Screen-printed Military Textiles for Wearable Energy Storage

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ABSTRACT

Textile-based supercapacitors incorporated into military uniforms enable the autonomy of wearable, physiological sensors that can be safer and more comfortable for the Warfighter. Previously, researchers have incorporated supercapacitor electrode components into common textiles such as cotton and polyester, but not in military-relevant textiles that have different fabric characteristics. In order to understand how current uniforms could be transformed into energy storage, a baseline for incorporating aforesaid components onto military textiles is needed. This paper describes how screen printing was used to assess the feasibility of the technique to incorporate electrode ink comprised of activated carbon and an acrylic binder onto military relevant textiles. Sheet resistance was used as a metric to evaluate the quality of screen prints, while electrochemical impedance spectroscopy and cyclic voltammetry were used to investigate the behavior of the most promising screen printed textile electrode using ionic liquid electrolyte (1-ethyl-3-methylimidazolium tetracyanoborate) and graphene foil as current collectors. It was found that the electrode ink favored military textiles that had a tighter weave and were partially composed of nylon. Screen printed spandex woven textiles were found to have the highest conductivity, attaining areal and gravimetric capacitances of 20 mF/cm^2 and 4.21 F/g$_{carbon}$, respectively.

INTRODUCTION

Today's dismounted Warfighter is being equipped with more wearable sensors to increase his/her situational awareness and thus effectiveness towards completing the mission. Environmental and physiological sensing is crucial to enhancing the Warfighter's effectiveness in the field. Some examples of currently fielded wearable sensors (according to the US Army Program Executive Office portfolio, [1]) include (1) the Generation II Helmet Sensor – which gathers data to characterize the effects of blast and head-impact events, and (2) the Remote Physiological Status Monitor – which "measures heart rate, respiration, core body temperature, activity and posture" [2]. Wearable sensor technologies – such as eyewear, smart textiles, tattoos, and 'jewelry' [3] – are evolving rapidly in the commercial and academic sector, and the military must find ways to leverage these advancements to improve the Warfighter's mission.

FIGURE 1. Examples of wearable sensor technologies (figure from Ref [3]).

In order to integrate such technologies into the Warfighter's wearable system, Army scientists recognize that power – in addition to size, weight, and cost – must be considered [4]. Additionally, these power sources must be safer for the Warfighter. The Army conformal battery, for example, "is designed to be worn in front of the Soldier's ballistic plate and must be disposed of if it is punctured" [5]. Wearable power sources based on textile supercapacitors offer a unique solution because "they can be made entirely of non-toxic materials [and] do not ignite if punctured" [6].

Textile-based supercapacitors have been developed by a few academic groups, as outlined in a recent review by Jost et al [7]. Based on preliminary electrochemical data presented, the most probable applications for textile supercapacitors are similar to ones for micro-supercapacitors – i.e, supercapacitors designed to serve as power sources/energy storage

units in low-powered microelectronic devices such as implantable biomedical devices, environmental sensors, and microelectromechanical systems [8]. Recently, a wearable, integrated physiological sensor/real-time wireless transmission system powered by a coin cell battery was demonstrated by Kassal et al [9] to detect uric acid in wounds. The wireless transmission component on which this integrated system is based is a rigid, printed circuit board (PCB) the size of a credit card [10]. Although the wireless transmission component in this system was wearable and portable, the user's ultimate interest for this kind of system will be comfort, a characteristic which the rigid PCB does not provide. Comfort is especially imperative for the Warfighter as he/she becomes increasingly burdened with physical weight during mission performance. Emerging durable fabric circuit boards and stretchable electrical interconnects [11] are enabling the reality of all flexible and wearable systems that can provide more comfort to the user. It is thus becoming evident that comfortable, flexible, and safe power solutions such as textile-based supercapacitors must be concurrently developed in order to provide wearable sensor autonomy.

