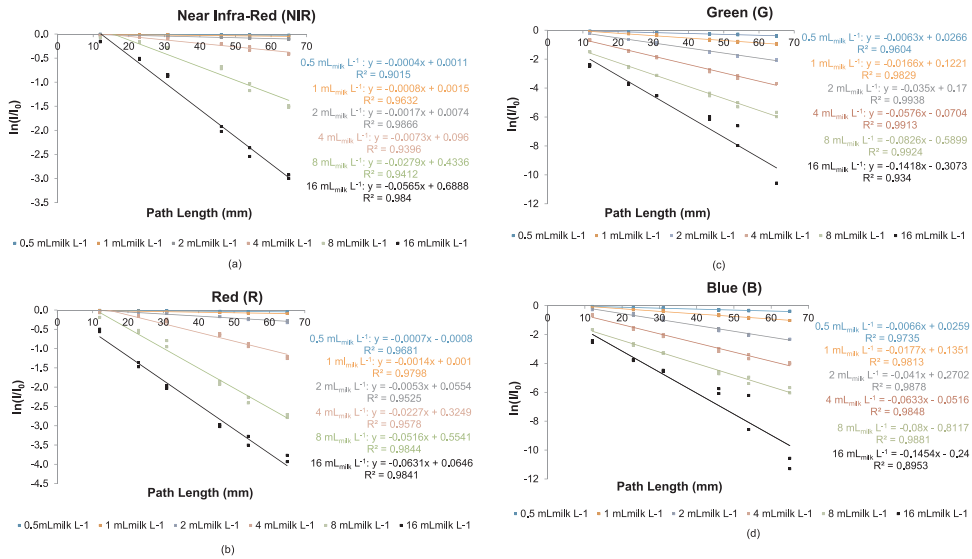


Fig. 2. a:  $\ln(I/I_0)$  vs optical path length ( $l$ ) (Near-Infrared). b:  $\ln(I/I_0)$  vs optical path length ( $l$ ) (Visible-Red). c:  $\ln(I/I_0)$  vs optical path length ( $l$ ) (Visible-Green). d:  $\ln(I/I_0)$  vs optical path length ( $l$ ) (Visible-Blue). (For interpretation of the references to color in this figure legend, the reader is referred to the web version of this article).

$$\frac{I}{I_0} = \frac{10 - OM_{SAMPLE}}{10 - OM_{DW}} \tag{2}$$

where:  $OM_{SAMPLE}$ : voltage measured with the fluid under test, and  $OM_{DW}$ : voltage measured with de-ionized water at the same wavelength.

The figure of 10 V in the equation arises from the operational design of the RAMS unit.

### 3.1. Determination of the dynamic range of turbidity

The natural logarithm of  $\frac{I}{I_0}$  was calculated and was plotted against the optical path length ( $l$ ) at all four wavelengths - see Fig. 2a to d.

In these figures, the measurements appear in pairs for each path length and dilution. The two data points of the pair correspond to the two orientations of the optical windows associated with the path length, as described in the methodology section. The figures show that changing the orientation has little effect on the measured absorption. The same is not the case for scatter measurements, due to geometric effects associated with the optical assembly, and is the reason the scatter measurements have not been analysed in this work.

The dilution series reported yields turbidity values  $\geq 20$  NTU, for which the data fits the Beer-Lambert model very well (mostly  $R^2 > 0.9$ ). The non-zero intercept can be attributed to instrumental noise. The values of the attenuation coefficient  $\alpha$  were then calculated from the slope of the fit for each dilution and LED. All calculated  $\alpha$ -values, together with off-line turbidity values, are presented in Table 1. As a conclusion, it can be said that variation in attenuation of light intensity for turbidity values up to 1700 NTU was successfully resolved by the optical sensor, and the more turbid the sample, the higher the calculated  $\alpha$ -value.

Turbidity values are next plotted against  $\alpha$ -values - see Fig. 3.

