UPCYCLING WASTE COTTON INTO CARBON REINFORCEMENTS FOR EPOXY COMPOSITES

MUHAMMAD AWAIS,[*] MUHAMMAD MOHSIN,[*] AAMER KHAN[*] and GIANLUCA CARCIA[**]

[*]*Department of Textile Engineering, University of Engineering and Technology, Lahore, 3.5 Km Khurrianwala, Makuana bypass Faisalabad, Pakistan*
[**]*Department of Applied Science and Technology (DISAT), Politecnico di Torino, Corso Duca degli Abruzzi, 24, 10129, Italy*
✉ *Corresponding author: M. Mohsin, muhammad.mohsin@uet.edu.pk*

Received March 14, 2026

This paper introduces a sustainable circular-economy plan of transforming waste cotton fabric into useful carbon materials through controlled pyrolysis and assesses their application as epoxy composite strengthening agent. Scanning electron microscopy and Raman spectroscopy confirmed that the semi-graphitic carbon had retained the fibrous morphology and porous surfaces of the cotton precursor. Composites with 1 wt% and 3 wt% of cotton-based carbon were compared to the composites with the commercial multi-walled carbon nanotubes (MWCNTs). Cotton-based carbon composites with 3 wt% loading exhibited about 90% greater ultimate tensile strength, 150% greater tensile toughness and a moderate increase in Young's modulus, compared to MWCNT composites. Tribological tests showed the presence of lower friction as a result of the fibrous morphology and consistent carbon-rich transfer layers. The electrical conductivity testing further indicated that carbon composites made of waste cotton came close to that of the MWCNT-based composites. These results indicate that carbon based on cotton is a low-cost, eco-friendly additive for achiving mechanically tough, electrically active, and tribologically improved polymer composites.

Keywords: carbon materials, mechanical properties, cotton recycling, waste derived composites

INTRODUCTION

The textile industry has been expanding tremendously in the past decades due to the growth of the population, changes in living standards, and the swift changes in fashion patterns.[1] This trend has been good for the economy, but has had serious impacts on the environment. One of the biggest effects is the large volume of textile debris that is produced across the world. Cotton waste is a by-product from post-consumer textiles, production off-cuts and fabrics with defects.[2] In the world, approximately 92 million tonnes of textile waste is generated annually,[3,4] of which, cotton makes a significant contribution. The current waste management system is still inefficient in the collection and recycling of waste materials, as well as in reintegrating waste into new garments.[2] Consequently, most of these wastes are disposed of in landfills or incinerated, leading to environmental problems.[1,5] In landfills, under anaerobic conditions, microorganisms slowly degrade cotton, releasing methane, a greenhouse gas that has a global warming potential (GWP) of approximately 84 times that of CO_2 over a 20-year time horizon.[6] Methane emissions from landfills are the third largest source of anthropogenic methane emissions in the world and account for about 18-20% of the total anthropogenic global emissions.[7] Although incineration reduces the volume of waste, it produces CO_2 and other harmful aerosols, such as toxic gases, particulate matter (PM2.5/PM10) and others.[8-10]

Fillers based on carbon have gained widespread popularity in the improvement of the performance of polymer composites.[11] Multi-walled carbon nanotubes (MWCNTs) are especially promising reinforcements, with individual MWCNTs having tensile strengths ranging from as low as 11 GPa up to near 100 GPa in the general literature on carbon nanotubes,[12,13] aspect ratios greater than 1000[14] and electrical conductivities from 10^3-10^7 S/m based on tube quality and dispersion.[15] Thanks to these properties, it is possible to enhance the mechanical, electrical and tribological properties of polymer composites at low filler contents.[11] The MWCNTs do tend to agglomerate via strong van der Waals forces, leading to stress concentrations and a decrease in the composite performance at higher MWCNT content.[12,16] Also, animal studies have shown that MWCNTs are associated with pulmonary inflammation, granuloma formation, and asbestos-like pathogenicity,[17,18,19] which results in strict

occupational exposure limits with NIOSH recommending 1 μg/m³ as an 8-hour time weighted average.[20,21]