Previous work on fabricating textile-based supercapacitors involved coating manufactured fabric with active materials ([12], [13]). While these textile-based supercapacitors show great promise for wearable energy storage, addressing the issue of manufacturability, scale-up, and ease of integration into the Warfighter's uniform is imperative. To be manufacturable, the way the textile supercapacitor is made must be amenable to existing industrialized techniques. To be scalable, the industrialized techniques should be ones that can meet the production demands of the military. Lastly, to be easily integrated, the wearable energy storage should be compatible with the Warfighter's existing wearable material. Jost et al. at Drexel University were the first to address the issue of manufacturability and scale-up ([12], [14]) by screen printing activated carbon ink on commonly used textiles such as cotton and polyester-based knit fabrics. Although these fabrics might be worn as casual wear or underwear, existing and developmental military textiles in the Warfighter's uniform are more appropriate substrates to consider for treatment. Krintz and Ervin at the Army Research Laboratory explored depositing graphene onto Kevlar, which is found in military protective armor [15]. However, the Warfighter will most likely be wearing his/her military uniform, which is usually a blend of different fibers.

This paper presents an assessment of the feasibility of screen printing on typical fabrics incorporated in military uniforms using cost-effective materials, as well as recommendations of ways screen-printed military textiles could be improved for wearable textile-based supercapacitor application. Screen printing is a popular technique that uses a mesh screen to transfer ink onto textiles. A squeegee is typically used to move ink across and through the openings of the mesh screen and onto the textile. The hope for this study is to provide insight on the applicability of printing active materials for supercapacitors on manufactured military fabrics before garment assembly.

EXPERIMENTAL

Military Textiles

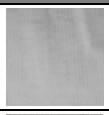
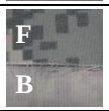
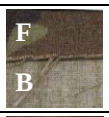
Seven military relevant textiles were selected for screen printing and are listed in *Table I*. Three are currently used in end items worn by the military (i.e., 50/50 NyCo, Spandex, and Defender™ M#14 Stretch). The 50/50 NyCo (50% nylon and 50% cotton) textile was manufactured by ITG Burlington and has a rip stop weave. It is the same type of fabric included in the Army Combat Uniform (ACU). The Spandex textile is a twill weave composed of a 95% nylon/5% elastane mix. This textile is the same type of fabric included in the outer shell fabric of the Army Extended Cold Weather Clothing System. Sigma 4 Star™ and Defender™ M – manufactured by ITG Safety Components and Tencate, respectively – are both textiles that meet the military test specification for the Flame Resistant (FR)-ACU (FR-ACU). They are both textiles that are comprised of nylon, rayon and aramids for flame resistance. The first two fabrics listed in *Table I* – 100% cotton and 100% polyester – are fabrics found commercially and were used as baselines. The last fabric listed in the table – Nonwoven 3NP – is one being considered for other developmental garment end items.

Activated Carbon Ink

Two formulations of carbon ink were synthesized, differing only in activated carbon type. The composition of each ink during initial mixing consisted of 95 wt% activated carbon and 5 wt% Liquitex® Gesso binder. The binder is a proprietary mix of calcium carbonate, acrylic polymer, and titanium dioxide, which agrees with energy dispersive X-ray spectroscopy data collected on a pure binder sample. Water was used as a carrier to facilitate mixing of ink ingredients. The formulation for Ink 1 utilized activated carbon – Maxsorb A – and was used to refine ink synthesis details for screen

printing. *Figure 2A* shows that particle sizes range from being <10μm to as large as 30-40μm. The formulation for Ink 2 utilized an activated carbon sample from Norit® – "Supra 30." The particle size distribution stated by Norit® is as follows – 2-4μm (D10), 5-10μm (D50), and 13-20μm (D90). This also agrees with the SEM (*Figure 2B*) collected on the plain particles. The reported Branauer, Emmett and Teller surface area was 1900 m^2/g.

TABLE I. Characteristics of military textiles used in this work. The front (F) and back (B) of each fabric is indicated in textiles that are not uniform throughout.

