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Comparative Study of Cyclic Voltammetry and Cycle Stability of Electropolymerized Poly (3, 4-ethylenedioxythiophene) Poly (sodium 4-styrenesulfonate) on Screen-Printed Electrodes in Aqueous Media

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Abstract:

Poly (3,4-ethylenedioxythiophene) poly(sodium 4-styrenesulfonate) (PEDOT:PSS) electropolymerized and deposited onto screen-printed carbon electrodes (SPCEs) and screen-printed platinum electrodes (SPPEs) was studied for anodic/cathodic peak current and cycle stability. Cyclic voltammetry (CV) shows that the redox ability of SPCEs electropolymerized with PEDOT:PSS (PEDOT:PSS/SPCEs) was significantly improved in comparison to SPPEs electropolymerized with PEDOT:PSS (PEDOT:PSS/SPPEs; the peak current difference of oxidation and reduction (ΔI) for PEDOT:PSS/SPCEs was ~3 times higher than that of PEDOT:PSS/SPPEs (ΔISPCE = 350 μA, ΔISPPE = 125 μA). Oxidation and reduction peak current of the CVs showed that both electrodes could maintain electrode integrity for over 30 days. The results suggest that the electropolymerized PEDOT:PSS had good adhesion to SPCE and SPPE surfaces. There was an insignificant change in the cycle stability curve after 3000 cycles compare to the initial cycle and an insignificant change in the cycle stability curve after 30 days in comparison to the first day for booth electrodes. The results suggest that electrode integrity of both PEDOT:PSS/SPCEx and PEDOT:PSS/SPPEx was maintained after repetitive CV cycles in aqueous media .

Keywords: *Screen-printed carbon electrodes, screen-printed platinum electrodes, cycle stability, aqueous media, PEDOT:PSS.*

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I. INTRODUCTION

Electrochemical devices include sensors for *in situ* monitoring of pollutant molecules for environmental applications [1]–[3], or energy storage devices such as capacitors and batteries [4]-[6]. Such devices require prolonged storage of electrodes in aqueous media. A stable electrode that can withstand repetitive CV cycles required for prolonged lifetime of electrochemical devices [7]. Repetitive CV cycles in phosphate buffered saline (PBS) were used to characterize the adhesion and stability of the material used for the modification of electrochemical-sensor electrodes in aqueous media [7], [8]. In the case of supercapacitors, a repetitive CV cycle was used to determine performance [5] and galvanostatic

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charge/discharge cycling to determine the capacitance of the coated electrodes [9], [10].

Carbon electrodes in Li-air batteries suffer from side reactions when charging at high voltage [11], [12]. Carbon electrode side reaction was suppressed by coating the electrodes with conducting polymers [13]. Besides batteries, our work shows that the repetitive CV cycle profile of screen-printed platinum electrodes (SPPEs) modified with conductive polymer maintained its stability for 300 cycles and also maintained a repetitive CV over 15 days in PBS, pH 7.1 [8]. These results suggest that coating electrodes with conductive polymers has the potential to enhance the lifetime of devices that operate based on electrochemical principles.

For device miniaturization and scaling-up purposes, screen-printed electrodes (SPEs) have considerable potential [14], [15]. Several materials are used in SPE fabrication: platinum and carbon are common ones. Platinum has efficient electron transfer, high charge/discharge stability in acidic media, and high exchange-current density (4 A/m^2) but is relatively more costly compared to carbon [16], [17]. Therefore, carbon replaces platinum for cost-effectiveness. Affordability of carbon electrode comes with its own limitations; the irregular physical structure of carbon leads to relatively larger electrical and charge-transfer resistance compared to noble metals [18]. Incorporation of a conductive polymer could reduce such resistance.

Deposition of conductive polymers onto electrodes has been extensively applied for transducers in electrochemical sensors owing to the ability to increase the rate of reduction/oxidation process [19], which is useful for electrochemical device fabrication. In addition, conductive polymers do not form an insulating oxide layer like nonnoble metals do, which makes them stable in air. Further, they have molecular porosity when swelled in a solvent, and hence a very high surface area [20]. Commonly used conductive polymers in electrode fabrication are polyaniline (PANI) [21], polypyrrole (PPy) [22], and poly(3,4-ethylenedioxythiophene) (PEDOT) [23]–[25]. Their good environmental stability and electrical conductivity, plus useful mechanical, optical, and electronic properties, make them suitable for various applications besides electrode fabrication [26].

