Microfluidic platform for multi-parameter water quality analysis

John Cleary, Damien Maher, Deirdre Cogan, Dermot Diamond

CLARITY Centre for Sensor Web Technologies, Dublin City University, Ireland

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Outline

• Introduction
• System design and development
• Phosphate sensor design and deployments
• Other parameters
• Future plans & Conclusions
Introduction

Motivation

• Monitoring of environmental water quality is predominantly based on manual sampling followed by laboratory analysis
• Expensive → Limited in terms of frequency and number of locations
• Not sufficient to satisfy the demands of environmental legislation (e.g. EU WFD)

Autonomous sensors

• Can provide high-frequency data from a larger number of locations with lower cost
System requirements

- Reliable and sensitive detection system
- Long deployable lifetime ⇔ Low power consumption
- Wireless communication of data to user
- Rugged and portable design
- Low cost, Low maintenance
Approach

- Colorimetric reagent chemistry
- Microfluidic technology
- LED-based detection systems
- Communication by GSM / short range radio (e.g. ZigBee)
Key factors in system design

Chemistry
• Sensitive detection (mg/L - µg/L)
• Reagent stability
• Simple methods (number of reagents/steps)
  → simple, robust microfluidic chips

Detectors
• LED & photodiode or Paired Emitter Detector Diodes (PEDDs)
• Sensitive, Low power consumption, Low cost

Communications
• Application dependent (range, frequency, power demands)
System design

- Communications (GSM / ZigBee)
- Sample port
- Reagent & standard storage
- Pumping system
- Microfluidic chip
- Detector (LED & photodiode)
- Waste storage

- Flash memory chip
- RS232 ports
- FET outputs
- Power supply
- Microcontroller
- FET outputs
- Power supply
- Microcontroller
- FET outputs
Phosphate analyser

3 stages of development
• Demonstration of method (chemistry & detector) in microfluidic manifold
• Development and field testing of prototype system
• Commercialisation (Episensor Ltd., Ireland)
Phosphate sensor

- Phosphate sensor
- Molybdenum yellow method
- Single, highly stable reagent
- Fast reaction

(1) Sample inlet;
(2) Control board and detection system;
(3) Dual channel peristaltic pumps;
(4) Reagent bags;
(5) IP68 enclosure
Specifications

- Minimum sample interval: 15 minutes
- Linear response range: 0-20 mg/L $\text{PO}_4^{3-}$
- Limit of detection: 0.20 mg/L $\text{PO}_4^{3-}$
- Up to 1000 assays before reagent/battery replacement
- Weight 1.8 kg with full reagent load
Deployments (i) Wastewater

• WWTP in Co. Kildare, Ireland
• Sensor installed in effluent discharge tank
• 30 min sample interval
• Autosampler collecting 24 samples/week for validation
Deployments (ii) Estuary

- Broadmeadow Water
- Enclosed estuary north of Dublin City
- Highly impacted - municipal wastewater + agricultural inputs
Deployments (ii) Estuary

![Graph showing phosphate sensor and manual sample data over time]

**Phosphate Sensor**
- Sensor value (mg/L phosphate)
- \( R^2 = 0.9706 \)

**Manual Sample**
- Sensor value (mg/L phosphate)
- \( R^2 = 0.908 \)

\[ y = 1.040x - 0.039 \]
Nitrite detection

Griess assay

- Single reagent can be incorporated into platform with minimal changes
- Detection at 540 nm
- Detection limit 5 µg/L

\[
y = 1.2944x + 0.0476
\]

\[R^2 = 0.9955\]
# Nitrite detection – blind test

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<th>Sample Reference/Type</th>
<th>Sample ID</th>
<th>Sample Concentration 17/02/2012 by IC</th>
<th>Sample Concentration 21/02/2012 by HACH (LL)</th>
<th>Sample Concentration 21/02/2012 by IC</th>
<th>DCU results 22/02/12</th>
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<td>0.870</td>
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</table>

Samples and external analyses supplied by TE Laboratories Ltd.
pH measurement

- Mixed indicator solution
- Dual LEDs with adjustable intensity
- Photodiode detector
Mixed indicator solution

- Colour change over range pH 4 - 10
- 3 options for detection wavelength
- Linear response using ratio of 430 and 570 nm peaks

![Graph showing absorbance vs wavelength and pH vs ratio Abs430/Abs570]
Chemical Oxygen Demand (COD)

- Standard COD method based on oxidation using potassium dichromate, conc. $\text{H}_2\text{SO}_4$, heating to $150^\circ\text{C}$ for 2 hours

- Colour change from chromate ion ($\text{Cr}_2\text{O}_7^{2-}$, orange) to chromic ion ($\text{Cr}^{3+}$, green)

- Monitor decrease in chromate peak at 430 nm
Detector output

Issues

- Strong acid → resistant materials → more expensive
- Heating → high power consumption
Nitrate detection

Chromotropic acid method

- Simplified version of literature method* in which reagents (CA + conc. H₂SO₄) added separately with cooling

- Reagents can be combined into single solution, cooling step eliminated

Calibration

- Calibration curves by UV-vis and LED-based detection
- Linear response to approx. 50 mg/L
- Suitable for environmental and drinking water applications
Future work

Phosphate
• Long term deployment of multiple sensors at sites of interest
• Recently installed at constructed wetlands treatment system

pH, Nitrite
• Field-testing of prototype units
Future work

COD
- Optimisation of detection method in microfluidic system using dual wavelength detection (430 and 600 nm)
- Development of prototype for wastewater monitoring

Nitrate
- Optimisation of chromototropic acid method
- Development of prototype (drinking water)
Conclusions

Deployable sensor for phosphate has been developed and successfully field tested in wastewater, freshwater and estuarine conditions.

Prototypes for nitrite and pH have been developed and are ready for field testing.

Methods for COD and nitrate are being optimised.
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