Turbidity is, in general, the measure of the collective optical properties of a water sample that cause the light to be scattered and

Table 1  
Summary of experimental data (Test Fluid 1).

| Concentration (mL <sub>milk</sub> L <sup>-1</sup> ) | Turbidity (NTU) | $\alpha$ (mm <sup>-1</sup> ) |        |        |        |
|---|-----------------|------------------------------|--------|--------|--------|
|   |                 | NIR                          | R      | G      | B      |
| 0.5   | 19 ± 4          | 0.0004                       | 0.0007 | 0.0063 | 0.0066 |
| 1   | 40 ± 8          | 0.0008                       | 0.0014 | 0.0166 | 0.0177 |
| 2   | 90.6 ± 19       | 0.0017                       | 0.0053 | 0.035  | 0.041  |
| 4   | 224 ± 46.5      | 0.0073                       | 0.0227 | 0.0576 | 0.0633 |
| 8   | 617 ± 128       | 0.0279                       | 0.0516 | 0.0826 | 0.08   |
| 16  | 1699 ± 352.5    | 0.0565                       | 0.0631 | 0.1418 | 0.1454 |

NIR: Near-Infrared, R: Visible-Red, G: Visible-Green, B: Visible-Blue.

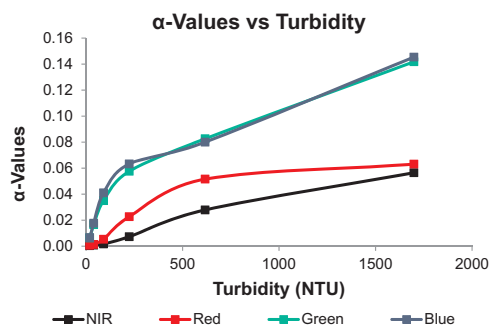


Fig. 3. Turbidity against  $\alpha$ -values. (For interpretation of the references to color in this figure, the reader is referred to the web version of this article).

absorbed rather than transmitted in straight lines [26]. Its measurements are then based either on the transmitted light (turbidimetry) or on the scattered light (nephelometry). In this work, the turbidity values, as provided by the bench-top turbidimeter, are nephelometric. Nephelometry, which relies on measurements of the intensity of scattered light, usually of  $90^\circ$ -scatter, as this detection angle is considered to be very sensitive to particle scatter [23], is the most-widely used technique with respect to laboratory turbidimeters. On the other hand, the attenuation coefficient  $\alpha$  as determined by transmission is strongly affected by absorption as well as scatter. The subject of the variation of  $\alpha$  with turbidity is not further pursued in this work, as the aim of the research was to determine the capability of the sensor within a range of concentration of suspended solids and determine its mathematical dynamic range in terms of turbidity. However, the variation in  $\alpha$ -value seen with turbidity and colour of light will be subject to further investigation with nephelometric measurements of  $180^\circ$ -scatter perhaps able to help with this.

From Table 1 and Fig. 3, it can additionally be concluded that, although the degree of absorption is within the dynamic range of the equipment at all four light colours for turbidity values up to 1700 NTU, the variation of  $\alpha$  with turbidity in the visible-green and visible-blue is larger. This may be expected as, in the blue and green region of the visible spectrum, suspended solids absorb larger portions of the incident radiation [21]. On the other hand, the near-infrared range is characterised by low reflectivity and absorptivity, and may be more suitable for the analysis of samples that are strongly light-scattering [5]. To investigate this future work will cover samples with turbidity greater than 1700 NTU. For these turbidities, it may at some point be seen that long optical paths may not be capable of providing values within the dynamic range even for the green and blue LEDs, so, in the future, a decision on the correct choice of an optical path based on the concentrations of suspended solids will be made as well.

As mentioned in the methodology section for very high dilution, low turbidity samples ( $< 0.5 \text{ mL}_{\text{milk}} \text{ L}^{-1}$ ,  $< 20 \text{ NTU}$ ) the effect of instrumental noise on absorption measurements was significant. As a result, if the measurements on high dilution samples are included in the data in Fig. 2a to d the  $R^2$  values are less than 0.9, and for this reason they have been excluded from the plots.