PEDOT has high electrical conductivity (300 S/cm), transparency, and excellent air stability [27]. Incorporation of PEDOT with dopant ions of poly(styrenesulfonate) (PSS) leads to the more stable PEDOT:PSS material [28]. However, the presence of PSS means that PEDOT:PSS is hydrophilic; this can lead to weak adhesion to the electrode surface, which results in film degeneration and peeling from the electrode [7], [29], [30]. For this reason, previous research has focused on enhancing PEDOT:PSS water stability and adhesion to electrodes by adding polyvinyl alcohol (PVA) [29], Nafion [30], or sodium carboxymethyl cellulose as a binding reagent [7]. In addition, our initial work demonstrated that deposition of PEDOT:PSS by electropolymerization can overcome the aforementioned problem [8]. However, few studies have been made on understanding the behavior of electropolymerized PEDOT:PSS on either SPE type after repetitive CV cycles with prolonged storage in aqueous media.

Therefore, in this study, the continuation of our work in [8], we report on comparison of CV performance and cycle stability of the electropolymerized PEDOT:PSS on both screen-printed carbon electrodes (SPCEs) and screen-printed platinum electrodes (SPPEs). Cyclic voltammetry of electropolymerized PEDOT:PSS on both SPCEs and SPPEs was conducted to look for changes in working electrode integrity, especially adhesion of PEDOT:PSS to electrode surfaces as well as repetitive CV cycles of electrodes in PBS, pH 7.1 to test stability. Our results provide an initial insight into the applicability of PEDOT:PSS-deposited electrodes for electrochemical devices that require long CV cycle stability in aqueous media.

II. Materials and Methods

A. Apparatus and Reagents

Screen-printed carbon electrodes and screen-printed platinum electrodes with working electrode (Ø = 2 mm) and a compact voltammetry cell-starter kit were purchased from PINE Research Instruments, Grove City, PA, USA. A PocketSTAT was used to perform electrochemical measurements of CV cycles (IVIUM Technologies, Eindhoven, the Netherlands). Monomer 3,4-ethylenedioxy -thiophene (EDOT) and poly(sodium 4-styrenesulfonate) solution (PSS), lithium perchlorate powder ($LiClO_4$), and potassium ferricyanide ($K_3[Fe(CN)_6]$) were purchased from Sigma-Aldrich, St. Louis, MO, USA. Phosphate

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buffer solution (PBS; pH 7.1) was prepared in the lab. Distilled water was used throughout the experiments. The materials used were adapted from previous work [8] with slight modification.

B. Modification of screen-printed carbon and screen-printed platinum electrodes

SPCEs and SPPEs were modified by electropolymerization deposition (EPD) of EDOT and PSS on the working electrodes (Fig. 1). EPD was performed under galvanostatic mode in an aqueous solution that contained 0.5 ml EDOT, 1 ml PSS, and 13.5 ml $LiClO_4$ (0.1 M). The EDOT/PSS solution was stirred for 24 hr before EPD. Scan rate, number of scans, current, and potential settings were 0.1 mA/s, 10 cycles, 100 µA, and 400 mV, respectively. The electrode modification process was adopted from previous work [8] with slight modification.

C. Electrochemical characterization of carbon and platinum electrodes electropolymerized with PEDOT:PSS

The modified electrodes PEDOT:PSS/SPCE and PEDOT:PSS/SPPE were characterized electrochemically using different CV tests. Figure 2 shows the electrochemical characterization conducted on electropolymerized electrodes. Electron transfer between the electropolymerized working electrodes and solution was analyzed by measuring the magnitude of the anodic/cathodic peak current (ΔI_p) of CV graphs. CVs were conducted in 0.05 M $K_3[Fe(CN)_6]$ with a 100 mV/s scan rate. To test electrode integrity in liquid media, repetitive CV cycles were performed in PBS, pH 7.1. The experiments were conducted under ambient temperature that fluctuated between 25 °C and 27 °C.