Given these drawbacks, it is obvious that the search is going on for alternative reinforcement materials that are more sustainable, less risky and can deliver the same functionality. Pyrolysing waste cotton textiles to obtain carbon has become an eco-friendly alternative to the use of conventional carbon nanofillers at the nanoscale level.[22,23] The amorphous cellulose structure of waste cotton can be transformed into a porous structure of partially graphitic carbon under oxygen-limited conditions in the temperature range of 400-800 °C, as evidenced by XRD, TGA and BET analyses of the biochar produced from waste cotton.[22,24] This facilitates easier penetration of the matrix, mechanical interlocking and enhanced interfacial adhesion in polymer composites.[23,25]

The advantage of cotton-based over nanoscale fillers is being able to control particle size. Cotton derived carbon is reported to be micron-sized and has been reported to have less agglomeration effects and more uniform dispersion in polymer matrices, as compared with nanoscale fillers.[25,26] Mechanical enhancements in biochar-reinforced epoxy and related polymer systems have been reported as achieving gains of approximately 8-35% from 1-9 wt% loading of various biochar-epoxy systems[26,27] and as high as ~180% at around 4 wt% loading in a broader biochar-filled polymer composite review.[28] Percolation phenomena have been reported for electrical systems based on similar types of carbon filled epoxy composites, starting at loadings as low as 1 wt%.[16]

The evidence presented is encouraging, but the direct comparison of waste-cotton-derived carbon and commercial MWCNTs in an epoxy composite, which would encompass mechanical, electrical and tribological properties under controlled conditions at the same time, has never been reported in the literature. Most of the research reported examines the synthesis and characterization of biochar, or the mechanical properties alone, and does not include both mechanical and electrical properties under controlled conditions. Thus, there exists a gap where informed decisions are limited in the selection of reinforcement materials in engineering practice. Furthermore, the utilization of real post-consumer waste textiles as the carbon source, compared with virgin cotton, has not been primarily studied either; this is an important gap for the practical implementation of circular economy in industry.

Although the scale of micron-sized carbon cotton is different from nanoscale MWCNTs, still, a direct comparison is scientifically valid since MWCNTs are considered as the industrial standard in carbon reinforcement of polymers. The aim is not to match the particle geometry, but the focus is to compare the mechanical, electrical and tribological properties of the low cost, sustainable carbon made from waste cotton to those of MWCNTs at identical filler loadings (1 and 3 wt%). This application-oriented benchmarking sets the real potential for cotton derived carbon as an alternative to MWCNTs that is safer and more sustainable.

In this regard, this paper explores carbon that is obtained using waste cotton fabrics as a green filler in epoxy composites. Two filler loadings (1 wt% and 3 wt%) were experimentally tested and compared with commercial MWCNT-reinforced systems to determine variations in mechanical, electrical and tribological performance. This work will help valorise textile waste by proving the potential of using cotton waste-derived carbon as a high-performance and eco-friendly reinforcement, while also helping to achieve the goals of the circular economy in the composites and textile industry. The results of the presented research are likely to have a pragmatic and evidence-based input to the creation of more environmentally friendly composite materials and contribute to the overall objective of decreasing the environmental footprint of textile waste disposal.

EXPERIMENTAL

Materials and methods

Cotton waste fabrics were obtained from Masood Textile Mills (Faisalabad, Pakistan) to serve as the carbon precursor. Commercial multi-walled carbon nanotubes (MWCNTs) (30-50 nm diameter, 10-20 μm length, >95% purity) were procured from Dongguan Sat Nano Technology Material Co., Ltd., Guangdong, China, and used as the benchmark nanofiller for comparative analysis.

The pyrolysis of the waste cotton was conducted in an inert environment with the help of a KJ T1600 horizontal tube furnace (Zhengzhou Kejia Electric Furnace Co., Ltd., Zhengzhou, Henan, China). In each pyrolysis run, 100 g of waste cotton fabric was loaded into the furnace. The temperature was kept at 200 °C for the first 30 minutes, and then 400 °C for the next 30 minutes to have a full vaporization of the volatiles, followed by final carbonization at 700 °C during the last 45 minutes (optimized conditions were found in previous literature to achieve high-purity char of cotton). This is because the yield of the carbonized cotton at the final

temperature (700 °C) was about 25% (variation of 3%) relative to the mass of the carbonized residue and the mass of the original (feed stock) cotton.