	Textile	Weave	Fabric Mass (mg/cm^2)	Fiber composition	Thickness (mm)	Military End Item
	Cotton	Knit	16.8	100% Cotton	0.53	-
	Polyester	Knit	19.5	100% Polyester	0.50	-
	50/50 NyCo	Woven (Ripstop)	21.7	50% Nylon, 50% Cotton	0.38	Army Combat Uniform
	Spandex	Woven	15.2	98.7% Nylon, 1.3% Elastane	0.30	Army Extended Cold Weather Clothing System
	Sigma 4 Star™	Woven	20.5	45% *m*-Aramid, 32% FR-Rayon, 17% Nylon 6% *p*-Aramid	0.35	-
	Defender™ M#14 Stretch	Woven (Twill)	19.3	64% Rayon, 25% *p*-Aramid, 10% Nylon, 1% Urethane	0.40	Flame Resistant – Army Combat Uniform
	Nonwoven 3NP	Nonwoven	15.4	Rayon, *p*-Aramid	0.34	-

The binder was diluted with a small amount of water to ease mixing with carbon. Water was then added until a creamy consistency was achieved.

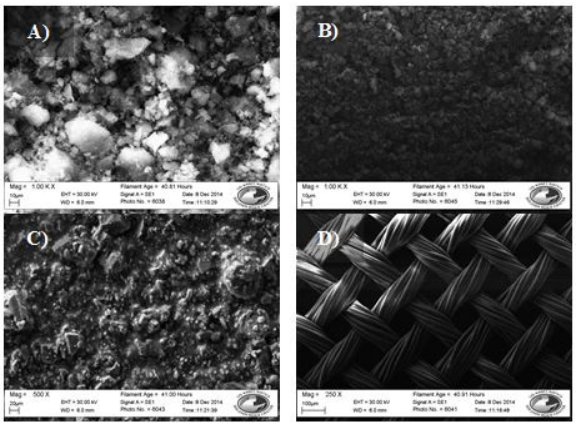


FIGURE 2. SEM images of ink components and screen mesh. (A) Maxsorb A, Ink 1 carbon (B) Norit® Supra 30, Ink 2 carbon. (C) Liquitex® Gesso, binder used both inks. (D) Screen printing mesh used to apply both inks.

Screen Printing

Sample squares (1 cm^2) of each textile were placed on a chemical resistant vinyl mat adhered to a level tabletop, and printed using a Speedball® screen printing kit. The mesh screen in the Speedball® kit was composed of a woven polyester monofilament and secured in a wooden frame to make it taut. It had an effective area of 10" x 14", with about 100µm x 100µm thread spacing (*Figure 2D*). The mesh screen was aligned on top of the textile squares, using C-clamps positioned at the wooden frame and edge of the tabletop to secure the screen during printing. Ink was poured at one of the shorter edges of the screen, and manually pushed through the screen with a 9" wide plastic squeegee. Pushing ink through the screen involved moving the squeegee longitudinally across the screen from one short edge to the opposite edge. A longitudinal movement of the squeegee is considered one screen print pass.

Characterization

Textile samples before and after screen printing were examined under scanning electron microscope (SEM) (Zeiss EVO 60). Screen printed textiles were not sputter-coated. Gravimetric analysis was performed on samples to determine carbon mass deposited by the screen printing process. Samples were weighed before screen printing and after drying for 16 hours in a vacuum oven at 60°C. The amount of carbon deposited on each textile was determined by Eq. (1):

$$m_{\text{carbon}} = \left(m_{\text{screen printed textile}} - m_{\text{unprinted textile}}\right) \times 0.95 \quad (1)$$

Resistivity measurements were taken with a 4-point probe using a Keithley 2400 Sourcemeter at room temperature using alligator clips to make electrical contact.

Supercapacitor Device Assembly and Testing

Figure 3 shows an exploded view of the layered sandwich configuration of the way in which devices were assembled. A few drops of ionic liquid (1-ethyl-3-methylimidazolium tetracyanoborate [EMI TCB]) were deposited onto the surface of each screen-printed textile, as well as on the Whatman filter paper, which acted as the separator. Ionic liquid was used as an electrolyte for its anticipated use as a safer supercapacitor electrolyte, while EMI TCB was chosen because of its relatively high ionic conductivity and wide electrochemical window. Each component soaked in ionic liquid for at least thirty minutes before assembly. Strips of graphene foil (obtained from Graphene Supermarket [https://graphene-supermarket.com/]) were used as current collectors. The whole assembly was sandwiched between two glass micro slides clamped with a small binder clip. PTFE thread tape was used to keep ionic liquid wetting from spreading to the area of the graphene foil where electrical contact was made (bottom left corner of *Figure 3*).

