



Article Development of Activated Carbon Textiles Produced from Jute and Cotton Wastes for Electromagnetic Shielding Applications

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Abstract: Increasing amounts of waste resulting from over-consumption carry substantial risks for human and environmental health, and disposing of this waste requires enormous amounts of energy. As a result, waste-to-wealth and circular economy approaches have gained attention in both academia and the commercial sector in recent years. Accordingly, this study aims to develop electromagnetic shielding materials by converting non-conductive waste textiles into conductive value-added product and porous fabrics by carbonizing the structure itself rather than by adding any conductive particles. To this end, the novel contribution of the present study is that waste textiles were converted into activated carbon in a shorter time and without compromising the integrity of the fibrous network via microwave pyrolysis without inert gas. Sulfuric acid was used as a dehydration and activation agent, suppressing the release of volatile organic substances and eliminating greenhouse gas emissions. This approach also increased product yield and reduced energy consumption and sample shrinkage. The structures of the activated carbon textile showed EMI shielding within 20-30 dB (99.9% attenuation) in the 1–6 GHz frequency range. The maximum SSE/t value of 950.71 dB·cm²·g⁻¹ was obtained with the microwave post-treated activated carbon textile. Micropores were dominant characteristics of these materials, and pore diameters increased with increased acid concentration. The maximum surface area of 383.92 m²/g was obtained with 8% acid. Ultrasound treatment reduced water-energy consumption and cost. Only 5 min of microwave post-treatment increased textile conductivity and thermal stability and contributed positively to electromagnetic shielding.

Keywords: textile recycling; nonwoven; microwave pyrolysis; self-purging; electromagnetic interference (EMI) shielding

1. Introduction

The increased use of electronic devices in the industrial, commercial, military, and consumer sectors and IoT devices has created a new form of pollution known as electromagnetic noise, radio frequency interference, electromagnetic (EM) radiation, or electromagnetic interference (EMI). Electromagnetic radiation can cause interference, malfunctioning of equipment, and serious diseases such as brain tumours, leukemia, insomnia, nervousness, and headache on exposure to electromagnetic waves [1,2].

EMI shielding is a method used to prevent the transfer of electrical and magnetic waves from one location to another. It is achieved by utilizing a conductive or magnetic material. The signals passing through a system can be minimized by EMI shielding, which involves the absorption or reflection of waves [3].

According to electromagnetic theory, EM shielding occurs in three ways: 1—reflection, which involves impedance mismatch (conductive metals); 2—absorption: attenuation of electromagnetic energy by magnetic, dielectric, and polarization losses; 3—multiple internal reflections, which occur when there is heterogeneous structure, a very large interfacial area, and voids and defects that can cause reflection [4].



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Copyright: © 2023 by the authors. Licensee MDPI, Basel, Switzerland. This article is an open access article distributed under the terms and conditions of the Creative Commons Attribution (CC BY) license (https:// creativecommons.org/licenses/by/ 4.0/). EMI shielding materials that reduce electromagnetic radiation are generally made of metal due to the high electrical conductivity of most metals, which allows the material to form a Faraday cage when encountering EM waves. However, metals are relatively heavy, are uneconomical to process, and have disadvantages in corrosion resistance. In addition, metals cause secondary radiation by reflecting EM waves. Recent research has focused on composite materials with good impedance matching, high green shielding indices, tunable reflection-absorption, light weights, corrosion resistance, flexibility, and easy processing [5,6]. Accordingly, research on carbon materials, textiles, and their composites has increased due to their excellent flexibility, electrical properties, environmental friendliness, and chemical inertness [2]. Textiles with EM shielding functions have a broad range of applications in daily and professional use. Particularly in military and civic applications, the importance of protection against EM radiation is very high [7].

Carbon-based textile materials are used not only for EMI shielding, but also to produce supercapacitors, batteries, and sensors. They are additionally used in photocatalytic degradation of organic pollutant-dyes in electronic textiles [8]. Carbon materials can be manufactured in any form, such as one-dimensional quantum dots, two-dimensional graphene, three-dimensional graphite, and amorphous materials like carbon black and activated carbon. Activated carbon (AC) can be combined with textiles to make a highly porous, relatively inexpensive product with a low density. The cost-effectiveness and large-scale production of AC materials make their wide use in EMI shielding more possible [9]. Carbon materials can also be easily produced through waste-to-wealth and circular economy processes. It is essential to be mindful of the waste produced, as it can negatively impact human and environmental health. Proper waste disposal also requires significant energy, which can further contribute to environmental issues. Therefore, obtaining carbon from waste is ecologically and economically more advantageous than using other materials.

Carbon materials in EMI shielding applications are generally used in conjunction with other materials, such as Mxenes, metals, and conductive polymers, to enhance performance [8]. Table 1 shows production methods and performance metrics for carbon-based textiles. As seen in Table 1, studies in which activated carbon produced by the pyrolysis method is used alone for EMI shielding are rare. Tables 1 and S1 compare other carbon-based EMI shielding materials and self-purging pyrolysis with the material produced in this study.

