

Article

Electromagnetic Interference Shielding Material from Grape Seeds: A Sustainable Pyrolysis Route

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Abstract

Grape marcs represent one of the most effectively exploited biowaste resources through cascade valorization approaches, in which byproducts are processed via multiple sequential steps such as extraction, bio-treatment, and pyrolysis. In this study, we present a novel route for producing graphitic carbon (GC) from grape seeds derived from exhausted marc via pyrolysis. We integrate hydrolysis and CO₂ methanation in a one-pot methodology to valorize both bio-oil and gaseous pyrolysis byproducts. The GC obtained through pyrolysis is evaluated in GC/Polytetrafluoroethylene (PTFE) composites as an electromagnetic interference (EMI) shielding material across the X-band frequency range (8–12 GHz). This work demonstrates a viable and eco-friendly pathway to upcycle abundant biomass into a lightweight, sustainable, and highly tunable material, which represents a promising candidate for effective EMI shielding while simultaneously mitigating process emissions.

Keywords: graphitic carbon; pyrolysis; methanation; grape marcs; electromagnetic interference shielding



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1. Introduction

In recent years, biowaste management for the production of high-value carbonaceous materials has attracted significant attention due to the high carbon content and renewable nature of biomass. Organic residues from the wine industry represent a well-established example of circular valorization, in which wastes are converted into industrial and energy products through integrated processes [1]. Specifically, grape pomace and wine lees are processed via distillation and extraction to obtain bioethanol, tartaric acid, and polyphenolic compounds, which find applications in food, pharmaceutical, and chemical industries. The remaining byproducts, particularly exhausted grape marcs, can serve as feedstock for anaerobic digestion to produce biogas and biomethane, as well as for composting, thereby

contributing to sustainable soil management and carbon cycling. Pyrolysis, a thermochemical conversion process carried out in an oxygen-free environment, represents an alternative valorization pathway for exhausted grape marc, enabling the recovery of syngas, bio-oil, and high-value solid carbonaceous materials, namely graphitic carbon (GC) [2,3]. Pyrolysis parameters (such as temperature, heating rate, additives, etc.) play a crucial role in tailoring the degree of graphitization, porosity, and electrical conductivity of biomass-derived GC suitable as electrode materials in supercapacitors and batteries, adsorption and catalytic applications [4]. In parallel, the process parameters strongly influence both the yield and the quality of gaseous and liquid bioproducts. In particular, pyrolysis under a high-pressure hydrogen atmosphere, namely hydrolysis, has been demonstrated to promote calorific value, chemical stability, and miscibility with conventional fossil fuels of pyrolysis bio-oils by reducing their oxygen-to-carbon ratio (O/C) [5].

Recently, there has been growing emphasis on developing electromagnetic interference (EMI) shielding materials using graphitic carbon (GC) produced via the pyrolysis of waste biomass [6–10]. Implementing large-scale EMI shielding solutions within urban environments is essential not only to reduce electrosmog exposure but also to strengthen cybersecurity by preventing electromagnetic interference from compromising sensitive electronic systems. Although traditional metals are successfully used in this application field, they are often associated with drawbacks such as high density, high cost, limited flexibility, and poor corrosion resistance [7]. Thus, conductive polymer composites incorporating micro- and nanoscale conductive fillers such as GC are increasingly explored as sustainable, lightweight alternatives exhibiting strong chemical resistance. The efficacy of GC as EMI shielding fillers is primarily attributable to its conductive properties [10]. When incorporated into polymer matrices (films and 3-D porous scaffolds [6]), these conductive carbon fillers establish networks that attenuate electromagnetic waves mainly through two mechanisms: (i) reflection loss (SE_R) caused by the interaction with mobile charge carriers (free electrons) on the material's surface; and (ii) absorption loss (SE_A) resulting from dielectric and ohmic losses within the material [10].

