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## The effect of plasma pre-treatment on the electrical resistance of MXene ( $Ti_3C_2T_x$ ) coated fabrics<sup>†</sup>

Ashleigh Naysmith, \*<sup>a</sup> Timothy Smith, <sup>a</sup> Naeem Mian<sup>b</sup> and Andrew Hewitt<sup>a</sup>

The electronic (e-)textiles market is forecast to increase to a value of \$11 billion in the coming decade with applications spanning industries from medical monitoring to personal thermal management. Achieving high-performance e-textiles requires conductive coatings that are continuous and well-adhered to facilitate efficient electron transport. However, this remains a challenge due to the inherently irregular microstructure of textile fibres, particularly in natural fibres. Plasma treatment is widely used in e-textile research. However, the effects of plasma treatment parameters on the electrical resistance of subsequently coated e-textiles has not been systematically investigated. This research is the first to comprehensively evaluate how plasma pre-treatment affects the electrical resistance of e-textiles, providing critical insights for optimising the fabrication process. A Taguchi design of experiment was used to examine four fabrics (linen, polyester, nylon, wool) subsequently coated with MXene, and several plasma treatment parameters (power, flow rate, time, gas). The study presents exceptional findings demonstrating the impact of optimally selected plasma treatment parameters on the subsequent electrical resistance of MXene-coated (MC) e-textiles. Each fabric type exhibits a highly significant reduction in electrical resistance when treated with the optimal plasma parameters compared to the MC-control samples. For example, MC-wool's electrical resistance was lowest using hexafluoroethane with a median electrical resistance of 345  $\Omega$ ; a 99.96% decrease in electrical resistance compared to the median electrical resistance of the control MC-wool fabric. Air treatment was optimal for Nylon demonstrating a median of 22  $\Omega$ –99.7% decrease in electrical resistance from the MC-Nylon control sample. The results of this study provide valuable insight into enhancing the electrical performance of MXene-based coatings using a quick, simple and environmentally-friendly method.

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## Introduction

E-Textiles can serve as flexible, low-cost sensors and actuators while providing comfort akin to conventional textiles.<sup>1</sup> State of the art within the field has presented e-textile applications within industries as diverse as structural monitoring, health and medical monitoring, soft robotics, clothing for extreme environments strain sensors, detection of biochemicals and personal thermal management (PTM).<sup>2–9</sup> There are four major material approaches to the development of e-textiles: metallic (both macro and nanoscale) such as silver nanowires and copper nanoparticles; conducting polymers such as poly(3,4-ethylenedioxythiophene polystyrene sulfonate (PEDOT:PSS)

and polypyrrole; carbonaceous (including graphene, reduced graphene oxide, and carbon nanotubes); and ceramics (such as MXene, zinc oxide, and boron nitride); and combinations thereof.<sup>10–16</sup> The implementation of nanomaterial-based coatings has demonstrated superlative results. For example, silver nanoparticle coated lycra attained conductivity of  $3.85 \times 10^4$  S ( $2.59 \times 10^{-5} \Omega$ ).<sup>17</sup>

MXenes, the early transition metal–carbides/nitrides, are growing in popularity in e-textile developments due to several advantageous properties. MXenes benefit from near metallic electrical resistivity ( $\approx 10^{-6} \Omega$  m) and excellent mechanical properties while retaining hydrophilic properties, allowing easy processability in aqueous solution.<sup>18</sup> For comparison, the comparable 2D material, graphene, can achieve electrical resistivity of  $\approx 10^{-8} \Omega$  m, but solution-based processing is challenging. MXene has the lowest resistivity of any solution-processed 2D material.<sup>18</sup> However, the surface terminations that aid MXene's adhesion to many textile substrates also hinders its environmental stability. Aqueous dispersions exposed to air can

<sup>a</sup> Department of Fashion and Textiles, University of Huddersfield, Queensgate, HD1 3DH, UK. E-mail: ashleigh.naysmith@hud.ac.uk

<sup>b</sup> Department of Engineering, University of Huddersfield, Queensgate, HD1 3DH, UK

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degrade within days, rapidly oxidising the MXene surface into  $\text{TiO}_2$ .<sup>18</sup>

Textile surfaces, particularly natural fibres, are notoriously complex to coat due to the inconsistencies and imperfections in their structure. When developing coated e-textiles, a continuous conductive network is essential to achieve an effective conductive fabric. Pre-treatment can enhance subsequent coating efficacy by enhancing the fabric structure and chemical composition. Plasma treatment, particularly cold plasma treatment, is an effective and environmentally friendly technology for the surface modification of textiles.<sup>19–21</sup> Plasma treatment benefits from a water-free process and relatively low energy consumption. A lifecycle analysis analysis by Parisi *et al.* (2015) comparing plasma pre-treatment to a conventional wet process treatment found that plasma treatment was environmentally advantageous on several counts, including consumption of abiotic resources, lower environmental acidification, lower global warming potential, lower toxicity to humans, and aquatic and terrestrial ecosystems.<sup>22</sup>

Plasma pre-treatment of textiles offers the potential for improving the adhesion and durability of conductive coatings, thereby benefitting from reducing the number of required active materials. For example, oxygen plasma treatment of linen before coating with silver nanoparticles produces a 225% increase in nanomaterial loading compared to the non-plasma-treated linen fabric.<sup>23</sup> Research by Xu *et al.* (2021) confirmed that oxygen plasma treatment of textile fabric promotes “strong interfacial interactions” between the surface terminations of the MXene and the activated functional groups of the textile and imbues long-term stability.<sup>24</sup> Plasma pre-treatment of textile fabrics has been demonstrated to decrease electrical resistance ( $\Omega$ ). For example, Petkeviciute *et al.* (2022) found a 90% lower  $\Omega$  in nitrogen plasma pre-treated PEDOT: PSS coated wool.<sup>25</sup> Numerous studies have used plasma pre-treatment to enhance the performance of their e-textile.<sup>26–28</sup> Unlike previous studies, which primarily used plasma treatment in a limited capacity, either as a development stage or only studied discrete parameters, this study systematically investigated multiple plasma pre-treatment parameters to achieve the lowest possible  $\Omega$  in MC-textiles. Table 1 details the state of the art presented in the literature where plasma pre-treatment has been used in the development of conductive fabrics. Garg *et al.* (2007) evaluated atmospheric plasma parameters on wool and polyester, reporting a decrease in  $\Omega$  was proportional to the number of passes of plasma treatment.<sup>29</sup> Rajasekaran *et al.* (2018), using DC glow discharge, found that polypyrrole-coated cotton showed higher conductance when pre-treated with Argon plasma than when pre-treated with nitrogen plasma.<sup>30</sup> Conversely, Vida *et al.* (2023) investigated plasma treatment on MC-silicon. They found that work function was positively correlated to oxygen-plasma treatment time, increasing due to the incorporation of oxygen and subsequent formation of  $\text{TiO}_2$ .<sup>31</sup> This related to decreased electrical conductivity.<sup>31</sup>

No previous research has systematically explored the effects of plasma pre-treatment on the  $\Omega$  of MC-fabrics. However, to aid the study design, previous studies of polypyrrole-coated fabric were reviewed. It is anticipated that the bonding

mechanisms of both polypyrrole and MXene with textile fabrics are similar. Polypyrrole bonds with fabric (particularly with cellulosic fabric) surfaces *via* hydrogen bonds and electrostatic forces. MXenes with copious surface terminations are also known to bond with textiles *via* hydrogen bonding and van der Waals forces (Fig. 1).<sup>43,44</sup> Therefore, a circumspect inference can be made between ppy-coated fabrics and the properties evident in the MC-fabrics.

Following an extensive literature review, to the best of the authors' knowledge, there has been no systematic investigation on how plasma pre-treatment affects the  $\Omega$  of MC-textiles. Herein, we investigate the effect of plasma treatment on the  $\Omega$  of MC-textile fabrics (Nylon, polyester, linen, and wool). By extending this study to include four contrasting fabric types, the research provides insight into the effects of differing textile substrates on the  $\Omega$  of MC-textiles, providing a valuable resource to the e-textile discipline. Additionally, this study is the first to examine impact of hexafluoroethane plasma treatment on e-textile  $\Omega$ . The thorough investigation of how varying plasma treatment parameters impact the  $\Omega$  of different fabric types provides a foundation for other research groups to efficiently improve their developments *via* utilising the results of this work.

The four fabric types were selected for their chemically and structurally different compositions. The study used a plant fibre (linen), a protein fibre (wool), a polyamide (Nylon) and a polyethylene (polyester). Polyester, composed of PET, is one of the most commonly used fibres worldwide and provides a useful contrast to Nylon.<sup>20,45</sup> Nylon, a hydrophilic polyamide, provided a comparison to wool in terms of chemical composition whilst contrasting in structure.<sup>20</sup> Wool is a polyamide-based fibre with a complex multi-layered structure with a hydrophobic fatty acid monolayer surface.<sup>20,46</sup> Wool's inner cortex is hydrophilic. Linen was used as the representative plant fibre textile due to its low energy requirements in production compared to petrochemical-based textiles.<sup>47,48</sup>

An extensive Taguchi study was designed using the initial study's findings to analyse each fabric type individually against the plasma treatment parameters for their effect on  $\Omega$ . A mixed level  $L^8 4^1 2^3$  Taguchi Design of Experiment was designed (Table 2). Each Taguchi parameter was repeated for each fabric type, leading to 96 plasma-treated experimental samples plus 12 control samples. The Taguchi methodology benefits from experimental efficiency and design robustness, but caution should be taken regarding interactions between factors.<sup>49</sup>

The literature review identified the most commonly selected parameters (as detailed in Table 1). Considering the most reasonable extreme range for each variable given the capacity of the available plasma machine, the following parameters were determined:

**Fabric type:** Nylon, linen, wool, polyester

**Gas:** Argon, air, hexafluoroethane, oxygen

**Power:** 100 W, 200 W

**Flow rate (SCCM):** 10, 30\*

**Time (s):** 30, 120

\*The Hexafluoroethane parameter could not attain an SCCM above 13 due to the pressure increase in the plasma instrument.