D. Statistical analyses

R software was used to test two null hypotheses [31]. The first states that there is no significant change between the initial cycle and the other cycles. The second states that there is no significant change between the cycle of day 1 and the cycles of other days. The statistical testing would be applied within each electrode (i.e., platinum and carbon). To decide whether a parametric or nonparametric approach is more suitable for our data, we investigated the homogeneity of variance using the Fligner-Killen test. If the variances are not significantly different, one-way ANOVA testing was used. Otherwise, the nonparametric Kruskal-Wallis statistical test is chosen.

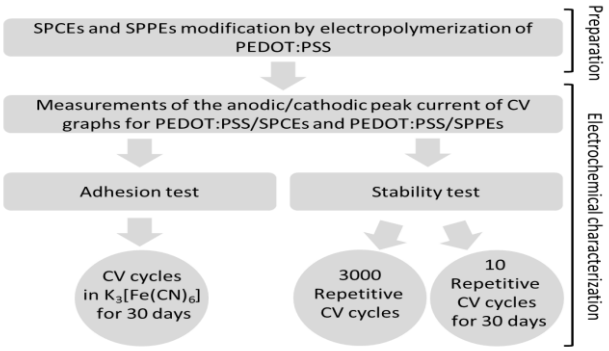

Fig. 2. Diagram showing electrochemical characterization conducted on PEDOT:PSS electropolymerized onto screen-printed carbon and platinum electrodes.

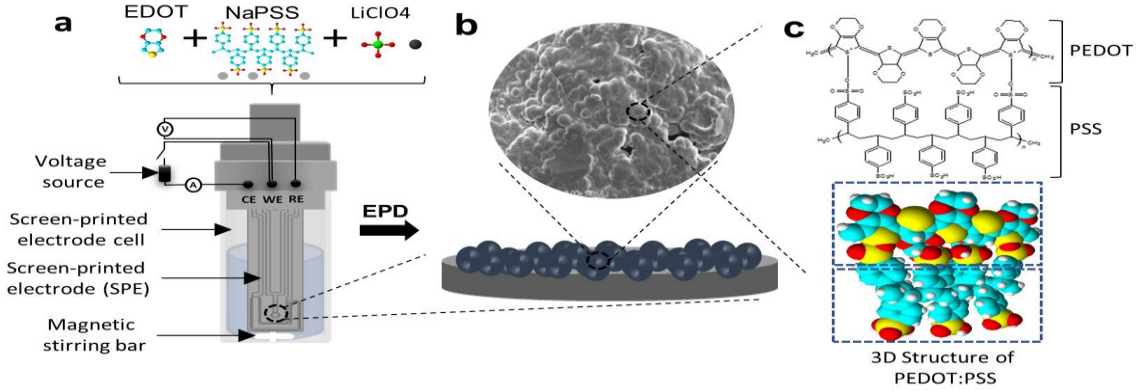

Fig. 1. Modification process of PEDOT:PSS electrodes by electropolymerization deposition (EPD) of EDOT/NaPSS/$LiClO_4$ solution using galvanostatic mode. (a) EPD setup. (b) Schematic diagram and FESEM

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image of PEDOT:PSS electropolymerized onto working electrode. (c) Chemical structure and 3D structure of PEDOT:PSS.

III. Results and Discussion

A. Redox Current of PEDOT:PSS/SPCEs and PEDOT:PSS/SPPEs

The peak current on a CV graph indicates the electron-transfer ability of an electrode; a higher peak current implies that more electrons are shuttled from the solution to the electrode and vice versa. Figure 3 illustrates the CVs of electropolymerized SPCEs and SPPEs in comparison to unmodified or bare ones. Peak current difference (ΔI_p) between the electropolymerized electrodes and the bare electrodes was determined. For both SPCEs and SPPEs, electropolymerized electrodes show increased oxidation and reduction peak current. The ΔI was ~3 times higher for SPCEs than for SPPEs ($\Delta ISPCE = 350\ \mu A$, $\Delta ISPPE = 125\ \mu A$). The results suggest that PEDOT:PSS can be used to enhance electron transfer for both electrodes, but it significantly improved SPCEs, probably as a result of increased surface area owing to carbon's initially having a rougher surface [32].

