SYNTHESIS OF CARBON BLACK FROM POST-CONSUMER TEXTILE WASTE AND ITS APPLICATION IN COMPOSITES

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Received July 2, 2025

Considering the worldwide trend of embracing recycling for a greener future, this study transforms cotton-based textile waste into eco-friendly carbon black. It reports an innovative synthesis of carbon black from 100% cotton-based post-consumer textile waste by pyrolysis in an inert nitrogen environment at three temperatures. The synthesized carbon black was applied as a reinforcing material in epoxy resin composites in five different concentrations. Commercial carbon black N-660 was used as a benchmark material. The best results were exhibited by carbon black when synthesized at 950 °C. Morphology analysis confirmed the agglomerate formation of commercial carbon black and the dispersed nature of the prepared carbon black. Raman analysis confirmed defect reduction and increased graphitization of carbon black by increasing pyrolysis temperature. Also, the epoxy composites exhibited improved mechanical properties after the addition of the cotton-based carbon black. Overall, the post-consumer textile waste was successfully converted into valuable textile composites with enhanced properties.

Keywords: post-consumer textile waste, pyrolysis, carbon black, upcycling, composites, mechanical properties

INTRODUCTION

Growing environmental concerns and the pressing need for sustainable practices have made it imperative to investigate environmentally friendly alternatives in many industrial sectors.[1] The creation of sustainable materials stands out among these industries as a hub for innovation, propelled by the need for long-term solutions. Carbon black, a black colour pigment used in inks, is a ground form of amorphous carbon. Due to its special qualities, carbon black is a highly adaptable material that is used in different sectors. It improves the conductivity and durability of goods like tires and vehicle parts as a vital reinforcing agent. The durability and efficacy of various rubber compounds are guaranteed in the rubber industry by carbon black's ability to increase tensile strength and abrasion resistance. Concurrently, it serves as a pigment and a filler in plastics, electrical cables, and packaging materials to impart color as well as conductivity, and UV resistance to them. Statistics show that the carbon black market grew to USD 24.10 billion in 2024, and with a compound annual growth rate of 5.75%, it will grow to USD 31.87 billion in 2030.[2]

Traditionally, carbon black is obtained from hydrocarbons, such as coal tar, ethylene cracking tar, and vegetable oils through incomplete combustion or pyrolysis under controlled conditions, compromising the sustainability of the environment, as these precursors are petrochemical based. Various methods can be used for their production, *i.e.*, furnace black, lampblack, channel black, and thermal black. Carbon black manufacturers are prompted to increase production because of the growing demand in various sectors. However, the main obstacles to meeting the market demand for carbon black are the precursor's availability, cost, and environmental implications. Above all, severe weather variations and a shortage of potable water are direct results of environmental contamination. The carbon footprint of traditional carbon black synthesis techniques highlights the need to find more environmentally friendly alternatives. Hence, recycling of carbon black is highly desirable.

On the other hand, the textile industry is growing tremendously. The demand for apparel and clothing is constantly increasing because of the growing population and the general interest in

fashion. Consequently, this sector is generating $1.2 trillion per year.[3] The literature estimates that the world consumes 62 million tons of textiles annually, which, by 2030, is expected to jump to 102 million tons.[4] Because of the use of environmentally harmful and unsustainable processes, this industry is severely harming the environment, thus contributing to pollution in all areas. The massive amount of post-consumer textile waste is increasing annually as the fashion industry is expanding. Certain approaches are available for recycling textile items, *i.e*., biodegradation, shredding techniques, and the recovery of synthetic fibers' monomers, but these are often impeded by the low final product quality or other limitations, such as fabrics' fiber blend composition. Consequently, there is still no long-term comprehensive recycling option for the enormous volume of post-consumer textile waste, especially for a good final quality product. Currently, only 1% of the textile waste is close loop recycled, while the rest either ends up in landfills, is burned or results in low-quality products.

Under these circumstances, using textile waste offers a viable path toward the production of sustainable carbon black. Textile waste, which results from the textile industry's manufacturing operations and consumer disposal, poses a considerable environmental burden. Worldwide brands and consumers are pushing the textile industry to adopt sustainable methods like recycling. This will reduce the environmental impact of textile waste by converting it into a high-value materials, for example, by using it as a feedstock for carbon black synthesis. Certain precursors, including lignite and biomass have been used to create carbon black.[5,6]

This study examines the viability and effectiveness of producing environmentally friendly carbon black from 100% cotton-based post-consumer textile waste and using this material in composites. This strategy aims to reduce energy and carbon emissions while simultaneously maximizing the value of textile waste by utilizing environmentally friendly synthesis of carbon black through a pyrolysis process at three temperatures.

The objective of this research paper is to replace commercial carbon black with a sustainable and recyclable material. Post-consumer textile waste is a serious problem for the environment. On the other hand, the synthesis of commercial carbon black is not an environmentally friendly process, as it is synthesized from hydrocarbons and certain less ecofriendly ingredients. This research involves upcycling 100% cotton-based post-consumer textile waste into carbon black through pyrolysis process at temperatures of 750 °C, 850 °C and 950 °C, and its characterization through SEM, Raman, and FTIR analyses, as well as the evaluation of its reinforcing capability in epoxy composites when used in 1%, 2%, 3%, 4% and 5% concentration. Commercial carbon black N-660 (CC) is also used as a benchmark material. The hypothesis behind this research is that carbonization and graphitization of carbon black is increased at high pyrolysis temperatures,[7] which causes reduction in defects and yields carbon black having better reinforcing capabilities. The novelty of this research is the synthesis of carbon black based on post-consumer textile waste and its application to epoxy composites as a reinforcing material. This research offers not only a sustainable solution for handling post-consumer textile waste, but also provides a valuable reinforcing material to be used in epoxy resin composites. This research aims to promote sustainable practices in material science and increase environmental stewardship by illuminating the possibility of environmentally friendly carbon black synthesis from textile waste for composite developments.

EXPERIMENTAL

Materials

100% cotton-based textile waste was obtained from Interloop Ltd., Pakistan. Epikote Resin 3.57 (based on bisphenol) and hardener were purchased from Hexicon, Sweden. Urea was obtained from Archroma Ltd., Pakistan. Commercial carbon black N-660 was obtained from Service Tires Ltd., Pakistan.

Pyrolysis of cotton-based textile waste

Textile waste material was cut into 100-gram samples. It was washed with distilled water and left to air dry for four hours at 105 °C in the oven. Afterward, this waste was pyrolyzed in a silica glass tube of a horizontal tube furnace (KJ–T1600) Zhengzhou Kejia Furnace Co. Ltd., China, available at the Recycling Laboratory of the Textile Department. Three different pyrolysis temperatures: of 750 °C, 850 °C and 950 °C, were assessed with the rate of 5 °C/min of heating. Pyrolysis was conducted in an inert nitrogen atmosphere. Nitrogen was provided in a controlled amount using a metered pump. Afterward, samples were allowed to cool at room temperature and collected from the furnace tube. Then, they were converted into powder using a Roll ball mill (QM-55), TENCAN, China, using a ball milling technique.

For calculating the yield of carbon black, an electric fine weighing balance was used. Scanning electron

microscopy (SEM), Raman, and Fourier transform infrared (FTIR) spectroscopy were performed on carbon black particles.