Table 1 The effects of plasma treatment parameters on conductive textiles published in the state of art

Textile substrate	Coating	Plasma parameters	Result	Ref.
Nylon 6	Polyaniline	Gas: oxygen, Argon, ammonia Power: 60 W Pressure: 100 Torr Time: 10 min	$O_2$ plasma treatment improved conductivity and durability. Other gases showed minor improvements	32
Nylon 66	Ppy	—	He + $O_2$ plasma pre-treated fabric showed strongest interfacial bonding and high conductivity.	26
Wool and polyester	Ppy	Type: atmospheric plasma Gas: helium, helium/acetylene (5%), helium/nitrogen (5%) Flow rate: 0.7–14 l min <sup>-1</sup> Time: 0.848 s (5–50 repeats) Power: 970 W frequency: 90 kHz	Electrical resistivity decreases proportional to the number of passes of plasma treatment. Helium/nitrogen mix resulted in the lowest resistivity.	29
Polyester	Amino functionalised carbon nanotubes	Gas: oxygen gas Flow rate: 20 sccm Pressure: 13 Pa Power: 150 W Time: 120 s	Electrical resistance decreased	27
Polypropylene	Reduced graphene oxide	Gas: oxygen Frequency: 26 kHz Pressure: 25 mPa Time: 5.0 min (per side)	No comparison of plasma treated and untreated	33
Silver plated Nylon Carbon cloth	MXene	Gas: oxygen plasma time: 5 min	No comparison of plasma treated and untreated	34
	MXene	Gas: Argon/oxygen Time: 10, 15, and 20 minutes Power: 110, 170, and 230 W Ratio (Argon/oxygen): 10:2, 9:3, and 8:4	Small improvement in CV curve for plasma pre-treated – chemically modified better	35
Linen	Silver nitrate	Type: atmospheric pressure dielectric barrier discharge reactor Gas: oxygen flow rate: 3 L min <sup>-1</sup> . Time: 45 s	Improved antibacterial performance	23
Nylon/ polyester	MXene	Gas: oxygen activated by oxygen Flow rate: 20 ml min <sup>-1</sup> Power: 150 W Time: 40 s	No comparison of plasma treated and untreated	28
Polypropylene and viscose	Ppy	Type: radiofrequency plasma Gas: Argon Frequency: 13.56 MHz Time: 2 min	No comparison of plasma treated and untreated	36
PTFE sheet & Nylon 6,6		Gas: Argon Flow rate: 10 mL cc <sup>-1</sup> Power: 150 W	Output performance of the T-TENG – 7.6 times enhancement from pristine to 12 min Ar plasma treated	37
Polyester	Monolayer graphene oxide	Time: 3 to 12 min (with steps of 3 min) Type: DBD Power: 3000 W	Plasma treatment decreased the number of coatings needed to achieve similar values of $R_e$ .	38
Cotton	Ppy	Type: DC glow discharge Gas: Argon, nitrogen Pressure: 0.2 mbar Applied potential: 300 V Time: 10 min and 20 min	Argon plasma pre-treatment shows higher conductance than the sample pre-treated with nitrogen plasma.	30
Aramid spun laced non-woven fabric “a substrate”	Mxene	Type: diener nano Time: 3 min Power: 800 W	No comparison of plasma treated and untreated	39
	MXene	Gas: air Flow rate: 400 sccm Pressure: 101 mbar Time: 0, 10, 20, and 30 min.	Enhances $NO_2$ gas absorption	40
Polyester and Nylon-lycra	Polypyrrole	Gas: Argon scanning electron microscopy used at a pressure of 0.05 mbar, with a power of 56 exposed to plasma time: 5 s	Decreased electrical resistance by 25%	41
65% PET and 35% cotton blend	None	Gases: air, Argon and $O_2$ Flow rate: 60 sccm Power: 200, 500 and 800 W Pressure: 0.3 mbar Time: 5, 10 and 15 min	Plasma treatment consistently reduced the surface and volume electrical resistivity of the fabrics. Oxygen had the most positive effect, followed by air and then Argon.	42

With Hexafluoroethane gas, the ignited plasma rapidly increased the pressure within the chamber. When the flow rate was taken

above 13 SCCM, the pressure exceeded the safety threshold set, thus automatically reducing the flow rate back to 13 SCCM.



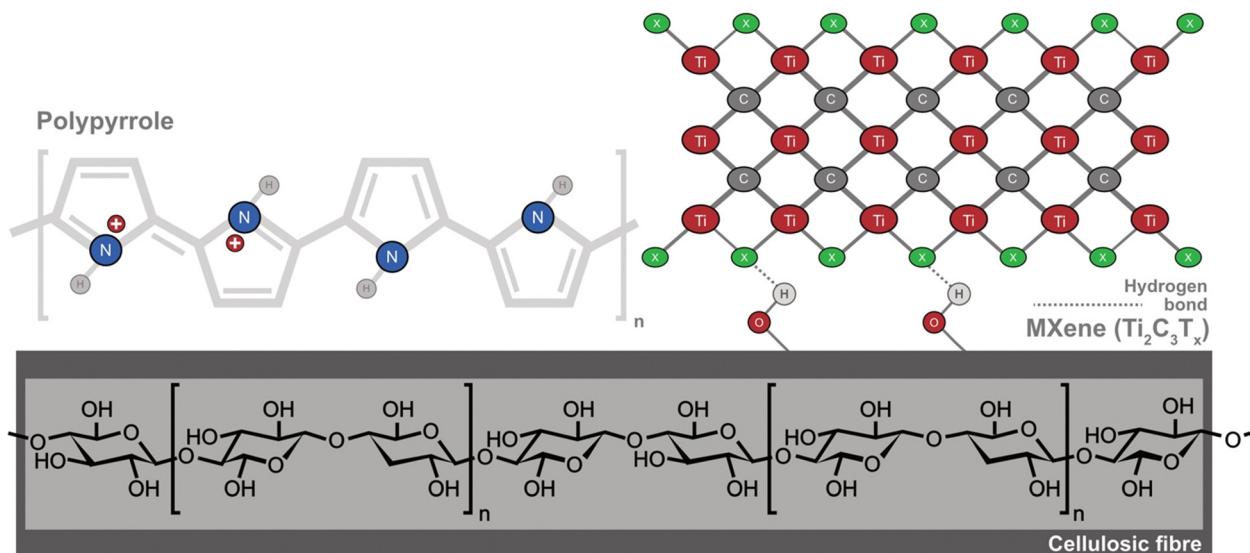


Fig. 1 Diagram showing bonding mechanisms between polypyrrole and MXene with cellulosic fibres.

Table 2 Taguchi parameter settings used for each sample set

Gas	Power (W)	SCCM	Time (s)
Argon	100	10	30
Argon	200	30	120
Air	100	10	120
Air	200	30	30
HxFE	100	30	30
HxFE	200	10	120
Oxygen	100	30	120
Oxygen	200	10	30

## Methodology

Air, argon, and oxygen plasma provide surface activation; removing contaminants, etching the fabric surface and producing chemically active sites for improved adhesion and wettability. Hexafluoroethane (HxFE) differs: HxFE deposits a thin film on the fabric's surface, which was anticipated to increase oleo- and hydrophobic properties.<sup>50</sup> Hence, HxFE provided an interesting contrast, particularly with the natural fibres that suffer from rough surfaces that can hinder electrical performance.

## Materials

Materials used in the MXene synthesis are described in the ESI,† S1. The following fabrics were used: Linen (Whaley's Bradford Valencia natural, even weave, approx. 240 gsm, spun yarn), Nylon (lab stock, satin weave, 78 gsm). Polyester (White Polyester Taffeta Fabric, Whaley's Bradford, 65 gsm), Cotton (Whaley's Bradford Kilberry White, plain weave, approx. 111 gsm), and Wool (Whaley's Bradford Voltaire Wool Fine Natural, even weave 230 gsm). All fabrics were kept under Standard Conditions (SC) (20 °C, 65% relative humidity) for a minimum of 24-hours before treatment and after treatment before  $\Omega$  testing.

## Scouring process

Fabrics were scoured prior to treatment using the following process. Sodium carbonate dissolved in tap water and then diluted in  $500.0 \pm 1.0$  ml of water in a 1000 ml beaker.  $1.0 \pm 0.05$  g of sodium carbonate was used as this was 3.5% of the fabric weight (liquor ratio =  $57.2 \text{ g l}^{-1}$ ). The fabric samples were placed in the beaker on a hot plate, brought to 100 °C, and stirred every 10 minutes. After 30 minutes, the fabrics were removed, rinsed with water, and left to dry. The fabrics were kept under SC for a minimum of 24 hours before treatment.

Samples were prepared to a weight of 0.064 g (S.D. = 0.002, range 0.061–0.068 g) with a minimum length of  $1.0 \pm 0.05$  cm. The initial weight of each fabric sample had a mean of  $0.064 \text{ g} \pm 0.005 \text{ g}$ , ranging from 0.061 g to 0.069 g (Table 3). The coating was maintained at a mean weight increase of  $77.2\% \pm 5.6\%$ , ranging from 71.6% to 82.0%. This gave a padded weight of  $0.114 \text{ g} \pm 0.009 \text{ g}$ .

## MXene synthesis

Titanium carbide ( $\text{Ti}_3\text{C}_2\text{T}_x$ ) MXenes were prepared following a minimally intensive layer delamination (MILD) method.<sup>51</sup> The synthesis method is described in the ESI,† S1. The MXene sediment was freeze-dried (Christ, Alpha 2–4 LSCbasic freeze dryer) at  $-88.8$  °C and 0.0378 mbar for 240 hours. The synthesised MXene powder was added to DI water to create MXene (2 M) suspension.

Table 3 Fabric weight and coating descriptive statistics (including control samples that were not plasma treated prior to MXene coating)

Descriptive statistics	N	Minimum	Maximum	Mean	Std. deviation
Initial weight	108	0.061	0.069	0.064	0.002
Coated weight	108	0.106	0.123	0.114	0.005
Weight increase (%)	108	71.6	82.0	77.15	2.66



### Treatment protocol

Fabric samples, prepared to a weight of  $0.084 \pm 0.003$  g, were left in SC for a minimum of 24 hours. The samples were placed in the plasma machine in sets of three. Each sample was treated under the specified plasma protocol (Table 2) using a PICO low-temperature, low-pressure plasma machine (Pico-AR-200-PC-c, Diener GmbH, Ebhausen, Germany (40 kHz). Samples were removed from the plasma machine using tweezers and placed on a weighing boat with the plasma-treated side facing up. Two to three drops of MXene suspension were syringed onto the sample, and then a mini rolling pin was used to pad the suspension on the fabric until a pick-up of 77% (S.D. 2.57, range 72–82%) was achieved. Additional MXene suspension was used if required to reach the target pick-up rate. These samples were left to dry in SC for 24 hours.

Additionally, three samples of each fabric type were coated with MXene (2 M) without prior plasma treatment and characterised as a control condition.

### Physicochemical characterisation

Elemental analysis was conducted using energy dispersive X-ray fluorescence spectrometer (EDX-XRF) (Shimadzu EDX-7000 XRF). XRF characterisation was undertaken using a selection of 11 samples representing each fabric type and gas type used. UV-visible spectrophotometry analysis was used to confirm MXene formation (UV/Vis/NIR Spectrophotometer, Jasco V-700 series). Scanning electron microscopy (SEM) analysis was conducted using a Zeiss (SEM EVO MA10) scanning electron microscope. Each sample was gold-coated prior to SEM analysis. The MXene samples were in powder form. Transmission electron microscopy (TEM) analysis was conducted using a Hitachi 9500 transmission electron microscope, operated at 300 keV, and the images were captured on a Gatan Oneview camera.