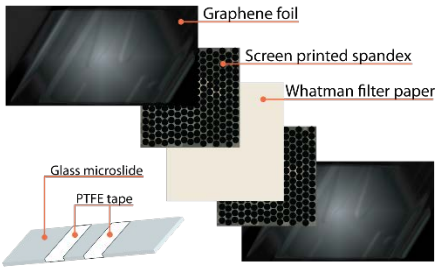


FIGURE 3. Supercapacitor set-up for testing screen printed military textiles.

RESULTS AND DISCUSSION

Military Textiles Require Several Screen Print Passes with Carbon Ink to be Conductive

Ink 1 was used as a surrogate ink to determine details of the screen printing procedure. A thicker consistency of ink made it easier to coat the textile fibers, whereas more 'watery' ink created an uneven dispersion of carbon. Secondly, it was found that military textiles were most amenable to being screen printed compared to the other textiles – specifically, Cotton, Polyester, and Nonwoven 3NP. For Cotton and Polyester, the knit structure was too loose to hold the carbon on top of the textile surface. Generally, the tighter the weave/knit, the better the screen print [16]. For the Nonwoven 3NP textile, the fibers resisted the carbon ink, suggesting that the acrylic polymer-based binder used in our ink formulation was not compatible with the Rayon/*p*-Aramid fiber blend.

In order to render the military fabrics conductive throughout (especially through the thickness of the fabric), screen printing was required on both sides due to the tightness of the weaves. *Figure 4* shows that the amount of carbon deposited onto the military textiles ranged from 1.66–6.28 mg/cm^2 when the number of screen print passes ranged from 2-10 per side of textile. Consistency in mass deposited was best achieved when four passes were made on each side of the fabric. It is possible that at higher screen print passes, additional passes may have affected prior passes.

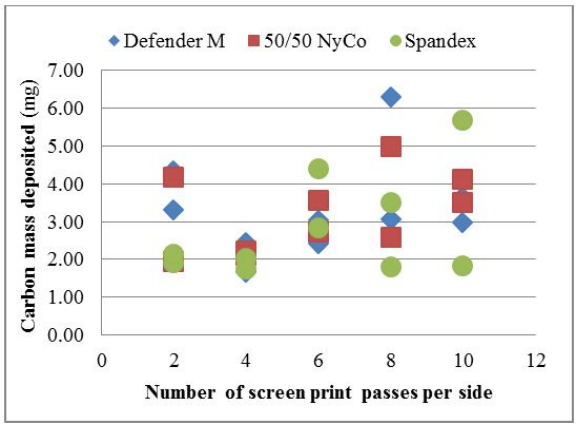


FIGURE 4. Carbon mass deposited on military fabrics vs. number of screen print passes per side using Ink 1.

Figure 5 shows the sheet resistance readings obtained for the same screen-printed textiles in *Figure 4*. Although mass consistency was better achieved at lower numbers of passes for all the textiles, the sheet resistance data in *Figure 5* show that at least eight or ten screen print passes per side are required to render all the military textiles conductive. The Spandex textile, however, seemed to produce conductive readings at any given number of screen print passes (i.e., 2-10 per side). Since all the military textiles had similar thickness and tight weave structures, this suggests that the fiber composition of the Spandex textile was most amenable to our ink.

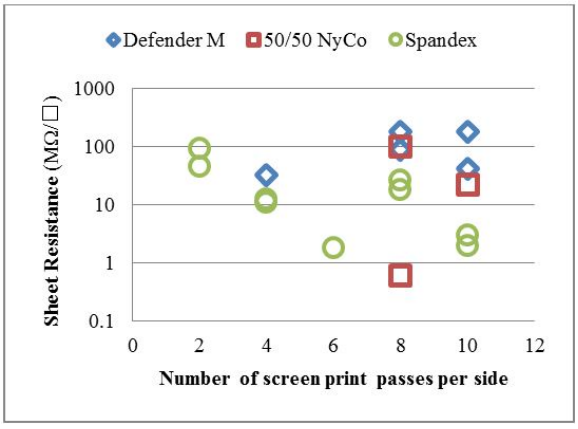


FIGURE 5. Sheet resistances of screen printed military fabrics vs. number of screen print passes per side using Ink 1. Spandex was the only military textile that had conductive readings at any given number of screen print passes