Material	Production Method	Max. EMSE Frequency (dB) (GHz)		Thickness (cm)	SSE/t dB cm ² g ⁻¹	Ref.
Carbon Fiber Polymethacrylimide	Polymerization: water bath at 50 °C for 4 days Foaming: 240 °C—1 h	Reflection Loss -53.2	2–18 GHz	0.2	-	[10]
NiCo ₂ O ₄ nanofiber Carbon	Electrospinning and calcination up to 700 °C—2 h in N2	13.44	2–18 GHz	-	-	[11]
Polyolefin-Graphene	Ultrasonication, extruding and 3D printing	35	8.2–12.4 (X-band)	0.2	244.9	[12]
Cellulose-CNT	Ultrasonication Vacuum filtration	26.67	5.85–8.2 (C-band).	0.0036	9944	[13]
CoFe/CoCu PDMS Carbon Cotton fabric	Hydrothermal reaction Calcination: 1000 °C—1 h in argon PDMS encapsulation	73.46	2–18	0.024	12,227.21	[14]
MXene-CNT- Cotton fabric	Spray-coating Mxene, CNT, PDMS	46.05	8.2–12.4 (X-band)	0.0138	6710	[15]

Table 1. Carbon-based materials used for EMI shielding.

Material	Production Method	Max. EMSE (dB)	Frequency (GHz)	Thickness (cm)	SSE/t dB cm ² g ⁻¹	Ref.
Carbon Fiber Poly (aryl ether nitrile)	Ultrasonication Coating layer-by-layer	37.76	8.2–12.4 (X-band)	0.2	543	[16]
Natural Rubber CNT Foam	Supercritical CO ₂ foaming	33.74	8.2–12.4 (X-band)	0.13	312.69	[17]
Activated Carbon- Copper Nonwoven	Electroless plating	70	1.5–10	0.04	11,162	[18]
Activated Carbon Aerojel	Sol-gel process and pyrolysis	20	8.2–12.4 (X-band)	1.1 0.2	-	[19]
Activated Carbon RGO PVA	Drop casting	9.7 21	8.2	0.08 0.2	-	[20]
Activated Carbon from acrylic fibrous waste	Pyrolysis in muffle furnace without inert atmosphere Most closely related study	28.29	2.45	1.16	-	[4]
Activated Carbon from jute-cotton waste	Pyrolysis in microwave without inert atmosphere	39.54	3 GHz	0.245	950.71	This Work

Table 1. Cont.

Activated carbon (AC) can be combined with textiles to form a highly porous, relatively inexpensive product with a low density. The cost-effectiveness and large-scale production of AC materials make their wide use in EMI shielding more possible [9]. There are four critical processes through which activated carbon is obtained: heating technology, activation method (physical, chemical or physicochemical), activators, and pyrolysis atmosphere (purging and inert gas).

Heating is the most energy-intensive process for carbonization and activation. Generally, conventional heating technologies have been used to obtain carbon, and the process times are relatively long [21–25]. Microwave heating has recently been preferred for carbonization due to faster, homogeneous heating and lower energy consumption [26]. In addition, microwave pyrolysis produces more solid products with higher surface areas and better pore volumes than does conventional pyrolysis [27,28].

Pyrolysis is a thermochemical process that occurs at high temperatures, without oxygen, in which large molecules turn into smaller molecules [29]. Inert gas is often used to create an oxygen-free environment for pyrolysis. Improvements in energy costs and the use of inert gases directly and significantly affect the final cost. Recently, some studies have been carried out using "self-purging" to eliminate inert gas [30-35]. Gases emitted during pyrolysis serve as the activating agents, so carbonization and activation are combined into one step. The CO_2 and H_2O in the pyrolysis gases can activate the carbon [34]. If water exists in the material, in conventional pyrolysis, the heating rate will drop due to absorption of heat by the water. However, in the microwave, the presence of water causes significant microwave absorption and increases the heating rate of wet materials compared with dry materials [36]. For this reason, water and sulfuric acid act as heating media and activators for microwave pyrolysis in the present study. Furthermore, sulfuric acid acts as a cleaning chemical to remove some impurities, serves as a dehydration and activation agent that suppresses the release of volatile organic substances, eliminates greenhouse gas by-products, increases product yield, and reduces energy use and sample shrinkage. The results obtained in this study align with those of a recent study [37] on the effects of sulfuric acid on carbon retention and yield.

This study aims to develop electromagnetic shielding materials by converting nonconductive waste textiles into conductive, porous value-added products by carbonizing the structure itself, rather than by adding conductive particles. The novel contribution of the present study is that a textile manufactured from jute-cotton waste was carbonized and activated in one step by microwave pyrolysis without inert gas and converted into activated carbon without compromising the integrity of the fibrous network (Supporting video). Tables 1 and S1 show the position of this study relative to the existing literature and its contribution.

2. Materials and Methods

2.1. Materials

Jute and cotton wastes were obtained from Ege University Textile Engineering Department. Sodium hydroxide was purchased from Smyras. Sulphuric acid (95–97%) and ethanol were purchased from Merck. Carbon black (CB, 99.9% S.A. 75 m^2/g) was obtained from Alfa Aesar (Ward Hill, MA, USA). All solutions were prepared with distilled water.