### Electrical characterisation

$\Omega$  analysis was conducted using a Rohde and Schwarz LCR Bridge 4-point probe, measuring capacitance and resistance using the parallel measurement setting with 1 kHz response frequency at 10.00 mm ( $\pm 0.05$  mm) point distance confirmed with callipers. The sample was held in place with a clamp stand to avoid movement during testing. Each sample was tested in three locations across each sample whilst held flat and then tested in a folded position twice (one way, then the reverse).

Samples are denoted by the treatment parameters as follows: Fabric/Gas/Power/SCCM/Time. Individual  $\Omega$  tests are denoted by run and test. For example, the parameter of linen fabric, treated with air plasma under 200 W power, 30 SCCM flow rate for 30 seconds is denoted Linen/Air/200/30/30. Run 1, electrical test 1 is denoted R1a, Run 1, electrical test 2 is R1b and so on.

### Statistical testing

The analyses in this research used the median responses from the data to inform the main effects and interaction plots.<sup>52</sup>

This was necessary due to the non-normality displayed in the data, requiring non-parametric statistical methods to be used. The median  $\Omega$  value was calculated from nine measurements. Each sample was measured three times in different locations across the sample at a  $1.0 \pm 0.05$  cm point distance.

Minitab<sup>®</sup> Statistical Software 21.4.2.0 was used to create the Taguchi design, main effects plots, interaction plots, Kruskal-Wallis and Mood's Median analysis. Further analyses were undertaken using IBM SPSS Statistics version 28.0.1.1 (14), including Friedman tests, Wilcoxon signed-rank tests using a Bonferroni adjustment and bootstrapped descriptive statistics.

A Mood's Median test was used to identify whether a type of gas (irrespective of specific treatment parameters) influenced the  $\Omega$  of the MC-fabric. A Friedman test identified whether parameter settings (*i.e.* parameters within the Taguchi design) influenced the  $\Omega$ . Thirdly, a *post hoc* analysis using Wilcoxon signed-rank tests was carried out to identify which specific parameter affected the  $\Omega$ . Finally, another Wilcoxon signed-rank test was conducted to evaluate the difference between an MC-control fabric with no plasma pre-treatment, and the plasma pre-treated MC-fabric with the lowest  $\Omega$ .

## Results and discussion

### MXene characterisation

The UV-visible spectrophotometry spectrum (ESI,<sup>†</sup> S2) confirms the characteristic absorption peaks for MXene at 325 nm and 753 nm. This was consistent with previously reported spectra therefore indicating successful MXene synthesis.<sup>53</sup> XRF elemental analysis of the MC-fabric further confirms the presence of MXene across the surface of the fabric, with high titanium content in all samples. However, as shown by Fig. 2 the detection of aluminium content suggests a proportion of unetched MAX phase. Whilst research has shown that a degree of residual aluminium content is anticipated when using the 24-hour MILD synthesis method, this was likely to have a negative impact on  $\Omega$ .<sup>54</sup> Recent studies refining the MILD method suggest that longer etching times (> 24 hours) support a higher degree of etching, highlighting a trade-off between synthesis time and product purity.<sup>55,56</sup> The full surface elemental composition is provided in ESI,<sup>†</sup> S3 also reveals an absence of lithium; indicating that the MXene was successfully washed before freeze-drying.

### Inorganic elemental surface composition

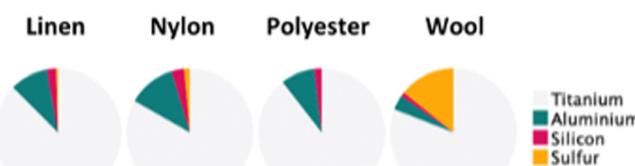


Fig. 2 Inorganic elemental composition main components (XRF data) showing Ti content that confirms successful MXene coating, but presence of Al indicates unetched MAX phase.



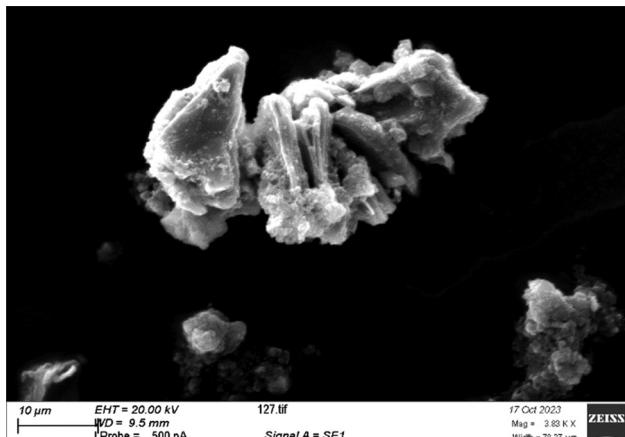


Fig. 3 SEM micrograph MXene  $\text{Ti}_3\text{C}_2$  powder showing formation of MXene sheets.

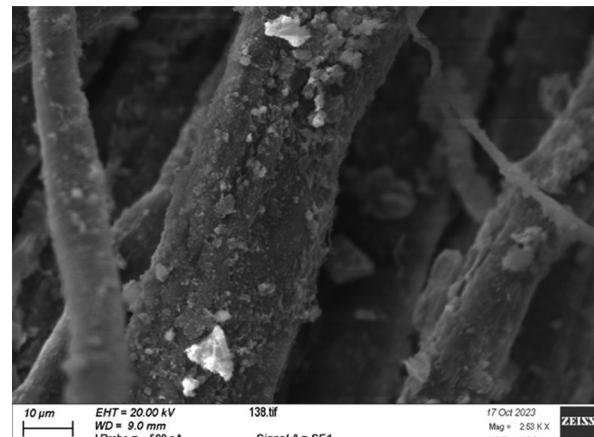


Fig. 5 MXene coating on linen pre-treated by argon gas (200 W, 30 SCCM, 120 s) showing MXene flakes adhered across the surface.

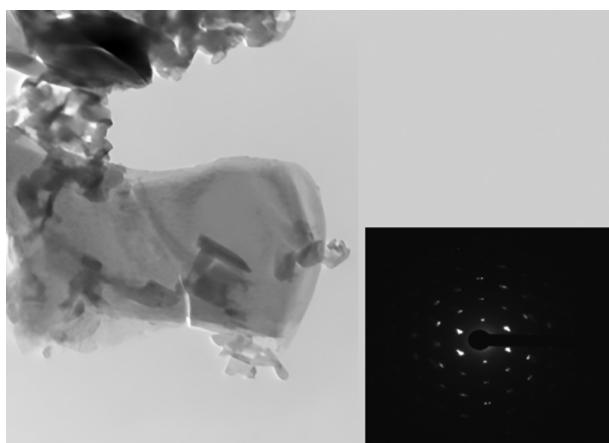


Fig. 4 TEM micrograph of MXene  $\text{Ti}_3\text{C}_2$  powder. Inset, corresponding diffraction pattern, indicating single layer MXene formation.

The SEM micrograph of the etched MXene powder (Fig. 3), presents the formation of the characteristic accordion-like structure of  $\text{Ti}_3\text{C}_2\text{Tx}$  MXene, indicative of successful exfoliation. TEM analyses (Fig. 4) show regions of monocrystalline MXene with thin flakes, confirming the exfoliation of single sheets. However, the imperfections to the accordion structure visible in the SEM, alongside segments of polycrystalline  $\text{Ti}_3\text{C}_2\text{Tx}$ , demonstrated by concentric rings visible in the TEM images, indicate a degree of oxidation on the surface with a transformation to anatase  $\text{TiO}_2$ .<sup>57</sup> This further underscores the importance of refining the etching and delamination process.

Fig. 5 shows the SEM micrograph of MC-linen fabric following plasma pre-treatment (Argon gas, 120 W, 30 SCCM, 120 s). It is evident that the MXene flakes have a thorough coating across the fibre. This is vital in ensuring the conductive pathway across the fabric to achieve the intended functional applications.

### Electrical resistance

Table 4 summarises the  $\Omega$  values for untreated control samples at a  $1.0 \pm 0.05$  cm point distance. The full descriptive statistics

Table 4 Summary: descriptive statistics of the  $\Omega$  of non-plasma-treated MC-fabric. Further data and bootstrapping criteria can be viewed in ESI S4 and S5

	Mean ( $\text{M}\Omega$ )	Median ( $\text{M}\Omega$ )	Std. Dev. ( $\text{M}\Omega$ )	Variance	Min ( $\text{k}\Omega$ )	Max ( $\text{M}\Omega$ )
Wool	556.08	1000.0	526.43	$2.77 \times 10^{17}$	44.9	1000.0
Nylon	0.632	0.007	1.40	$1.97 \times 10^{12}$	0.52	4.20
Polyester	0.003	0.001	0.002	$3.76 \times 10^6$	0.45	0.005
Linen	0.109	0.014	0.282	$7.94 \times 10^{10}$	1.42	0.860

of the MC-control samples  $\Omega$  are provided in ESI,<sup>†</sup> S6. The kurtosis displayed in all the samples were outside the range of statistical normality; in wool and polyester, the distribution was too flat. In Nylon and linen, the distributions were extremely peaked. Table 5 provides the full  $\Omega$  results of MC-fabrics for each parameter tested (further results, ESI,<sup>†</sup> S7). Each value was derived from a  $1.0 \pm 0.05$  cm point distance at three placement locations per sample. Despite the identical treatment conditions, the resulting  $\Omega$  varied widely in identical samples (Fig. 6). The variance values between different fabric types vary by several orders of magnitude. For example, the variance of polyester is  $3.76 \times 10^6$ , and wool displays a variance of  $2.77 \times 10^{17}$ . This variability is in line with previous works.