We present a sustainable strategy to convert grape seed from exhausted marc waste into GC suitable for EMI shielding applications. Grape seeds from exhausted marc are suitable for enhanced graphitization due to their higher content of aromatic lignin compounds [11], which promote the ordering of C-sp² atoms into graphitic clusters [12]. The promoted development of graphitic domains in biomass-derived carbon significantly enhances electrical conductivity and interfacial polarization, leading to improved EMI shielding effectiveness [9]. Although the primary objective is the synthesis of high-quality GC, a distinctive feature of our biomass pyrolysis route is the one-pot combination of experimental approaches typically applied to the valorization of bio-oil and gaseous pyrolysis byproducts: the use of (i) hydrolysis conditions [5] and (ii) a methanation catalyst [13]. Indeed, molecular hydrogen can be exploited in methanation reaction, also known as the Sabatier reaction, to reduce the CO₂ footprint of biomass pyrolysis [13].

We provide experimental results on quantitative and qualitative characterizations of GC, as well as of gaseous and liquid byproducts obtained by this sustainable strategy. Moreover, GC is validated as a sustainable and cost-effective filler for polymer composites for electromagnetic interference shielding applications. Polytetrafluoroethylene (PTFE, or Teflon) is specifically selected as the polymer matrix due to its easy processability, low density, and crucial chemical stability and corrosion resistance, which are highly sought after for robust and reliable EMI shielding applications. Measurements are conducted across the X-band frequency range (8–12 GHz), utilizing two distinct GC loadings across a range of thicknesses from 0.2 mm to 1.8 mm.

2. Results and Discussion

Figure 1 shows the autoclave reactor setup and the picture of crushed grape seeds before and after pyrolysis treatment. According to several experimental protocols on biomass pyrolysis reported in the literature [3], grape seeds were thermally treated by a preliminary *carbonization* step at 500 °C, followed by *graphitization* at a higher temperature (800 °C). Both stages were tested in H₂ ambient with and without the methanation catalyst. The catalyst was loaded into the autoclave using dedicated crucibles to avoid direct contact with biomass feedstock. Specifically, we use a catalyst based on non-noble metal, Ni(0)/CeO_y/MK10 [14] to activate both H₂ and CO₂ reactants for promoting methanation process at relative low temperature with high yield and selectivity.

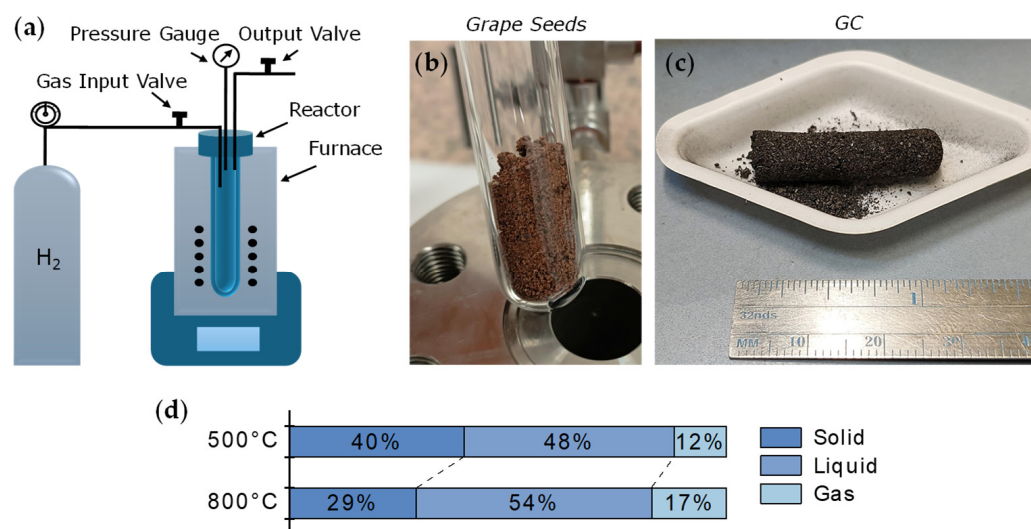


Figure 1. (a) Sketch illustrating the autoclave reactor setup. (b) Digital photographs of the exhausted grape seed feedstock and (c) GC after graphitization. (d) Biomass yields conversion (wt.%) to solid, liquid and gas products resulting from the two-step pyrolysis at 500 °C (carbonization) and 800 °C (graphitization) in H₂ atmosphere.