The as-pyrolyzed cotton char (CC) was not uniformly dispersed in the stirring process because of its fibrous structure. Therefore, it was dry-pulverized in a high-speed pulverizer (Silver Crest SC-350G, (Neckarsulm, Germany) 25,000 rpm) for 2 min (batch size ≤50 g) to ensure a fine and homogeneous powder that could be uniformly dispersed in the epoxy and compared to the powder-form MWCNT filler.

Epoxy resin (Epilox 19 36/700 T) and epoxy resin hardener (Epilox Hardener H 10 31) were purchased from LEUNA Harze GmbH (Saxony-Anhalt, Germany). The carbon filler made from cotton was added to the epoxy filler in two concentrations 1 wt% and 3 wt% to determine the effect of filler concentration on the performance of the composite. The filler in the resin-hardener mixture was dispersed by means of a 79-1 heating magnetic stirrer (Canfort Laboratory and Education Supplies Co., Ltd., Guangdong, China) and subjected to constant agitation until it was uniformly dispersed. The ready mixtures were poured into dog-bone shape molds in compliance with ASTM D638-04 in tensile testing. The other specimens were electrical and tribological testing specimens. All the cast samples were allowed ambient curing of 24 hours, and post-curing at 60 °C for 4 hours to achieve full crosslinking. Five replicates of each experimental formulation were prepared to achieve statistical reliability and reproducibility of the data.

Characterization

Yield of cotton char

The yield of cotton char was obtained by following the formula given in Equation (1):

$$\boldsymbol{Yield}\ \% = \frac{\boldsymbol{Mass\ of\ pyrolyzed\ solid\ residue}}{\boldsymbol{origional\ mass\ of\ material}} \times \mathbf{100} \qquad (1)$$

Morphological characterization

The morphology of the subject materials was analysed using a scanning electron microscope (AIS-1800C) in the magnification range of 10,000X to 50,000X.

Raman analysis

Raman spectroscopy (Renishaw H43662 equipped with a green laser having 514 nm wavelength) was carried out to determine the structure of the subject carbon materials.

Mechanical analysis of composites

Mechanical analysis was carried out using a Testometric Material Testing instrument (M500-50AT) at a constant traverse speed of 1.0 mm/min. Tensile properties were obtained from the load-elongation curves and compared among the various samples.

Frictional analysis

The friction properties were measured on a pin-on-disk tribometer (Anton-Paar). An AISI 420 steel ball of 6 mm diameter was used with a load of 5 N to erode the composite surface. The amplitude of the ball was kept at 3.27 mm, and a linear speed of 0.185 m/s was used during the whole measurement. The friction coefficient (μ) for each sample was deduced from the ratio of frictional force parallel to the composite surface to the normal load. The friction coefficient values were plotted and compared among one another.

Conductivity analysis

The electrical measurements were performed in the microwave frequency range utilizing an open-ended coaxial sensor (Agilent 85070D) and a network analyser (Agilent E8361A). The instrument was calibrated by exposing the probe to open air (open circuit), then running a measurement in short-circuit conditions, and finally in de-ionized water. Afterward, the composite specimen was measured in the frequency range of 2~10 GHz. The electrical conductivity in S/m was deduced, plotted against the frequency for each specimen, and compared among each other.

RESULTS AND DISCUSSION

Morphology of carbon materials

SEM analysis revealed that the carbonized cotton fibers exhibited diameters ranging from 5 to 12 μm and retained their characteristic fibrous morphology after carbonization, including distinct surface serrations inherited from the precursor fibers. These surface features are expected to enhance interfacial adhesion with the polymer matrix by increasing mechanical interlocking and available surface area, consistent with previous reports.[1] In contrast, the reference MWCNTs appeared as entangled clusters or bundles, with individual tube diameters between 30 and 50 nm, reflecting their multi-walled nanostructure (Fig. 1). This comparison highlights that cotton-derived carbon fibers

maintain a macroscale fibrous architecture with serrated surfaces, which may offer improved load transfer and polymer interaction compared to nanoscale MWCNTs.

