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Monitoring chemistry in situ with the Smart Stirrer—a magnetic stirrer bar with an integrated process monitoring system

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Abstract

Inspired by the miniaturisation and efficiency of the sensors for telemetry, we have developed a device that provides the functionalities of laboratory magnetic stirring and integrated multi-sensor monitoring of various chemical reaction parameters. The device, called "Smart Stirrer", when immersed in a solution, can *in situ* monitor physical properties of the chemical reaction such as the temperature, conductivity, visible spectrum, opaqueness, stirring rate, and viscosity. This data is transmitted real-time over a wireless connection to an external system, such as a PC or smartphone. The flexible open-source software architecture allows effortless programming of the operation parameters of the Smart Stirrer in accordance with the end-user needs. The concept of the Smart Stirrer device with an integrated process monitoring system has been demonstrated in a series of experiments showing its capability for many hours of continuous telemetry with fine accuracy and a high data rate. Such a device can be used in conventional research laboratories, industrial production lines, flow reactors, and others where it can log the state of the process to ensure repeatability and operational consistency.

The modern level of microelectronics and communications has enabled precise telemetry and high-performance data analysis using low-cost miniature devices that require little power. Such devices are widely adopted by many industries where precision in measurements and miniaturisation is essential. The use of microcontrollers and systems on a chip (SoC) integrated circuits has greatly expanded and simplified measurements in research laboratories—high-fidelity monitoring of various parameters has never been easier[1]. With the automation of experimental procedures and remote reaction monitoring comes improved safety, opening of new reaction pathways, improved repeatability, and reduced experimenter resource requirement[2–5].

Wireless communication for data transfer further extends the use of SoC in labs. For instance, a wearable device comprising ion-sensitive field-effect-transistor (ISFET) based pH and printed temperature sensor can monitor sweating and skin temperature[6]. Real-time continuous monitoring of sweat metabolites such as glucose or lactate have been realised using a skin-mounted electrochemical microchip flow detection system, transferring the data via Bluetooth[7]. A bio-optoelectronic system for the compensation of Adenosine Triphosphate (ATP) levels has been developed[8], comprising a reaction chamber, photosensor, Mbed microcontroller, relay and syringe pump. ATP is constantly depleted due to the presence of an ATP-hydrolysing enzyme, while a real-time monitoring system measures ATP and compensates automatically for the depletion. In another study, a transducer device was shown to control the operation of liquid-liquid extraction to a mass spectrometer enabling long-term monitoring of dynamic processes such as drug dissolution[9]. A portable electrochemical system with an integrated sensor based on the ISFET with improved detection limit has been developed for potentiometric tests[10]. Gate Scientific provides a hotplate with smartSENSE Stirbar features continuously relaying the temperature or pH of the liquid in which it is immersed via RFID wireless communication to the hotplate[11].

A wireless endoscopy capsule is a great example of the integration of miniature SoC and sensors, and the powerful functionality of the wireless data transfer[12]. A pill-shaped electronic capsule ingested by a patient collects the data from the entire gastrointestinal tract. This electronic capsule is integrated with an image sensor (CMOS camera), illumination optics, processing unit, a wireless communication module, and a battery[13]. The data are compressed and processed in the unit to be sent to the data recorder through the radiofrequency channel for immediate detection of bleeding[14], or some diseases[15]. A capsule with a gas sensor was used to obtain information about the chemical composition of the gut[16].

The integration of such devices with the Internet of Things (IoT) technology may greater enhance their functionality[17–19]. The remote sensor networks can be linked with distributed analytical chemical services, centralized laboratories, cloud storage, and cloud computing. The efficiency of a fully cloud-based system has been demonstrated in conducting catalytic reactions in which a fixed volume of reaction solution is passed multiple times through a small volume of catalyst contained within a packed column[20]. The Particle Photon Wi-Fi module (Particle Inc, USA), an out of the box solution that includes low-cost Wi-Fi microcontrollers and development kit to build smartphone or web apps, have been used for remote operation and monitoring of long-term pH-oscillating reaction and acidification due to microbial fermentation (spoilage of milk)[21]. The cloud provides service for integration of the data stream into a database of choice. A portable magnetoresistive testing system has been demonstrated for influenza virus detection[22]. Being fully compatible with modern mobile health platforms it can transmit data to a cloud-based infrastructure.