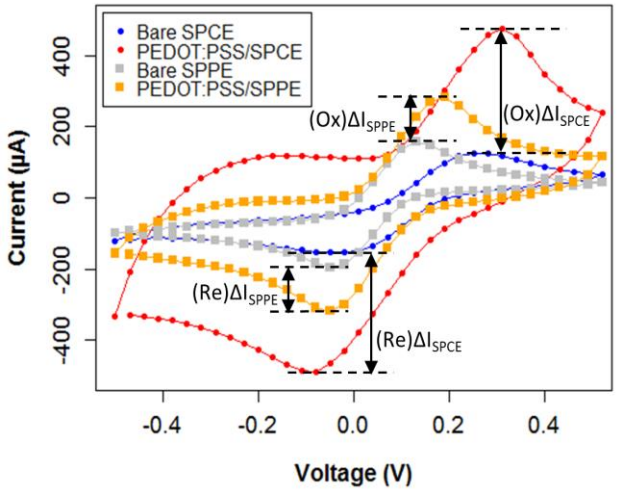

Fig. 1.CVs showing change of peak current for electropolymerized SPCEs and SPPEs, and unmodified ones in 0.05 M $K_3[Fe(CN)_6]$. Scan rate 100 mV/s. (Ox: oxidation, Re: reduction).

B. Adhesion of PEDOT:PSS to SPCEs and SPPEs

Adhesion of electropolymerized PEDOT:PSS to working electrodes of SPCEs and SPPEs is important to determine electrode integrity and therefore lifetime in aqueous media such as water or other electrolytes. To test for lifetime, electropolymerized PEDOT:PSS/SPCEs and PEDOT:PSS/SPPEs underwent repeated CV cycles in 0.05 M $K_3[Fe(CN)_6]$ over 30 days. As can be seen in Figure 4, both electrodes retained their initial oxidation and reduction peak current over 30 days, suggesting that PEDOT:PSS has good adhesion to carbon and platinum working electrodes. And as stated previously, since SPCEs have a higher peak current, thanks to PEDOT:PSS, and are more cost effective, they can be used to replace SPPEs in applications with electrodes involved in repetitive CV cycles in aqueous media.

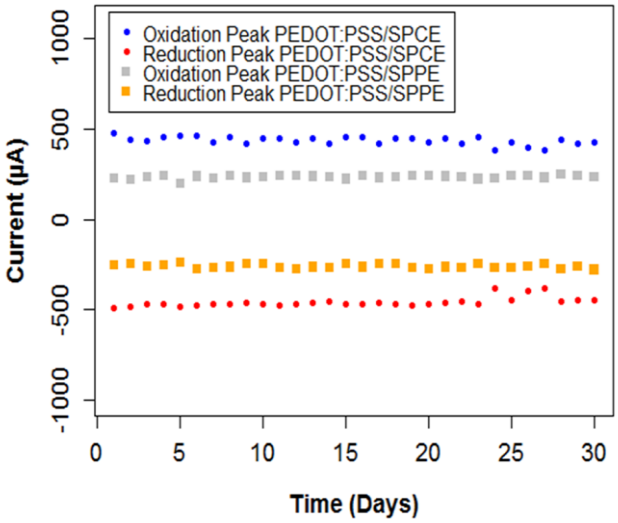

Fig. 2.Oxidation and reduction CV peak current for electropolymerized electrodes in 0.05 M $K_3[Fe(CN)_6]$. Scan rate 100 mV/s with measurements made daily for 30 days.

C. CV Cycle Stability of PEDOT:PSS/SPCEs and PEDOT:PSS/SPPEs in Buffer Solution

Electrode cycle stability means minimal change in CV profiles when repetitive CV measurements are conducted in liquid media over several days. First, repeated CV cycles were conducted up to 3000 cycles in PBS, pH=7.1, as shown in Figures 5 a and b. The first measurement aims to see the effect of repeated cycles on the integrity of both electrodes. Then, similar electrodes underwent 10 CV cycles conducted over times (30 days), as shown in Figures 6 a and b. For 30 days, the electrodes were

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continuously immersed in PBS, pH 7.1. The second measurement aims to see the effect of prolonged electrode storage in PBS, pH 7.1, on CV cycle stability.