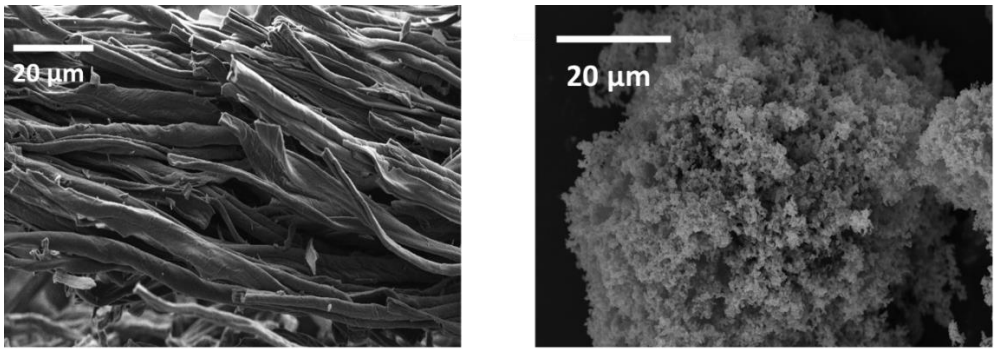


Figure 1: Morphology of (a) Cotton char, and (b) MWCNTs

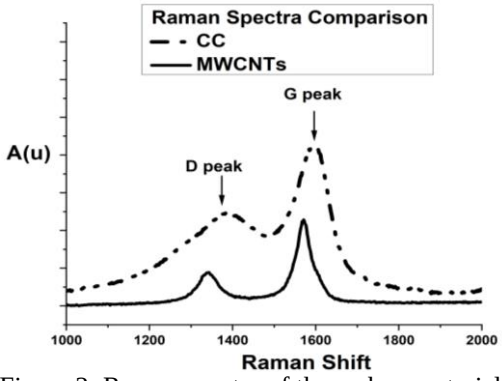


Figure 2: Raman spectra of the carbon materials

Table 1
Raman peak position and I_D/I_G ratio comparison

S.no	Sample	D-peak position (cm^{-1})	G-peak position (cm^{-1})	I_D/I_G
1	MWCNTs	1343	1571	0.59
2	Cotton char	1363	1590	0.73

Raman analysis

The Raman spectra of the MWCNTs and cotton-derived carbon are presented in Figure 2, revealing the typical characteristics of carbon-based materials. A Gaussian Lorentzian function was used to fit the spectra and calculate the I_D/I_G ratios of the integrated areas of the D and G bands. The D band is attributed to disordered C-C bonds, and the G band is attributed to ordered graphitic C=C bonds, therefore, the I_D/I_G ratio is used to indicate the extent of structure disorder. MWCNTs commercial products had sharp D and G peaks with a low I_D/I_G ratio, which means a high level of crystallinity and the absence of numerous structural defects characteristic of the nanotube morphology.

Conversely, the carbon obtained by the transformation of cotton presented wider D and G bands with slightly increased I_D/I_G ratios, indicating less structure of graphitic planes and more disorder. This semi-crystalline or partially amorphous nature could increase toughness and ductility in epoxy composites, as it is evident in the tensile performance section. Table 1 summarizes the peak values and the I_D/I_G ratios of the two materials.

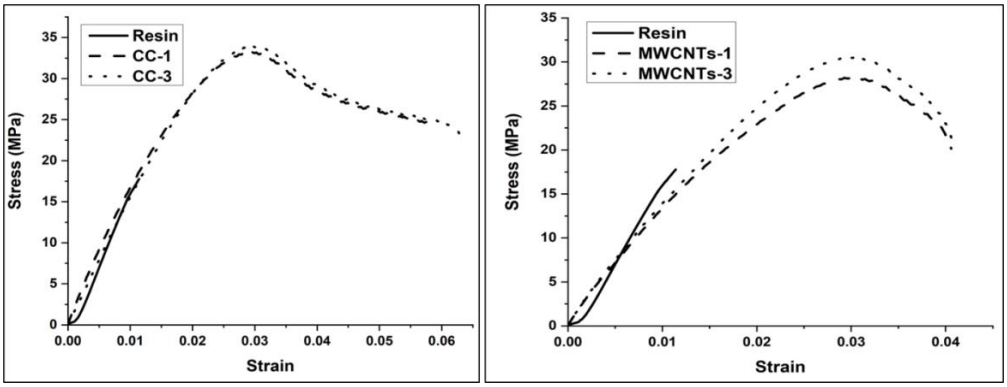
Tensile testing

The tensile properties of neat epoxy resin and carbon-filled composites are presented in Figures 3-5. The neat epoxy was brittle in nature and had breaking strength of 17.76 MPa and strain of 1.1%,

which is common to this highly crosslinked amorphous network. This behavior was significantly ductile, and its tensile strength increased nearly twofold by the addition of carbon fillers. The carbon made from cotton (cotton char) was the most effective among the formulations used in this research. It also had a maximum tensile strength of 33.92 MPa (Sample CC-3, with 3 wt% filler loading), which represented nearly 90% more than that of the neat resin and 10.5% greater than that of the MWCNT-filled composite with the identical loading (30.57 MPa).