There are a few commercial implementations of the IoT platforms that focus on the data-driven lab. TetraScience Inc (USA)[23] collaborates with equipment manufacturers and software vendors to integrate cloud connectivity that will allow 24/7 experimental data collection from lab instruments. Deepmatter Group Plc (UK)[24] provide the concept of digitizing chemistry, where obtained data are processed in the cloud using Artificial Intelligence and

Machine Learning *to make better molecules and provide insights into processes.* Thermo Fisher Connect[25] (Thermo Fisher Scientific Inc, USA) is a cloud-based system designed to monitor and process data from six different types of equipment, including a genetic analyzer, real-time polymerase chain reaction, next-generation sequencing system, thermocyclers and mass spectrometers. It also allows connection with a community inside the system, peers and collaborators, and access to scientific content libraries.

These examples show that SoCs with sensors can be easily integrated into almost any object and adapted for various applications. However, there is an acute problem in an affordable device with a maximum range of measured parameters that is easy to operate and requires no or little setup. Conventional real-time monitoring systems for chemical processes is performed with the external devices, often with provided single functionality, and therefore they need to be combined along with external agitation systems. Setting up external probes into the reactor may increase the chance of contamination and creates an additional reactor dead volume where impurities may accumulate over time. In the present work, we introduce an affordable solution for *in-situ* monitoring of parameters of a chemical reaction or the state of the reactor. The Smart Stirrer system is based on a Bluetooth module and sensors providing functionality for temperature, colour, rotation inertia and conductivity monitoring. Such a multi-functional device works as a conventional magnetic stirrer bar analytical system based on an open-source code, which allows easy customisation of the functionality for the required user needs.

Experimental Section

A: Device Fabrication. Two complementary implementations of the Smart Stirrer have been produced: one based on an ATmega328p (Atmel) 8-bit microcontroller with an RN4871 Bluetooth module (Microchip Technology Inc., U.S.A.), and the other based on an nRF52840 SoC (Nordic Semiconductor ASA, Norway). The first implementation benefits from an easy-to-program Arduino-compatible environment; the second provides an all-in-one system with an advanced Bluetooth 5 protocol, TX Power 8 dB, 64× faster 32-bit CPU, and 10-fold lower energy consumption [26]. Further details on the power consumption are provided in the Supplementary Information.

The stirrer contains a modular architecture with stackable sensor boards that allow rapid adjustment of sensor functions for particular applications (Figure 1a). Details of the sensor are provided in the Supplementary Information with the design and firmware files[27]. The sensors include an inertia module (three-axis accelerometer, gyroscope and magnetometer), an ambient light sensor, thermistor and medium conductivity recorder realized using two external copper-plated electrodes. The Smart Stirrer is powered with either a coin cell battery (36 mAh) or a rechargeable Li-polymer battery (120 mAh) capable of continuous data monitoring for over 100 h.

The housing of the Smart Stirrer is either a screwed 3D-printed poly(methyl methacrylate translucent capsule for quick access to the electronics or a sealed fluorinated ethylene propylene (FEP) pouch for excellent chemical resistance.

B: Receiver. A PC and/or a Raspberry Pi 3 B+ with built-in Bluetooth Low Energy (BLE) has been used to receive and monitor the data from the Smart Stirrer during the tests and experiments. Custom Matlab and/or Python codes have been used to connect to the Smart Stirrer, to send information to start/stop sensor reading, for data transfer, to record to the file, and for plotting the readings real-time. When the Smart Stirrer is powered, it starts the Bluetooth advertising transmission that broadcasts the device information. The program on the central device (receiver) sends a request to the Smart Stirrer to start getting data from sensors, sending it over BLE with a predefined interval, typically 1 s. The data from the sensors were sent by the Stirrer as a package of the hexadecimal numbers further converted to the ASCII text file by the central device. Additionally, the data from the Smart Stirrer can be monitored using free mobile app nRF Toolbox and UART service.