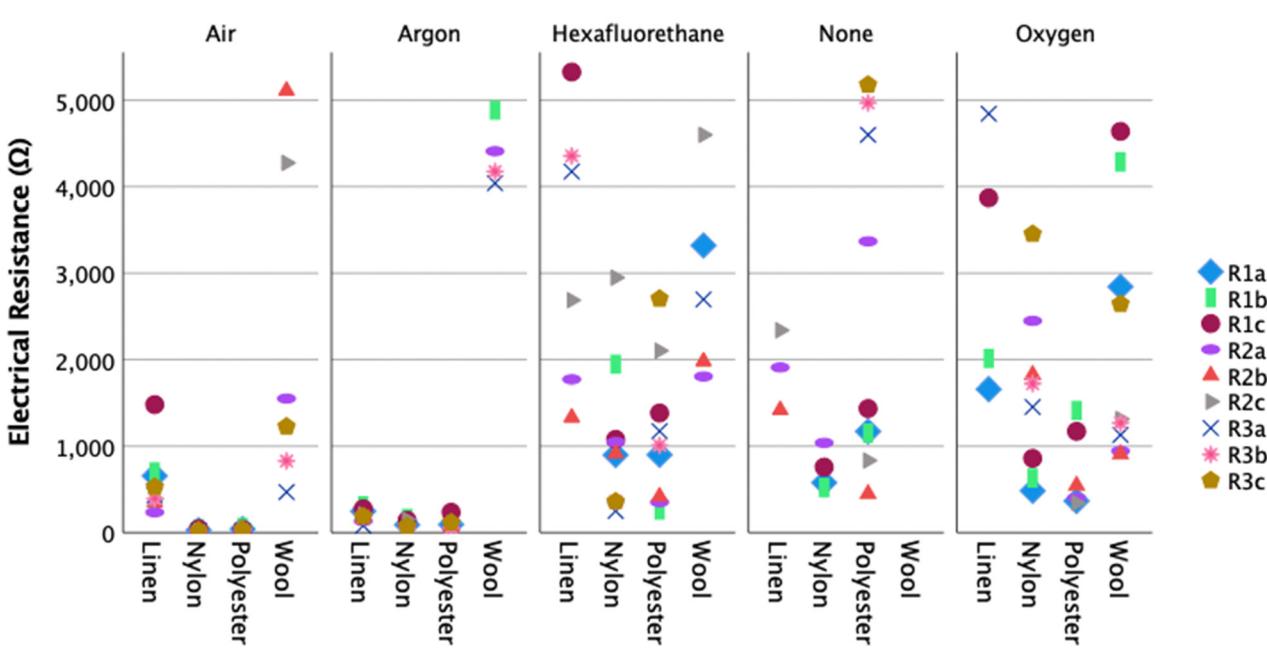
A recent study found that the weave pattern and interlacing coefficient of satin weave fabrics significantly impacted the electrical conductivity of graphene-coated fabrics.<sup>58</sup> Where the coating thickness was insufficient to overcome the elevation discrepancy between peaks and valleys on the fabric surface, the continuity of the coating was negatively impacted. As a result, the conductivity was severely hindered.<sup>58</sup> Research by Liu *et al.* (2023) discovered that fibres with the capacity for hydrogen bonding and van der Waals attractive forces, particularly those with “high planarity”, were predisposed to lower  $\Omega$  following a coating with polypyrrole, including polycotton, wool and Nylon.<sup>59</sup> Kaynak *et al.* (2021) demonstrated polypyrrole-coated polyester to have a lower average  $\Omega$  than polypyrrole-coated Nylon-lycra.<sup>41</sup> The authors hypothesised that surface structures could be the fundamental cause for this difference.

Table 5  $\Omega$  descriptive statistics by plasma treatment parameter

Fabric	Gas	Power (W)	Flow Rate (SCCM)	Time (s)	Median ( $\Omega$ )	Range ( $\Omega$ )	Skewness	Sample numbers
Linen	Air	100	10	120	1127	5202	1.66	1, 3, 5
Linen	Air	200	30	30	467	2317	1.83	2, 4, 6
Nylon	Air	100	10	120	22	52	1.01	7, 9, 11
Nylon	Air	200	30	30	33	143	3.18	8, 10, 12
Polyester	Air	100	10	120	46	116	1.2	13, 15, 17
Polyester	Air	200	30	30	26	59	1.96	14, 16, 18
Wool	Air	100	10	120	2600	15 772	2.42	19, 21, 23
Wool	Air	200	30	30	6900	33 789	1.8	20, 22, 24
Linen	Argon	100	10	120	402	2692	2	25, 27, 29
Linen	Argon	200	30	30	200	819	1.42	26, 28, 30
Nylon	Argon	100	10	30	164	624	1.54	31, 33, 35
Nylon	Argon	200	30	120	65	150	1.8	32, 34, 36
Polyester	Argon	100	10	30	22	35	0.81	37, 39, 41
Polyester	Argon	200	30	120	190	926	2.64	38, 40, 42
Wool	Argon	100	10	30	12 100	448 680	3.78	43, 45, 47
Wool	Argon	200	30	120	7165	15 540	1.22	44, 46, 48
Linen	Oxygen	100	30	120	3610	37 530	1.89	49, 51, 53
Linen	Oxygen	200	10	30	25 600	2 497 550	3.41	50, 52, 54
Nylon	Oxygen	100	30	120	1040	3212	1.2	55, 57, 59
Nylon	Oxygen	200	10	30	3180	47 926	2.71	56, 58, 60
Polyester	Oxygen	100	30	120	1340	30 199 705	3.69	61, 63, 65
Polyester	Oxygen	200	10	30	595	4444	1.48	62, 64, 66
Wool	Oxygen	100	30	120	1688	7932	2.43	67, 69, 71
Wool	Oxygen	200	10	30	1870	13 220	1.74	68, 70, 72
Linen	HxFE	100	10	120	4200	46 243	2.89	82, 84, 86
Linen	HxFE	200	30	30	7020	5 298 090	3.82	83, 85, 87
Nylon	HxFE	100	10	120	2030	233 690	3.5	88, 90, 92
Nylon	HxFE	200	30	30	478	6171	3.07	89, 91, 93
Polyester	HxFE	100	10	120	1270	6524	1.09	94, 96, 98
Polyester	HxFE	200	30	30	1500	11 973	2.79	95, 97, 99
Wool	HxFE	100	10	120	502	5185	2.2	100, 102, 104
Wool	HxFE	200	30	30	14 400	77 570	1.11	101, 103, 105

Electrical Resistance ( $\Omega$ ) of Mxene coated fabrics by gas and fabric type to a maximum of 5000  $\Omega$ 

Gas

Fig. 6  $\Omega$  of MC-fabrics by gas type to 5000  $\Omega$ , demonstrating the significant variation depending on fabric type, gas type, and plasma parameters.

Furthermore, the research of Komolafe & Torah (2021) found that fabric surface roughness and porosity influenced the  $\Omega$  of

the subsequent e-textiles. The authors noted that primer layers are often required in e-textile developments to maintain a low  $\Omega$



without requiring high quantities of (often expensive) conductive coatings.<sup>60</sup> Whilst the variance in the present study restricts the potential extrapolation of the results, it was concurrent with previous work. Above all, the unmistakable trends in relative  $\Omega$  differences remain evident.

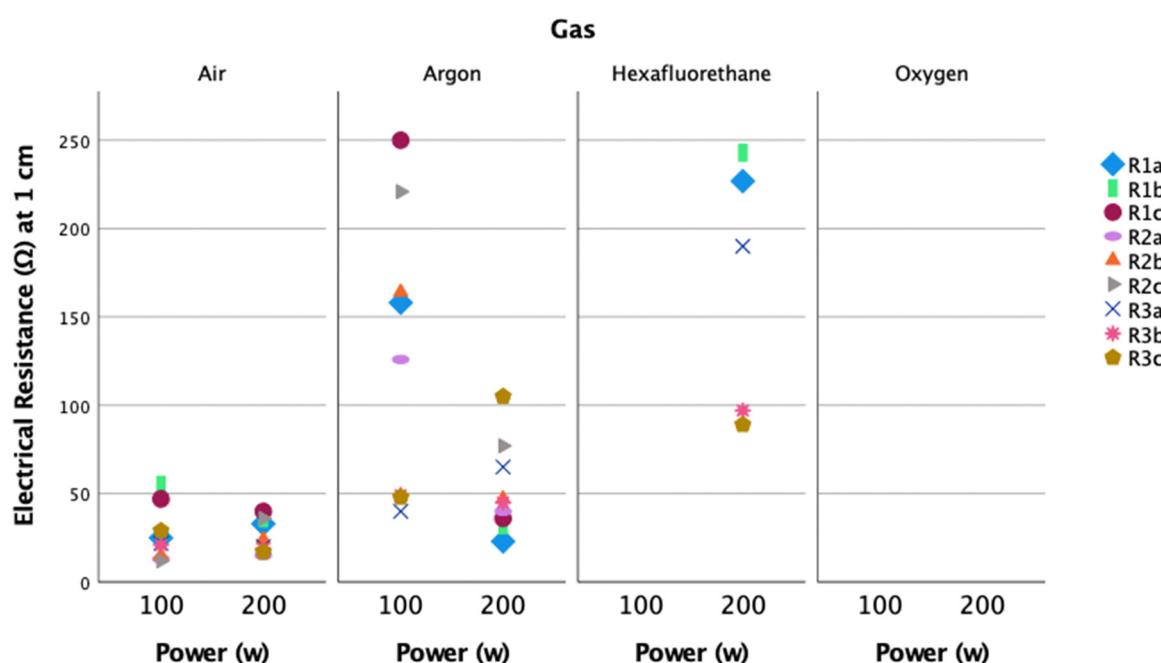
With the MC-fabric types isolated, it is evident from the main effects plots (Fig. 8, 9, 11 and 12) that the parameters of plasma treatment have significant effects on the  $\Omega$ . Additional graphs demonstrating the spread of  $\Omega$  results across the MC-fabrics can be found in the ESI,<sup>†</sup> S8–S11. The differing responses to plasma parameters by fabric type are striking: MC-wool displays its lowest  $\Omega$  following oxygen plasma treatment, whereas the  $\Omega$  of Nylon and linen is highest following oxygen. Argon-treated MC-fabrics consistently demonstrate lower  $\Omega$ , except once again for wool. This study's lowest  $\Omega$  values are found in synthetic fabrics pre-treated with argon plasma. These results will be examined and discussed in the proceeding sections. The power level of the plasma treatment has a stronger effect on natural fibres than on synthetic fibres, with 100 W power leading to lower  $\Omega$  than 200 W power. Considering the main effects plots, a higher flow rate was typically associated with lower  $\Omega$  in all MC-fabrics. This effect is more pronounced in natural fibres than in synthetic fibres. The effect of longer treatment time was most significant in Nylon. Polyester and wool displayed lower  $\Omega$  with shorter treatment times.

## Nylon results and discussion

The bootstrap specifications, full bootstrapped statistics, response table and the signal-to-noise ratios can be found in ESI,<sup>†</sup> S11–S14. The general trends in the effect of gas and power on the  $\Omega$  of MC-Nylon are illustrated in Fig. 7. The highest-performing plasma treatment (Air/100/10/120) demonstrates a highly significant improvement to  $\Omega$  in comparison to the untreated MC-Nylon ( $p$ -value = 0.008), as confirmed by the Wilcoxon signed rank test result (ESI,<sup>†</sup> S15). The median of untreated MC-Nylon was 7000  $\Omega$  (range: 522  $\Omega$ –4.2 M $\Omega$ ). Nylon pre-treated with air plasma exhibited an extraordinary median of 22–24  $\Omega$ , representing a marked decrease in  $\Omega$  from 7000  $\Omega$  without plasma treatment. Argon treatment presented improved  $\Omega$ , although less consistently than Air. HxFE results were highly specific to the parameters. Oxygen plasma pre-treatment was ineffective, resulting in a median of 750 or 2700  $\Omega$ , dependent on the specific parameters, indicating only marginal improvement compared to untreated MC-Nylon. The statistical analyses conducted on the Nylon results are shown in Table 6.

