Sample Tube pH Monitoring via Passive Powering and Communication

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***Abstract*—This paper presents the application of a simple, inductively-coupled potentiometric pH measurement system into the cap of standard laboratory sample tube caps, thus enabling continuous, wireless monitoring of a bevy of samples. The system is powered by a compact Class E amplifier using inductive coupling via a designed resonance frequency of 1 MHz. Signals are transmitted back via load modulation at frequencies a fraction of the power carrier frequency, thus allowing for extraction of the signal frequency. Results are demonstrating which clearly show that modulation frequency tracks closely with open circuit potential, and the system as a whole features good sensitivity and linearity, as well as holds promises for a host of applications.**

***Keywords*—pH, iridium oxide, inductive coupling, wireless power transfer, load modulation**

I. INTRODUCTION

Sensing of pH in real-time across a multitude of lab samples is a labor intensive task, wherein the researcher may have to manually measure each sample container or, at a minimum, fabricate a measurement apparatus for each sample. It also exposes samples to varying environmental conditions with each measurement. For example, it has previously been found that varying oxygen levels can cause differences in gene expression [1]. Oxygen content is a condition which could vary in local atmosphere due to heterotrophic microbial respiration, human respiration, or other reaction pathways, and should be controlled for in pH monitoring of cultures. Other applicable scenarios can be listed as, for example, water samples being monitored over time: exposure to CO_2 will cause the creation of carbonic acid [2] and alter pH readings assumed to be the result of other factors. Obviously, there are a variety of biological and environmental samples in which chemical monitoring should be performed in atmospheres where humidity, temperature, and gas content are controlled.

pH monitoring in enclosed environments is currently possible but either costly, difficult to arrange into an apparatus, or still requires manual input. The most common device used is the glass style, dual junction, Ag/AgCl sensors; these feature low drift and high accuracy but take up considerable space and, if fitted individually to each sample container, would be prohibitively expensive. A researcher can choose to purchase these in bulk and fit them to a batch of samples to be monitored simultaneously; however, there are only several real-time monitoring solutions available, most feature only a few of channels. Further, these systems are quite expensive.

In this work, we present a reusable sample container cap with swappable pH transducers which can be screwed on to a standard sample tube and fitted to a base station monitoring an array of containers. A basic layout of this system and the cap-integrated receiver (RX) side are shown in ***Fig. 1***. This device uses inductively coupled coils for power transmission and communication (by load modulation - LM), sampling an iridium oxide (IrOx) pH probe. An LM PCB, with solenoid antenna, is fitted to the cap and interfaces with disposable IrOx/AgCl electrodes on polyimide. There are several benefits of this design. First, IrOx probes are simple and inexpensive to produce as well as being high performance materials [3]. Second, these caps can be designed to fit a variety of pre-existing tube designs and capacity (20, 30, 50 and 100 ml). And third, the passive wireless system reduces device complexity and cost, and the live/continuous data recording allows users to both keep their samples out of ambient environmental conditions and perform analysis on the fly. Given the simplicity of both the transmitting and receiving sides, the system can easily be reconfigured to communicate continuously with large sets of samples or can include linear/rotary mechanical systems for rapid discrete measurements of that same sample set. We speculate that this

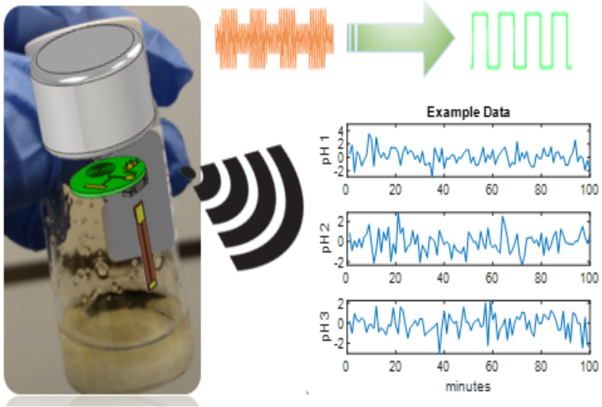

Fig. 1 – Eventual complete system: base station, data transmission, and probe in cap.

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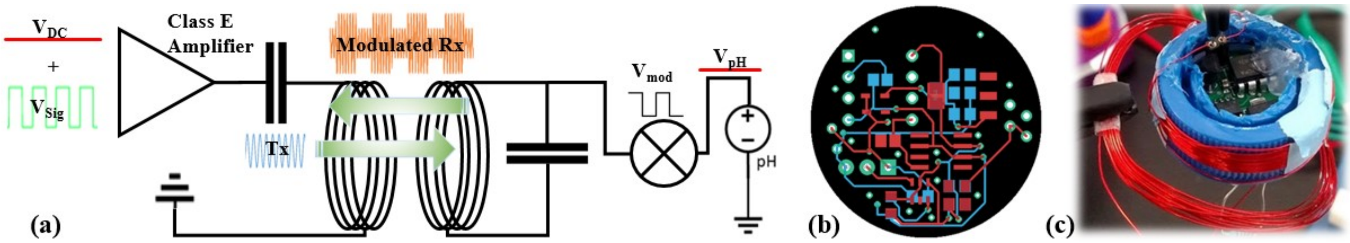

Fig. 2 – (**a**) A simplified schematic demonstrating that power is inductively coupled into the cap side and a load modulation-based communication signal is sent back. (**b**) Cap PCB design, dual layer and measuring in at 28 mm in diameter. (**c**) PCB and coil mounted into a demo cap, with a solenoid coil mounted externally. One of many possible TX-side coils is shown beneath it; in this work, a nearly identical coil was placed beneath the PCB coil.

device will yield significantly improved data collection in a myriad of lab sciences.