Activated Carbon Ink And Nylon In Military Textiles

To ensure low sheet resistance (and more conductive textiles), ten passes of Ink 2 were screen printed on each side of each military textile. Since Ink 2 utilized a more conductive carbon, differences in sheet resistances enabled a better understanding of carbon particle interconnectivity within each military textile.

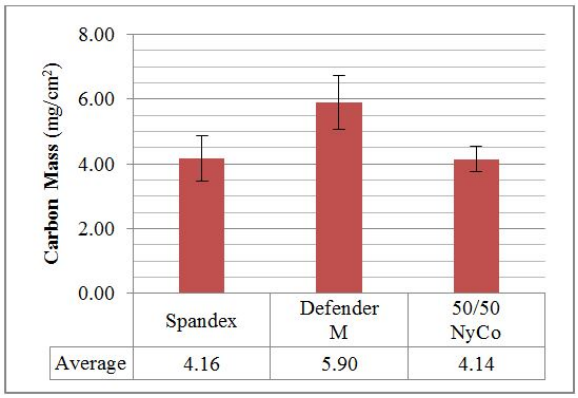


FIGURE 6. Average mass (from eight replicates) of activated carbon deposited onto military textiles with Ink 2. Error bars indicate standard error.

Figure 6 and *Figure 7* show gravimetric and sheet resistance results, respectively. Generally, the gravimetric uptake of carbon was higher (~4-6mg/cm^2) with Ink 2 than with Ink 1. This can be attributed to the different particle size distribution of each activated carbon. *Figure 6* shows that similar masses of carbon were screen printed on 50/50 NyCo and Spandex. SEM images in *Figure 8A-C* suggest that this may be due to the two textiles' similar weave structures, but SEM images of the coated textiles (*Figure 8D-F*) paint a different story: Spandex and Defender M have a visible sheet of carbon in ("caking") and on the surface of its weave. The 50/50 NyCo textile appears to have minimal carbon treatment but is conductive enough for SEM imaging and sheet resistance measurements.

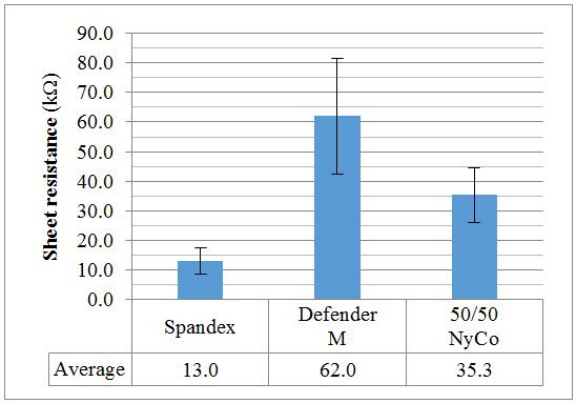


FIGURE 7. Average sheet resistance (from eight replicates) of military textiles screen printed with Ink 2. Error bars indicate standard error.

The sheet resistances in *Figure 7* exhibit a trend consistent with fiber composition of military textiles screen printed with Ink 2: the higher the nylon content, the more conductive the textile. This can be explained by the fact that, in the wood finishing industry, nylon paintbrushes are generally recommended for use with latex and acrylic paints, whereas natural-based materials will generally absorb water before adhering to the actual paint [17]. Since the other half of 50/50 NyCo fiber composition is cotton, this fact may explain the non-adherence of Supra 30 particles on the surface of the textile: it is possible the liquid precursor of Ink 2 was drawn into fiber bundles of the NyCo textile before curing.