2.2. Textile Production

The textile used in the experiments consists of 5 layers: 2 nonwoven layers made from 25% jute and 75% cotton wastes and woven cotton fabric fixed between two layers. Production of the nonwovens was carried out in Ege University Textile Engineering Department according to the needlepunch production method. *Needle punching* is an environmentally friendly method used to produce nonwoven by mechanical entanglement of the fibers alone, without using any chemicals. The fibers were processed into nonwovens using a Dilo needling machine with an automatic feeding system. First, single-layered nonwovens were produced by needle punching by blending jute and cotton wastes at a ratio of 25% to 75%, respectively. Then, the nonwovens were combined with woven fabric to increase the dimensional stability of the structure, again by the needlepunch method, without using any chemicals. A schematic drawing of the structure of the combined textile is shown in Figure 1. Production parameters of the needlepunch process are given in Table 1.



Figure 1. Schematic drawing of the nonwoven–woven–nonwoven textile obtained by needle punching.

2.3. Pyrolysis System and Experiments

A 700 W microwave oven (Arçelik MD 674, Bolu, Turkey) operating at a frequency of 2.45 GHz was used. Porcelain was used as a reactor (5 cm height, 7 cm inner diameter, 5 mm thickness). Porcelain has a high resistance to temperature (up to 1400 $^{\circ}$ C) and chemicals. High-temperature PTFE was used as a soft-flexible sealant and relief valve.

The flow chart of the experimental system is depicted in Figure 2. Two methods were tried: in one, NaOH was used to remove impurities and increase penetration by sulfuric acid; in the other, ultrasound treatment was used without pre-cleaning to reduce energy and water consumption. Microwave post-treatment was used to remove oil and tar from the activated carbon textile and to increase conductivity.



Figure 2. Flow chart of experimental system and methods.

Preliminary tests were conducted to determine the experiment parameters (time, power, H_2SO_4 concentrations) due to a lack of reference studies on microwave pyrolysis of textiles. After approximately 1 h, porcelain slowly evolves from microwave transparency to microwave absorbency, compromising the safety of the work. More than 1 h in the microwave was needed to produce the desired product. When the microwave was started at high power values, the porcelain exploded. Slow increases in microwave power were thus used for safety and to mimic slow pyrolysis.

2.4. Characterizations

Tests included Raman spectroscopy, X-ray diffraction (XRD), X-ray photoelectron spectroscopy (XPS), Fourier-transform infrared spectroscopy (FT-IR), thermogravimetric analysis (TGA), scanning electron microscopy (SEM), elemental CHNS, Brunauer–Emmett–Teller (BET) analysis, 4-probe conductivity testing, and measurement of electromagnetic shielding. The characterizations, measurements, and methods used are described in Table 2.

Table 2. Production parameters of the needlepunch process.

Production Parameters							
Needle penetration depth	1 cm						
Folding-belt speed	2.8 m/min						
Needle penetration depth	2.5 mm						
Needle penetration depth for joining nonwovens and woven fabric	1.2 cm						

Electromagnetic shielding effectiveness was tested in the 1–6 GHz frequency range. Tests were performed by using an anechoic chamber test system according to the TS EN50147-1 standard [38]. The TS EN 50147-1 standard covers measurements of the shield-

ing attenuation of shielded enclosures (chambers, rooms) in the frequency range of 9 kHz to 40 GHz. This standard aims to establish a common measurement procedure for verifying the effectiveness of shielding of a shielded enclosure. During the measurements, the sample was positioned between a signal generator and a receiver. The signal generated was amplified before it was transmitted to the antenna. The antenna directed EM radiation towards the sample. The sample allowed some radiation to pass through it to the receiving antenna, while the rest was reflected. Figure 3 shows a schematic drawing of the test system. Based on the fundamental principle of the EMSE test, the process involves measuring the number of transmitted signals, then calculating the EM field that is blocked by the sample. To measure the shielding effectiveness, the power received by the antenna was compared under conditions with and without the sample. The EM wave was measured in dBmV. The reduction of the EM wave was then determined by calculating the difference between the two measurements in dB.



Figure 3. Schematic drawing of the system for testing the effectiveness of electromagnetic shielding.

3. Results

Raman spectroscopy and XRD analysis were performed to determine the type of carbon composing the developed material. Charged surface functional groups cause dipole losses and provide absorption-based EMI shielding. Therefore, XPS and FT-IR analyses were performed to determine the nature of surface functional groups. As the roughness and porosity of the surface cause internal reflections in EMI shielding, SEM and BET analyses were performed. CHNS analysis is a characteristic analysis because it gives the percentage of carbon in carbon materials. TGA analysis was conducted to elucidate the thermal stability of the material. The aim of these analyses was to elucidate the features that directly or indirectly affect the performance of the developed product.