### 3.2. Distinguishing between apparent and true colour

To address Question 2 the attenuation coefficient  $\alpha$  was determined both for filtered and unfiltered Test Fluid 2 samples. As expected, turbidity after filtration is negligible because of the suspended solids having been removed, leaving only the dissolved component. Indeed, as seen in Table 2, the bench-top turbidimeter detects only noise. It can also be seen in Table 2 that even before filtration, recorded turbidity values are low ( $\leq 20 \text{ NTU}$ ) as the settleable fraction of the suspended solids has been removed.

Due to the consequent high scatter in the measured transmission data, in particular for the near-infrared and the red, only the plots for visible-green and visible-blue are presented below in Fig. 4a to d.

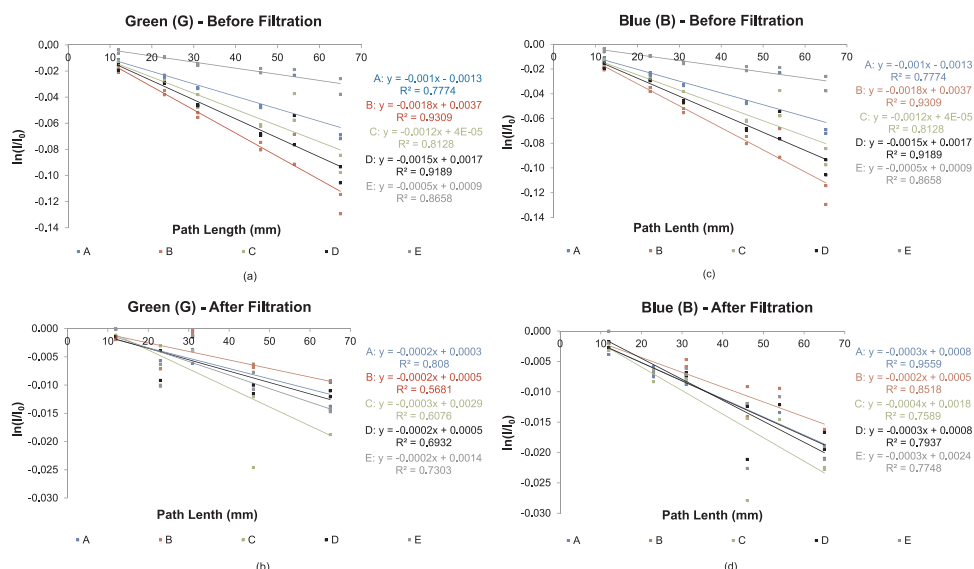
However, in general, the tests showed that the effect of filtering out suspended solids was clearly identified at all four colours with the turbid unfiltered samples exhibiting significantly higher  $\alpha$ -values than the turbidity-free filtered samples.

Finally, the  $\alpha$ -values obtained in the green and the blue are compared to measurements of true and apparent colour in Table 3. True colour includes colour from dissolved matter (turbidity has been removed by filtration or centrifugation) and apparent colour includes colour both from suspended and dissolved matter [6].

As can be seen in the table, the turbid unfiltered samples (apparent colour) exhibit significantly higher  $\alpha$ -values than the turbidity-free filtered (true colour) samples. True colour in water samples is, in general, attributed to dissolved matter derived from

**Table 2**  
Measurements of Turbidity.

| Samples | Before filtration<br>Turbidity (NTU) | After filtration |
|---------|--------------------------------------|------------------|
| A       | $9.5 \pm 2.4$                        | $0.56 \pm 0.14$  |
| B       | $15.6 \pm 3.9$                       | $0.56 \pm 0.14$  |
| C       | $11.9 \pm 3.0$                       | $0.76 \pm 0.19$  |
| D       | $14 \pm 3.6$                         | $0.51 \pm 0.13$  |
| E       | $3.4 \pm 0.9$                        | $0.56 \pm 0.14$  |



**Fig. 4.** a:  $\ln(I/I_0)$  vs optical path length ( $l$ ) (Visible-Green - Before Filtration). b:  $\ln(I/I_0)$  vs optical path length ( $l$ ) (Visible-Green - After Filtration). c:  $\ln(I/I_0)$  vs optical path length ( $l$ ) (Visible-Blue - Before Filtration). d:  $\ln(I/I_0)$  vs optical path length ( $l$ ) (Visible-Blue - After Filtration). (For interpretation of the references to color in this figure legend, the reader is referred to the web version of this article).