C: Demonstration. An acid–base titration experiment[28,29] was conducted to determine the concentration of an approximately 1 M NaOH solution. A Smart Stirrer was placed into a conical flask with 100 mL of distilled water and the magnet rotor of the hot plate was set to 240 rpm. Afterwards, 2 mL of the NaOH solution, prepared from 98% NaOH pellets (Sigma-Aldrich, UK) and a droplet of thymol blue indicator (Sigma-Aldrich, UK) were added. Hydrochloric acid (HCl) (Fischer Scientific, UK) of 0.1 M was dosed into the flask using a syringe pump at a flow rate of 4 mL min^{-1}. With increasing acid content, the colour, temperature and conductivity of the solution were recorded by the Smart Stirrer.

Once the exact concentration of the NaOH solution was determined, it was used to titrate approximately 1.7 M HCl solution. Here 25 mL of the NaOH solution was added into 100 mL of water with thymol blue indicator followed by dosing the HCl solution at a flow rate of 4 mL min^{-1}. During this experiment, the temperature, colour and conductivity of the solution changed in a broader range compared to the previous experiment. For a reference, temperature and conductivity were monitored externally with a K-type thermocouple immersed into the solution and a Keithley 2401 electrometer. For the sake of demonstration, both implementations of the Smart Stirrer were used in the titration experiments.

An oscillation reaction (a manganese-catalysed oxidation of malonic acid by bromate)[29,30] was performed to study the response times of the unit. The stirrer was placed into 150 mL of 1.5 M sulphuric acid (H_2SO_4) solution (Sigma-Aldrich, UK) cooled using an ice bath. Then 1.8 g of malonic acid $CH_2(CO_2H)_2$ (Fisher Scientific, UK) and 1.6 g potassium bromate $KBrO_3$ (Fisher Scientific, UK) were added. After dissolution, 0.61 g of manganese nitrate hexahydrate $Mn(NO_3)_2 \cdot 6H_2O$ (Fisher Scientific, UK) was added, starting the reaction that was recorded with a video camera and the Smart Stirrer.

Viscosity evaluation was carried out using a set of polyvinyl alcohol (PVA) 80% hydrolysed, $M_w = 9000 - 10000$ gmol^{-1} (Sigma-Aldrich, UK) aqueous solutions with different concentrations of PVA. The Smart Stirrer was placed into a beaker with the PVA solution, rotated with a laboratory plate till a constant angular velocity. Afterwards, the laboratory plate was removed rapidly and the stirrer slowed down under the influence of the viscous media with the angular rate measured by the stirrer. Oscillation reaction and viscosity measurements were carried out using Smart Stirrer with nRF52 chip (Nordic).

Results and Discussion

Figure 1 shows the ATmega and nRF52 implementations of the Smart Stirrer. The design is modular and contains stackable boards that allow adding the required sensors for the intended application. The ATmega unit is compatible with the Arduino software development platform allowing for a simple start in developing the unit. The nRF52 unit is though significantly more computationally powerful and energy-efficient. The main layer contains the microcontroller, a wireless communications module and an optical sensor to put the device into a sleep mode. The second layer contains an inertia sensor and an analogue-to-digital converter for temperature and conductivity measurements. The layer connections include power and data lines that allow addition of many layers independently. The Nordic nRF52 implementation of the Smart Stirrer contains a single board with a SoC that contains both the microcontroller and the wireless communication module. The materials and components included in the Smart Stirrer are readily available and cost below $20.