Composites preparation

Epikote Resin 3.57 (based on bisphenol) and hardener were used to prepare composites. A control sample was prepared using epoxy resin (E.R.) and hardener in a 5:1 ratio. The dispersion of the prepared carbon black was made by adding it to epoxy resin and hardener for all composite formulations in a concentration of 1% ~ 5% by mixing and stirring with a magnetic stirrer. The recipe for the composites is exhibited in Table 1. Then, they were placed in a silica mold, and dog-bone-shaped samples were prepared. The mixture was left at room temperature for one day to settle in the mold and subsequently cured at 70 °C for 3 hours in a precision oven (Y-902), Fangyuan Instrument (DG) Co., Ltd., China, available in the laboratory. Commercial carbon black N-660 was used to prepare benchmark samples in the same concentration range and by the same recipe. After that, the composites were carefully removed from the mold, and testing procedures were carried out for further analysis, *i.e.*, morphology and mechanical analyses. The concentration of carbon black in composites is presented in Table 1. The whole process is schematically represented in Figure 1.

Characterization

The prepared carbon black and composites were subjected to the analysis explained below.

Yield of carbon black

Utilizing the formula provided below in Equation (1), the yield of carbon black produced at various temperatures was calculated:

Yield % = (Mass of pyrolytic solid residue/ Original mass of specimen) *100 (1)

Raman analysis of carbon black

The Raman spectroscope (H-43662), Renishaw, United Kingdom, was used to study the structural properties of prepared carbon black. The green laser source employed had a 514 nm wavelength.

Table 1
Denotation of samples

S.R. No	Sample code	Material	
1.	E.R.	Epoxy resin	
2.	CC	Commercial carbon black	
3.	CC-1%	Composite reinforced with 1% commercial carbon black	
4.	C 750-1%	Composite reinforced with 1% cotton-based carbon black synthesized at	750 °C
5.	C 850-1%		850 °C
6.	C 950-1%		950 °C
7.	CC-2%	Composite reinforced with 2% commercial carbon black	
8.	C 750-2%	Composite reinforced with 2% cotton-based carbon black synthesized at	750 °C
9.	C 850-2%		850 °C
10.	C 950-2%		950 °C
11.	CC-3%	Composite reinforced with 3% commercial carbon black	
12.	C 750-3%	Composite reinforced with 3% cotton-based carbon black synthesized at	750 °C
13.	C 850-3%		850 °C
14.	C 950-3%		950 °C
15.	CC-4%	Composite reinforced with 4% commercial carbon black	
16.	C 750-4%	Composite reinforced with 4% cotton-based carbon black synthesized at	750 °C
17.	C 850-4%		850 °C
18.	C 950-4%		950 °C
19.	CC-5%	Composite reinforced with 5% commercial carbon black	
20.	C 750-5%	Composite reinforced with 5% cotton-based carbon black synthesized at	750 °C
21.	C 850-5%		850 °C
22.	C 950-5%		950 °C

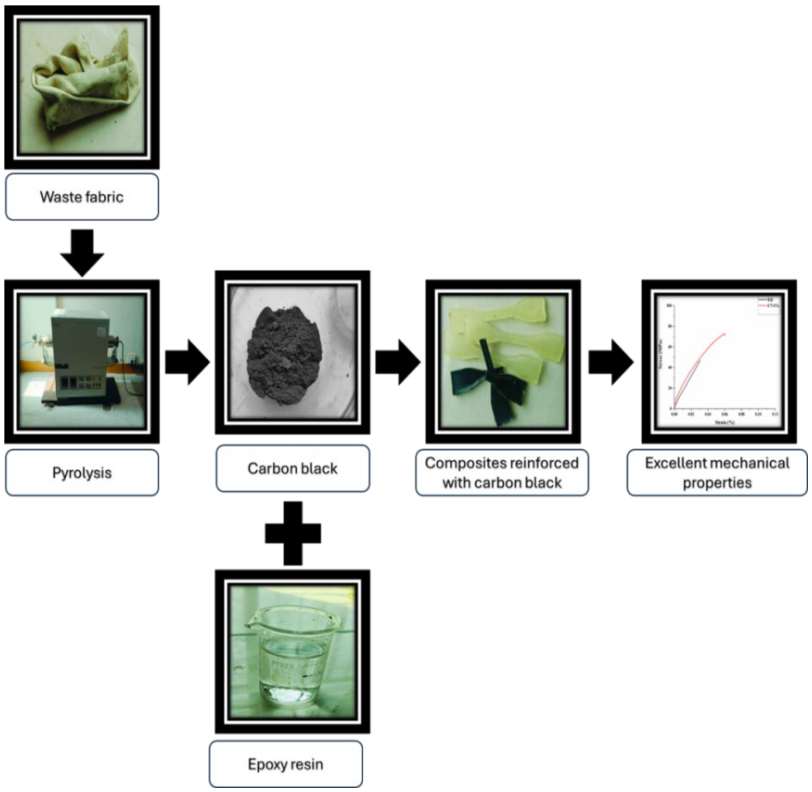

Figure 1: Schematic representation of the preparation process

FTIR analysis of carbon black

A Fourier transform infrared spectroscope (Equinox 44), Bruker, Germany, was used to examine the chemical composition of various functional groups found on the surface of carbon black. The spectra were recorded in the frequency range from 400 to 4500 cm^{-1}, at the resolution of 2 cm^{-1}. FTIR data were ATR corrected through Poor Man's ATR correction formula given in Equation (2):

$$A_{ATR} = A \times (\nu_o / \nu) \quad (2)$$

where A_{ATR} = ATR corrected absorbance, A = measured absorbance, ν= actual wavenumber (cm^{-1}) of each data point, ν_0= reference wavenumber (a fixed value used as normalization point).

Morphological analysis of carbon black and composites

The synthesized carbon black's morphology and surface topography were examined using a scanning electron microscope (AIS-1800C), Seron Technologies Inc., Korea.

The carbon black was sputtered with gold to improve the conductivity. Scanning electron microscopy was also used to examine the morphology of carbon black reinforced composite materials. To halt molecular mobility, the epoxy composites were carefully broken into fragments in liquid nitrogen. The pieces were then coated in gold using a sputter coater to stop the charging effect during SEM analysis.

Mechanical properties of composites

Tensile testing equipment (M500-50 AT), United Kingdom, was used to test composite materials mechanically by applying the standard test method specified in ASTM D-638. Dog bone shape samples, with dimensions of 115 mm × 25 mm, were prepared following the test technique. The jaws were moved at a rate of 5 mm/min.

RESULTS AND DISCUSSION

Yield of carbon black at different pyrolysis temperatures

Table 2 shows the yield percentage of synthesized carbon black. A yield of 25% was obtained for carbon black synthesized at 750 °C (C 750), of 21.5% – at 850 °C (C 850), and 19.5% – at 950 °C (C 950). According to the results obtained, when the precursor was exposed to high temperatures, more bonds broke because of the release of higher volatile matter content, gases, and oil as compared to low pyrolysis temperatures, due to thermal cracking. As a result, a low percentage of carbon black was obtained.