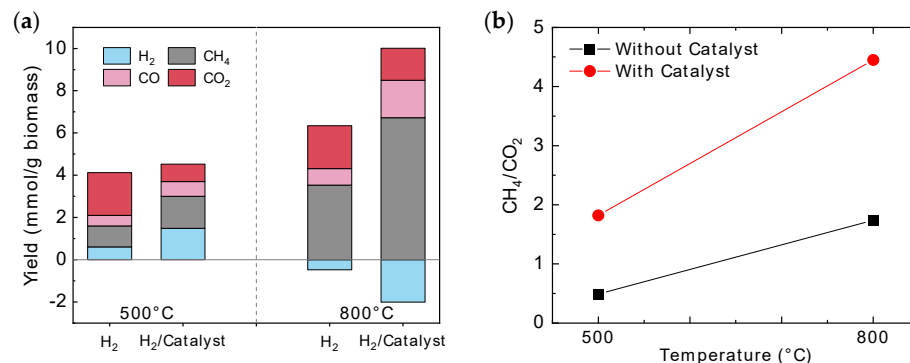
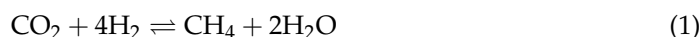
Figure 1d shows the weight percentages (wt.%) of biomass conversion into solid, liquid and gas products after *carbonization* and subsequent *graphitization* pyrolysis processes. Thermal treatment at 500 °C induces significant degradation of the intrinsic oils in grape seeds and promotes bond cleavage and structural rearrangement in their lignocellulosic fraction, with lignin undergoing partial aromatization and cellulose and hemicellulose largely decomposed [15,16]. Grape seed pyrolysis mainly gives rise to liquid products with an important percentage of solids that decreases from 40% to 29% after the higher-temperature pyrolysis stage. No substantial changes are observed in solid, gaseous and liquid product yields when pyrolysis is carried out with or without a catalyst.

The main products detected in the extracted organic phase of liquid byproducts are long-chain hydrocarbons and carboxylic acids (C > 9), derived from the seeds' lipid fractions and phenols present in the lignin portion. Table 1 reports the comparison (measured as relative area) between the main families of products in the organic fraction obtained at 500 and 800 °C, in the presence or absence of a catalyst. Interestingly, the presence of the catalyst is reflected in an important decrease in carboxylic acid fractions both at 500 and 800 °C. This indicates an important role of the catalyst in the deoxygenation reaction of uncondensed products reaching its surface during annealing.

Table 1. Comparison between the relative amount of the main families in the oil obtained under different pyrolysis experimental parameters.

Entry	Temp. [°C]	Gas	Catalyst	Linear and Cyclic Long Chain Hydrocarbons	Long Chain Carboxylic Acids	Phenols	Others
1	500	H ₂	No	24.4%	32.1%	20.7%	22.8%
2	500	H ₂	Yes	29.8%	22.3%	21.6%	26.3%
3	800	H ₂	No	27.3%	17.3%	25.6%	29.8%
4	800	H ₂	Yes	29.6%	13.2%	26.1%	31.1%

