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Chronoamperometry Analysis of Sweat Sensor Technology

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Chronoamperometry Analysis of Sweat Sensor Technology

Evan Fritz

Department of Chemical and Biomolecular Engineering

Honors Research Project

Submitted to

The Honors College


Approved:

 Date 4/24/19
Honors Project Sponsor (signed)

Dr. Chelsea Monty-Bromer
Honors Project Sponsor (printed)

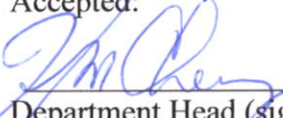
 Date 4/24/19
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Dr. Chelsea Monty-Bromer
Reader (printed)

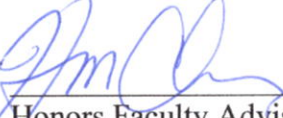
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Executive Summary

The main motivation of the research performed in Dr. Chelsea Monty-Bromer's group is to create a product for athletes, especially runners, marathoners, and triathlon participants, giving them the ability to monitor their hydration levels and know when they are becoming dehydrated or overheated. The vision for the technology is for it to eventually connect to a smart watch via Bluetooth to provide real-time feedback to the athlete. The end goal of this project is to create a functional product that can be sold on the market to address the desire for athletic monitoring for overall athlete wellness and performance. In the future, this product can be further utilized in various biomedical applications to assist cystic fibrosis patients and many others in monitoring daily health and bodily function.

The sensors were fabricated using small electrospun nylon-6 polymer mats that were then dip-coated in a solution of multi-walled carbon nanotubes and TritonX-100 to increase conductivity. The nanotubes on the sensor mat were functionalized using cyclo-oligomeric calixarene dissolved in toluene, allowing for the sensor to determine sodium ion concentrations. The fully functional sensors viability is verified using chronoamperometry experiments. These experiments involve exposing the sensor to an electric current and various solutions of differing sodium ion concentration while maintain constant voltage to measure change in current.

For the chronoamperometry tests, the expected result of an ideal sensor is a linear relationship between sodium ion concentration and change in current. Viable sensors are those that perform with an R-squared value of greater than or equal to 0.80 during linear regression analysis. Various sensors were produced to determine reproducibility of viable sensors and verify the chosen ideal sensor parameters.

One aspect of the research was to determine the ideal sensor parameters for the final prototype design. Such parameters were CNT variety, concentration, and dip coat time. Solutions of 0.25, 0.5, and 1 mg/mL concentrations of a certain CNT with 0.3 wt.% TritonX-100 surfactant were tested. The CNT varieties were CNT, CNT with an oxide function group (CNTOH), and CNT with a carboxylic acid functional group (CNTCOOH). Finally, two different dip coat times were analyzed, 1 minute and 2 minutes in both CNT/TritonX-100 solution and DI water each.

Chronoamperometry analysis along with various surface morphology testing yielded results that concluded that CNT was preferred carbon nanotube variety moving forward as well as 0.25 and 0.5 mg/mL solutions of carbon nanotube to TritonX-100 solution further created the best performance in sensors. Surface morphology experiments were completed by others within the research group, and as such were not within the scope of this research paper. This paper focuses primarily on the chronoamperometry testing and results, though decisions and discussion presented throughout reflect the sum of all research group experiments and results. It was also later determined that a 2-minute dip coat of the nylon fiber mat in carbon nanotube/TritonX-100 solution method further increased consistent sensor viability and performance. Reproducibility experiments determined that out of 68 function sensors of known parameters (nanotube, concentration, nylon coat weight, dip coat time), 44 sensors performed well in either the high sodium ion solution (40 mM – 150 mM) or the lower sodium ion solution (10 mM – 60 mM) chronoamperometry experiments. This shows that approximately 64.7% of the sensors produced had an R-squared value greater than 0.8 in chronoamperometry experiments. To further develop this trend and prove its consistency, more sensors should be produced under the same conditions and tested to determine viability. Further work into reproducibility would greatly reduce wasted resources and time spent on producing nonfunctional sensors.

Future work should be devoted to standardizing as much of the sensor production and chronoamperometry testing as possible. Being able to verify which parts of the production process that are creating the most error and variability in the sensor chronoamperometry results could help increase the reproduction of viable sensors and reduce the loss of time and resources. Other work could be done to further optimize the sensor parameters to produce even more efficient and consistent sensors. Future research could also be performed on making nylon with different coat weights to determine the effects of different fiber diameters in sensor performance and response.

This research has allowed me to utilize much of the chemistry, statistics, and experimental design knowledge that has been touched on in my coursework. I gained more experience in the field of prototyping and the creation of a new products through the channels of research and development. This project has offered me many great hands-on experiences through the making and testing of prototypes to really apply the knowledge and skills that I have honed through my coursework. I also learned much about electrochemistry, performing experiments through chronoamperometry, and laboratory experimental design.

Introduction

Dr. Monty-Bromer's research group has developed various biosensors for the detection of sodium ion concentration found in human sweat. For this project, chronoamperometry experiments were conducted as one of the characterization methods to determine the concentration of sodium ions in the solution. The goal of this report is to detail the variables and methodology used to determine the ideal sensor parameters and the work towards consistently producing functional sensor prototypes with great reliability.

The overall goal of this research is to create highly reliable sensor prototypes to help endurance athletes and marathon and ultra-marathon runners to monitor their hydration levels and to increase performance during training races. The sensor has the capability of detecting in real-time the sodium ion concentration found in an athlete's sweat, which can be used to determine hydration levels during intense training [1]. This information can be incredibly helpful to coaches and trainers who will be able to monitor this information to keep athletes safe and optimally hydrated, especially in more dangerous training scenarios such as intense heat or long duration workouts. These sensors can also help the solo athlete, marathon runner, etc. as the information can be directly fed into a smartwatch, cellphone, or equivalent technology to monitor such key aspects of health and exercise. The prototype would be worn in either the athlete's sock/shoe, wristband, or headband. The sensor would then gather the desired information and feed it into a monitoring system as discussed prior. Ultimately, the sensor will help to alleviate the dangers and symptoms of heat exhaustion and heat stroke through advanced monitoring and detection and help athletes to prevent other potential issues of dehydration such as cramping or nausea. As there are currently no in-situ electrolyte sensors currently on the

market, development of the prototype into a functional product will create a great business opportunity [2].