In situ Temperature, Conductivity and Colour Monitoring

Titration was carried out with 0.1 and 1.7 M HCl solutions monitoring colour, conductivity and temperature in-situ shown in Figure 2. The data from the light sensor (red, blue, and green colour channels) were used to construct the RGB colour profile, a horizontal colour bar. In both cases, the initial blue colour of the thymol blue indicator changed close to the equivalent point. In the case of 0.1 M acid, the blue colour changed to yellow (indicator colour in the pH region of 2.8 to 8.0). In the case of 1.7 M acid, the colour almost immediately changed further to red (pH below 2.8). The visual changes in the solution colour agreed

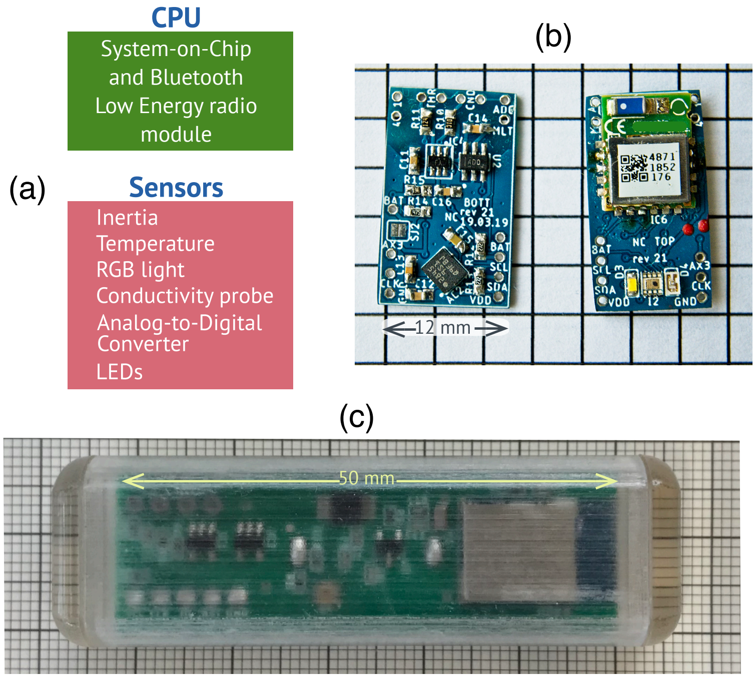

Figure 1: (a) Smart Stirrer hardware platform. (b) Photograph of the circuit board of the Smart Stirrer ATmega, and (c) 3D-printed capsule with the Smart Stirrer nRF52.

with the readings of the Smart Stirrer. However, it is worth noting that the colour readings changed over time (± 0.1 a.u.) due to the variations of the overall illumination entering the sensor due to the spinning of the Smart Stirrer, although not affecting the RGB profile. The Smart Stirrer, therefore, provides both mixing and colour data and the exact determination of the equivalence point for acid-base titration.

In addition to colour measurements, the Smart Stirrer also provided information on the temperature and conductivity of the medium. During the 1.7 M HCl titration, exothermic neutralisation reaction was accompanied by the increase of the solution temperature by approximately 2 degrees (Figure 2c). The same dynamics in the temperature change was recorded by the Smart Stirrer (Figure 2b). The drastic change in the conductivity occurs due to the change in ion concentration[28,29]. The combination of multiple physical parameters measured generate complementary data in the reaction behaviour and provide more accurate information not available from a single instrument. Moreover, putting the Smart Stirrer into the reaction enables collecting data to ensure repeatability and consistency of the process