Histograms in Figure 2a indicate the moles of the four main gaseous species (H₂, CH₄, CO₂, CO) produced after pyrolysis at 500 °C and 800 °C, normalized per gram of biomass feedstock. In the absence of catalyst, CO₂ is the most abundant gaseous species produced at 500 °C. Indeed, CO₂ is a common product in lignocellulosic biomass pyrolysis [17], and oxidation reactions cannot be excluded due to molecular oxygen adsorbed on the feedstock. When the catalyst is introduced into the autoclave, a slight increase in the total moles of gaseous species is observed, as indicated by pressure measurements. This effect can be ascribed to a secondary catalyzed pyrolysis stage of uncondensed products reaching the catalyst surface. The graph in Figure 2b shows that the CH₄/CO₂ ratio exhibits a nearly fourfold increase when the catalyst is used (from 0.5 to 1.8). According to ref. [14], this experimental evidence supports the occurrence of the catalyzed Sabatier reaction, where the high concentration of molecular hydrogen shifts the equilibrium toward CH₄ and H₂O formation (Equation (1)):

**Figure 2.** (a) Histograms of the moles of the main gaseous species produced during the pyrolysis process per gram of biomass during the carbonization step at 500 °C and the graphitization step at 800 °C in a H₂ atmosphere with and without the methanation catalyst. (b) Ratio between the CH₄ and CO₂ moles produced during pyrolysis at different temperature of 500 °C and 800 °C, with and without catalyst.

Despite the H₂ consumption to promote CO₂ conversion, the overall hydrogen balance (the difference between the number of H₂ moles in the autoclave before and after pyrolysis) is positive after pyrolysis at 500 °C. Indeed, hydrogen is one of the main products of steam and dry reforming of bio-oil and hydrocarbons during thermal treatment, and these processes can be further enhanced by the presence of a catalyst [17].

Conversely, the second pyrolysis step at 800 °C (*graphitization*) leads to a negative hydrogen balance since higher temperatures promote H₂ consumption by hydrodeoxygenation reactions of bio-oils, as expected for hydrolysis processes [18].

Figure 3 reports the morphological, structural and Raman characterizations of the solid product of pyrolysis at 500 °C and 800 °C with catalyst. SEM images show the presence

of charging effects in the sample after *carbonization* at 500 °C (Figure 3a), which are not observed after pyrolysis at 800 °C (Figure 3b). This evidence can be correlated to improved conductive properties of the solid product after thermal treatment at higher temperature. A comparison of the TEM images of GC after carbonization (Figure 3c) and graphitization (Figure 3d) reveals the formation of characteristic layered structures typical of graphitic materials following the second pyrolysis step at elevated temperature. Figure 3d shows areas of the sample with higher and lower contrast images corresponding to, respectively, thicker and thinner layers of stacked graphene foils.