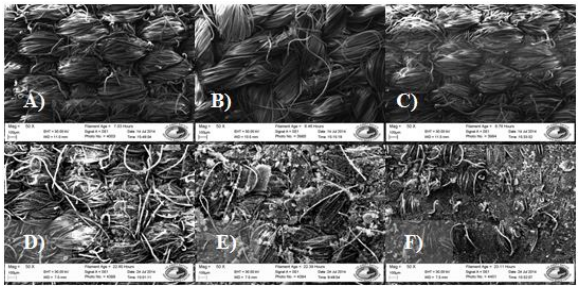


FIGURE 8. SEM images of military textiles selected for Ink 2 screen prints. Top row (A-C) is untreated military textiles – from left to right: 50/50 NyCo, Defender M#14, and Spandex. Bottom row (D-F) is military textiles screen printed ten times on each side with Ink 2 – from left to right: 50/50 NyCo, Defender M#14, and Spandex

Screen-Printed Spandex Textile as an Electrode in a Supercapacitor Device

After military textiles were screen printed and dried, flaking of activated carbon occurred during physical handling, indicating poor carbon adhesion. Devices were initially assembled in a coplanar set-up utilizing an ionogel (ionic liquid gel) electrolyte, Ink 2 electrodes and no current collector as in ref [14]. While wax treatment and ionogel promoted better carbon adhesion in the resulting device, the combination of coplanar electrode configuration and direct electrical contact onto screen-printed textiles yielded highly resistant devices. A parallel plate – "sandwich" – set-up was thus employed using graphene foil current collectors, which provided better electrical contact. Results in *Figure 9* are of devices fabricated in this set-up with neat ionic liquid (EMI TCB) and filter paper as a separator. For comparison, untreated Spandex "electrodes" were used as a control. Screen-printed electrodes experienced a loss of carbon during final device assembly.

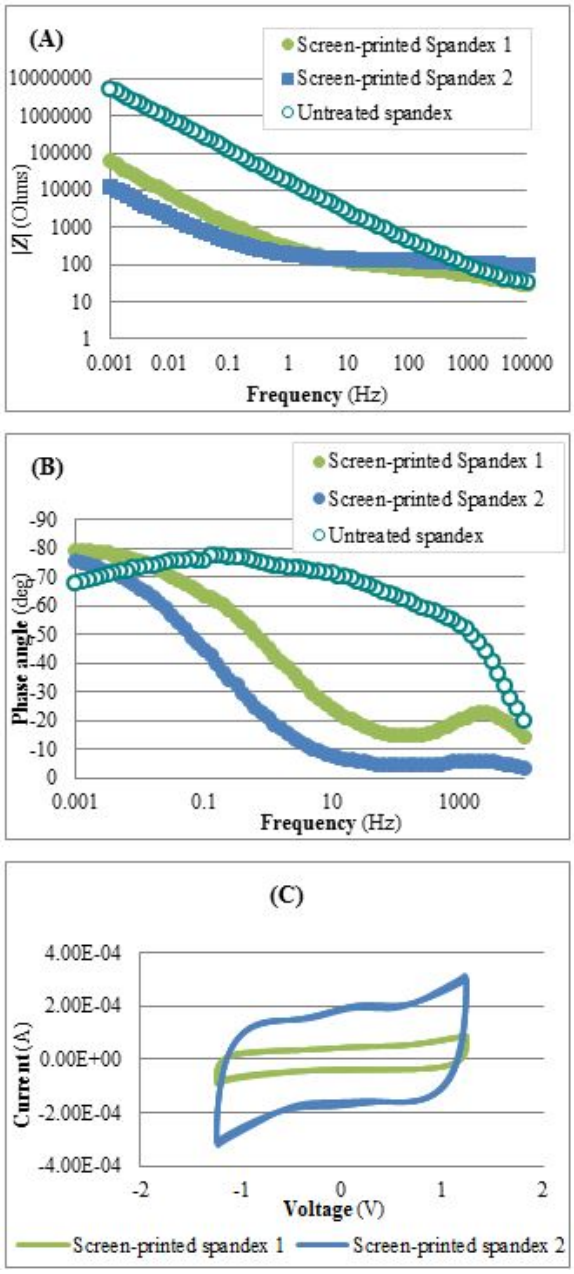


FIGURE 9. Supercapacitor device data: (A) Magnitude of *Z* (|*Z*|) from EIS. (B) Phase of *Z*, from EIS. (C) Cyclic voltammogram, taken from -1.25 to +1.25 V at a scan rate of 10mV/s. Three replicates of the untreated spandex were performed using different pieces of untreated spandex and the same value for capacitance was achieved.