Various scientists reported similar results, indicating higher bond breakage, with higher removal of oils and gases, and thus, a lower amount of residue.[8] It could be explained by the fact that high temperatures might have provided more kinetic energy to break organic bonds.[9] During the

process of thermal decomposition, kinetic energy of polymeric chains of 100% cotton-based textile waste increases. At the molecular level, vibrations of chains also intensify by increasing kinetic energy along with rotational motions. This increases the chances of cleavage of C-C, C-O and C-H bonds and results in acceleration of primary reactions, such as decarboxylation and dehydration.[10] Secondary reactions are also accelerated by this kinetic energy, which removes oxygen containing functional groups. The rearrangement of molecular chains is facilitated by this elevated kinetic energy at high pyrolysis temperature, which results in increased aromatization and graphitic ordering. This mechanism could be the reason that carbon black synthesized at high temperatures of pyrolysis (950 °C) shows higher graphitization, low structural defects and superior reinforcing capabilities, when used as filler in epoxy composites.

Depending on the temperature used for pyrolysis, a blend of three different species: char, bio-oil, and biogas, is commonly generated, which is mentioned in Table 2. Pyrolysis performed at lower temperature produces carbon black with comparatively large amounts of oxygen containing functional groups, poor graphitic ordering and comparatively low yield of carbon.[11] When this carbon black is used in composites as a reinforcing material, it is less effective.[12] In this research, the aim was to obtain high quality graphitized carbon black with less structural defects, which could provide high strength to epoxy resin composites, and this could be obtained by pyrolysis at high temperature. This idea is also in line with reported literature.[13] Therefore, this is the reason behind selecting these high pyrolysis temperatures – for ensuring complete carbonization of cotton-based post-consumer textile waste, removal of volatile organic content with aromatization and dehydrogenation, and structural ordering of cotton-based carbon black similar to that of commercial carbon black. Thus, we were able to investigate the impact of higher temperatures on the reduction of defects and graphitization in a systematic way, and then confirm the results by Raman analysis.

In this work, post-consumer textile waste was pyrolyzed, starting from ambient temperature to final pyrolysis temperatures, *i.e.* 750 °C, 850 °C, and 950 °C. It is imperative to distinguish the thermal stages during this pyrolysis process. Initially, when temperature rises, primary devolatization occurs, which involves depolymerization, dehydration and generation of oxygenated volatile matters from 200 °C to 600 °C.[14] Afterwards, high pyrolysis temperature favors aromatization, dehydrogenation and tar cracking, along with reorganization of structure towards graphitic/polyaromatic ranges. Therefore, when yields of carbon black synthesized at 750 °C, 850 °C and 950 °C were compared, it was observed that secondary reactions might have occurred and removal of non-condensable gases (*e.g.*, CO_2, CO, CH_4, H_2) enhanced as compared to initial depolymerization of cellulose.[15]

Moreover, volatile matter refers to the sum of volatile fractions, produced during the process of pyrolysis (including permanent gases and condensable vapors). On the other hand, pyrolytic oil is the condensable liquid fraction, which is obtained after condensation of vapors. So, changes in volatile matter are not the same as changes in the yield of oil as non-condensable gases can be released as volatile matter. Literature shows that the yield of biochar and bio-oil decreased by increasing pyrolysis temperature,[16] and that of gas increased by increasing pyrolysis temperature. The percentage yields mentioned in **Error! Reference source not found.** are in accordance with the literature.[17,18] The yield of carbon black is reduced by increasing pyrolysis temperature. Also, the difference between 25% and 19.5% is relevant. However, higher pyrolysis temperature was chosen to obtain carbon black of high quality, having enhanced structural and functional characteristics. Carbon black synthesized at 950 °C exhibited the lowest yield among all, but it exhibited lower structural defects and high graphitization. In addition, it was in single particle form, instead of clusters of agglomerates, and it exhibited enhanced reinforcing capabilities when used as filler in epoxy resin composites when compared to carbon black synthesized at lower pyrolysis temperatures. Therefore, the slight decrease in yield was compensated by the high quality of the carbon black (with lower structural defects and enhanced graphitization). Also, spending extra energy to reach and maintain the higher processing temperatures produced superior carbon black, which was suitable for superior performance.

Table 2
Yield of carbon black synthesized at different temperatures

Sample	Pyrolysis temperature	Percentage yield of carbon black	Bio-oil
C 750	750 °C	25.0±1%	30±1%
C 850	850 °C	21.5±1%	21±1%
C 950	950 °C	19.5±1%	8±1%

Raman analysis of carbon black

Raman analysis of commercial carbon black and cotton-based carbon black synthesized at 750 °C, 850 °C, and 950 °C was performed, and the results are exhibited in Figure 2. In Raman analysis, the D-peak exhibits defects in the material, while the G-peak exhibits graphitic content in the material made of carbon, which is associated with the sp^2 coordination of materials. The origin of the D peak is the boundary zones that are present in sp^2-coordinated regions. The G-peak is the representative of the stretching of in-plane sp^2 carbon bonds. Fitting of G and D-peaks was performed with the mixed Gaussian-Lorentzian shape (GauLor) function for estimation of the area, which was present under both peaks. The I_D/I_G ratio is exhibited in Figure 2.

Commercial carbon black had a considerable number of defects and less graphitization. Carbon black synthesized at 750 °C exhibited an I_D/I_G ratio of 0.92. By increasing the pyrolysis temperature of carbon black, D peaks became slightly less broad, and G peaks became sharper, indicating lower defects and a more ordered structure.[19] A shift in D peak from 1360 cm^{-1} to 1356 cm^{-1} was observed in the case of cotton-based carbon black when pyrolysis temperature increased from 750 °C to 950 °C. This decrease in vibrations was the indication of few amorphous regions in cotton-based carbon black. It could be because of the removal of defects, *i.e*., C-H, C-O groups, when the secondary pyrolysis process might have taken place. Comparing carbon black pyrolyzed at 850 °C and 950 °C to carbon black pyrolyzed at 750 °C, the G band shrank from 1583 cm^{-1} to 1581 cm^{-1}. This decline is correlated with the temperature at which pyrolysis occurs. When pyrolysis temperature increases from 750 °C to 950 °C, the removal of oxygenated function groups might have occurred, and the structure of carbon black might have become more aromatic.[15] The graphitization of the material is achieved as the temperature rises. These results are in line with those reported in the literature.[20]

The ratio of I_D/I_G also decreased with increasing pyrolysis temperature. It could be because of the expulsion of inorganic compounds,[21] and increased graphitization of carbon black at higher temperatures. Carbon black synthesized at 850 °C exhibited an I_D/I_G ratio of 0.90, while carbon black synthesized at 950 °C possessed an I_D/I_G ratio of 0.82. At higher temperatures, healing of defects and rearrangement of carbon black into a more stable structure may have resulted in fewer defects.[22] Defect reduction leads to improved mechanical properties of epoxy composites reinforced with carbon black by making stronger bonds with epoxy resin. Carbon black synthesized at 950 °C exhibited slightly a lower I_D/I_G ratio as compared to commercial carbon black, which means it has fewer structural defects and high graphitic order. From this, it can be concluded that secondary reactions, *i.e*., aromatization and deoxygenation, could be promoted by high temperature pyrolysis,[15] which might have resulted in more ordered sp^2-hybridized carbon domains in C 950.