Background

The research of these sensors has led to many refinements for the creation of functional prototype that can be used in real-time experiments. The main components of the sensor include a nylon-6 base, multiwalled carbon nanotubes (MWCNT), and cyclo-oligomeric calixarene to functionalize the surface of the sensor for selective detection [3]. Upon further research, carbon nanotubes (CNTs) were added into the nylon fiber for further testing to increase conductivity [4]. The nylon-6 base mats were created through electrospinning to generate various thin fibers that create an excellent medium for the addition of the carbon nanotubes. For the experiments to determine ideal sensor parameters, 14%, 17%, and 20% nylon-6 were tested for sensor functionality. Furthermore, three different types of carbon nanotubes including MWCNT, MWCNT with an oxide functional group (MWCNT-OH), and MWCNT with a carboxylic acid functional group (MWCNT-COOH) were used while also varying their concentrations found in solution. Due to the goal of this research being product development of a functional prototype, reproducibility and the creation of many ideal sensors became the focus after ideal experimental conditions were determined.

The bulk of the experimental testing detailed in this report is electrochemical analysis through chronoamperometry. These experiments were performed on a Potentiostat/Galvanostat (Model CHI600D Electrochemical Analyzer, Interface 1000 Gamry Potentiostat) via direct-current (DC) amperometry with an applied voltage from the potentiostat of 2 V against the open-circuit potential (VOC) of the sensor and measuring resulting current. After the addition of sodium ions to the sensor, the ions will bind with the calixarene found on the functionalized

carbon nanotube/nylon-6 base which will then close the circuit that is created bringing forth an electric current across the sensor [3]. The monitoring instrument will then detect the magnitude of current leading to the determination of sodium ion concentration. **Fig. 1** shows the setup used to analyze each sensor and the instrument used in these experiments.



Figure 1: Pictured on top is the setup used for analyzing each sensor by attaching it the electrodes and dropping solutions of varying sodium ion concentration.

Experimental Methods

Sensor Fabrication

The nylon-6 fiber mats were created through an electrospinning process. The electrospinning process used throughout the experiments was not the focus of this report but is greatly detailed in other sources [5]. The mats are cut into 1 cm by 1 cm squares to create the base for each sensor sample. Solutions of 0.25, 0.5, and 1 mg/mL of a certain CNT variety were created with 0.3 wt.% TritonX-100 surfactant solution in an accommodating vial. The CNT varieties as stated before are CNT, CNT with an oxide function group (CNTOH), and CNT with a carboxylic acid functional group (CNTCOOH). This solution of TritonX-100 and CNT variety are then sonicated for 1 hour to allow for dispersion of CNTs into the solution. This entire process is detailed in **Fig. 2**.

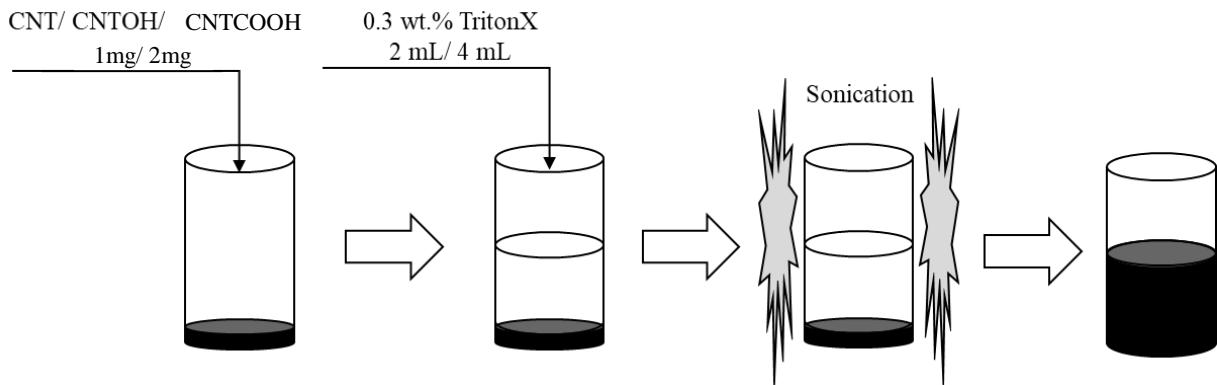


Figure 2: Detailed procedure to produce CNT variety/TritonX-100 surfactant solutions. It contains weights and volumes that produce the different compositions (0.25, 0.5, and 1 mg/mL) of solutions used in the sensor production.

Immediately after the solution is produced, a dip coating procedure is implemented to allow the CNTs to adhere to the surface of the nylon-6 base square. To follow this procedure, the nylon-6 square mentioned prior is placed into the CNT variety/TritonX-100 solution for approximately 1 minute. The square is then placed immediately into DI water for approximately 1 minute to rinse the excess CNTs. The dip-coating time was doubled for some of the samples in

the reproducibility experiments performed after ideal sensor parameters were determined. The samples are then placed in a covered petri dish to allow for a safe environment for overnight drying. This procedure is further illustrated in **Fig. 3**.