Fig. 8 presents the main effects plot for MC-Nylon. The Taguchi analysis confirmed that gas type is the dominant factor affecting  $\Omega$ , while longer treatment times generally lead to lower  $\Omega$  values. SCCM and power had minimal impact.

**Electrical resistance ( $\Omega$ ) of Mxene coated Nylon by gas type and power to maximum of 250  $\Omega$**



**Fig. 7** Electrical resistance of MC-Nylon by gas and power parameters up to 250  $\Omega$ , demonstrating the consistently excellent results of air treated MC-Nylon and the consistently poor results of oxygen treated MC-Nylon.

Table 6 Statistical tests run on MC-Nylon

Statistical test	Purpose	P-Value	Null hypothesis	Null hypothesis rejected?	Appendix number for full results
Mood's median test	To explore the gas type's effect on the MC-Nylon's $\Omega$ .	$p$ -Value (0.046)	$H_{N10}$ : Plasma gas type used has no effect on the $\Omega$ of MXene-coated Nylon.	Yes, rejected	ESI S20 and S21
Friedman test	To identify whether individual plasma parameters affect MC-Nylon's $\Omega$ .	$p = <0.001$	$H_{N20}$ : specific plasma gas treatment type parameters have no effect on the $\Omega$ of MC-Nylon.	Yes, rejected	ESI S22 and S23
Post hoc analysis with Wilcoxon signed-rank tests	To isolate which treatment parameters affect the $\Omega$ of MC-Nylon. – A Bonferroni correction was applied; thus, the significance level was set at $p < 0.0125$ .	$p = 0.008$	$H_{N[3-6]0}$ : the plasma treatment parameters have no effect on the $\Omega$ of treated MC-Nylon.	Yes, reject null in case of HxFE. Other gases, null retained.	ESI S24
Wilcoxon signed rank test vs. untreated MC-Nylon	Analysis of the effects of Air10010 120 (the most beneficial plasma parameters)	$p = 0.008$	$H_{N70}$ : air plasma gas treatment with parameters (P100, S10, T120) has no effect on the $\Omega$ of MC-Nylon.	Yes, null rejected.	ESI S25–S27

The narrow confidence intervals suggest high repeatability of the excellent  $\Omega$  results. For example, the MC-Nylon with parameter Air/200/30/30 has a 95% confidence interval of 20.8–32.9  $\Omega$  with a mean of 26.7  $\Omega$  and median of 24.0  $\Omega$  across the nine measurements. The extraordinary  $\Omega$  result achieved from the air treatment of MC-Nylon likely results from the mild etching enabling MXene to adhere to the surface, without roughening the surface to an extent that hinders electron transport. There is also the potential of nitrogen-containing functional groups being introduced, which can interact beneficially with the amide groups in the Nylon surface structure.

Power and flow rate shows a minor effect, but these trends should be interpreted with caution – the impact of plasma parameters must be evaluated in the context of each gas type, as the differences in gas properties significantly influence the  $\Omega$  outcomes. Holding gas type as constant, it is evident that the parameter levels have a significant effect on MC-Nylon's  $\Omega$  under argon and HxFE treatment (ESI,† S18). Considering the limitations in achieving the HxFE target flow rate, it is likely that HxFE flow rate has a more amplified effect than is demonstrated within this dataset.

Plasma treatment primarily modifies the fabric's surface (approx. 10  $\mu\text{m}$ ) retaining the bulk fibre properties. However, surface characteristics are crucial in attaining a uniform conductive coating.<sup>61</sup> While the subsequent coatings (polypyrrole vs. MXene) in previous research differ from that of the present study, the surface functional bonding groups are comparable. The findings in this present study contradict the findings of Deogaonkar-Baride & Palaskar (2022).<sup>26</sup> Their research posited that the surface roughness of a fabric correlated to the successful adhesion of a conductive coating (in this case, polypyrrole) to a Nylon fabric.<sup>26</sup>

There appears to be a delicate balance between surface roughness providing a solid foundation for good adhesion between the fabric surface and a conductive coating, and causing irregularities on the surface that hinder a continuous layer required for a reliable conductive coating. Their research found that helium

treatment provided no enhancement to surface roughness, whereas argon treatment caused a significant increase in surface roughness, resulting in high  $\Omega$ . However, the gas flow used in this research was 5000 SCCM, significantly higher than the flow rate used in the current study.

Our work contrasts with the findings of Kyung Wha Oh *et al.* (2001) who showed that oxygen plasma treatment was most conducive to high electrical conductivity, whereas argon and ammonia showed negligible change. This contrasts our results in which oxygen plasma treatment had the least impact on reducing the  $\Omega$  of MC-Nylon. The present study's high  $\Omega$  values from oxygen pre-treatment may be attributed to the more aggressive ablating effect of oxygen treatment, overly-etching the Nylon, producing voids and cracks on the surface, and potentially breaking down the polymer chains.<sup>61,62</sup> Previous work proposed that the conductivity of polyaniline-Nylon is dependent upon the deposition of conducting material on the surface and interstices, differentiating between physisorption and diffusion into the fibre. The etching of oxygen plasma introduced acidic functional groups, –OH and –OOH, to Nylon fabrics has been shown to enable the polymerisation of polyaniline on Nylon, correlating to improved electrical conductivity in polyaniline-Nylon composites.<sup>32</sup> These contrasting results can be explained by the differing adhesion mechanisms of MXene and polyaniline. The oxygen plasma treatment of Nylon prepared the surface to enable covalent bonding of polymerised polyaniline-Nylon composites, creating a highly uniform surface which is likely to be additive in charge transport processes. This contrasts from the non-covalent bonding of MXene with Nylon in which the surface roughness created by oxygen is not coupled with the potential for covalent bonding, and therefore likely hinders charge transport over the fabric surface.

## Wool results and discussion

Fig. 9 shows the main effects plot for MC-Wool. The bootstrap results and specifications, signal-to-noise ratios and response table for means can be found in ESI,† S16–S19. To note, the



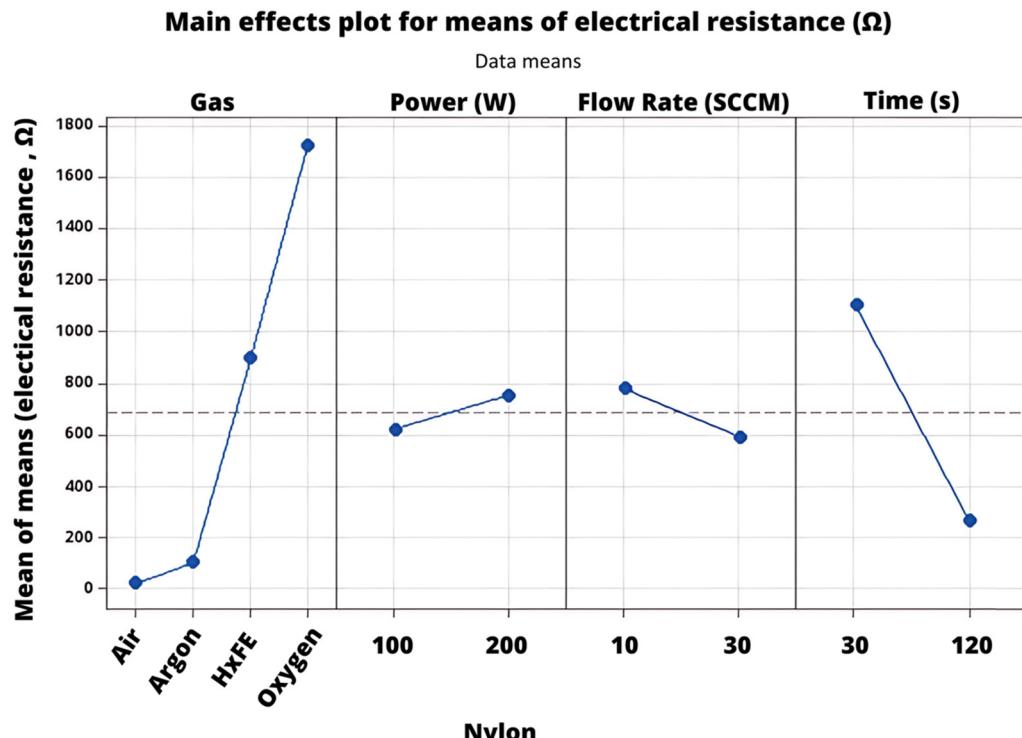


Fig. 8 Main effects plot of MC-Nylon demonstrating the large effects of gas type and treatment time, and more negligible effects of power and flow rate.

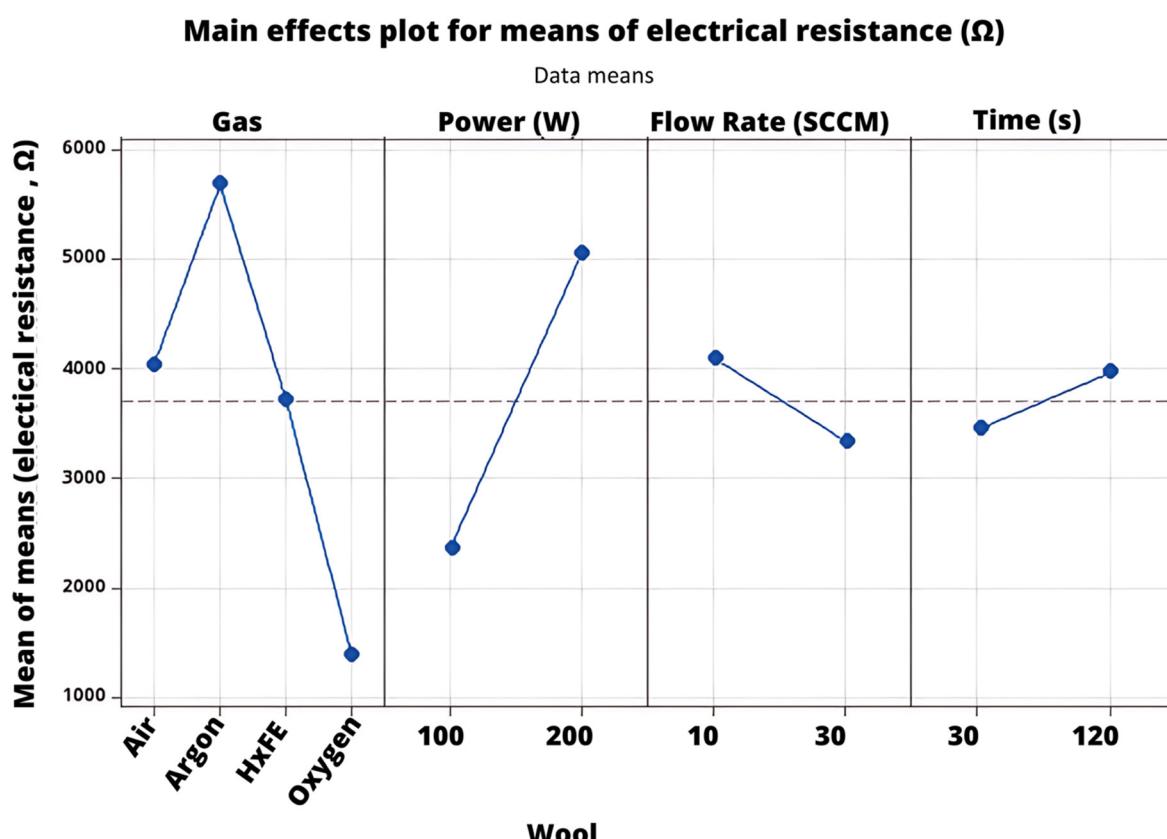


Fig. 9 Main effects plot of MC-wool demonstrating the significant effects of gas type and power on the  $\Omega$ .