FTIR analysis of carbon black

The presence of different functional groups on the surface of carbon black is exhibited in FTIR spectra. Figure 3 illustrates the decrease of oxygen and hydrogen-containing functional groups as the pyrolysis temperature increased. Functional groups were represented by the peak at the wavenumber of 3361 cm^{-1}, indicative of hydroxyl group-containing compounds, and C-H groups were represented in the range of 2860 cm^{-1} to 3000 cm^{-1}, characteristic of cotton species.[23] The peak at the wavenumber of 1136 cm^{-1} is related to the C–O bond, which is present in the spectra of cotton.[24] The peak at the wavenumber of 1675 cm^{-1} was an indication of bending vibration of H–O–H in the spectrum of cotton.[25] The peak that appeared in the spectrum of C 750 at the wavenumber of 1002 cm^{-1} was related to alkoxy compounds that contained C-O bonds.[26] At higher temperatures, this peak disappeared, which meant that oxygen-containing functional groups could be removed at higher

temperatures; that's why the spectra of C 850 and C 950 did not show this peak. CC did not have any functional group on its structure, so no peak was exhibited by it. The peak, which appeared in the wavenumber range from 1531 cm^{-1} to 1672 cm^{-1}, was related to C=C stretching vibrations in the spectrum of C 750, also confirmed by literature.[27] The spectra of C 850 and C 950 did not exhibit that peak, which means that these functional groups could be removed at higher temperatures from carbon black.

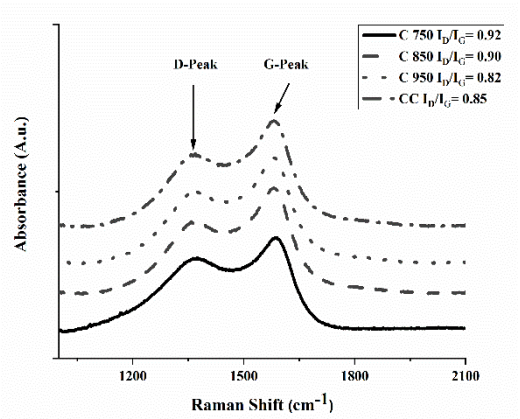

Figure 2: Raman spectra of commercial (CC) and cotton-based carbon black synthesized at different temperatures

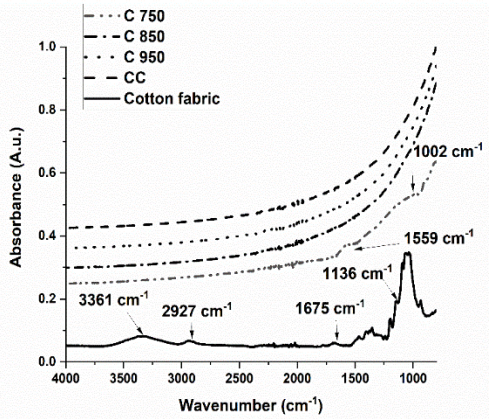

Figure 3: FTIR spectra of cotton, commercial, and cotton-based carbon black synthesized at different temperatures

Morphological analysis of carbon black and of composites

Morphological analysis of two types of carbon black, *i.e.*, commercial carbon black N-660 and cotton-based carbon black, was performed. This analysis aimed to compare carbon black's physical structure and evaluate its potential applications in composites. Results revealed that commercial carbon black was spherical, with a diameter of around 200 microns, as shown in Figure 4 (a). This regular spherical shape is a characteristic attribute of commercial carbon black, which influences its interaction with other materials, *e.g.*, with epoxy resin in the case of composites. Morphology analysis confirmed the formation of agglomerates of particles because of the presence of van der Waals forces.[28] Because of the formation of agglomerates of commercial carbon black, it might not have adhered well to the epoxy resin and exhibited less load-bearing capacity in composites,[29] which will be discussed in detail below. These agglomerates also behaved as a weak point in the structure of epoxy composites, which might have impacted the mechanical properties of composites negatively. Further additional trials were conducted to increase dispersion of benchmark material, *i.e.*, commercial carbon black. Ball milling was performed under similar conditions. It indicated that the particles of commercial carbon black had strong van der Waals forces, which were present even after mechanical processing. This is also evident from the literature.[30]

On the other hand, cotton-based carbon black had no definite shape, as exhibited in Figure 4

(b). Its diameter was around 200 microns, but unlike commercial carbon black, cotton-based carbon black presented dispersed particles and had a high specific surface area. It could be due to the breakdown of the original structure of cotton at high pyrolysis temperatures. FTIR analysis confirmed the elimination of functional groups at high pyrolysis temperatures and supported these morphological changes – the disappearance of functional groups led to the undefined shape of particles of carbon black. Therefore, the difference between the formation of agglomerates, in the case of commercial carbon black, and the separate particle nature of carbon black from post-consumer textile waste remains valid. Owing to its separate-particle state, the adhesion of cotton-based carbon black with epoxy resin might have increased, which might have dissipated stress, thereby increasing the load-bearing capacity of composites. Moreover, the irregular morphology of cotton-based carbon black facilitates its interlocking within the matrix of epoxy. Higher surface area is provided by this morphology for the epoxy to physically adhere to the filler. This leads to stronger interfacial adhesion, which improved

the mechanical properties of the composites, as also evident from literature.[31] When load is applied, this morphology contributes to dissipating stress. To interpret the performance of composites, this competing impact was considered.

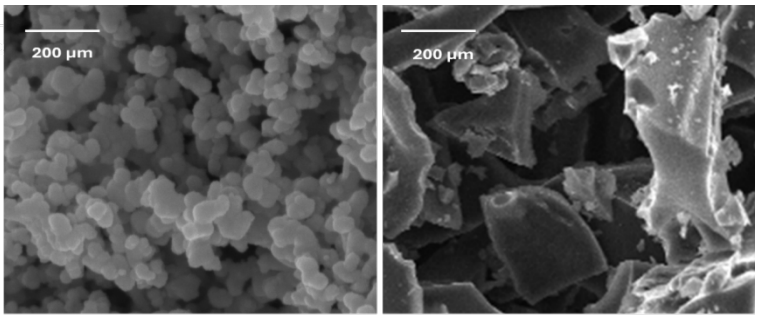

Figure 4: Morphology of (a) commercial carbon black (CC), and (b) cotton-based carbon black C 950

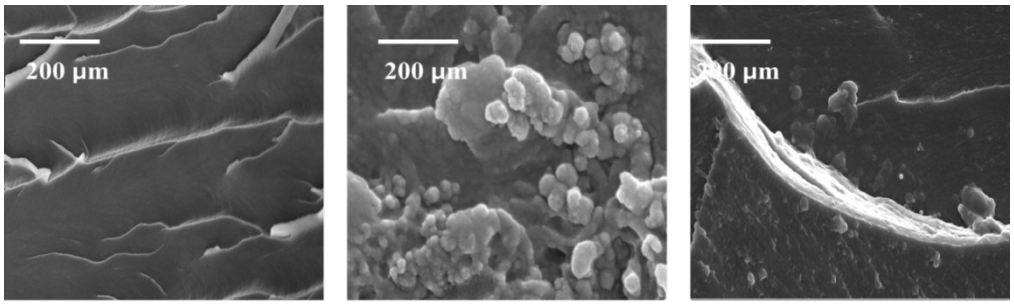

Figure 5: Morphology of (a) epoxy resin, (b) CC-4% composites, and (c) C 950-4% composites