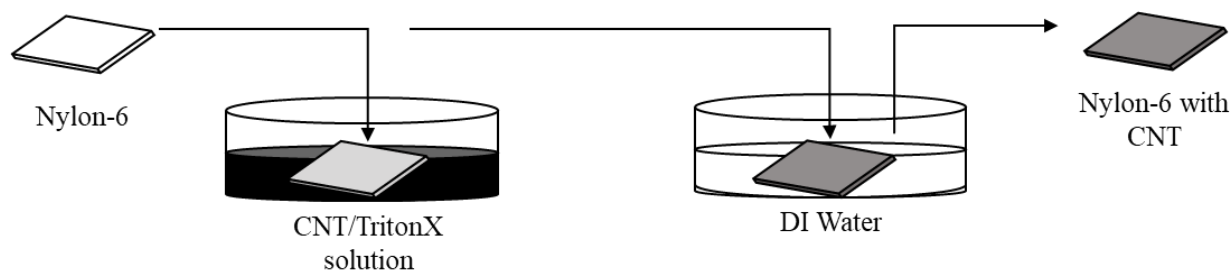


Figure 3: Procedure detailing dip coating method for samples to attach CNTs from solution to nylon-6 square.

The next day, another solution was prepared with cyclo-oligomeric calixarene and toluene which was then mixed for approximately 15 minutes to completely dissolve the calixarene in the toluene. The samples from the other day were then placed directly into the solution and allowed to sonicate for approximately 5 minutes. The samples in solution were left overnight for the reaction of nylon and CNT. This procedure is detailed in **Fig. 4**. The samples are then removed the very next day to dry at normal atmospheric conditions.

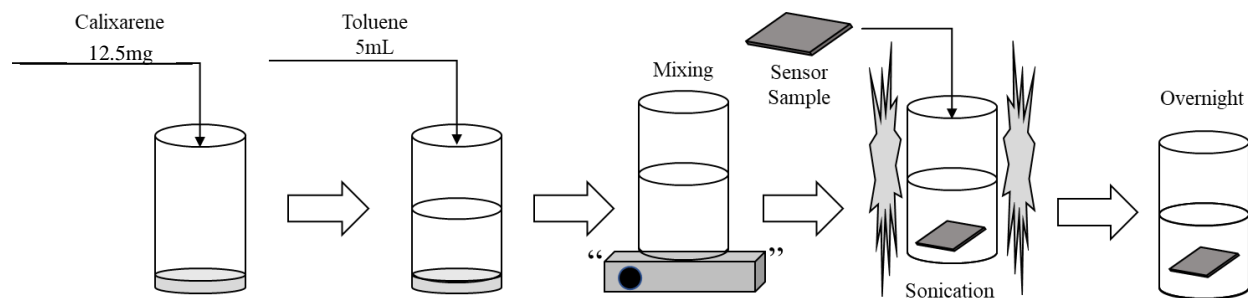


Figure 4: Detailed procedure for the creation of calixarene/toluene solution for overnight sample reaction.

A detailed list of all samples created to determine ideal sensor parameters can be found in **Table 1**. A detailed list of sensors created for the reproducibility experiments categorized by number can be found in **Table 2**.

Table 1: Detailed list of all sample created for the experiments performed to determine the ideal sensor parameters for future experiments.

% Nylon	MWCNT	Concentration (g/L)
14	CNT	0.25
14	CNT	0.5
14	CNT	1
14	CNTOH	0.25
14	CNTOH	0.5
14	CNTOH	1
14	CNTCOOH	0.25
14	CNTCOOH	0.5
14	CNTCOOH	1
17	CNT	0.25
17	CNT	0.5
17	CNT	1
17	CNTOH	0.25
17	CNTOH	0.5
17	CNTOH	1
17	CNTCOOH	0.25
17	CNTCOOH	0.5
17	CNTCOOH	1
20	CNT	0.25
20	CNT	0.5
20	CNT	1
20	CNTOH	0.25
20	CNTOH	0.5
20	CNTOH	1
20	CNTCOOH	0.25
20	CNTCOOH	0.5
20	CNTCOOH	1

Chronoamperometry

The chronoamperometry experiments involved hooking up the sensor to the electrodes on the instrument as discussed prior in the background section of this report. One is the working electrode and the other is the counter electrode combined with reference electrode. These electrodes were hooked up by placing the sensor on a glass slide and then clamping in into place with two micro-alligator clips [Nickel-plated steel] on each side of the sensor with the electrodes

then attached to these clips. A couple of drops of Milli-Q water were dropped onto the sensor to wet it. The instrument was then turned on to allow for an electric current to be passed through the sensor, monitoring the magnitude of said current. Several solutions of sodium chloride and Milli-Q water were prepared prior to act as sample sweat and provided the sodium ions necessary for the experiment. These solutions were prepared for a low sodium ion level of 10, 20, 30, 40, 50, and 60 mM and a higher sodium ion level of 40, 50, 80, 100, 125, and 150 mM used mostly for the reproducibility experiments. First, a 30 μ L drop of the lowest sodium ion concentration from a set was dropped on the sensor. After a period of time to allow the instrument to register the magnitude of the current, the next highest concentration was dropped on the sensor with this process repeating until the highest concentration has been dropped on the sensor. Every time a drop is placed on the sensor, the potential of the electrode changes, which alters the magnitude of the current as it is being recorded by the monitoring instrument as a function of time. The magnitude of current is observed for each drop and the differential change in current for each solution concentration is recorded to determine its effectiveness. This procedure is further illustrated in **Fig. 5**.