As can be seen from Figures 5 a and b, both PEDOT:PSS/SPCEs and PEDOT:PSS/SPPEs have insignificant difference in their repetitive CV cycle profile from initial cycle to 3000 cycle. However, a statistical check on the data suggested that the variance varies significantly for PEDOT:PSS/SPCEs and PEDOT:PSS/SPPEs, with a *p*-value of 2.2×10^{-16} and 2.7×10^{-10}, respectively. Therefore, the Kruskal-Wallis test was applied for data shown in Figures 5 a and b (*p*-values 0.1 and 0.9, respectively). Furthermore, the results imply that both electrodes with electropolymerized PEDOT:PSS have cycle stability in the buffer solution. Although carbon is known to possess irregular features that can limit repetitive charge/discharge owing to high electrical resistance and high charge-transfer resistance [18], electropolymerization of PEDOT:PSS on carbon working electrodes enables the electrodes to improve with respect to both properties, thus enabling the repetitive CV cycles required in electrochemical devices for sensing and energy storage applications. For sensor applications in aqueous media, PEDOT:PSS could be an effective electrochemical transducer owing to its good adhesion to and cycle stability.

To further investigate the cycle stability with prolonged storage of the electropolymerized electrodes in aqueous media, PEDOT:PSS/SPCEs and PEDOT:PSS/SPPEs were soaked in PBS, pH 7.1, and for 30 days the electrodes underwent 10 CV cycles each day. As can be seen from Figures 6 a and b, PEDOT:PSS/SPCEs maintained CV cycle stability for 30 days storage, whereas PEDOT:PSS/SPPEs displayed a slight difference in the current compared to the day-1 cycle. Data analyses showed that the variance varies significantly for both electrodes, with *p*-value = 3.3×10^{-12} for PEDOT:PSS/SPCEs and *p*-value = 5.4×10^{-8} for PEDOT:PSS/SPPEs. The results obtained from the Kruskal-Wallis test applied to both sets of data show that the *p*-values are above 0.05 (*p*-values = 0.9 and 0.1, respectively). The results demonstrate that both electrodes have promising cycle stability in liquid media. For electrochemical devices such as sensors and energy storage devices where electrodes are constantly in aqueous media such as buffer solution or electrolytes, ability to maintain stability over prolonged storage is deemed important for device operation.

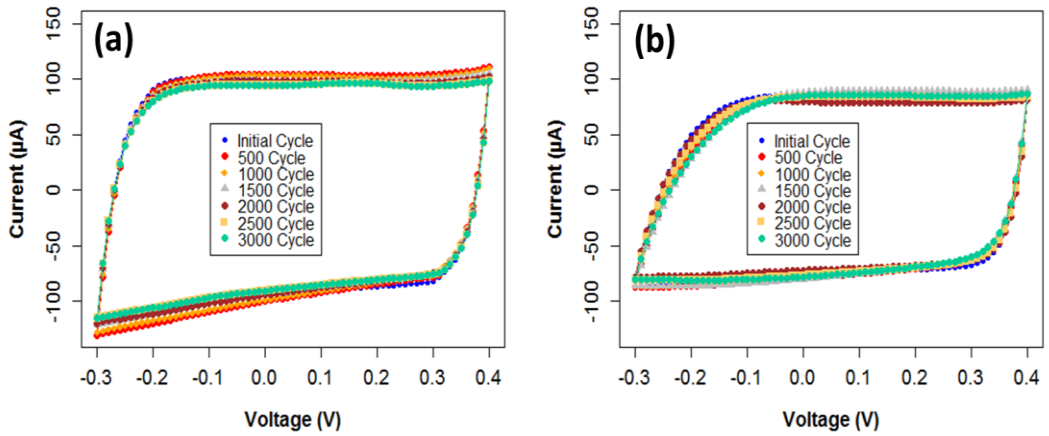

Fig. 3. Cycle stability profile for initial cycle, 500, 1000, 1500, 2000, 2500, and 3000 cycles in PBS pH=7.1. Scan rate 100 mV/s, for (a) PEDOT:PSS/SPCE, (b) PEDOT:PSS/SPPE.