The morphology of neat epoxy, composites with commercial carbon black, and cotton-based carbon black was analyzed, and the results are exhibited in Figure 5. According to the results, neat epoxy was in a layered form, as shown in Figure 5 (a). Commercial carbon black formed agglomerates and did not disperse well in the epoxy matrix, as shown in Figure 5 (b). These agglomerates of commercial carbon black created stress concentration points and did not dissipate stress uniformly upon application, for this reason, these composites exhibited inferior mechanical properties. On the other hand, cotton-based carbon black dispersed into separate particles due to the removal of inorganic matter and other functional groups, as shown in Figure 5 (c). It dispersed uniformly in the matrix and helped in micro-mechanical interlocking of particles with epoxy resin; therefore, it distributed stress uniformly from the matrix to particles. It prevented slippage of layers when stress was applied and resisted crack propagation, thereby exhibiting improved mechanical properties.[32] FTIR analysis further confirmed the removal of functional groups.

Comparatively smaller particles of the prepared carbon black were observed in Figure 5 (c). The partial fragmentation of cotton-based carbon black could be the possible reason for the breakdown into smaller particles during mechanical mixing. During pyrolysis at high temperature, volatile organic matter and species other than carbon might be removed from carbon black, leaving behind a less compact and more porous structure.[33] When shearing force was applied, carbon black could be broken down into small and fine particles. This might have enhanced the uniform dispersion of particles within the epoxy resin matrix, resulting in high mechanical properties of epoxy composites.[34] Moreover, these small particles exhibited highly graphitic structure, as observed by Raman analysis, which might have ensured that mechanical integrity and reinforcing capability were not compromised. In addition, interfacial contact might have improved,[35] which contributed to increasing the mechanical properties of the epoxy composites. The composites with commercial carbon black and cotton-based carbon black were produced under strictly identical conditions for ensuring fair comparison. The dispersion of both fillers was ensured using a mechanical stirrer under heating for achieving uniform distribution of filler particles. Further stirring and mild sonication was

performed during preparation of composites with commercial carbon black. It was observed that there was no significant difference in mechanical performance or quality of dispersion, which confirmed the agglomerate formation of carbon black as its intrinsic morphology.

Analysis of mechanical behavior of composites

Mechanical analysis of composites reinforced with carbon black is displayed in Figure 6. The results indicate that when carbon black was added to the epoxy resin, the nature of the epoxy composite transformed from brittle to ductile. Blank epoxy resin exhibited brittle behavior. The utilization of commercial carbon black in 1% to 5% concentration in composites increased the breaking strength up to a certain limit, and afterward, it exhibited a decreasing trend. An increment of about 21%, 35%, and 2% was seen for 1% ~ 3% filler loading, after that it reduced to about 60% and 35% using 4% and 5% commercial carbon black when compared with blank epoxy. It could be because of the formation of agglomerates, which deteriorated tensile properties at higher filler concentration.

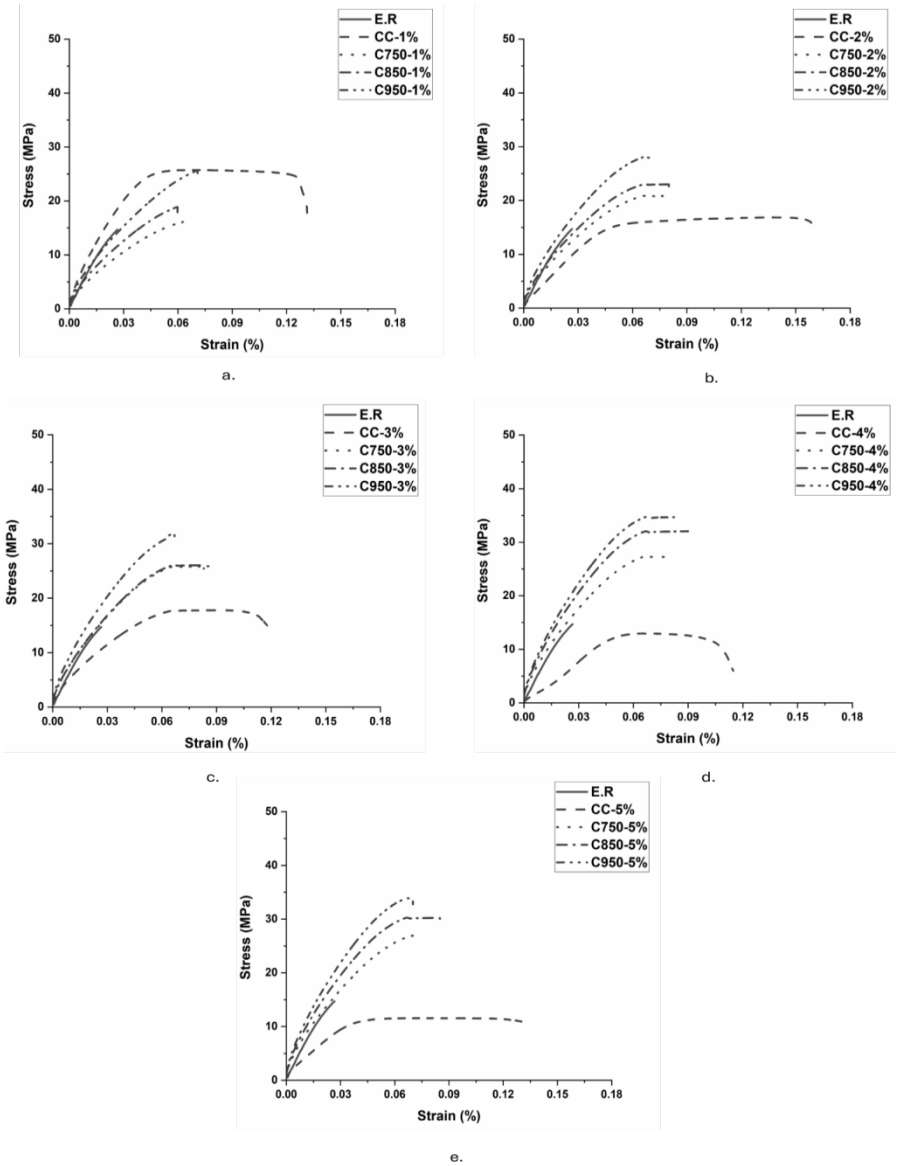

Figure 6: Stress v/s strain diagrams of blank epoxy and composites reinforced with commercial and cotton-based carbon black synthesized at different temperatures