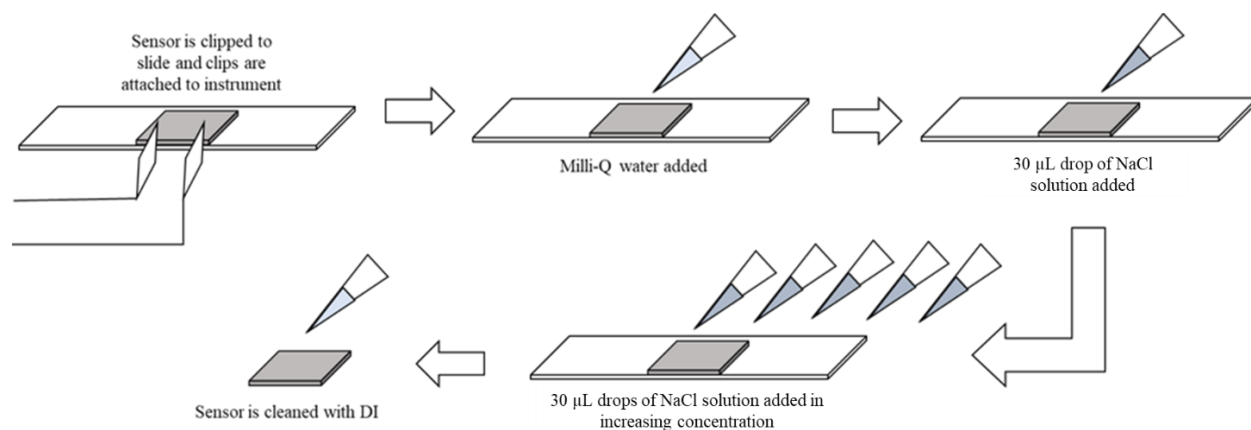


Figure 5: Detailed procedure for chronoamperometry experiments on fabricated sensors.

Data and Results

Chronoamperometry

Using the procedure detailed prior in the experimental methods portion of this report for chronoamperometry, various graphs were produced looking at sodium ion concentration of the solution versus the change in current observed by the instrument. Change in current was determined by taking the derivative of the drop in current recorded by the instrument and then recording the magnitude of change at the time the drop was observed. Examples of these graphs can be seen below in **Figs. 6** and **7**. A good sample would produce a significant linear trend ($R^2 \geq 0.80$) with a negative slope. Many times, one of the data points in a series would be omitted as an outlier to better analyze the linearity of the results as can be seen in **Fig. 7**. These two graphs depict one for each of the two different sets of sodium ion concentration solutions use (OS for the lower concentration set and NS for the higher concentration set) that were used to analyze the concentration range of the sensor.

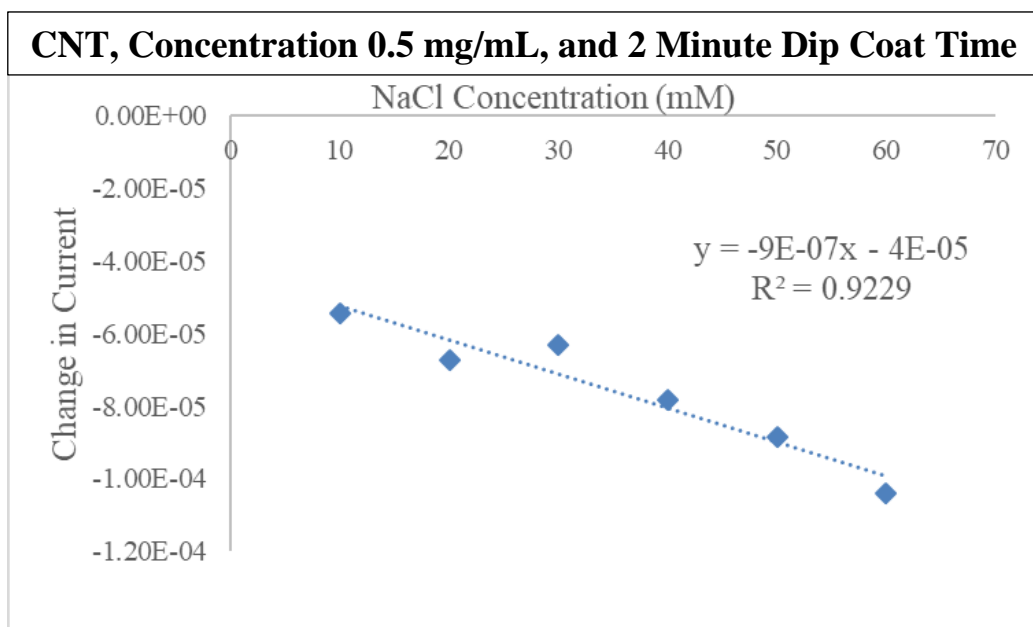


Figure 6: Graph depicting change in current versus sodium ion concentration for the chronoamperometry test on a given using the lower sodium ion concentration solution set (10 - 60mM).