3.1. Raman Analysis

There are two distinct bands in the spectrum between approximately 1350 and 1584 cm⁻¹, which are attributed to carbon-specific D and G bands in Raman spectra of the textiles (Figure 4). The G-band is the primary vibrational mode representing the planar configuration and corresponding to the stretching vibrations of sp² carbon. The D band highlights the defects in the crystal structure and reflects the presence of irregular carbon atoms.

Relatively low ID/IG (range 0.78–0.86) ratios and narrow FWHM (D-G) in the range of 81.86–226.47 indicate carbon formation, low numbers of defects, and less deformation associated with the acid ratio (Table S2). The ID/IG ratio increases up to 4%, and there is no significant increase after that point. Figure 4 shows shifts in the positions and intensities of the G and D bands depending on the acid concentration. The change in G band intensity increases linearly with the number of graphene layers [39]. The decreasing intensities of the G and D bands as the acid concentration increases can be attributed to the decreasing number of layers. The maximum G band intensity was obtained in the experiment with



only water. The G band position shifted slightly to higher wavenumbers (blue) as the acid concentration increased.

Figure 4. Raman spectra of activated carbon textile structures impregnated with water and different H_2SO_4 concentrations and Raman spectra of activated carbon textile structures microwave post-treated for 5 min.

The G band and D band positions and intensities are sensitive to electron or hole doping. The Raman D-band intensity increases when the Fermi level approaches half of the excitation energy. The decrease in the D-band intensity indicates hole doping. Hole doping reduces the electrons, resulting in phonon frequency blue shift [40,41]. In the present study, oxygen functional groups at the surface of the activated carbon textile structures provided hole doping. They caused decreasing G-D band intensities and blue shifts in the G band positions. The change in G band intensity increases linearly with the number of graphene layers [39]. The decrease in G band intensity observed in the present study can be attributed to the exfoliating effect of sulphuric acid on the graphene layers of activated carbon samples.

The calculations used to derive crystallite size along the basal plane L_a , per Tuinstra and Koenig, are given in Supplementary Materials as Equations (S1) and (S2). L_a values calculated from these equations are given in Table S2.

3.2. XRD Analysis

Figure 5 shows the XRD pattern of activated carbon textile structures produced at different sulfuric acid concentrations. Two diffraction peaks were found at ~20°, which confirmed (002) diffraction of irregular-amorphous carbon and is consistent with the literature [4,42]. The 2 theta peak position systematically shifted slightly to smaller degrees and showed increased distance between crystals as the acid concentration increased (Table S3). The average distance (calculated by Bragg's Law in the Supplementary Materials, Equation (S3)) between the layers is ~0.4 nm, greater than that of graphite (0.335 nm). This distance can be attributed to the presence of oxygen functional groups [43] and the activation agent (sulfuric acid) [44], which can penetrate the layers on exposure to microwave energy.





The size and lattice parameters of the crystal structure are given in the Supplementary Materials as Equations (S3)–(S5). The parameters of the crystal structure values calculated from these equations are given in Table S3.

3.3. XPS Analysis

Figure 6 shows the XPS survey analysis in the wide range of 0–1400 eV for carbonized and microwave post-treated samples. The dominant binding energies belong to carbon and oxygen. The presence of sulfur, which is not in the starting material, can be interpreted as sulfuric acid residue. The oxygen peak at 530.95 eV in the starting material may be associated with NaOH and indicate the presence of unwanted residues.



Figure 6. XPS Survey analysis of activated carbon textile structure.

Figures 7 and 8 show that microwave post-treatment removes some oxygen functional groups from the surface. The increase in the number of C=C bonds in the activated carbon textile structure corroborates the increase in conductivity following the 5–min microwave post-process, which may depend on the increase in conjugation.



Figure 7. XPS diagrams showing C1S of raw nonwoven-woven textile structure (**a**), activated carbon textile structure (**b**), microwave post-treated activated carbon textile structure (**c**).



Figure 8. XPS diagrams showing O1S of raw nonwoven-woven textile structure (**a**), activated carbon textile structure (**b**), microwave post-treated activated carbon textile structure (**c**).

3.4. FT-IR Analysis

Figure 9 depicts the FT-IR spectrum of the activated carbon textile structure. The peak at ~3440 cm⁻¹ indicates O-H stretching, indicating the presence of water and hydroxyl functional groups in the FTIR spectrum. Peaks at ~2930–2850 cm⁻¹ indicate CH₂ stretching vibrations. The peak at ~1640 cm⁻¹ indicates the presence of C=O and conjugated C=C groups. C=O and C=C groups are characteristic groups in activated carbons [45]. The peak at ~1398 cm⁻¹ corresponds to bending vibrations of the CH₃ groups. The peak at ~1043 cm⁻¹ can be attributed to the C-OH stretch. The peak at ~876 cm⁻¹ can be associated with the C–O bond [46]. These results are in agreement with the XPS results.

3.5. TGA Analysis

Figure 10 and Table 3 show TGA analysis of raw nonwoven-woven textile structure and the activated carbon (AC) textile structures obtained in this work.



Figure 9. (a) FT-IR Spectra analyses of activated carbon textile structures (b) Comparative FT-IR analyses of activated carbon textile structure (8% sulfuric acid) with and without microwave post-treatment for 5 min.