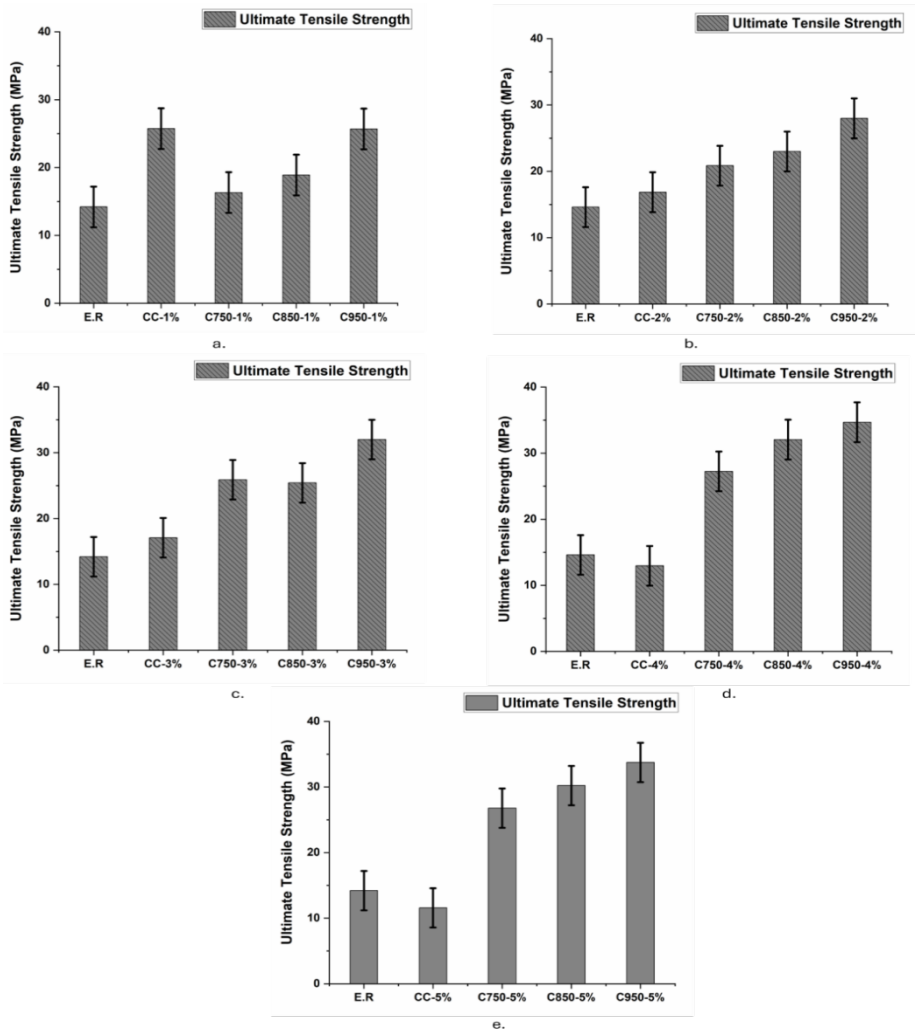

Figure 7: Ultimate tensile strength of blank epoxy and composites reinforced with commercial and cotton-based carbon black synthesized at different temperatures

Maximum mechanical properties were exhibited by cotton-based carbon black synthesized at 950 °C, when compared with those synthesized at 750 °C and 850 °C. The addition of carbon black synthesized at 750 °C to the composite led to increased breaking strength by 7.4%, 39%, 42.5%, 82%, and 79%, when utilized in concentrations of 1-5%, respectively. This increment was 22%, 52%, 73%, 113.13%, and 101% when utilizing carbon black synthesized at 850 °C; and 70%, 86%, 111%, 130%, and 122% when utilizing carbon black synthesized at 950 °C, in the concentration range of 1% ~ 5%. The elevated temperature led to the formation of many micro-pores and voids in the structure of carbon black by removing volatile matter content. Due to these pores, epoxy resin had the possibility of better entanglement with the carbon black, resulting in an increase in micro-mechanical interlocking. An increment in micro-interlocking of fillers with epoxy resin would require more force to break the composites. These results are also evident from the literature.[36] Other authors also reported that high pyrolysis temperature might have increased the pore density of carbon black,[37] which then resulted in composite tensile strength enhancement.

Ultimate tensile strength properties of composites reinforced with carbon black were examined. When using commercial carbon black, ultimate tensile strength exhibited an increment at first, for concentrations of 1% ~ 3%. This rise was 76%, 15%, and 17%, respectively, as compared to the blank epoxy, as shown in Figure 7. Afterwards, it showed a decrease of about 11% and 21% using 4% and 5% concentration. Composites reinforced with cotton-based carbon black exhibited an improvement in tensile properties when increasing the concentration of carbon black from 1% to 4%. An increase of about 12%, 43%, 77%, 87% and

83% was observed using C 750; of 29%, 57%, 74%, 120% and 107% for C 850 and of 76%, 91%, 119%,137%, and 131% for C 950, in comparison with the blank epoxy, as evident from Figure 7. Carbon black might have increased the interfacial area of composites by increasing concentration. Similar results were reported in the literature.[38] The reason behind the inclusion of carbon black as a filler is to increase the epoxy composites' strength. It the presence of carbon black particles might have obstructed the propagation of cracks.

Three stages occur in the mechanical deformation mechanism as described in the literature.[39] Initially, interfacial debonding causes the deformation of composites. Carbon black particles act as sites for stress concentration. The stress state of the polymer matrix changes because of these stress concentration sites. When the applied force is increased, stress concentration rises, which causes volume to dilate and voids to be produced. Afterward, debonding actions come into play, producing cavitation and composite failure/fracture. The capability of composites to bear stress could be enhanced by increasing the amount of filler particles up to a certain loading. Also, an increase in filler concentration enhanced crosslinking. The movement of molecules might be blocked due to more crosslinking, because of which the strength would be increased.[40] Beyond the optimum filler concentration, an increment in viscosity of matrix composites was observed, which might have caused improper mixing and the formation of agglomerates.[41]

Young's modulus of composites reinforced with carbon black was tested, and the results are exhibited in Figure 8. Carbon black as a reinforcing material enhanced Young's modulus of epoxy composites, as compared to the blank epoxy, with increasing filler loading in the composites.