Table 7 Statistical tests run on MC-Wool

Statistical test	Purpose	P-Value	Null hypothesis	Null hypothesis rejected?	Appendix number for full results
Mood's Median test	To explore the gas type's effect on the MC-wool's $\Omega$ .	0.261	$H_{W10}$ : plasma gas type used has no effect on the $\Omega$ of MC-wool.	No, retained	ESI S29 and S30
Friedman test	To identify whether individual plasma parameters affect MC-wool $\Omega$ .	<0.001	$H_{W20}$ : specific plasma gas treatment type parameters have no effect on the $\Omega$ of MXene coated wool.	Yes, rejected	ESI S31 and S32
<i>Post hoc</i> analysis with Wilcoxon signed-rank tests	To isolate which treatment parameters affect the $\Omega$ of MC-wool. – A Bonferroni correction was applied; thus, the significance level was set at $p < 0.0125$ .	$p = 0.008$	$H_{W[3-6]0}$ : the plasma treatment parameters have no effect on the $\Omega$ of [GAS] treated MC-wool.	Yes, reject null in case of HxFE. Other gases, null retained.	ESI S33 and S34
Wilcoxon signed rank test <i>vs.</i> untreated MC-wool	To analyse effects of HxFE100/10/120 (the most beneficial plasma parameters) <i>vs.</i> untreated MC-wool.	$p = 0.008$	$H_{W70}$ : HxFE plasma gas treatment with parameters (P100, S10, T120) has no effect on the $\Omega$ of MC-wool.	Yes, null rejected.	ESI S35 and S36

main effects plot does not isolate the competing effects within a parameter. For example, the HxFE mean is  $7516 \Omega$  across all parameters. However, the mean  $\Omega$  of the low power parameters is  $583 \Omega$ , whilst the mean of higher power parameters reaches  $14\,448 \Omega$ . Therefore, the individual parameters demonstrate a significant effect. The statistical analyses conducted on the wool results are shown in Table 7.

Once again, the highest performing plasma treatment (HxFE/100/10/120) demonstrates a highly significant decrease in  $\Omega$  in comparison to the untreated MC-wool ( $p$ -value = 0.008). MC-wool without plasma pre-treatment had a median  $\Omega$  of  $1000 \text{ M}\Omega$ .

As illustrated by Fig. 10, HxFE treatment offers the most consistent low  $\Omega$  in MC-wool and the significance of plasma parameters. Wool pre-treated with HxFE plasma demonstrated significant variation dependent on the parameters, with median  $\Omega$  of  $345 \Omega$  and  $7100 \Omega$ . The HxFE treatment parameters of 100 W power, 10 SCCM flow rate and 120 s treatment time led to an  $\Omega$  representing a  $1.0 \times 10^9$  reduction in  $\Omega$  from the non-plasma-treated MC-wool. Wool treated with polymerising HxFE has a smoother surface, leading to better coating uniformity. These positive results likely relate to wool's microstructure. Research has shown that conductive coatings are more effective on a smooth

### Electrical resistance ( $\Omega$ ) of Mxene coated wool by gas type and power to a maximum of $500 \Omega$

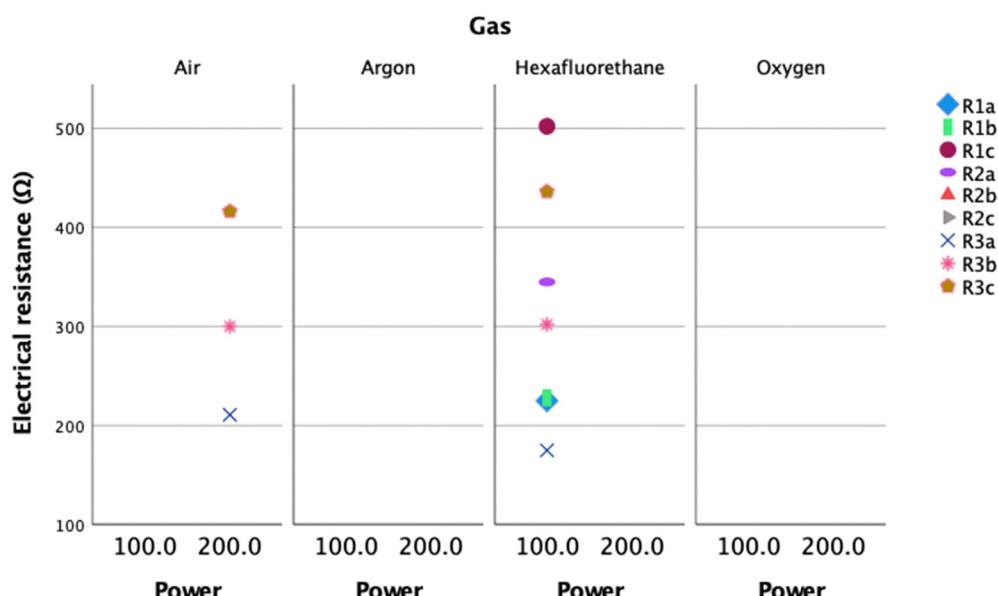


Fig. 10  $\Omega$  of MC-wool by gas type and power up to  $500 \Omega$  demonstrating the strong effect of HxFE low power treatment, and highlighting the negligible effects of other treatment parameters.



surface, and often, when printing circuits on textiles, a thick layer of conductive material is required to "overcome surface roughness and fibre porosity".<sup>63</sup> The HxFE treatment polymerises on the wool surface, creating a more uniform, smooth surface.<sup>64</sup> It is likely that this enables the conductive MXene coating to be retained at the surface, rather than absorbed into the wool fibre, which provides a continuous electron transport pathway.

In contrast, the activation plasma processes, such as oxygen treatment, create hydrophilicity on the naturally hydrophobic wool surface, potentially facilitating the diffusion of MXene through the wool fibre into the hydrophilic cuticle/cortex. It is plausible that this diffusion could dilute the conductive pathways, leading to increased incomplete pathways creating barriers to the electrical conductivity. This would result in effective absorption of the MXene in the wool fibre under oxygen, air and argon treatment, but result in less effective electrical properties.

Oxygen plasma treatment has been one of the most commonly utilised gases for the plasma treatment of wool to enhance dye uptake.<sup>20</sup> In comparison to the untreated control sample, our results showed that wool pre-treated with oxygen plasma led to improved  $\Omega$  of 1340 or 1450  $\Omega$ , dependent on the specific parameters. These results indicate that oxygen plasma treatment is reliable for facilitating lower  $\Omega$  in MC-wool, regardless of the parameters, but the mechanism does not lead to optimum low  $\Omega$ .

Oxygen plasma treatment has been shown to have an etching effect that introduces "grooves along the [wool] fibre axis" and

oxidises the wool surface, imparting hydrophilicity and encouraging the uptake of dyes and polymer adhesion.<sup>65</sup> Oxygen plasma treatment can cause transformation within wool fibre's keratin Amide I from its alpha-helical structure to beta-pleated sheet and remove the main constituent of wool's fatty acid surface, 18-methyl eicosanoic acid (MEA), uncovering the hydrophilic epicuticle and leading to increased dye uptake.<sup>19,20</sup> Disulphide bonds can also become cleaved resulting in higher cysteic acid content which reduces the wool's surface barrier.<sup>19,65</sup>

The enhanced adhesion typically observed with oxygen plasma treatment is attributed to the removal of wool's hydrophobic monolayer and surface etching, which decreases light reflection and enhances colour depth.<sup>20</sup> However, MXene behaves like a pigment dye.<sup>66</sup> Plasma treatment has been shown to enhance wool's absorption of highly water-soluble dyes and therefore dyes with lower solubility, such as pigment dyes, plasma treatment may be less effective.<sup>20</sup>

More aligned with the results of our study, Molina *et al.* (2005) found air plasma treatment to oxidise and etch the surface of the wool fibre, superior to the effects of pure oxygen plasma treatment.<sup>67</sup> Fig. 10 shows the  $\Omega$  results using Air2003030 parameters. However, these positive results derive from a single sample suggesting that the results are likely outliers.

#### Polyester results and discussion

Fig. 11 shows the main effects plot for MC-polyester. The MC-polyester analysis bootstrap specifications, full bootstrapped