Figure 10. Comparative TGA analysis of the structures of the raw nonwoven-woven textile, the activated carbon textile with 8% sulfuric acid and the microwave post-treated activated carbon textile.

Table 5. Characterizations, measurements, equipment, and methods	Table 3.	Characterizations,	measurements, e	equipment, and	d methods.
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Analysis	Equipment	Analysis Method
Raman Carbon Fingerprints	Renishaw (Renishaw, Wotton–under–Edge, UK)	Laser λ = 532 nm, power = 2.5 mW, grating: 2400 g mm ⁻¹ . The magnification of the microscope optics for the collection was set to ×20. D and G bands were calculated from a Gaussian peak-fitting function using the original data without any smoothing. 400–3000 cm ⁻¹
XRD Diffraction Crystal Structure	Rigaku Ultima IV X-Ray Diffractometer (Rigaku, The Woodlands, TX, USA)	Bragg–Brentano geometry, Cu K α (λ = 0.15443 nm), 10° \leq 2 $\theta \leq$ 70°, 20 kV-40 mA, slit:10 mm, scan speed: 2°, scan step: 0.06°
FT-IR Surface Chemistry	Thermo Scientific Nicolet iS50 (Thermo Fisher Scientific, Madison, WI, USA)	400–4000 cm ^{-1} , scanning resolution is 0.5 cm ^{-1} .
XPS Surface Chemistry	Thermo Scientific K-Alpha (Thermo Fisher Scientific, East Grinstead, UK)	X-ray source: Al K α Monochromatic (1486.68 eV) X-ray dimension: 250 μ m, sample area: 60 \times 60 mm, analyzer: 180°, energy: 50 eV, scanning: 10

Analysis	Equipment	Analysis Method
TGA Thermal Stability	TA Instruments SDT Q600 V20.9 (TA Instruments, New Castle, DE, USA)	From room temperature to 900 $^\circ C$ (temperature ramping 10 $^\circ C/min$) under N_2 atmosphere (100 mL/min). Pan: Alumina
BET Porosity	Micrometrics 3 Flex (Micromeritics, Nor-cross, GA, USA)	Brunauer Emmet Teller (BET) method, N_2 adsorption-desorption isotherms at 78.39 K, relative pressure range P/P0 from 0.01 to 1, outgassed 300 °C for 5 h under N_2 gas
SEM Morphology	Carl Zeiss 300VP (Carl Zeiss, Jena, Germany)	All sample surfaces were sputtered with gold.
4-Probe Conductivity	Lucas Signatone Pro4 (Lucas Signatone Corp., Gilroy, CA, USA)	4-probe method. The measurements were done via a computer-controlled Keithley based on the average of 5 measurements.
EMI Shielding	Consept Test and Technology System (Consept Test and Technology System, Gebze, Turkey)	Effectiveness of the 3D activated carbons at EMI shielding was tested in an anechoic chamber and a frequency range of 1 to 6 GHz by the EN50147-1 standard

Table 3. Cont.

The TGA curve depicts two main phases. With the lower mass loss, the first stage can be attributed to moisture elimination. The second broad phase is pyrolysis. The broad curve for the second stage can be attributed to the decomposition of inorganic materials and impurities and the degradation of functional groups. The decrease in moisture content after microwave post-treatment indicates that this treatment may have reduced the functional groups on the surface. The percentages of oxygen functional groups found by the XPS and Raman analyses also supports this assumption. The microwave post-treatment increased thermal stability. Supporting details and figures are given in Supplementary Materials (part S4).

3.6. SEM Analysis

Scanning electron microscopy (SEM), which makes images with accelerated electrons under 5 kV voltage, was conducted before and after carbonization. The fiber structure of the textile was seen to remain intact. Surface roughness increased, and a porous structure formed with carbonization, as seen in Figure 11c,d.

Images were analyzed with the FIJI-ImageJ image-processing program. A total of 25 fibers were sized and averaged. The dimensional analysis determined that the fibers carbonized with water were ~60% thinner than those carbonized with 8% acid. This result can be interpreted as showing that sulfuric acid reduced the dimensional reduction in the fibrous structure by increasing carbon retention in the material, and this result is consistent with results published the literature [37].

3.7. Yield, BET, Elemental-CHNS and Conductivity

The dominant structures of the activated carbon textile are micropores. In addition, as the acid concentration increases, the ratio of micropores to total pores decreases in connection with the increasing size of the pores. The red-colored third column, titled "micropore area" in Table 4, is the ratio of total pore area to micropore area as a percentage. Table 4 depicts that as the acid concentration increases, the surface area increases and the pores expand, as expected.



Figure 11. (a) Textile raw material produced from jute-cotton waste, magnified $500 \times$ (b) Structure of the carbonized textile produced with 8% sulfuric acid, magnified $500 \times$ (c) Textile raw material produced from jute-cotton waste, magnified $10,000 \times$ (d) Structure of the carbonized textile produced with 8% sulfuric acid, magnified $10,000 \times$.