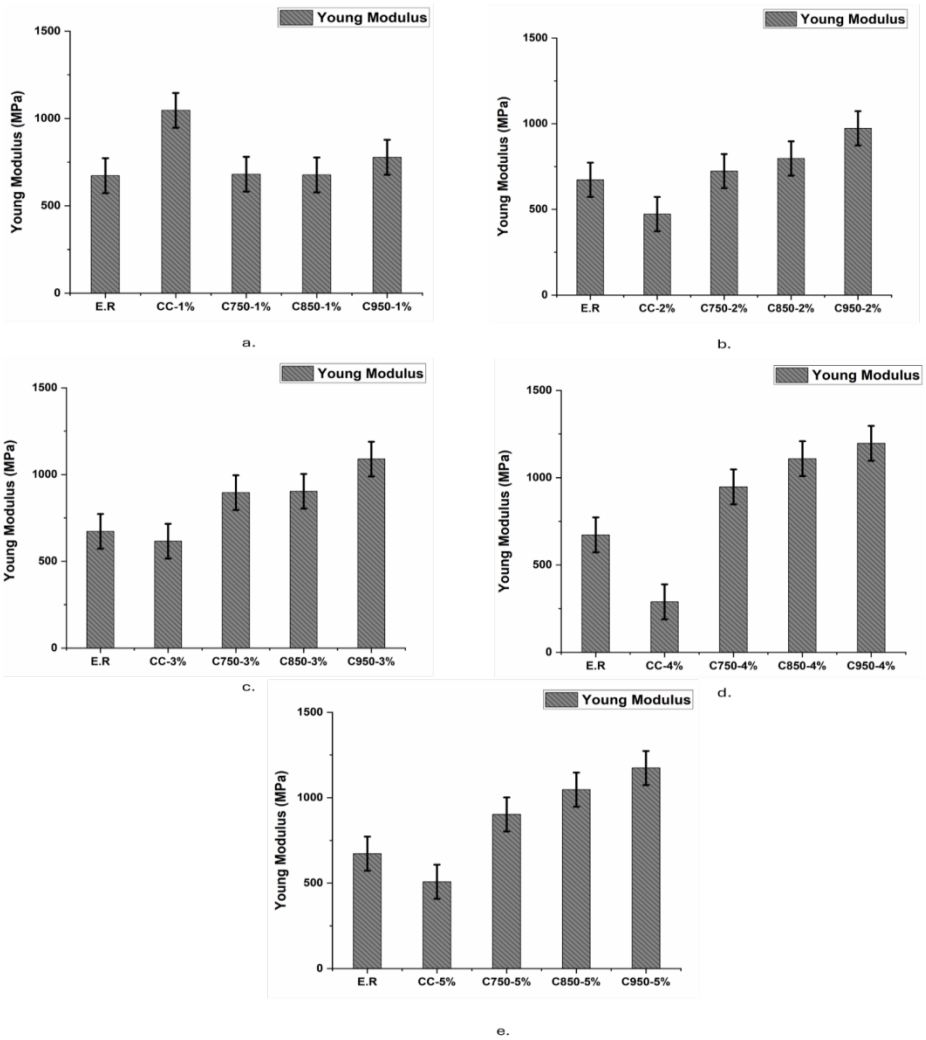

Figure 8: Young's modulus of blank epoxy and composites reinforced with commercial and cotton-based carbon black synthesized at different temperatures

The commercial carbon black improved Young's modulus of epoxy composites initially, when used in 1% concentration, by about 56%, then exhibited a decreasing trend: by 29%, 9%, 57%, and 25%, when using 2% ~ 5% concentration of carbon black. Non-uniform dispersion of the particles might be the reason for this decrement at higher concentrations, as evident from Figure 8. When C 750 was used, Young's modulus increased by about 1%, 8%, 33%, 41%, and 34.5% for 1% ~ 5% concentrations of the filler, respectively. C 850 increased Young's modulus by about 0.63%, 18%, 34%, 65%, and 56%. In the case of C 950, when used in 1% concentration, it increased Young's modulus by about 77%, also, when used in 4% concentration, it enhanced Young's modulus to a maximum level on a comparative basis. Overall, the Young's modulus increasing trend was of about 16%, 45%, 62%, 78%, and 75% for concentrations of 1% ~ 5%, respectively, when compared with blank epoxy. Increased filler concentration up to a certain level improved the composite's rigidity.[42] By increasing the filler concentration, the capacity of the composites to bear load might have been enhanced. These results are also in agreement with the literature.[43] However, an overly high filler loading leads to improper mixing, non-uniform dispersal, and formation of agglomerates.

The region under the stress-strain curve from the origin to the yield point is termed resilience. It describes the material's ability to absorb energy in the elastic zone. The resilience of composites reinforced with carbon black fillers improved, as evidenced by Figure 9.

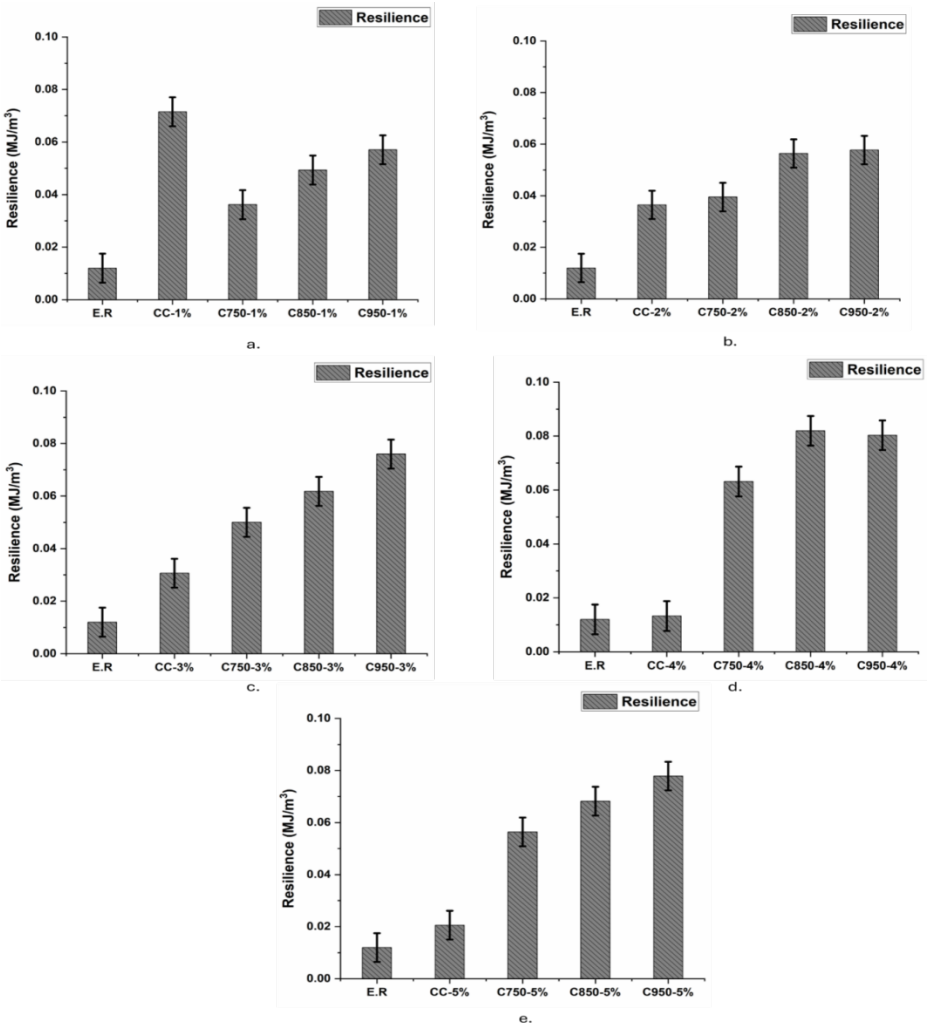

Figure 9: Resilience of blank epoxy and composites reinforced with commercial and cotton-based carbon black synthesized at different temperatures

By using commercial carbon black, resilience improved by about 6, 3, 2.5, 1.1, and 1.7 times, as compared to blank epoxy. Increments of 3.0, 3.2, 4.1, 5.2, and 4.7 times were observed by the addition of C 750 fillers in 1% ~ 5% concentration, respectively, when comparison was made with the blank epoxy composite. This increase was 4.1, 4.6, 5.1, 6.8, and 5.8 times using C 850, and 4.7, 4.8, 6.3, 6.6, and 6.4 times when C 950 was used in the amount of 1% ~ 5%, respectively. The molecular motion in composites would be halted as there was an increment in cross-linking between the filler and epoxy. Because of this, the properties of the materials to resist deformation might have been enhanced, which exhibited improved resilience.[44] The addition of the C 950 filler exhibited maximum resilience at 4% concentration. For commercial carbon black, high resilience was initially obtained, as compared to blank epoxy.