II. DEVICE DESIGN

A. *pH Measurement*

Measurement of pH is done by open-circuit potential (OCP) between an IrOx working electrode (WE) and any suitable reference electrode (RE). In most cases, silver chloride (AgCl) paste is used; for the purposes of isolating IrOx performance and device output from RE potential drift, a commercial RE was used in this case (see *Test Procedure*). A simple voltage buffer is used to ensure that current flow, thus faradaic reactions, does not occur. This configuration and its fabrication is discussed previously in [3], in which a simple voltage follower with significantly high input impedance and appropriate gain (~2.5) can allow for OCP measurement without appreciable Faradaic reactions in the IrOx. In this work, no gain was required at the voltage follower.

B. *Inductively-Coupled Power Transmission and Communication*

This group had previously built a base platform for testing of inductively-coupled power transmission and communication by load modulation [4]; while the initial demonstration focused on measurement of ECG signals, we subsequently demonstrated an open circuit potential measurement with the system for the purposes of pH measurement [5]. To briefly review the system shown in ***Fig. 2a***, the system essentially places a low frequency modulation signal atop a high frequency carrier signal (2 – 25 kHz and 1 MHz, respectively) based on a DC-to-FM conversion of the measurement signal by voltage-controlled oscillator (VCO). The modulated signal is sampled at the carrier transmitting solenoid coil (TX) and the modulation frequency could be easily measured atop the carrier; the previous multi-stage Sallen-Key bandpass filter and envelope detector was deemed unnecessary given the frequencies of interest and possible noise sources. The modulated square wave is then triggered on and a simple frequency measurement is taken via oscilloscope, for the purposes of this demonstration. In this case, the TX side L was 8.6 µH with a series C of 10 nF, while the RX side L was 9.5 µH with a parallel C of 2.7 nF; in practice, the complete system resonated close to 980 kHz. The most notable difference in this work is that the single modulation MOSFET was replaced with two opposing MOSFETs to ensure that the body diodes did not allow current passage unless the gate voltages were raised, regardless of drain-source relationships in these amplitude ranges.

The Class-E amplifier was modified from previous work. In order to achieve as efficient a design as possible, an empirical design guide was used [6]. Once again, a 1 W power output was targeted for the Class E amplifier to ensure testing functionality. L_1, the choke inductor, was chosen to be 150 µH and the other component values followed from there. The final amplifier configuration featured only 3 passive components outside the inductive coil. It was tuned as close to purely inductive as possible, thus the series C_2 of 10 nF. The parallel C_1 was chosen to be 470 pF and a load resistor of 18 Ω was used. The transistor used was an STP24NF10 (*STMicroelectronics, Geneva, Switzerland*).

III. TESTING, RESULTS, AND DISCUSSION

A. *Testing Materials*

The probe geometries used are featured in ***Fig. 3***. Two 1×1 mm^2 probes, one for the WE and one for the RE, are separated by 0.45 mm edge-to-edge; only the desired 1×1 mm^2 pad is left exposed, with the traces and surrounding area being covered by an additional layer of photoresist. Metal layer thicknesses and fabrication procedures follow [3]. Contact pad geometries were designed to match the internal pins of a 1.25 mm pitch commercial flexible printed circuit (FPC) connector (*39532045, Molex, LLC, Lisle, IL*).

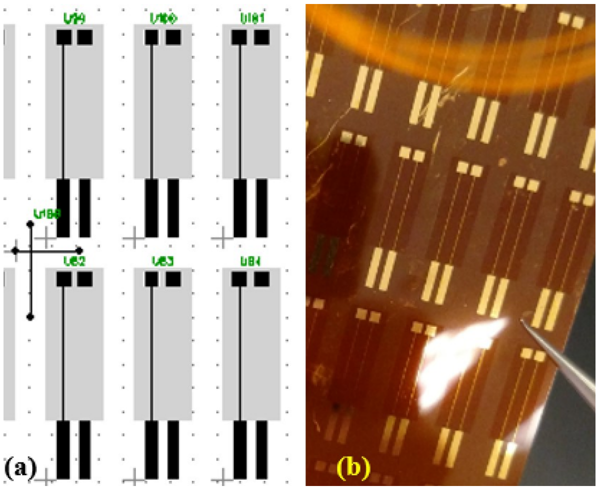

Fig. 3 – (**a**) pH probe mask design. (**b**) pH probes as fabricated.