Table 4. TGA data analysis of the structures of the raw nonwoven-woven textile and the activated
carbon textile.

Material	~Moisture%	T _{onset} (°C)	Decomposition Temperature (°C)	T _{endset} (°C)	Residue %
Raw Nonwoven + Woven Textile Structure	7.21	335.74	355.31	372.56	5.86
AC Textile Structure, 8% Acid	10.86	382.99	531.01	673.45	48.76
AC Textile Structure, 8% Acid Microwave Post-treated	7.95	434.77	585.55	697.96	62.70

The 5-min microwave post-treatment resulted in pore collapse (closing). The pore diameter decreased with microwave treatment from 2.13 to 2.11 nm for the sample treated with 8% acid. BET surface area decreased from 383.92 to 382.33 m²/g. However, this collapse increased conductivity, as seen in Table 5, as it eliminated some impurities and gaps that form electron barriers. No conductivity increase after microwave post-treatment was seen for the sample treated with 8% acid. This difference can be attributed to increased brittleness associated with microwave post-treatment and breaking of the inter-fiber connection, which created resistance for electrons.

Activator	Surface Area (m²/g)	Micropore Area (m²/g)	Micro Pore Volume (cm ³ /g)	Total Pore Volume (cm ³ /g)	V _{mic} /V _{tot} %	Pore (nm) Diameter
WATER	380.7	355.39 (93%)	0.1632	0.1796	90.8686	1.72
2% H ₂ SO ₄	246.66	222.31 (90%)	0.1199	0.1242	96.53784	2.01
4% H ₂ SO ₄	353.29	307.55 (87%)	0.1612	0.186	86.66667	2.11
8% H ₂ SO ₄	383.92	276.42 (72%)	0.1349	0.2008	67.18127	2.13
8% H ₂ SO ₄ , Microwave-treated	382.33	256.15 (67%)	0.1245	0.1995	62.40602	2.11

 Table 5. Pore structures of activated carbons prepared with different acid concentrations.

NaOH-cleaned samples were excluded due to the water-energy consumption associated with their production. In Figures and Tables, WATER means that nonwoven was carbonized with water alone. Figure 12a and Table 5 show that as the acid concentration increases, the surface area and percentage of carbon increase. Figure 12b shows pore size distributions in the activated carbon textile structures prepared with 8% acid. Interestingly, the surface area obtained with water alone is close to 8%. However, the product yield obtained with water is quite low. Product yields were calculated using Equation (1).







3.8. Measurements of Electromagnetic Shielding

Activated carbon textile structures are effective in EM shielding over a 20–30 dB range (99.9% attenuation) at 1–6 GHz. A 700-watt microwave post-treatment for 5 min has a positive effect on EM shielding, as also shown by 4-probe conductivity measurements. This improvement can be explained by the fact that this post-treatment partially removes oil, tar, and some other impurities. Specific EM effectiveness (SSE) and absolute shielding effectiveness (SSE/t) values given in Table 6, Figures 13 and 14 show EMI shielding effectiveness for the 1–6 GHz range and average EMI shielding values. All relevant formulas are given in the Supplementary Materials (Equations (S6)–(S9)).

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	D A 147	WAT	ſER	2% H ₂ SO ₄		4% H	$4\% H_2SO_4$		l_2SO_4
	KAW	UT *	MW *	UT *	MW *	UT *	MW *	UT *	MW *
Product Yield		14.	40	41.48		43.80		46.73	
BET m ² /g		380.70		246.66		353.29		383.92	
Resistivity		1.1×10^{5}	9.62	3.3×10^{7}	2 16	2.4×10^{7}	1 1 2	2.5×10^{7}	2.2×10^{6}
(Ohm-cm)		1.1 × 10	7.02	5.5×10	2.10	2.4 × 10	1.12	2.3×10	2.2×10
Carbon	43.55	60.83	66.33	65.80	67.36	68.60	85.49	70.34	86.94
Hydrogen	6.58	2.53	1.94	2.93	2.63	2.63	1.69	3.02	1.48
Nitrogen	0.31	1.41	2.08	0.55	0.47	1.32	0.76	0.89	0.40
Sulfur	0.84	0.28	4.25	0.59	0.32	0.28	0.14	0.34	0.05

Table 6. Yield, BET, Elemental-CHNS and Conductivity co-assessment.

* UT: Ultrasound treated; MW: Microwave post-treated.



Figure 13. EM-shielding effectiveness of materials produced with different treatments in the range of 1–6 GHz.



Figure 14. Average electromagnetic shielding values of the samples produced with different treatments.

The ultrasonically treated, acid-impregnated sample produced with no pre-cleaning shows better EM shielding than the sampled pre-cleaned with NaOH. This result can be explained by the NaOH residues found in the XPS results. Rinsing very well results in much more water consumption. Therefore, it is desirable to use acidic ultrasonic treatment instead of water in pre-cleaning if very high protection and conductivity are not required. The maximum SSE/t value, 950.71 dB·cm²·g⁻¹, was obtained from the microwave post-treated, activated carbon textile produced with 8% acid.