A material's toughness consists in its capacity to absorb energy before breaking. It can be evaluated by calculating the area under the stress-strain curve from the origin to the fracture point. The tensile toughness of composites reinforced with carbon black was enhanced, as exhibited in Figure 10.

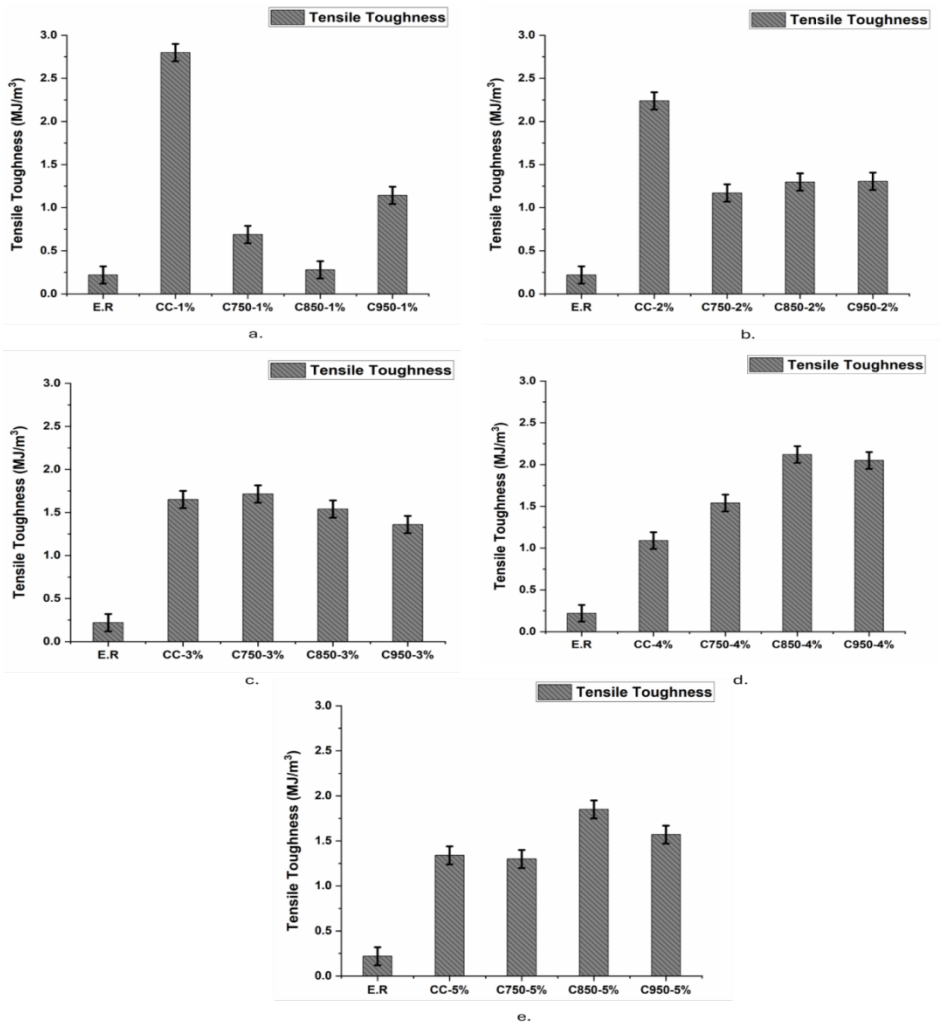

Figure 10: Toughness of blank epoxy and composites reinforced with commercial and cotton-based carbon black synthesized at different temperatures

For commercial carbon black, an increment of about 12.7, 10.1, 7.5, 4.9, and 6.0% in toughness properties for 1%~5% concentrations was observed when compared with blank epoxy. When C 750 was used as filler, an increase of 9.5%, 22.3%, 34.9%, 68%, and 40.4% was observed for 1%~5% concentrations. For C 850, toughness increased by about 27%, 37%, 39%, 69%, and 40%, and for C 950 – by 18%, 41%, 49%, 81%, and 70%, for the concentration range of 1%~5%, respectively. High tensile toughness indicates that the material is less responsive to stress intensity.

Owing to that property, the materials can bear high stresses. Moreover, the higher the ductility of the material, the greater will be the tensile toughness.[45] Carbon black fillers are thermally conductive materials, which, when incorporated in composites, dissipate heat rather than concentrating it. It is the reason why materials exhibit more stress-bearing capability before they reach their glass transition temperature.[46] The decrement in toughness value after a certain filler loading can be explained by agglomerate formation and a non-homogeneous mixture of epoxy and carbon black as filler.[47]

CONCLUSION

Successful synthesis of carbon black from cotton-based textile waste was carried out at three pyrolysis temperatures of 750 °C, 850 °C, and 950 °C. Commercial carbon black N-660 was used as a benchmark material. The characterization of carbon black was performed through yield, morphology, Raman, and FTIR analysis. The yield of cotton-based carbon black decreased as increasing pyrolysis temperature led to the release of volatile organic compounds, and the yield was in the range of 25% ~ 19.5%. Morphology analysis revealed the dispersed nature of the prepared carbon black, while Raman analysis exhibited that by increasing pyrolysis temperature, there were fewer defects in the cotton-based carbon black as the graphitic content increased because of the removal of inorganic matter. FTIR analysis exhibited the presence of functional groups in the case of the carbon black synthesized at 750 °C, *i.e*., alkoxy compound related to C-O bonds at the wavenumber of 1002 cm^{-1} and C=C bonds at the wavenumber in the range of 1531 cm^{-1} to 1672 cm^{-1}. These functional groups disappeared when the pyrolysis temperature increased to 850 °C and 950 °C, which supported morphology and Raman analysis.

The prepared carbon black was then used in the concentration range of 1~5% to reinforce epoxy resin composites. Cotton-based carbon black exhibited good particle dispersion into the epoxy resin. Due to this, the carbon black particles ensured good micro-mechanical interlocking with the epoxy resin, thereby increasing the mechanical properties of the composites. The results were compared with those of composites prepared using commercial carbon black N-660. The cotton-based carbon black exhibited superior performance, with an increase of 130% in breaking strength, 78% in Young's modulus, 81% in tensile toughness, as well as 6.6 times increase in resilience of the composites when using carbon black synthesized at 950 °C. It could be explained by the possible formation of pores due to the elimination of O and H-containing species from the material at high pyrolysis temperature, as evident from morphology, Raman, and FTIR analyses. Therefore, it can be concluded that cotton-based textile post-cosumer waste was succesfully converted into valuable and superior quality composites, with great commercial potential.

ACKNOWLEDGMENTS: The authors are thankful to the Pakistan Science Foundation for providing funding under the project named "PSF/CRP/T-Helix (28)" to carry out the research activities. The authors express their gratitude to Mr. Imran Shahid and Mr. Shakeel Ahmed for their assistance with the sample preparation and testing.

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