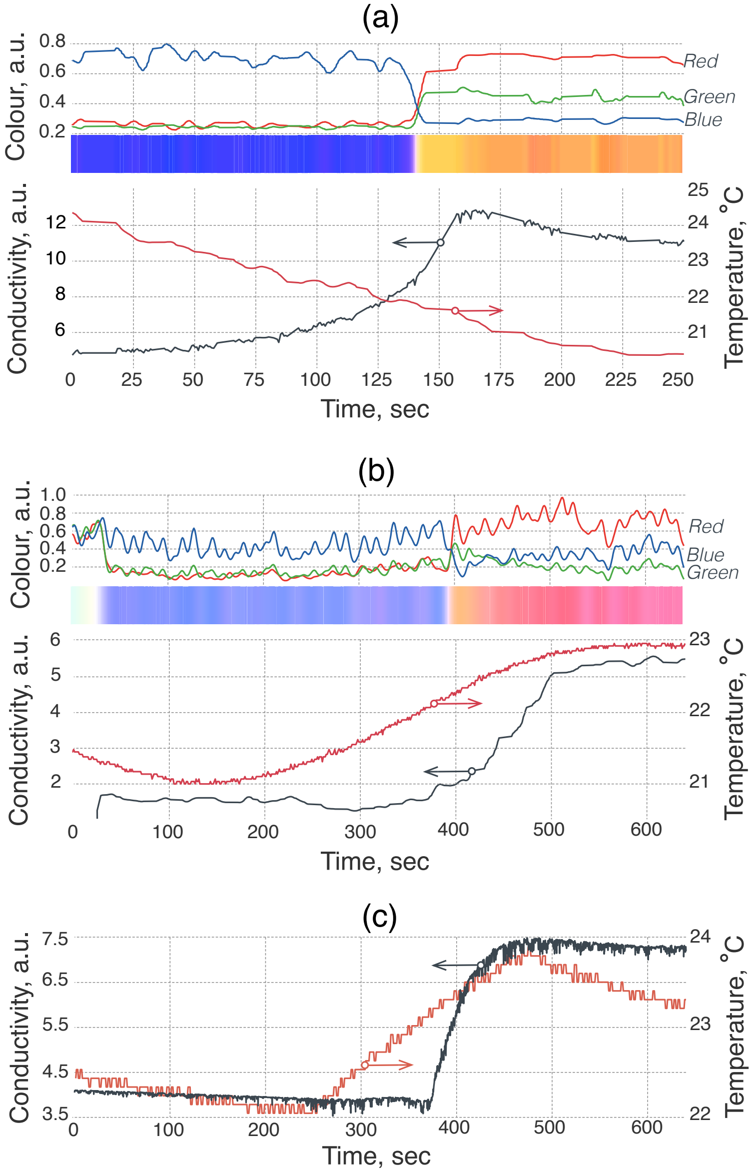

Figure 2: The experimental data received from the Smart Stirrer during the titration experiments of (a) 2 mL 1M NaOH solution with 0.100 M HCl (addition rate 4 mL·min^{-1}); and (b) 25 mL 1M NaOH solution with 1.7 M HCl (addition rate 4 mL·min^{-1}). Ambient red, green, and blue channels light (top), colour profile (middle horizontal bar), conductivity (blue curve at the bottom), and temperature (red curve at the bottom). (c) The temperature and conductivity for the experiment in (b) measured using the external thermocouple temperature logger and source meter, respectively. (a) and (b) were recorded using ATmega328p and nRF52 Smart Stirrers, respectively.

performed.

Figure 3a demonstrates the use of the Smart Stirrer to monitor the changes in the solution colour during the oscillation reaction. The horizontal colour bar in Figure 3 presents the