The effects of pore structure, acid concentration and microwave post-treatment on EMI shielding can be summarized as follows:

- As acid concentration increases, pore numbers and surface roughness increase, which increases the degree of multiple internal reflection and thus increases EMI shielding.
- A 700-watt microwave post-treatment for 5 min positively affects conductivity and thus EM shielding.

4. Discussion

4.1. The Role of Sulfuric Acid in the Process and Carbonization Mechanism

Sulfuric acid is used not only for demineralization, but also for dehydration. The dehydration process reduces harmful greenhouse gas emissions and almost doubles the carbonization efficiency by increasing carbon retention [37,47–50].

In order to understand the role of sulfuric acid in this process, carbonization with only water was conducted. Carbonization efficiency was 14.40% with water and 46.73% with 8% sulfuric acid. Product yield increased with increasing acid concentration. These results are consistent with those found in the literature [37]. At the beginning of the process, water and sulfuric acid serve as microwave absorbers. They are removed over time.

Meanwhile, above a critical threshold, the material becomes a microwave absorber as it turns into activated carbon. The process is accelerated by positive feedback. The role of sulfuric acid and the mechanism by which it affects the process are shown in Figure 15.



- temperature
- · Increases product yield
- · Reduces sample shrinkage
- · It is a polar molecule that ensures the homogeneous transfer of microwave energy to the material.

an inert atmosphere. CO2 gas, water vapor, and sulfuric acid contribute to the cooperation of carbonization and activation processes.

into activated carbon, it absorbs more microwaves and sparks occur. After a critical threshold, the material becomes a microwave absorber as it turns into activated carbon. A much more accelerated process with feedback occurs.

Figure 15. The role of sulfuric acid in the process and the mechanism of carbonization.

4.2. Mechanism of EMI Shielding and the Importance of Surface Chemistry and Physics

Activated carbon textiles obtained by this method showed electromagnetic shielding based on absorption and multiple internal reflections, as explained below:

1—Absorption: Surface functional groups are essential to inducing dipole polarization. In particular, functional groups such as -O and -OH cause unequal distribution of electrons and polarization, causing dispersion loss of EM waves [51].

2—Multiple internal reflections: Multiple internal reflections occur due to the roughness, heterogeneous structure, and random fiber orientation of the carbonized nonwoven. The nano-sized pores create interfaces that absorb large EM waves at minimal thicknesses [52].

The presence of micro-mesopores causes multiple reflections of EM waves at the interfaces between carbon and air, increasing shielding efficiency [19]. The impedance matching of the porous structure is close to that of air. Most EM waves are absorbed rather than reflected by this type of material. In addition, when EM waves encounter such a porous structure, multiple scattering and multiple reflections occur. These interactions are beneficial in trapping EM energy and converting it to heat [10].

Another critical issue is the surface roughness. There is an indirect positive correlation between roughness and EM shielding. This correlation can be explained by mechanisms such as internal reflection and trapping of EM waves between the gaps formed due to the roughness [53]. Higher surface roughness results in higher magnetic loss and permeability resistance and thus in higher EM shielding [54].

Considering all these factors, the dominant mechanisms of EM shielding are absorption and internal reflection and can be attributed to the presence of oxygen functional groups, the formation of many interfaces due to the high surface roughness, the micropore structure, heterogeneity, and the random fiber orientation of the carbonized nonwoven (Figure 16).



Figure 16. EMI shielding mechanism, principally absorption and multiple internal reflection.

4.3. Green Metrics of the Study

Table 7 and Figure 17 show that the specific energy and specific water values decrease as the acid concentration increases. As a result (Table 8), ultrasound treatment decreased the total cost compared to NaOH pre-cleaning. The use of sulfuric acid decreased water and energy consumption and cost.

Process mass intensity (PMI) is the total mass of materials used to produce a specified product mass. Materials include reactants, reagents, solvents used for reaction and purification, and catalysts. PMI is not a waste-based metric. It is an efficiency-based metric, and its value is relative. Based on the PMI, the proposed product seems to be commercially viable in terms of sustainability [55].

$$PMI = \frac{M_{Input}}{M_{P}}$$
(2)

where M_{input} = total mass used in process and M_p = mass of product [55].