A similar behaviour was observed in elongation at break, the CC composites reaching 6.2% strain, which is by far more than MWCNT composites did and seven times greater than neat epoxy, which is evidently a considerable enhancement in ductile properties. This shift to ductile behaviour as opposed to brittle behaviour is a sign of greater energy absorption and fracture resistance, which can be defined as the ability to take up elastic energy and regain shape, which is desirable in structural applications.

A combination of several interfacial and structural factors makes cotton char composites exhibit excellent tensile behaviour. The tensile loads exerted by char fibrous structure of cotton provide an interlacing network of reinforcing structures that effectively diffuse the stress concentration sites. The partially crystalline structure allows the formation of an ordered polymer filler interface, and the microporous surface allows the infiltration of the epoxy resin to give the highest level of mechanical interlocking.[29] These properties contribute to the fact that loads are well transferred and the possibility of voids or poor interfaces is reduced.



(a) (b)

Figure 3: Tensile testing of epoxy composites reinforced with (a) cotton char, and (b) MWCNTs

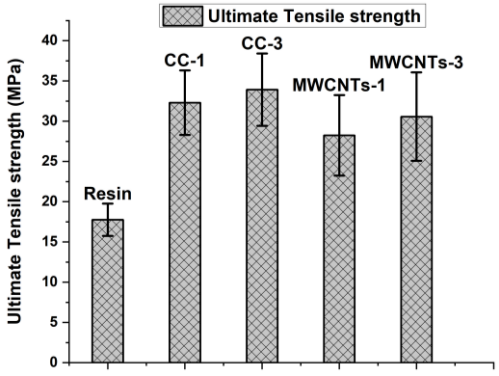
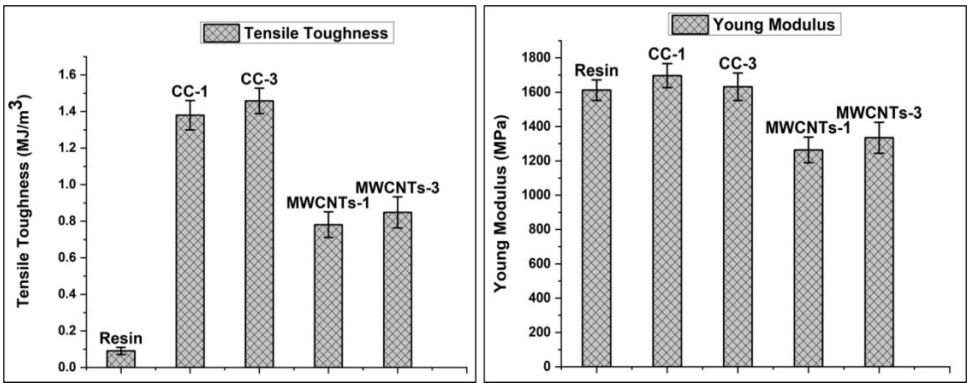


Figure 4: Ultimate tensile strength of composites



(a) (b)

Figure 5: (a) Young's modulus, and (b) tensile toughness of composites

On the other hand, MWCNTs tend to form entangled bundles and agglomerates, thus reducing the effective surface area and reinforcement efficiency. Moreover, the fibrous char forms a crack-bridging network, which blocks the crack propagation and re-distribution of the stress amongst the microcracks. The absorbed energy in the crack propagation is proportional to the area below the stress strain curve, as shown in the tensile toughness values. A loading of 3 wt% cotton char in the composite led to a toughness value of 1.45 MJ/m^3, which is substantially greater than that of the MWCNT composite (0.84 MJ/m^3) and by 150% greater than that of the neat epoxy, meaning that it has better fracture behaviour and higher energy absorption.