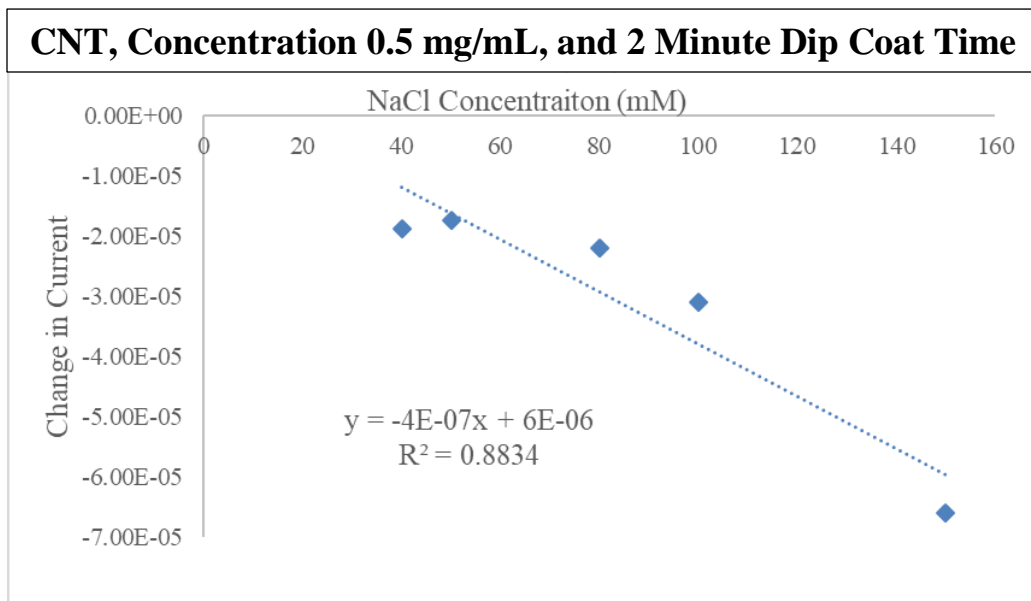


Figure 7: Graph depicting change in current versus sodium ion concentration for the chronoamperometry test on the same sensor as **Figure 6** using instead the higher sodium ion concentration solution set (40 - 150mM).

Reproducibility

After determination of the ideal sensor parameters, various sensors of these types were created to determine how reproducible consistently good sensors were. The totality of these experiments can be seen below in **Table 2**. Each of these sensors were recorded according to sensor identification number, coat weight of the nylon-6, concentration of nanotube (CNT) to TritonX-100 in mg/mL, time that the nylon-6 mats were coated in the nanotube solution, date of the chronoamperometry test, name of the test according to number and sodium ion solution set, and R^2 value from the resulting chronoamperometry test and following analysis as discussed prior. Gray boxes in the R^2 column indicate a sensor of $R^2 \geq 0.80$ representing a good sensor and all these sensors can viewed in **Table 3**. Dark gray coloring and white lettering of the test name indicates a sensor with a slope that has the opposite magnitude of what is desired. Dark gray coloring and white lettering in the sensor number column indicates a sensor that has a performance of $R^2 \geq 0.80$ on a lower sodium ion concentration set and a higher sodium ion

concentration set and all these sensors can be viewed in **Table 4**. Light gray coloring in the sensor number column indicates a sensor that has performance of $R^2 \geq 0.80$ on either the low sodium ion concentration set or the high sodium ion concentration set.

Table 2:Table detailing all the experiments of the sensors created for the reproducibility analysis.