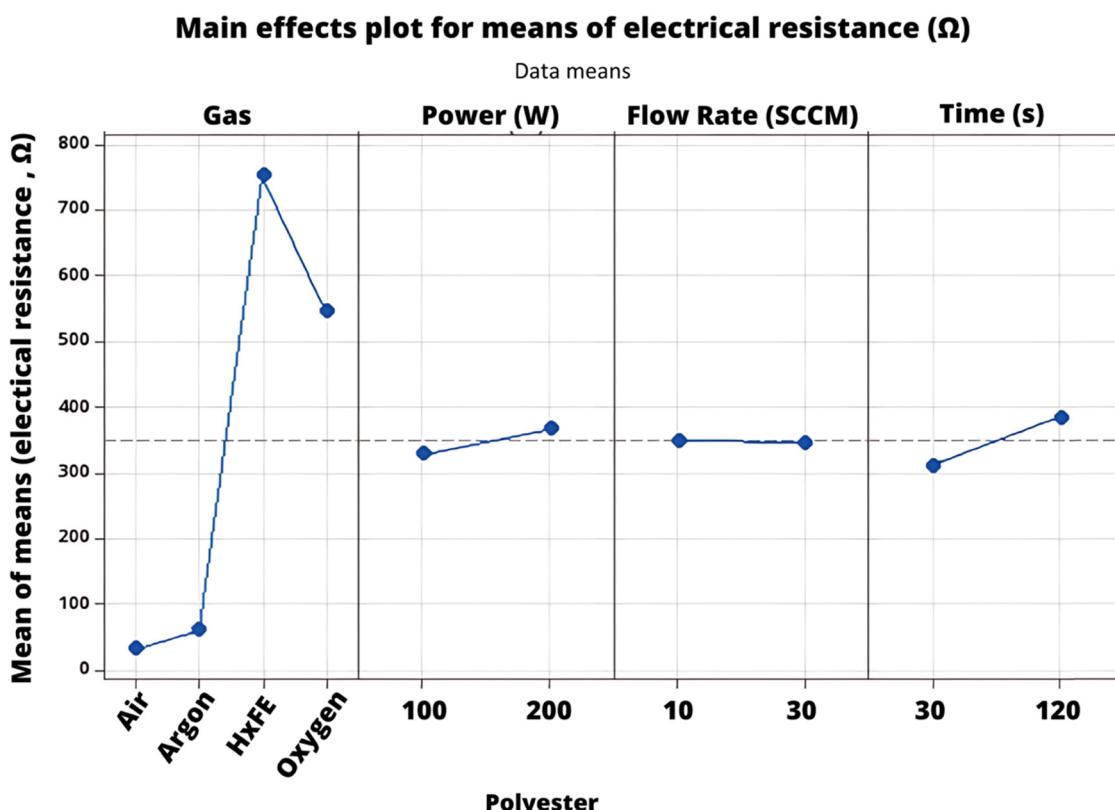
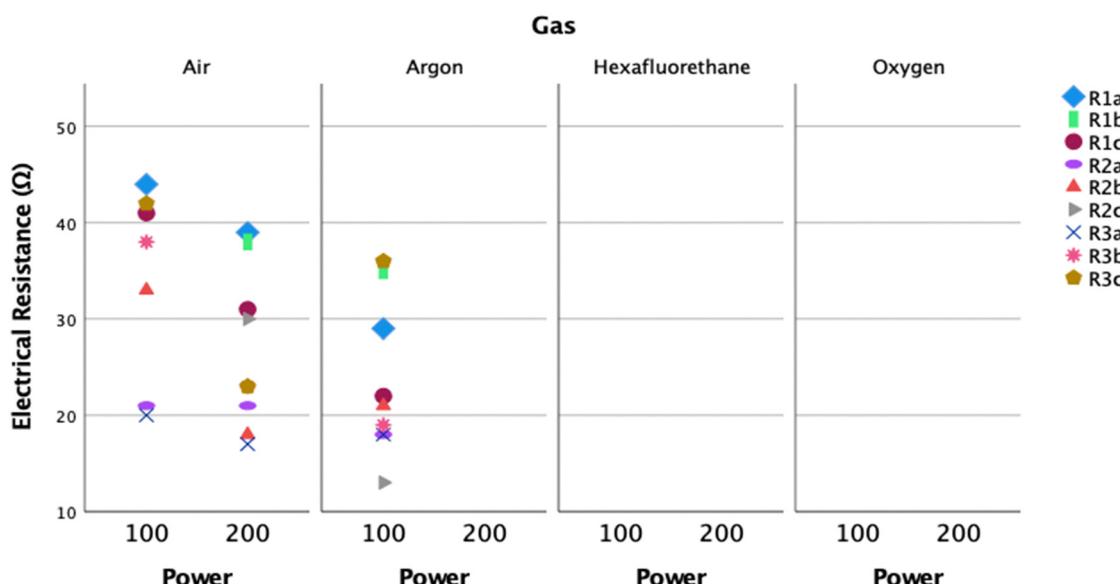


Fig. 11 Polyester main effects plot showing the significant effect of gas type and negligible effects of other parameters.



## Electrical Resistance ( $\Omega$ ) of Mxene coated polyester by gas type and power to a maximum of 50 $\Omega$



**Fig. 12**  $\Omega$  of MC-polyester by gas type and power to 50  $\Omega$ . The results here highlight that although the main effects overall are not significantly responsive to power, there are interactions which are not detected. MC-polyester's  $\Omega$  is lower under argon with low power treatment than with argon under high power treatment.

statistics, Taguchi response table for means and the signal-to-noise ratio are provided in ESI,† S28–S31. Fig. 12 illustrates the general trends of gas and power on the  $\Omega$  of MC-polyester. The statistical analyses conducted on the polyester results are shown in Table 8.

MC-Polyester without plasma pre-treatment had a median  $\Omega$  of 1437  $\Omega$ , the lowest of the control fabrics. Polyester pre-treated with air plasma exhibited a  $\Omega$  of 23–41  $\Omega$ , dependent on the

specific parameters, and argon pre-treatment led to a median of 21–105  $\Omega$ . These treatment parameters' exceptionally low variance and low  $\Omega$  make them promising routes for further development. Statistical analysis confirmed that gas type significantly affected the  $\Omega$  of MC-polyester and treatment parameters significantly influenced  $\Omega$  in air and argon-treated samples. Crucially, there is a highly significant ( $p$ -value = 0.008) reduction in the  $\Omega$  of the MC-polyester treated with the

**Table 8** Statistical tests undertaken on MC-polyester

Statistical test	Purpose	P-Value	Null hypothesis	Null hypothesis rejected?	Appendix number for full results
Mood's Median test	To explore the gas type's effect on the MC-polyester $\Omega$ .	$p = 0.046$	$H_{P10}$ : plasma gas type used has no effect on the $\Omega$ of MC-polyester.	Yes, rejected	ESI S32 and S33
Friedman test	To identify whether individual plasma parameters affect MC-polyester $\Omega$ .	$<0.001$	$H_{P20}$ : specific plasma gas treatment type parameters have no effect on the $\Omega$ of MC-polyester.	Yes, rejected	ESI S34 and S35
<i>Post hoc</i> analysis with Wilcoxon signed-rank tests	To isolate which treatment parameters affect the $\Omega$ of MC-polyester. – A Bonferroni correction was applied; thus, the significance level was set at $p < 0.0125$ .	Air and argon treatment parameters have a statistically significant effect at $p = 0.012$ , and $p = 0.008$ , respectively	$H_{P3-60}$ : the plasma treatment parameters have no effect on the $\Omega$ of [GAS] treated MC-polyester.	Yes, reject null in case of air and argon. Other gases, null retained.	ESI S36 and S37
Wilcoxon signed rank test vs. untreated MC-polyester	Arg1001030 (the most beneficial plasma parameters) vs. untreated MC-polyester.	$p = 0.008$	$H_{P70}$ : Argon plasma gas treatment with parameters (P100, S10, T30) has no effect on the $\Omega$ of MC-polyester.	Yes, null rejected.	ESI S38–S40



Argon/100/30 compared to the untreated MC-polyester control sample.

Oxygen and air plasma treatment have been shown to increase the roughness and surface energy of polyester, leading to better dye adsorption.<sup>20</sup> Our findings partially align with this trend, showing air to have a positive effect on  $\Omega$  of MC-polyester whereas oxygen treatment did not yield substantial improvements. However, as determined in previous sections, dye uptake does not necessarily correlate directly to improved  $\Omega$  since the continuity of the conductive pathway is critical and is dependent upon fabric microstructure, absorption, and bonding mechanisms.

Both air and argon are activation plasma treatments expected to break ester bonds in the polyester surface, generating radicals that can create various acidic functional groups on the surface.<sup>38</sup> These provide excellent points for hydrogen bonding or van der Waals forces between the polyester and the MXene coating. The relatively poor performance of oxygen plasma treatment in this study may be attributed to excessive surface etching, leading to a surface roughness that disrupts the conductive pathways, thereby hindering charge transport.

MC-Polyester pre-treated with HxFE plasma demonstrated a median  $\Omega$  of 762–1260  $\Omega$ , dependent on the parameters. Whilst Fig. 11 illustrates the minimal impact of parameter levels overall, the interaction plots indicated that there was a strong interaction between power and gas flow, suggesting complex dependencies which cannot be detected by the Taguchi analysis. A full factorial design could disentangle these

interactions to elucidate a more optimised setting of power and flow rate.

### Linen results and discussion

The bootstrap specification, full bootstrapped statistics are provided, Taguchi response table for means and signal-to-noise ratio in ESI,† S41–S44. Fig. 13 shows the main effects plot for MC-linen. Beyond the extreme effects of different gas types evident in Fig. 14, the substantial impacts of each plasma parameter are striking. Linen is the only fabric under test that was strongly affected by each plasma parameter. Lower  $\Omega$  was achieved with the lower power setting, higher flow rate, and longer treatment time. Interactions between parameters were minimal in linen.

Untreated MC-linen had a median of 14 300  $\Omega$ . Air and argon consistently produce lower  $\Omega$  in MC-linen. Linen pre-treated with argon plasma exhibited a  $\Omega$  of 131–277  $\Omega$ , dependent on the specific parameters, and air pre-treatment led to a median  $\Omega$  of 359–442  $\Omega$ . Conversely, linen pre-treated with oxygen plasma demonstrated a median  $\Omega$  of 2730–8900  $\Omega$ , dependent on the parameters.

The linen was exceptionally responsive to the specific parameters of oxygen plasma, favouring 100 W power, high flow rate and longer treatment time. This responsiveness is likely due to the linen's cellulosic hydroxyl groups being highly sensitive to the oxygen radicals induced by the plasma treatment. However, since the results from oxygen treatment are among the worst for  $\Omega$  of MC-linen, optimisation of this parameter may be

### Main effects plot for means of electrical resistance ( $\Omega$ )

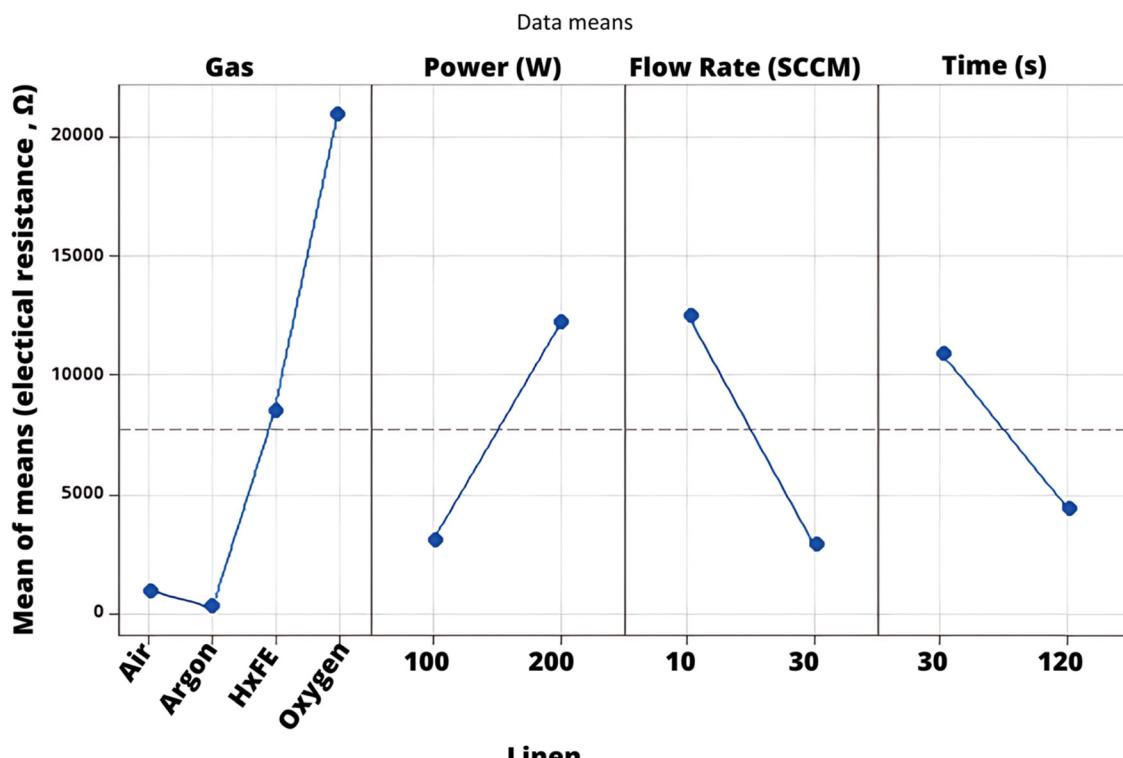


Fig. 13 Linen main effects plot demonstrating linen's sensitivity to all plasma parameters under test.