The addition of cotton char to the composites also increased the modulus of elasticity of the composites (Young's modulus). It enhanced the modulus of neat epoxy (1612 MPa) to 1697 MPa and 1631 MPa with 1 wt% and 3 wt% of char loading, respectively. Also, it was observed to be better than that of the MWCNT composites (1263-1335 MPa). This increment may be attributed to the fact that the high aspect ratio and the fibrous structure of cotton char assist in the uniformity of the stress distribution, good filler and matrix adhesion, and mechanical interlocking with the epoxy matrix, subsequently, removing interfacial slippage.[30] Thus, the fibrous morphology, microporosity and partial crystallinity of the char act synergistically, contributing to the enhancement of tensile strength, ductility, toughness, resiliency and stiffness of the composites.

Conductivity analysis

Polymer composites are mostly affected by intrinsic characteristics of carbon fillers, which determine their conductivity. The maximum conductivity was achieved at 0.69 S/M when using 1 wt% of MWCNTs in the composite, as shown in Figure 6. MWCNTs exhibit high conductivity that is attributed to effective charge conductivity that accompanies its tubular graphitic structure, which offers continuous conduction channels to the movement of the electrons. Unexpectedly, the conductivity of cotton char was 0.61 S/m, which was almost the same as that of MWCNTs. This result is explained by the fact that there are graphitic spaces in the cotton char, which help delocalize the electrons along the cotton char microstructure, based on its carbon-rich body to conduct charge effectively.

The electrical performance was also enhanced with the 3 wt% filler loading: MWCNT composites were found to exhibit the conductivity of 0.84 S/m and cotton char composites – the conductivity of 0.78 S/m. The reason behind this augmentation by supplementing the filler loading is largely explained by more conductive contact points and greater continuous electron pathways in the matrix, which would enhance the conductive network. A comparison of the results led to the conclusion that cotton char contributes significantly to electrical conductivity, and its performance is as good as that of MWCNTs.

The dielectric behaviour measuring the permittivity (εr) of the composites was tested within the frequency range of 2-10 GHz, using the neat epoxy matrix as control, as shown in Figure 7. The permittivity at 2 GHz in cotton char and MWCNT composites at 1 wt% loading increased by 75% and 200%, respectively. These values reached 100 and 250% for the 3 wt% loading, respectively. The higher electrical conductivity of MWCNTs and their ability to form an interlinked network in the matrix enable their composites to be superior in permittivity.[31] Still, cotton char also enhances the

dielectric properties, a feature that indicates its usefulness as a conductive filler. This characteristic suggests its potential application as an environmentally friendly substitute for conventional nanomaterials in areas where electrical and dielectric performance is important.

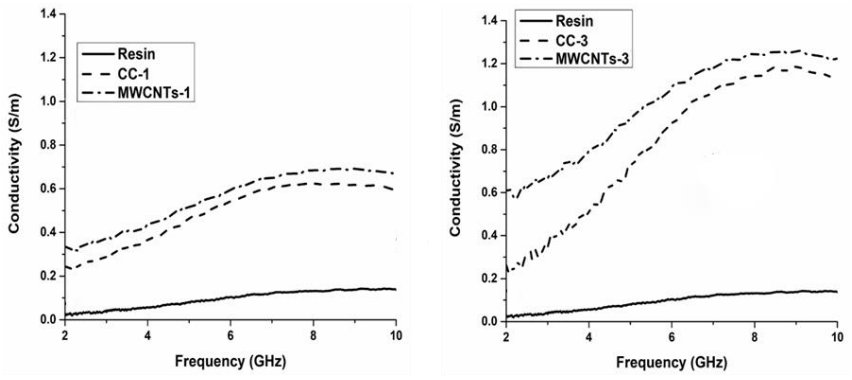


Figure 6: Conductivity of the various carbon filler composites for (a) 1 wt% and (b) 3 wt% filler loading

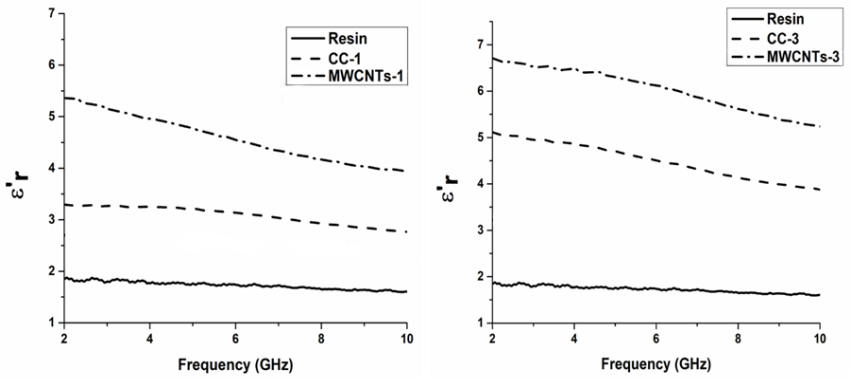


Figure 7: Real part of permittivity for (a) 1 wt% and (b) 3 wt% filler loaded composites