Sensor #	Coat Weight	Nanotube Concentration	Time Soaked (Sec)	Day Chrono Tested	Test Name	R2	Day Chrono Tested	Test Name	R2	Day Chrono Tested	Test Name	R2
20	?	?	?		20	0.8483						
26	?	?	?		26	0.7338						
27	?	?	?		27RR(OS)	0.915	10/1/2018	27RR	0.5886	?	27	0.5354
31	?	?	?	9/18/2018	31	0.4038	10/1/2018	31R	0.206			
32	?	?	?	9/18/2018	32	0.621						
33	?	?	?	9/18/2018	33R	0.7232						
34	?	?	?	10/1/2018	34RRR (OS)	0.684	10/1/2018	34RRR	0	9/18/2018	34RR	0.6041
35	?	?	?	10/1/2018	35RRR (OS)	0.5099	10/1/2018	35RRR	0.8907	9/18/2018	35RR	0.2522
36		0.25	60	11/6/2018	36 (OS)	0.3756	11/6/2018	36(NS)	0.3421			
37	26	0.25	60	11/6/2018	37 (OS)	0.5809	11/6/2018	37 (NS)	0.6325			
38	26	0.25	60	11/13/2018	38 (OS)	0.879	11/13/2018	38 (NS)	0.822			
39	26	0.25	60	11/13/2018	39 (OS)	0.928	11/13/2018	39 (NS)	0.959			
40	26	0.25	60	11/13/2018	40 (OS)	0.884	11/13/2018	40 (NS)	0.968			
41	26	0.25	60	11/13/2018	41 (OS)	0.984	11/13/2018	41 (NS)	0.814			
42	26	0.25	60	11/13/2018	42 (OS)	0.829	11/13/2018	42 (NS)	0.418			
43	26	0.25	60	11/13/2018	43 (OS)	0.932	11/13/2018	43 (NS)	0.769			
44	26	0.25	60	11/13/2018	44 (OS)	0.681	11/13/2018	44 (NS)	0.889			
45	26	0.25	60	11/13/2018	45 (OS)	0.807	11/13/2018	45 (NS)	0.454			
46	26	0.25	60	11/13/2018	46 (OS)	0.635	11/13/2018	46 (NS)	0.213			
47	26	0.25	60	11/27/2018	47 (OS)	0.894	11/27/2018	47 (NS)	0.79			
48	26	0.25	60	11/27/2018	48 (OS)	0.505	11/27/2018	48 (NS)	0.538			
49	26	0.25	60	11/27/2018	49 (OS)	0.733	11/27/2018	49 (NS)	0.116			
50	26	0.25	60	11/27/2018	50 (OS)	0.892	11/27/2018	50 (NS)	0.879			
51	26	0.25	60	11/27/2018	51 (OS)	0.607	11/27/2018	51 (NS)	0.941			
52												
53	25.3	0.5	60	10/15/2018	53(OS)	0.0608	10/19/2018	53R(OS)	0.615	10/23/2018	53 (NS)	0.0056
54	25.3	0.5	60	10/15/2018	54(OS)	0.0369	10/19/2018	54R(OS)	0.81	10/23/2018	54 (NS)	0.4285
55	25.3	0.5	60	10/15/2018	55(OS)	0.0383	10/19/2018	55R(OS)	0.365	10/23/2018	55 (NS)	0.5538
56	25.3	0.5	120	10/15/2018	56(OS)	0.838	10/19/2018	56R(OS)	0.899	10/23/2018	56 (NS)	0.8741
57	25.3	0.5	120	10/15/2018	57(OS)	0.0184	10/19/2018	57R(OS)	0.177	10/23/2018	57 (NS)	0.2016
58	25.3	0.5	120	10/15/2018	58(OS)	0.0007	10/19/2018	58R(OS)	0.478	10/23/2018	58 (NS)	0.009
59	25.3	0.25	90	10/15/2018	59(OS)	0.3573	10/19/2018	59R(OS)	0.518	10/23/2018	59 (NS)	0.2983
60	25.3	0.25	90	10/15/2018	60(OS)	0.0602	10/19/2018	60R(OS)	0.636	10/23/2018	60 (NS)	0.2271
61	25	0.5	120	10/29/2018	61 (OS)	0.8263	10/29/2018	61 (NS)	0.9844			
62	25	0.5	120	10/29/2018	62 (OS)	0.9271	10/29/2018	62 (NS)	0.1185			
63	25	0.5	120	10/29/2018	63 (OS)	0.912	10/29/2018	63 (NS)	0.891			
64	25	0.5	120	10/29/2018	64 (OS)	0.8613	10/29/2018	64 (NS)	0.1708			
65	25	0.5	120	10/29/2018	65 (OS)	0.8949	10/29/2018	65 (NS)	0.6513			
66	25	0.5	120	10/29/2018	66 (OS)	0.8292	10/29/2018	66 (NS)	0.8816			
67	25	0.25	60	10/29/2018	67 (OS)	0.6053	10/29/2018	67 (NS)	0.7142			
68	25	0.25	60	10/29/2018	68 (OS)	0.8528	10/29/2018	68 (NS)	0.8376			
69	25	0.25	60	10/29/2018	69 (OS)	0.6195	10/29/2018	69 (NS)	0.4596			
70	25	0.25	60	10/29/2018	70 (OS)	0.9204	10/29/2018	70 (NS)	0.5214			
71	25	0.25	60	10/29/2018	71 (OS)	0.9869	10/29/2018	71 (NS)	0.6304			
72	25	0.25	60	10/29/2018	72 (OS)	0.607	10/29/2018	72 (NS)	0.6038			
73	25	0.25	60	11/20/2018	73 (OS)	0.784759	11/20/2018	73 (NS)	0.693555			
74	25	0.25	60	11/20/2018	74 (OS)	0.891333	11/20/2018	74 (NS)	0.852886			
75	25	0.25	60	11/20/2018	75 (OS)	0.941429	11/20/2018	75 (NS)	0.649738			
76	25	0.25	60	11/20/2018	76 (OS)	0.87656	11/20/2018	76 (NS)	0.917705			
77	25	0.25	60	11/20/2018	77 (OS)	0.879215	11/20/2018	77 (NS)	0.897092			
78	25	0.25	60	11/20/2018	78 (OS)	0.21582	11/20/2018	78 (NS)	0.281177			
79	25	0.5	120	11/20/2018	79 (OS)	0.772044	11/20/2018	79 (NS)	0.64372			
80	25	0.5	120	11/20/2018	80 (OS)	0.822868	11/20/2018	80 (NS)	0.311897			
81	25	0.5	120	11/20/2018	81 (OS)	0.857329	11/20/2018	81 (NS)	0.012764			
82	25	0.5	120	11/20/2018	82 (OS)	0.803307	11/20/2018	82 (NS)	0.905468			
83	25	0.5	120	11/20/2018	83 (OS)	0.98407	11/20/2018	83 (NS)	0.911608			
84	25	0.5	120	11/20/2018	84 (OS)	0.922866	11/20/2018	84 (NS)	0.88341	11/20/2018	84 (NS) R	0.20105
85	25	0.5	120	12/4/2018	85 (OS)	0.761	12/4/2018	85 (NS)	0.251			
86	25	0.5	120	12/4/2018	86 (OS)	0.695	12/4/2018	86 (NS)	0.55			
87	25	0.5	120	12/4/2018	87 (OS)	0.978	12/5/2018	87 (NS)	0.737			
88	25	0.5	120	12/4/2018	88 (OS)	0.894	12/6/2018	88 (NS)	0.019			
89	25	0.5	120	12/4/2018	89 (OS)	0.839	12/7/2018	89 (NS)	0.803			
90	25	0.5	120	12/4/2018	90 (OS)	0.962	12/8/2018	90 (NS)	0.55			
91	25	0.5	120	12/4/2018	91 (OS)	0.992	12/9/2018	91 (NS)	0.605			
92	25	0.5	120	12/4/2018	92 (OS)	0.957	12/10/2018	92 (NS)	0.406			
93	25	0.5	120	12/4/2018	93 (OS)	0.927	12/11/2018	93 (NS)	0.851			
94	25	0.5	120	12/4/2018	94 (OS)	0.91	12/12/2018	94 (NS)	0.736			
95	25	0.5	120	12/4/2018	95 (OS)	0.911	12/13/2018	95 (NS)	0.824			
96	25	0.5	120	12/4/2018	96 (OS)	0.934	12/14/2018	96 (NS)	0.459			

Table 3: Table detailing the experiments of the sensors created for the reproducibility analysis that passed at least one chronoamperometry test with $R^2 \geq 0.80$.