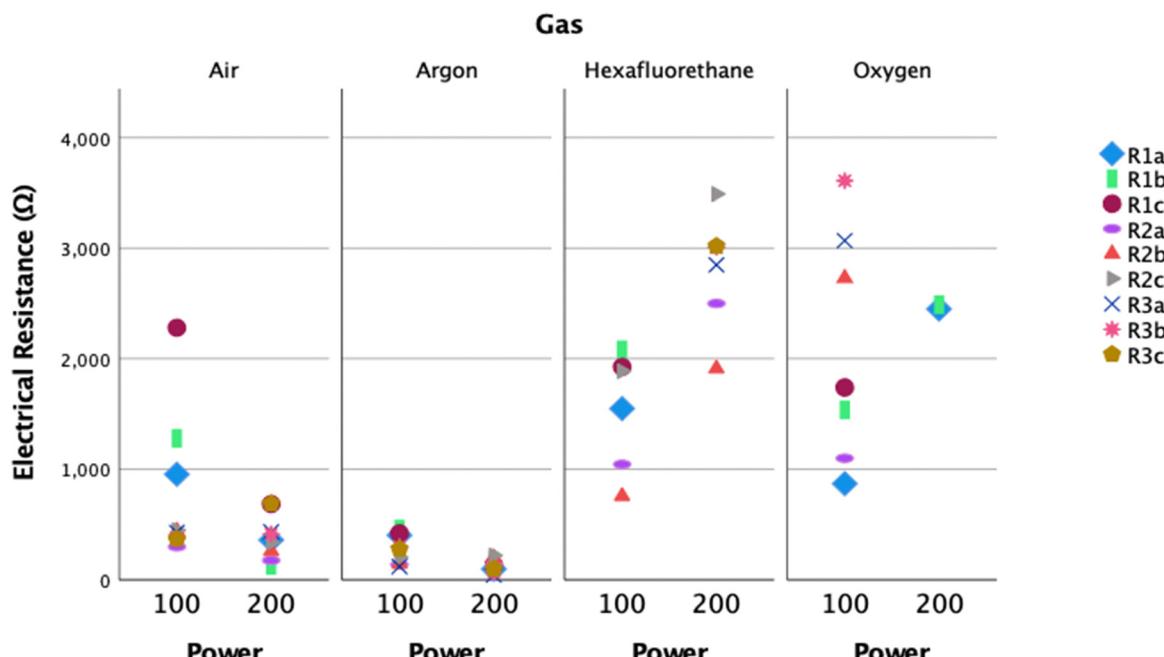
Electrical resistance ( $\Omega$ ) of Mxene coated linen by gas type and power to a maximum of 4000  $\Omega$ Fig. 14  $\Omega$  of MC-linen by gas type and power up to 4000  $\Omega$  highlighting argon's consistently positive effect on MC-linen's  $\Omega$ .

Table 9 Statistical tests undertaken on MC-linen

Statistical test	Purpose	P-value	Null hypothesis	Null hypothesis rejected?	Appendix number
Mood's median test	To explore the gas type's effect on the MC-linen $\Omega$ .	$p = 0.046$	$H_{L10}$ : plasma gas type used has no effect on the $\Omega$ of MC-linen.	Yes, rejected	ESI S45 and S46
Friedman test	To identify whether individual plasma parameters affect MC-linen $\Omega$ .	$<0.001$	$H_{L20}$ : specific plasma gas treatment type parameters have no effect on the $\Omega$ of MC-linen.	Yes, rejected	ESI S47 and S48
Post hoc analysis with Wilcoxon signed-rank tests	To isolate which treatment parameters affect the $\Omega$ of MC-polyester. – A Bonferroni correction was applied; thus, the significance level was set at $p < 0.0125$ .	Oxygen treatment parameters statistically affect the $\Omega$ at $p = 0.008$ . Argon parameter effects are notable but do not support rejecting the null hypothesis	$H_{L[3-6]0}$ : the plasma treatment parameters have no effect on the $\Omega$ of [GAS] treated MC-linen.	Yes, reject null in case of oxygen. Other gases, null retained.	ESI S49 and S50
Wilcoxon signed rank test <i>vs.</i> untreated MC-linen	Untreated MC-linen and one of the highest achieving parameters, Argon (P200, S30, T120).	$p = 0.008$	$H_{L70}$ : Argon plasma gas treatment with parameters (P200, S30, T120) has no effect on the $\Omega$ of MC-linen.	Yes, null rejected.	ESI S51–S53

ineffectual. The higher power parameter of oxygen comprises a collection of results of high  $\Omega$ .

It is clear from this graph that HxFE parameters effect the  $\Omega$  of MC-linen and that this range is comparable to the low power parameter of oxygen treatment. Fig. 13 presents the responses up to 4000  $\Omega$ , exemplifying the strong effect of argon pre-treatment for lowering the  $\Omega$  of MC-linen, particularly when the high-power parameter is used.

The results of the statistical analyses conducted on the linen are shown in Table 9. The effect of the best-performing plasma

treatment (Argon 200/30/120) was demonstrably effective at reducing the  $\Omega$  of MC-linen in comparison to the untreated MC-linen with a high significance value ( $p = 0.008$ ).

## Conclusion

The study used an L8 4<sup>1</sup>2<sup>3</sup> Taguchi experiment design to systematically investigate the impact of plasma treatment on the  $\Omega$  of MC-textile fabrics (Nylon, polyester, linen, and wool).



Table 10 Optimal plasma treatment parameters by fabric type

MXene coated fabric	Best parameter for achieving low $\Omega$				Median $\Omega$ achieved ( $\Omega$ )	Median electrical resistance of control MC-fabric
	Gas	Power (W)	Flow rate (SCCM)	Duration (s)		
Nylon	Air	100	10	120	22	7000 $\Omega$
Wool	HxFE	100	10	120	345	1000 M $\Omega$
Polyester	Argon	100	10	30	20	1437 $\Omega$
Linen	Argon	200	30	120	131	14 300 $\Omega$

This work is the first to demonstrate how fine-tuning the parameters of plasma pre-treatment of MC-fabrics can optimise the resulting  $\Omega$ . As the e-textile industry expands, particularly the growing use of MXene for textile functionalisation, these findings will provide a valuable foundation for researchers, manufacturers and developers to enhance the electrical properties of their e-textiles in a more efficient and environmentally friendly manner.

Significantly, the  $\Omega$  of each fabric type improved to a statistically significant degree under a specific set of plasma parameters. The most substantial improvement was seen in wool where the MC-control sample exhibited an  $\Omega$  eight orders of magnitude higher than that of the optimally treated sample (HxFE/100/10/120). The best results for each fabric type occurred under the conditions illustrated in Table 10. The complete set of hypotheses tested and statistical output are provided in ESI,† S54.

General trends indicated that natural fibre fabrics exhibited lower  $\Omega$  with higher flow rates and lower power settings. Synthetic fabrics tended to be less sensitive to the specific plasma parameters – except for Nylon responding favourably to longer treatment times. However, these overarching trends do not translate into universal optimisation strategies, since the optimal parameters differ significantly across fabric types. The results demonstrate that the  $\Omega$  of MC-fabrics can be precisely tuned through the strategic selection of plasma treatment parameters. Among the gases under test, oxygen plasma treatment typically yielded the least improvement, with wool the sole but notable exception to this trend. Oxygen-treated MC-wool exhibited significantly lower  $\Omega$  than the untreated control sample, highlighting the fabric-specific interaction. In contrast, Argon treatment consistently demonstrates lower  $\Omega$  in treated fabrics, except for wool. The most substantial improvements in  $\Omega$  were observed in synthetic fabrics pre-treated with argon plasma, with argon-treated MC-Nylon and argon-treated MC-polyester attaining means of 101  $\Omega$  and 90  $\Omega$ , respectively. These findings underscore the critical role of strategic parameter selection in optimising the electrical properties of MC-fabrics.

### Statistical tests

Wool was the most striking fabric under test due to its perverse behaviour compared to the other fabrics; HxFE treatment was the most advantageous. Linen was particularly sensitive to the specific plasma treatment parameters. Plasma treatment of MC-linen can lead to significantly lower  $\Omega$ , with argon treatment being the most advantageous. This was a surprising

result, given the popularity of oxygen plasma treatment for cellulosic fabrics across the literature. MC-Polyester is sensitive to the treatment parameters for argon and air but relatively indifferent to oxygen and HxFE treatment parameters. Both latter are disadvantageous for producing low  $\Omega$  in MC-polyester. There is a strong interaction between power and flow rate for MC-polyester, which is masked in the main effects analysis. However, lower  $\Omega$  can be achieved with low power and low flow rate or high power and high flow rate.

This study raises several key questions that warrant further research. Is fabric microstructure or chemical composition the dominant factor influencing the results of plasma treatment on  $\Omega$ ? To what extent does MXene diffuse through the wool cuticle under different plasma conditions? Can the variation in  $\Omega$  be solely attributed to this diffusion? A cross-sectional study could provide valuable insights into these mechanisms. Future work could explore different coating methods or potentially automate the coating process to reduce extraneous variables and increase the capacity to extrapolate the results. The results reported in the present study reiterate the need for uniformity in conductive coatings across the fabric surface to achieve high-performance conductive fabrics. Gaps in the conductive pathway cause a barrier for charge carrier transport leading to increased  $\Omega$ . Addressing these challenges will be invaluable in further advancing the practical applications of MC-e-textiles.

In summary, this work established optimised plasma treatment protocols by fabric type. The findings can benefit the large-scale production of e-textiles and the optimised use of MXene in textile applications.

### Author contributions

Ashleigh Naysmith: conceptualisation, methodology, validation, formal analysis, investigation, data curation, writing – original draft, writing – review & editing, visualisation, project administration. Tim Smith: methodology, validation, resources, writing – review & editing. Andrew Hewitt: supervision, resources, project administration, writing – review & editing. Naeem Mian: supervision, writing – review & editing.

### Data availability

The data supporting this article have been included as part of the ESI,†



## Conflicts of interest

There are no conflicts to declare.

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