Calculated capacitances from electrical impedance spectroscopy (EIS) and cyclic voltammetry (CV) data are shown in *Table II*. Capacitance from EIS and CV was calculated using Eq. (2) and Eq. (3), respectively.

$$C_{\text{EIS}} = \frac{1}{(2\pi f|Z|)} \quad (2)$$

$$C_{\text{CV}} = \frac{I_{V=0}}{\left(\frac{dV}{dt}\right)} \quad (3)$$

Areal capacitance refers to capacitance calculated in the device footprint. This can be viewed as the amount of capacitance that would occupy a particular footprint on a textile/garment/suit. Gravimetric capacitance is the calculated capacitance normalized by the total amount of active material (carbon) on each electrode. Since the devices tested in this study had an area of 1cm^2, these gravimetric capacitances are equivalent to Jost's definition of areal capacitance, which "depends [on] (1) mass loading of active material per area, and (2) the intrinsic capacitance of the selected active material" [11]. Areal capacitance (based on EIS and CV) due to graphene current collectors was <2% (evidenced by data with control device). Inconsistency between the two screen-printed devices is apparent and assumed to be from carbon loss. The benefit of the screen printed carbon can be seen in the gravimetric capacitances based on CV, which are 1.5x the gravimetric capacitance based on EIS; the untreated Spandex, on the other hand, achieves a lower capacitance when subjected under voltage. Nonetheless, supercapacitor behavior from each screen-printed device was achieved with the aid of graphene foil current collectors and demonstrates the feasibility of military textiles as a suitable substrate for active material printing as an electrode for supercapacitors.

TABLE II. Calculated capacitances from EIS and CV in *Figure 9*. The gravimetric capacitance incorporates the theoretical mass of the graphene foil current collector available in the screen printed devices.

		Areal capacitance (mF/cm^2)		**Gravimetric capacitance** (F/g$_{carbon}$)	
	Carbon mass (mg)	EIS*	CV	EIS*	CV
Device 1	5.99	2.7	4.2	0.29	0.44
	3.45				
Device 2	2.19	12.96	20	2.73	4.21
	2.56				
Untreated Spandex device	0.005	0.03	0.02	3.18	1.8
	0.005				

*Taken at f = 1 mHz

CONCLUSION AND RECOMMENDATION

High sheet resistances were generally achieved for screen-printed textiles. Improvement in conductivity was found using more screen prints, but caking in the bulk of the fabric occurred, which possibly led to carbon loss during physical handling.

Textile affinity to the activated carbon ink can possibly be attributed to the presence of nylon. It was found that the higher the nylon content in a military textile, the lower the four-point probe sheet resistance. Meanwhile, results suggest that weave type may have had an involvement with the amount of carbon mass that could be deposited (i.e, similar twill weaves in NyCo and Spandex held the same amount of carbon for a similar number of screen print passes).

Though the acrylic polymer binder used in this study provided non-optimal adhesion of carbon to military textiles, its mixture with water provided a suitable liquid carrier system for carbon delivery during screen printing. Ink reformulation with a better binder along with fewer screen print passes is recommended for improving carbon adhesion to fibers. Future carbon inks printed on military textiles can then be validated by performing standard testing (such as ghost tests and AATCC 8 ["Colorfastness to Crocking"] to visually assess its uniformity on military textiles. Mechanical durability of future screen printed ink on military textiles can be validated by performing ASTM D2136 ["Coated Fabrics – Low-Temperature Bend Test"], which would involve assessing cracks in coating after bending and flexing screen printed textiles.

Despite physical loss of carbon, supercapacitor behavior was still achievable in the final device architecture. Furthermore, it was seen that post-treatment of screen printed textiles with a curable liquid (wax, ionogel) could stabilize poorly bound carbon. Encapsulating assembled screen-printed textiles and graphene foil with a flexible, curable liquid is a recommended avenue for imparting more durability.

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