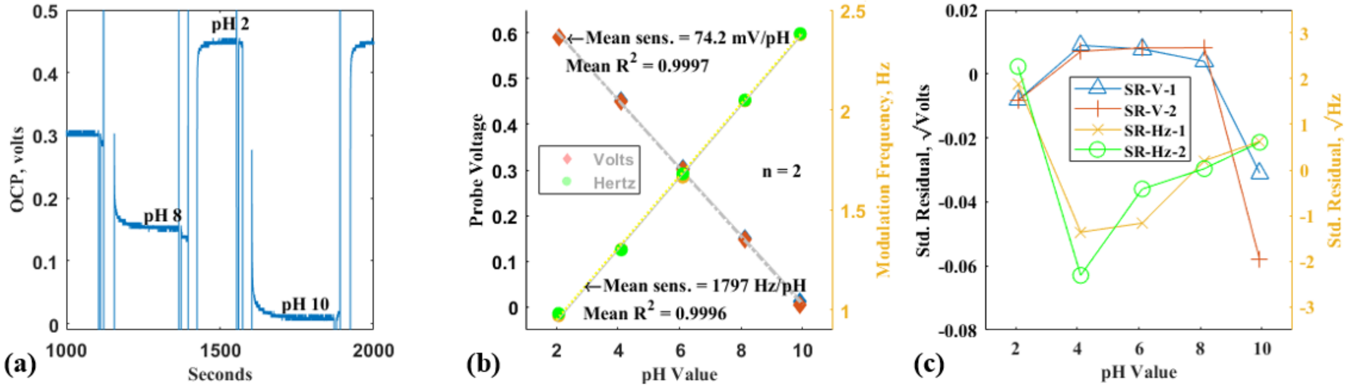

Fig. 4 – Data as recorded with coated IrOx probes. (**a**) A portion of the raw open circuit potential (OCP) as measured directly at the RE/WE pair. (**b**) Linear calibrations for both the OCP data and the measured frequency values from cap modulation. (**c**) Standardized residuals for both the OCP data and the measured frequency from cap modulation.

The pH panel used is known as a Britton-Robinson (B-R) buffer, a mix of a stock mixture of acetic, phosphoric, and boric acids and varying amounts of sodium hydroxide to determine the pH level; ratios and recipes were taken from [7]. The B-R panel was chosen due to the absence of chlorine ions and ethylenediaminetetraacetic acid, both of which will alter RE potential and potentially cause redox reactions with the IrOx. Five baths were made between pH 2 and 10 and reference measurements were made beforehand using a standard commercial dual-junction glass pH meter and electrode.

B. *Test Procedure*

The test procedure followed two parallel stages: first, the OCP between an IrOx-coated WE and commercial AgCl RE (*CHI111, CH Instruments, Austin, TX*) was measured, across the 5-point pH buffer panel, via direct data acquisition with probes attached to the PCB. While this measurement was taking place, the modulation frequency was measured at the transmitted inductive signal. Calibration curves were calculated as in [3], and standard residuals (S.R.'s) were calculated as:

$$S.R. = \frac{observed - expected}{\sqrt{expected}} \quad (1)$$

The expected values are interpolations based on the linear regression fit taken for the calibration curve. The standard residuals represent the relative deviation of each data point from the overall dataset's linear fit.

C. *System Output and Discussion*

Shown in ***Fig. 4*** are the results of testing. ***Fig. 4(a)*** features a portion of the raw OCP data being measured, while ***4(b)*** and ***4(c)*** are calculated values. Specifically, ***4(b)*** contains the calibrations and linear fit for both the potentiometric and modulation-frequency-based outputs; modulation frequencies are within manufacturer specification for an N_{DIV} value of 32. Probe potentiometric sensitivities and modulation calibrations featured highly similar Pearson coefficients, indicating that insignificant distortion of the probe signal was caused by format conversion. In ***Fig. 4(b)***, S.R.'s is used to explore the linearity of both measurements. The OCP measurements show bias in the basic pH region, which is to be expected to an extent with IrOx coatings [8]. Modulated frequencies also feature linear bias as the baths trend towards basic pH values, though the cause is to be determined. It is possible that the system noise injected into the VCO's SET pin introduced uncertainty into the frequency measurement, or that measurement uncertainty may come from noise-induced phase instability.

IV. CONCLUSIONS

We have demonstrated a simple system for load modulation-based communication of a potentiometric pH signal, as measured by a compact device powered via resonant inductive coupling. As this device features the applicable form factor and size to be located inside a sample tube cap, it can be easily expanded into a system which greatly expands the measurement capabilities of wet-lab researchers and a multitude of samples to be measured. Future work include miniaturization of the entire system and development of a buffer/envelope detector paired with gain which can allow for automated measurement. We expect that this wireless, continuous chemical measurement system can be implemented across a wide range of laboratories doing biological, biochemical and ecological investigations.

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