Sensor #	Coat Weight	Nanotube Concentration	Time Soaked (Sec)	Day Chrono Tested	Test Name	R2	Day Chrono Tested	Test Name	R2
20	?	?	?		20	0.8483			
27	?	?	?		27RR(OS)	0.915	10/1/2018	27RR	0.5886
35	?	?	?	1/0/1900	35RRR (OS)	0.5099	1/0/1900	35RRR	0.8907
38	26	0.25	60	11/13/2018	38 (OS)	0.879	11/13/2018	38 (NS)	0.822
39	26	0.25	60	11/13/2018	39 (OS)	0.928	11/13/2018	39 (NS)	0.959
40	26	0.25	60	11/13/2018	40 (OS)	0.884	11/13/2018	40 (NS)	0.968
41	26	0.25	60	11/13/2018	41 (OS)	0.984	11/13/2018	41 (NS)	0.814
42	26	0.25	60	11/13/2018	42 (OS)	0.829	11/13/2018	42 (NS)	0.418
43	26	0.25	60	11/13/2018	43 (OS)	0.932	11/13/2018	43 (NS)	0.769
44	26	0.25	60	11/13/2018	44 (OS)	0.681	11/13/2018	44 (NS)	0.889
45	26	0.25	60	11/13/2018	45 (OS)	0.807	11/13/2018	45 (NS)	0.454
47	26	0.25	60	11/27/2018	47 (OS)	0.894	11/27/2018	47 (NS)	0.79
50	26	0.25	60	11/27/2018	50 (OS)	0.892	11/27/2018	50 (NS)	0.879
51	26	0.25	60	11/27/2018	51 (OS)	0.607	11/27/2018	51 (NS)	0.941
54	25.3	0.5	60	10/15/2018	54(OS)	0.0369	10/19/2018	54R(OS)	0.81
56	25.3	0.5	120	10/15/2018	56(OS)	0.838	10/19/2018	56R(OS)	0.899
61	25	0.5	120	10/29/2018	61 (OS)	0.8263	10/29/2018	61 (NS)	0.9844
62	25	0.5	120	10/29/2018	62 (OS)	0.9271	10/29/2018	62 (NS)	0.1185
63	25	0.5	120	10/29/2018	63 (OS)	0.912	10/29/2018	63 (NS)	0.891
64	25	0.5	120	10/29/2018	64 (OS)	0.8613	10/29/2018	64 (NS)	0.1708
65	25	0.5	120	10/29/2018	65 (OS)	0.8949	10/29/2018	65 (NS)	0.6513
66	25	0.5	120	10/29/2018	66 (OS)	0.8292	10/29/2018	66 (NS)	0.8816
68	25	0.25	60	10/29/2018	68 (OS)	0.8528	10/29/2018	68 (NS)	0.8376
70	25	0.25	60	10/29/2018	70 (OS)	0.9204	10/29/2018	70 (NS)	0.5214
71	25	0.25	60	10/29/2018	71 (OS)	0.9869	10/29/2018	71 (NS)	0.6304
74	25	0.25	60	11/20/2018	74 (OS)	0.891333	11/20/2018	74 (NS)	0.852886
75	25	0.25	60	11/20/2018	75 (OS)	0.941429	11/20/2018	75 (NS)	0.649738
76	25	0.25	60	11/20/2018	76 (OS)	0.87656	11/20/2018	76 (NS)	0.917705
77	25	0.25	60	11/20/2018	77 (OS)	0.879215	11/20/2018	77 (NS)	0.897092
80	25	0.5	120	11/20/2018	80 (OS)	0.822868	11/20/2018	80 (NS)	0.311897
81	25	0.5	120	11/20/2018	81 (OS)	0.857329	11/20/2018	81 (NS)	0.012764
82	25	0.5	120	11/20/2018	82 (OS)	0.803307	11/20/2018	82 (NS)	0.905468
83	25	0.5	120	11/20/2018	83 (OS)	0.98407	11/20/2018	83 (NS)	0.911608
84	25	0.5	120	11/20/2018	84 (OS)	0.922866	11/20/2018	84 (NS)	0.88341
87	25	0.5	120	12/4/2018	87 (OS)	0.978	12/5/2018	87 (NS)	0.737
88	25	0.5	120	12/4/2018	88 (OS)	0.894	12/6/2018	88 (NS)	0.019
89	25	0.5	120	12/4/2018	89 (OS)	0.839	12/7/2018	89 (NS)	0.803
90	25	0.5	120	12/4/2018	90 (OS)	0.962	12/8/2018	90 (NS)	0.55
91	25	0.5	120	12/4/2018	91 (OS)	0.992	12/9/2018	91 (NS)	0.605
92	25	0.5	120	12/4/2018	92 (OS)	0.957	12/10/2018	92 (NS)	0.406
93	25	0.5	120	12/4/2018	93 (OS)	0.927	12/11/2018	93 (NS)	0.851
94	25	0.5	120	12/4/2018	94 (OS)	0.91	12/12/2018	94 (NS)	0.736
95	25	0.5	120	12/4/2018	95 (OS)	0.911	12/13/2018	95 (NS)	0.824
96	25	0.5	120	12/4/2018	96 (OS)	0.934	12/14/2018	96 (NS)	0.459

Table 4: Table detailing the experiments of the sensors created for the reproducibility analysis that passed both chronoamperometry tests with lower and higher sodium ion solution sets with $R^2 \geq 0.80$.