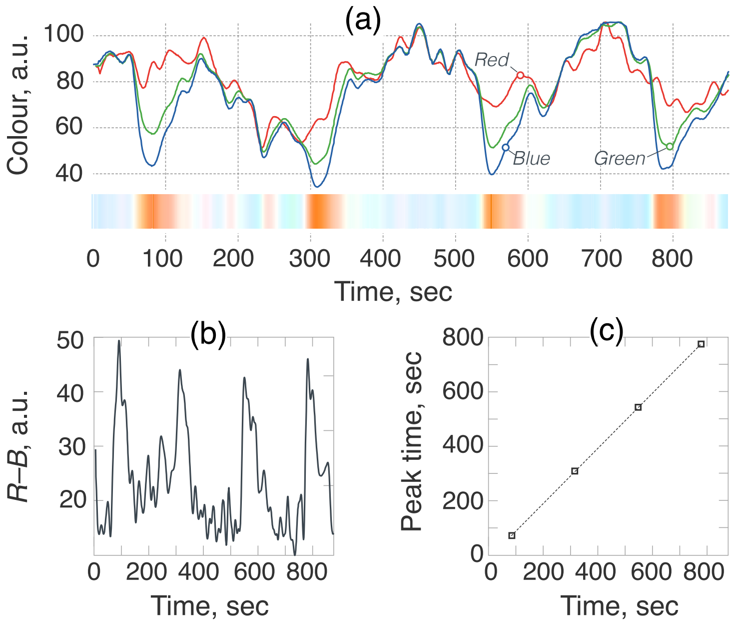

Figure 3: (a) Ambient light readings obtained by the Smart Stirrer during the oscillating reaction; (b) the difference in red R and blue B channel readings over time; (c) change in the R-B peak position with time.

ambient colour during the reaction as it recorded by the Smart Stirrer. While conductivity and temperature show no variation, the colour of the solution oscillates rapidly during the abrupt colourless-to-red revolution and gradually during the gentle red-to-colourless change. Figure 3b shows the difference in red-blue channels highlighting the oscillation reaction period of 230 s (Figure 3c). Comparison with the video of the experiment shows no response lag demonstrating the ability of the Smart Stirrer for measurements in real-time.

Viscosity Measurement

The inertia sensor built in into the Smart Stirrer was used for quantitative evaluation of the solution viscosity. For the test, PVA aqueous solutions with various concentrations were used. The solution viscosity depends on the molecular weight of the polymer, concentration, degree of hydrolysis, and temperature, and ranged between 1 to 1500 mPa·s[31].

The Smart Stirrer demonstration was performed by spinning the stirrer to about 200 rpm until a constant angular rate was achieved. Afterwards, the beaker with the stirrer was rapidly removed from the hot plate to exclude the effect of the magnet rotor on the Smart Stirrer (the magnetometer sensor in the Smart Stirrer allows recognising of the hot plate removal). Once the hot plate was removed, the spinning stirrer decelerates due to the frictional force F being proportional to the viscosity η (Stoke's law). Therefore, for the simplified model of a stirrer bar with a rod-like shape with length l and mass m, spinning around its centre mass, the angular acceleration (deceleration) of the stirrer is proportional to the viscosity of the solution:

$$\alpha \propto \eta \frac{l}{I} \tag{1}$$

where I is the rotational inertia.

Figures 4a and 4b shows the angular velocity ω of the Smart Stirrer when it was removed from the hot plate, and show the moment when the rotation was stopped due to friction for PVA solutions with various concentration (viscosity) for $\omega_1 = 142$ rpm (Figure 4a) and $\omega_2 = 183$ rpm (Figure 4b). The angular rate decreases linearly in agreement with the expectations, and the rate of deceleration strongly depends on the PVA concentration in the solution. Figure 4c presents the dependence of the mod of the angular acceleration $|d\omega/dt|$ as a function of PVA concentration (dotted lines) and specific viscosity η_{sp} (solid line) obtained using truncated Huggins equation:[32]

$$\eta_{sp} = [\eta]c + k[\eta]^2c^2, \tag{2}$$

where $[\eta] = 0.238$ mL/g is the PVA ($M_w \approx 9500$ gmol^{-1}, 80% hydrolised) intrinsic viscosity[33], $k = 0.688$ is referred to as the Huggins dimensionless constant, and c is the solution concentration.

Thus, the angular acceleration depends on the PVA concentration and does not depend on the initial spin rate of the stirrer, and can be used as a qualitative evaluation parameter for estimation of the dynamic viscosity. To a large extent, the viscous force on the stirrer depends on the viscosity of the solution, shape of a stirrer bar, its mass and rotation inertia.