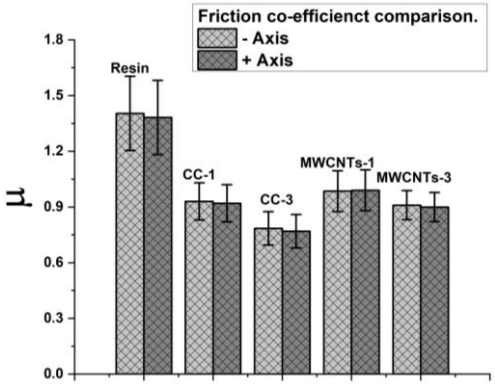


Figure 8: Coefficient of friction of the various composites

Friction analysis

Tribological performance of the epoxy composites was tested in a pin-on-disk set-up under the same testing conditions, as presented in Figure 8. The neat epoxy resin had the highest coefficient of friction, as because of its brittle nature, it could not depend on interfacial shear forces in its contact with the moving body. The introduction of carbon fillers decreased the coefficient of friction, which is a sign of better wear resistance and surface stability. Composites that were made with cotton carbon had the lowest value of friction in comparison with all formulations, especially at 3 wt% loading. The improvement is explained by the presence of the fibrous morphology and porous microstructure of cotton char, which stimulates the equal distribution of stress at the point of contact and helps create a

stable layer of transfer during sliding. The existence of a carbon-rich transfer layer will reduce the amount of direct asperity-to-asperity contact between the composite surface and the counter face, consequently, will minimize frictional resistance. Moreover, the improved mechanical integrity and toughness of cotton char composites allow maximum absorption of the stresses caused by friction, which reduces the destruction of the surface by repeated sliding. Even though MWCNT-based composites demonstrated lower friction than neat epoxy, the performance of these composites was lower than the one of cotton char composites. This action can be attributed to nanotube agglomeration and inefficient redistribution of stress under sliding loads. All in all, this study proves that the carbon derived out of cotton is an effective and sustainable filler to enhance the tribological performance of epoxy composites, without necessarily depending on nanoscale lubricating mechanisms.

CONCLUSION

This research demonstrates a viable and sustainable path of valorizing waste cotton textiles into useful carbon materials through the controlled pyrolysis and their further use as reinforcement in epoxy composites. Structural examination indicated that carbon of cotton origin maintained a fibrous, porous morphology of semi-graphitic nature, which is a major contributor to its reinforcing performance. When used as a filler in an epoxy matrix, cotton-derived carbon has been found to be greatly beneficial for the mechanical performance of the composites, specifically tensile strength, toughness and ductility, causing a noticeable shift of failure behaviour towards ductility instead of brittleness. The composites with a filler loading of 3 wt% showed a nearly 90% increase in ultimate tensile strength and 150% enhancement in tensile toughness, compared to neat epoxy. Also, they showed better performance than MWCNT-reinforced composites in terms of energy absorption and fracture resistance. The increased mechanical response can be explained by the good stress transfer properties, crack-bridging, and high interfacial adhesion of the fibrous and porous structure of the cotton-based carbon. Moreover, tribological experiments revealed a significant decrease in the coefficient of friction, which indicates that cotton-derived carbon can be applied to the surface to create stable transfer layers and increase the level of wear resistance. Electrical conductivity testing further indicated that carbon composites made of cotton came close to that of the MWCNT-based systems, indicating the formation of uniform conductive circuits throughout the matrix. Based on the results, waste cotton-derived carbon can be a competitive and sustainable alternative to traditional carbon nanofillers, which would enable the valorization of textile waste and the advanced polymer composite applications, incorporating the idea of the circularity.

ACKNOWLEDGEMENTS: The authors would like to acknowledge the lab technician Mr. Imran Shahid for performing the SEM and Mr. M. Maqsood Ahmad for the support during the experimental work.

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