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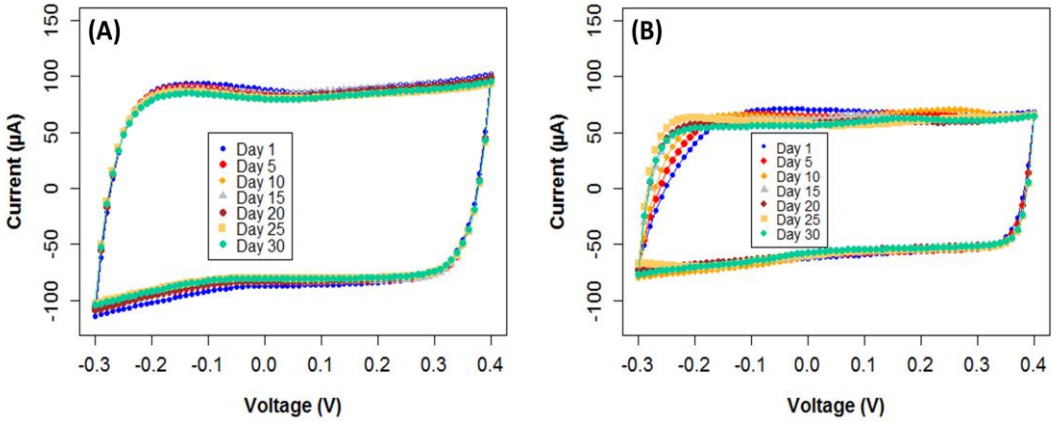

Fig. 4. Cycle stability profile for measurements made at day 1, 5, 10, 15, 20, 25, and 30 for (a) PEDOT:PSS/SPCEs, and (b) PEDOT:PSS/SPPEs in PBS, pH=7.1, at a potential scan rate of 100 mV/s.

IV. CONCLUSIONS

Carbon and platinum screen-printed electrodes electropolymerized with conductive polymer PEDOT:PSS were studied for determination of anodic/cathodic peak current and potential, as well as cycle stability in aqueous solutions. For CV peak current in 0.05 M $K_3[Fe(CN)_6]$, PEDOT:PSS/SPCEs showed peak current three times higher than that of PEDOT:PSS/SPPEs, suggesting that carbon-electrode redox current can be amplified by electropolymerization with PEDOT:PSS. For electrochemical devices to have a long lifetime, cycle stability is a requirement. Both electrode types demonstrate stable CV cycles up to 3000 cycles, suggesting that both are suitable to improve electrodes for electrochemical devices. Furthermore, a test of cycle stability in buffer solution was conducted every day for 30 days, demonstrating that both electrodes maintained their repetitive CV cycle profile without significant deviation from the initial cycle. These results suggest that electropolymerized carbon and platinum electrodes can improve integrity of carbon and platinum SPEs. We intend to apply these electrodes in sensor applications that require prolonged use in aqueous media, such as water-quality monitoring and physiological sensing. In addition, these electrodes can be applied as anodes in microbial fuel cells and energy storage devices.

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Afidalina Tumian, Dr Afidalina is an assistant professor at the Department of Electrical and Computer Engineering. She completed her engineering degree (electronics engineering majoring in computer science) at Multimedia University in 2002. She then continued her M.S.c studies at the University of Surrey for a year before joining the Kulliyyah of Engineering, IIUM, as a lecturer. Two years later, she went to the University of Oxford for her D.Phil. studies under the Life Sciences Interface Doctoral Training Centre programme. Having graduated from the University of Oxford with a D.Phil. in bioinformatics, her research interest is in data analytics and computational statistics, she has experience working with both structured and unstructured data. Her D.Phil. thesis was to understand the evolutionary aspect of recombination processes across mammalian species; she has had some of her work published in *Science*, *Nature*, and *e-Life*. Presently, she is actively engaged in several cross-disciplinary research projects in healthcare, biotechnology, and genetics, while teaching several undergraduate-level courses, including programming courses, probability and statistics, and some mathematics courses. She favors open-source tools like R and python.

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