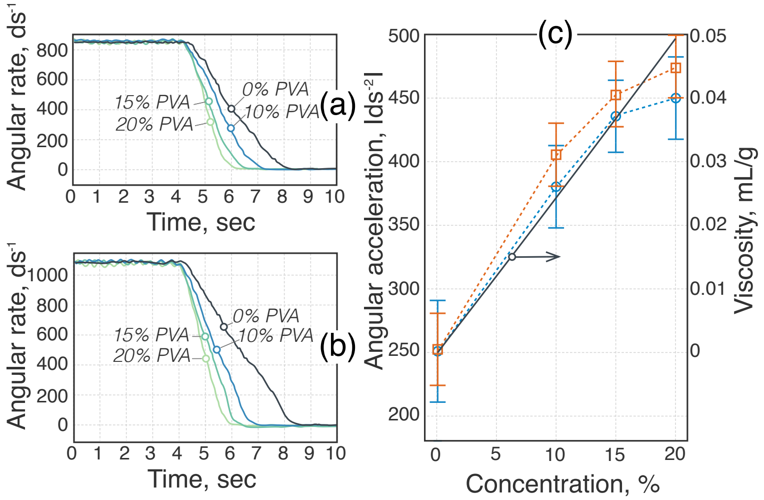

Figure 4: The angular velocity as a function of time measured by the Smart Stirrer during viscous deceleration in the PVA solutions starting from $\omega_1 = 142$ rpm (a), and $\omega_2 = 183$ rpm (b). The mod of angular deceleration (c) as a function of PVA concentration for corresponding ω_1 (blue squares) and ω_2 (orange circles) initial angular rates (the error bars shown are standard deviations for 6 measurements performed). Solid line represents the theoretical specific viscosity η_{sp} for PVA solution as a function of concentration (Equation 2).

Therefore, the obtained results indicate that with a careful calibration of the system stirrer-shape/mass/beaker-size it is plausible to use the Smart Stirrer for qualitative prompt and low-cost measurements of the viscosity.

In general, the use of wireless technology to transmit data on the state of a chemical reaction can greatly simplify the process control in the synthesis of materials. We believe that the Smart Stirrer with its presented concept will be in the near future a "must-have" laboratory device. The open-source code can be easily modified (or used as-is) for specific purposes in analytical chemistry. The Smart Stirrer is also a coherent complement to the concept of Telechemistry[21,34] and automation in chemical synthesis[18–20]. A wide range of low-cost sensors, along with the custom sensors and high performance of microprocessors, opens up unlimited possibilities for new applications, such as in-situ dynamic spectroscopy, pH and potentiometry, particle analysis, etc. The intrinsic rotation of a stirrer device may be used to harvest energy for powering electronic components makes it possible to use the device in arbitrarily long measurements. Perhaps the only limitation is a narrow temperature range (−40°C ÷ +85°C) inherent for the majority of digital electronics.

Conclusion

A low-cost and high-performance autonomous multi-sensor laboratory device that can be utilised as an intelligent magnetic stirrer bar, "Smart Stirrer", has been developed and demonstrated. The Smart Stirrer contains a small printed circuit board with an integrated system on a chip, a low-energy Bluetooth module, and an easily expandable range of sensors including ambient light, inertia, temperature, and conductivity. The Smart Stirrer shows many hours of continuous data transfer at a high rate.

In contrast to the commercial system such as of Gate Scientific that only operates in link with a proprietary hotplate (with a market price above $2000), the presented device comprises an affordable solution, and an open-source programming platform for a customizable functionality such as the power mode, sensors used, sensor resolution, sampling rate, and data transfer rate. The application of the Smart Stirrer was demonstrated in acid-base titration, and an oscillating reaction. Additionally, it was shown that the Smart Stirrer can be used for a qualitative evaluation of the viscosity thus making this device a low-cost alternative to the existing solutions.

The sensors demonstrated could form a platform for digitizing chemistry in research laboratories as well as industrial manufacturing. Connecting stirrers into the networks, Internet of Things, and wireless power will expand its application even further.

Acknowledgement

NC is grateful for the EPSRC Dial-a-molecule network for the proof-of-concept grant and the Institution of Chemical Engineers (IChemE) for the Andrew Fellowship. SB acknowledges the WMG Summer Internship Programme.

Supporting Information Available: The following files are available free of charge. SmartStirrerSupplementalMaterials.pdf. The Supporting Information contains of technical details of the Smart Stirrer circuit configuration, used sensors, and power consumption.

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