	Pre-Post Treatment			EMS	E (dB)			Average EMSE	Maximu	ms	Minim	ıms	Specific EMSE	SSE/t
	ireatilient	1 GHz	2 GHz	3 GHz	4 GHz	5 GHz	6 GHz	dB	dB GH	z	dB GH	Iz	SSE	
	WATER	28.36	14.18	24.31	25.77	30.45	35.40	26.41	35.40	6	14.18	2	186.04	759.34
WATER	Microwave	32.93	27.06	33.41	23.71	15.89	27.02	26.67	33.41	3	15.89	5	187.86	766.76
	NaOH	24.09	10.30	35.34	15.88	16.87	26.34	21.47	35.34	3	10.30	2	151.23	617.26
2% H ₂ SO ₄	Ultrasound	34.73	15.12	21.39	21.77	20.87	23.31	22.87	34.73	1	15.12	2	161.06	657.37
	Microwave	35.24	18.56	29.61	27.29	21.28	29.41	26.90	35.24	1	18.56	2	189.47	773.33
	NaOH	28.37	8.85	39.54	16.38	16.44	26.21	22.63	39.54	3	8.85	2	159.41	650.66
4% H ₂ SO ₄	Ultrasound	31.79	14.65	26.33	23.09	18.08	25.51	23.24	31.79	1	14.65	2	163.71	668.20
	Microwave	33.25	24.39	29.90	33.50	35.56	38.49	32.52	38.49	6	24.39	2	229.03	934.81
	NaOH	27.79	12.82	26.81	17.14	19.11	25.30	21.50	27.79	1	12.82	2	151.41	617.98
8% H ₂ SO ₄	Ultrasound	33.49	16.79	22.18	26.23	24.11	28.93	25.29	33.49	1	16.79	2	178.12	727.04
	Microwave	38.60	26.84	32.69	37.92	31.67	30.69	33.07	38.60	1	26.84	2	232.93	950.71





Figure 17. Specific energy and specific water of materials produced with different concentrations of sulfuric acid.

Table 8. Green metric data for ultrasound-treated sample	es.
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ACTIVATOR	PMI	Specific Water mL/g	Specific Energy kWh/g	Average EMSE dB	Unit Cost \$/g
WATER	90.28	83.333	1.44	26.41	0.2274
2% H ₂ SO ₄	49.35	27.736	0.49	26.90	0.0817
4% H ₂ SO ₄	41.05	25.714	0.46	32.52	0.0849
8% H ₂ SO ₄	49.13	24.000	0.45	33.07	0.0975

5. Conclusions

Increasing amounts of waste pose a risk to human and environmental health, and disposing of this waste requires enormous energy. Therefore, obtaining carbon from waste is ecologically and economically more advantageous than using other materials. In the present study, a multi-layered textile was produced from waste fibers by the environmentally friendly needlepunch method, which uses purely mechanical entanglement, without the need for any chemicals. In the next stage, the textile structure was carbonized and activated in one step and converted into activated carbon without compromising the integrity of the fibrous network (supporting video) by microwave pyrolysis without inert gas. This last procedural step is novel in the literature.

Water and sulfuric acid were heat absorbers for microwave radiation, as well as activation agents. The use of sulfuric acid eliminated the production of greenhouse gases, increased product yield, and reduced water-energy consumption and sample shrinkage.

Activated carbon textiles obtained by this method were capable of electromagnetic shielding, with the dominant mechanisms being internal reflections and absorption, in

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the 20–30 dB range (99.9% attenuation) at 1–6 GHz. The maximum SSE/t value of 950.71 dB·cm²·g⁻¹ was obtained from the microwave post-treated activated carbon textile.

As acid concentration increases, pore numbers and surface roughness increase, which increases multiple internal reflections and thus contributes to EMI shielding. A 700-watt microwave post-treatment for 5 min positively affects conductivity and thus EMI shielding. The use of ultrasound treatment instead of NaOH cleaning reduced water-energy consumption and cost. In addition, selected green metrics, namely specific energy-water, process mass intensity (PMI), specific shielding effectiveness (SSE), and absolute effectiveness related to thickness (SSE/t), are presented to draw attention to the importance of these issues.

In the future, the carbonized textiles developed in this study can be used as composite reinforced materials or combined with other materials for EMI shielding applications. Other potential applications of the developed materials include green buildings, reinforced composites, ohmic heating, CO_2 capture, and energy harvesting and storage. Material strengthening will be undertaken in the second stage of this study. This aim can be achieved by combining carbonized textiles with higher-strength materials (woven, knitted, nonwoven, wood products, and paper) by lamination, needle punching, and sewing. In addition, polydimethylsiloxane (PDMS) encapsulation to create a protective layer is promising as a method for strengthening carbonized textiles.

Supplementary Materials: The following supporting information can be downloaded at: https://www. mdpi.com/article/10.3390/fib11120110/s1, Figure S1: TGA-DTG analysis of a five-layer nonwoven fabric consisting of waste cotton and jute fibers; Figure S2: TGA-DTG analysis of activated carbon textile obtained with 8% acid; Figure S3: TGA-DTG analysis of microwave post-treated activated carbon textile manufactured with 8% acid; Table S1: Studies with similarities and differences relative to this study and the originality of this study; Table S2: Raman analysis of activated carbon fabrics; Table S3: XRD data analysis [56–58].

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Abbreviations

AC	Activated Carbon
BET	Brunauer-Emmett-Teller
CHNS	Elemental Carbon, Hydrogen, Nitrogen, Sulphur
EMI	Electromagnetic Interference
FT-IR	Fourier-Transform Infrared Spectroscopy
FWHM	Full Width Half Maximum
MW	Microwave post-treated
PDMS	Polydimethylsiloxane
PTFE	Politetrafloroetilen or Teflon
PVA	Polyvinyl Alcohol
UT	Ultrasound treated

WATER	Nonwoven was	carbonized	with	only water
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- XPS X-ray photoelectron spectroscopy
- XRD X-ray diffraction

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