Sensor #	Coat Weight	Nanotube Concentration	Time Soaked (Sec)	Day Chrono Tested	Test Name	R2	Day Chrono Tested	Test Name	R2
38	26	0.25	60	11/13/2018	38 (OS)	0.879	11/13/2018	38 (NS)	0.822
39	26	0.25	60	11/13/2018	39 (OS)	0.928	11/13/2018	39 (NS)	0.959
40	26	0.25	60	11/13/2018	40 (OS)	0.884	11/13/2018	40 (NS)	0.968
41	26	0.25	60	11/13/2018	41 (OS)	0.984	11/13/2018	41 (NS)	0.814
50	26	0.25	60	11/27/2018	50 (OS)	0.892	11/27/2018	50 (NS)	0.879
56	25.3	0.5	120	10/15/2018	56(OS)	0.838	10/19/2018	56R(OS)	0.899
61	25	0.5	120	10/29/2018	61 (OS)	0.8263	10/29/2018	61 (NS)	0.9844
63	25	0.5	120	10/29/2018	63 (OS)	0.912	10/29/2018	63 (NS)	0.891
66	25	0.5	120	10/29/2018	66 (OS)	0.8292	10/29/2018	66 (NS)	0.8816
68	25	0.25	60	10/29/2018	68 (OS)	0.8528	10/29/2018	68 (NS)	0.8376
76	25	0.25	60	11/20/2018	76 (OS)	0.87656	11/20/2018	76 (NS)	0.917705
77	25	0.25	60	11/20/2018	77 (OS)	0.879215	11/20/2018	77 (NS)	0.897092
84	25	0.5	120	11/20/2018	84 (OS)	0.922866	11/20/2018	84 (NS)	0.88341
93	25	0.5	120	12/4/2018	93 (OS)	0.927	12/11/2018	93 (NS)	0.851
95	25	0.5	120	12/4/2018	95 (OS)	0.911	12/13/2018	95 (NS)	0.824

Statistical analysis was performed upon the data set to determine significant statistical difference between the two concentrations created during the reproducibility experiments, 0.25 and 0.5 mg/mL, and between the two chronoamperometry test solution sets, higher sodium ion concentration set and lower sodium ion concentrations set. The t-test performed upon the concentration data can be seen in **Table 5**. The t-test performed upon the chronoamperometry test solution sets can be seen in **Table 6**.

Table 5: Statistical t-test performed on the data set to determine significant difference between 0.25 and 0.5 mg/mL concentration sensors

	<i>Variable 1</i>	<i>Variable 2</i>
Mean	0.691713	0.661131
Variance	0.055219	0.102155
Observations	60	60
Pooled Variance	0.078687	
Hypoth. Mean Diff.	0	
df	118	
t Stat	0.597137	
P(T<=t) one-tail	0.27578	
t Critical one-tail	1.65787	
P(T<=t) two-tail	0.55156	
t Critical two-tail	1.980272	

Table 6: Statistical t-test performed on the data set to determine significant difference between the high sodium ion solution set and the lower sodium ion solution set used in the chronoamperometry tests

	<i>Variable 1</i>	<i>Variable 2</i>
Mean	0.715991	0.62806
Variance	0.081532	0.071394
Observations	66	54
Pooled Variance	0.076979	
Hypoth. Mean Diff.	0	
df	118	
t Stat	1.727175	
P(T<=t) one-tail	0.043376	
t Critical one-tail	1.65787	
P(T<=t) two-tail	0.086753	
t Critical two-tail	1.980272	

Discussion

From this research, it was determined that CNT was the preferred carbon nanotube as it gave the best results for chronoamperometry. Chronoamperometry is one of the most important tests at this point in prototype development as it tests the sensors response in a similar way to the workings of the functional product. Further development yielded that 0.25 and 0.5 mg/mL carbon nanotube to TritonX-100 solution created the best and most consistent results in many of

the chronoamperometry and surface morphology tests that were performed on the sensors. Finally, a 2-minute dip coat procedure for the nylon sample in solution was chosen for the same preferred results. The final decision, after some of the reproducibility experiments and further surface morphology analysis, was that the prototype sensors moving forward would use the 0.5 mg/mL CNT with the 2-minute dip coat method.

The other focus of this research was testing reproducibility of the ideal sensor parameters as the goal of the project is to produce functional and marketable products out of consistent prototypes. According to **Table 2**, a total of 68 sensors of known creation parameters were produced all using either 0.5 or 0.25 mg/mL solutions with 2-minute or 1-minute dip coat. The results from the chronoamperometry experiments performed on these sensors yielded 44 sensors that were functional with either both or one set of sodium ion solutions, a success rate of 64.7%. Furthermore, only 15 sensors performed ideally with both sodium ion solutions sets, a success rate of 22.1%. For the 0.25 mg/mL solution with 1-minute dip coat sensors (28 sensors in total), 18 were functional with either both or one set (higher or lower) of sodium ion solution sets (64.3%) and 8 were functional with both higher and lower sodium ion solution sets (28.6%). For the 0.5 mg/mL solution with 2-minute dip coat sensors (27 sensors in total), 22 were functional with either both or one set (higher or lower) of sodium ion solution sets (81.5%) and 7 were functional with both higher and lower sodium ion solution sets (25.9%). From the statistical analysis, with a t-stat value of 0.597, there appears to be no significant statistical difference between the chronoamperometry data for the sensors created from 0.25 mg/mL solutions and from those created from 0.5 mg/mL solutions. Also, with a t-stat value of 1.727, there appears to be a significant statistical difference between data collected from the lower sodium ion concentration solution set and data collected from the higher sodium ion concentration solution

set. This indicates that the high sodium ion solution set used in the chronoamperometry testing was more selective than the lower sodium ion concentration solution set. From the consistent results for the 0.5 mg/mL, 2-minute dip coat sensors, these sensor parameters were chosen going forward for prototype creation and testing. The result was further verified in later research on fully fabricated prototypes that proved that the 0.5 mg/mL CNT with a 2-minute dip coat showed better results overall.

Future work should be devoted to standardizing as much of the sensor production and chronoamperometry testing as possible. Being able to verify which parts of the production process that are creating the most error and variability in the sensor chronoamperometry results could help increase the reproduction of viable sensors and reduce the loss of time and resources. This approach would aid greatly in the project's next steps in creating function prototypes and eventually a working product as it would reduce the need for quality control and reliance on the chronoamperometry experiment results. Other work could be done to further optimize the sensor parameters to produce even more efficient and consistent sensors. Furthermore, more work must be implemented into scaling up the sensor fabrication process to move forward the business plan. Future research could also be performed on making nylon with different coat weights to determine the effects of different fiber diameters in sensor performance and response.

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