**Table 3**  
Summary of Experimental Data (Colour).

| Samples | Before filtration       |                                       | After filtration |                     |                                       |        |
|---------|-------------------------|---------------------------------------|------------------|---------------------|---------------------------------------|--------|
|         | Apparent colour (Hazen) | $\alpha$ -values ( $\text{mm}^{-1}$ ) |                  | True colour (Hazen) | $\alpha$ -values ( $\text{mm}^{-1}$ ) |        |
|         |                         | G                                     | B                |                     | G                                     | B      |
| A       | 96 ± 24                 | 0.001                                 | 0.0012           | 21 ± 5.3            | 0.0002                                | 0.0003 |
| B       | 162 ± 41                | 0.0018                                | 0.002            | 22 ± 5.6            | 0.0002                                | 0.0002 |
| C       | 103 ± 26                | 0.0012                                | 0.0015           | 29 ± 7.3            | 0.0003                                | 0.0004 |
| D       | 138 ± 35                | 0.0015                                | 0.0017           | 20 ± 5.0            | 0.0002                                | 0.0003 |
| E       | 68 ± 17                 | 0.0005                                | 0.0005           | 23 ± 5.8            | 0.0002                                | 0.0003 |

certain organic and inorganic sources. For these samples, it is mainly due to coloured dissolved organic matter (CDOM), e.g. humic and fulvic acids from soil, which strongly absorbs short wavelengths like visible-green and visible-blue. Consequently,  $\alpha$ -values can be resolved in the filtered samples, albeit at the limit of experimental resolution, at these two colours.

The ability of the optical instrumentation to deal with colour is quite beneficial, considering that foods outside the range of acceptable colour run the risk of being rejected by consumers, as colour is not only related to the physical and chemical properties of the product but also to the consumer's perception of what product quality is acceptable [15].

### 3.3. Potential uses of the instrumentation

The capability of in-line sensors to characterise water samples chemically cannot be expected to match that of off-line, laboratory-based methods. This drawback is offset by the speed of response, and hence the capability of indicating variation of contaminant concentrations with time. The potential uses of the optical instrumentation are therefore in managing water usage, and in characterising the effects of changes to manufacturing processes and systems designed to improve water efficiency. An example would be monitoring the quality of a wastewater stream destined for reuse to ensure it does not exceed safety limits. This would help overcome one of the barriers to wastewater reuse, particularly in food manufacturing [3], which is variability in the content of the stream. For a more detailed discussion of applications see the work by Webb et al. [25].

## 4. Conclusions

The capability of optical instrumentation to give in-line, real-time water turbidity and colour analysis was investigated. Two types of test fluid, i.e. synthetic high-turbidity water prepared in the laboratory and used process water collected in different places within a brickyard, were used. In-line measurements of the attenuation of the intensity of light were made at four colours (visible-red, visible-green and visible-blue, and near-infrared) with a commercial optical product monitor in use in the food industry. These

measurements were supported by off-line water quality parameter measurements (turbidity and colour). Variation in attenuation of the light intensity with optical path length for turbidity values up to 1700 NTU was successfully resolved by the in-line instrument. For turbidity values > 20 NTU, the optical data fits the Beer-Lambert model well ( $R^2 > 0.9$ ), so the values of the attenuation coefficient  $\alpha$  were calculated from the fits for each dilution and colour. The effect of filtering out suspended solids in the used process water was also clearly identified in optical measurements. The attenuation coefficient could be resolved in the filtered samples, albeit at the limit of experimental resolution, for the two colours visible-green and visible-blue. This is consistent with true colour absorption in the used process water being from coloured dissolved organic matter, which strongly absorbs short wavelengths. The optical instrumentation appears then to have the potential to be extensively used within the food industry where rapid water content measurements of colour and turbidity are required, as a measure that will help it to enhance its water sustainability strategy. Future work will concentrate on further investigating the variation in  $\alpha$ -value seen with turbidity and colour of light, on attempts to increase the dynamic range of the instrument, on contractual improvements so that the behaviour of running fluids is tested and on applications out of the food industry.

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