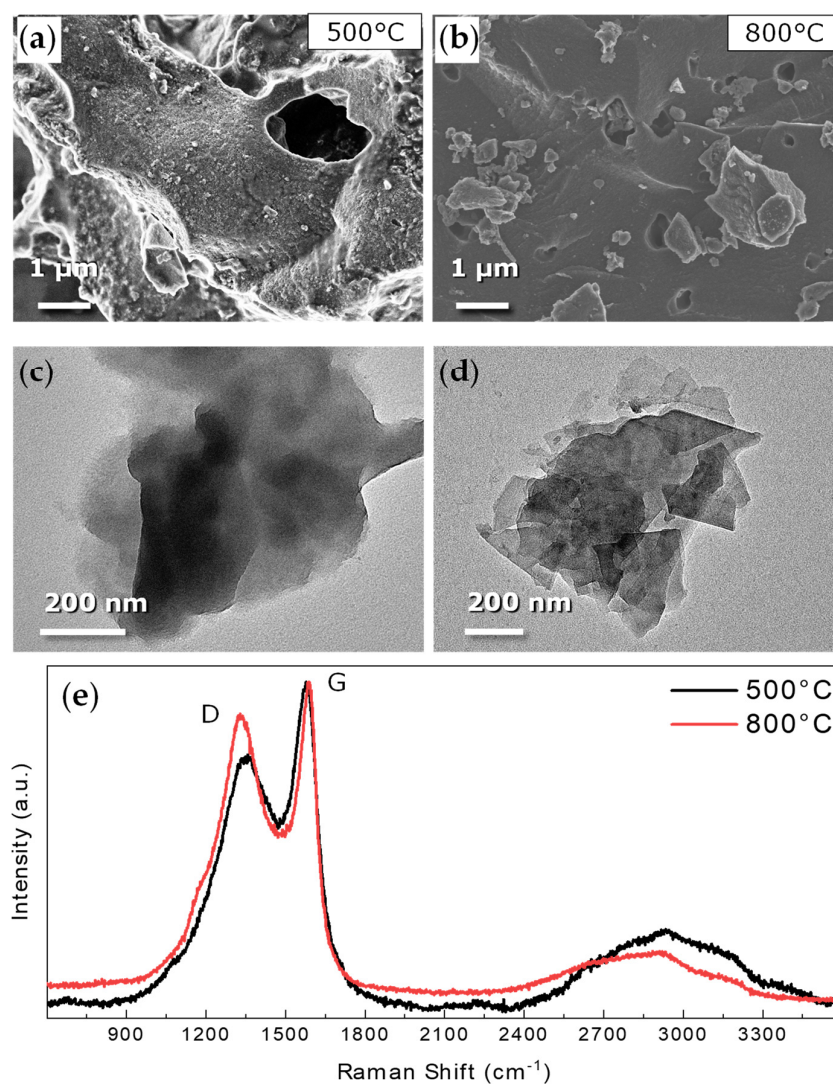


Figure 3. (a,b) SEM Image of GC after carbonization at 500 °C (a) and after graphitization at 800 °C (b). (c,d) TEM of GC after carbonization at 500 °C (c) and after graphitization at 800 °C (d). (e) Raman spectra of solid products obtained at 500 °C and subsequent graphitization at 800 °C.

Direct evidence of graphitic phase evolution during the two pyrolysis stages is provided by the Raman analysis [19]. Raman spectra in Figure 3e are mainly characterized by the first-order D and G modes of graphite around, respectively, 1350 cm⁻¹ and 1580 cm⁻¹. The D band is associated with the breathing modes of C-sp² rings. This mode is Raman inactive in perfect graphite since it requires a double-resonance process involving an elastic scattering with structural defects. The G mode involves the in-plane bond-stretching of two sp² atoms, and it can also be found in aromatic and olefinic chains since, in contrast

to D mode, it does not require the presence of sixfold aromatic rings. This forms the basis of the phenomenological three-stage model, defined as the “amorphization trajectory”, developed by Ferrari and Robertson, which correlates the evolution of the ratio between intensities of the D and G peaks with the transition from an amorphous carbon material to a graphitic one [20,21]. Thermal treatment at 800 °C favors clustering of the sp^2 phase into ordered rings, thus increasing the relative intensity of the D peak [22]. In parallel, the increasing graphitic structural order leads to a stiffening of the G phonon mode, evidenced by a shift in its peak position from 1578 cm^{-1} to 1587 cm^{-1} [20–22].

We evaluated the EMI shielding effectiveness (SE) of GC composites fabricated using PTFE as the polymer binder, a material that has been successfully used with biochar in related studies [23]. Figure 4a,b respectively show the total shielding effectiveness (SE_{tot}) in the 8–12 GHz frequency range for the GC/PTFE composites with 30 and 60 wt.% GC loading level, and with thicknesses in the range of 0.2 to 1.8 mm. The lower-loaded composites (30 wt.%) exhibited SE values ranging from 1 dB to 5 dB, whereas doubling the filler concentration to 60 wt.% significantly enhanced performance, yielding SE values from 8 dB to 14 dB across the same thickness range (Figure 4c). Table S1 of the Supplementary Materials provides a comparison between the performance measured in this work and data reported in the literature for EMI shielding composites based on both biomass-derived and fossil-derived graphitic carbon [6,8,10,24–30].

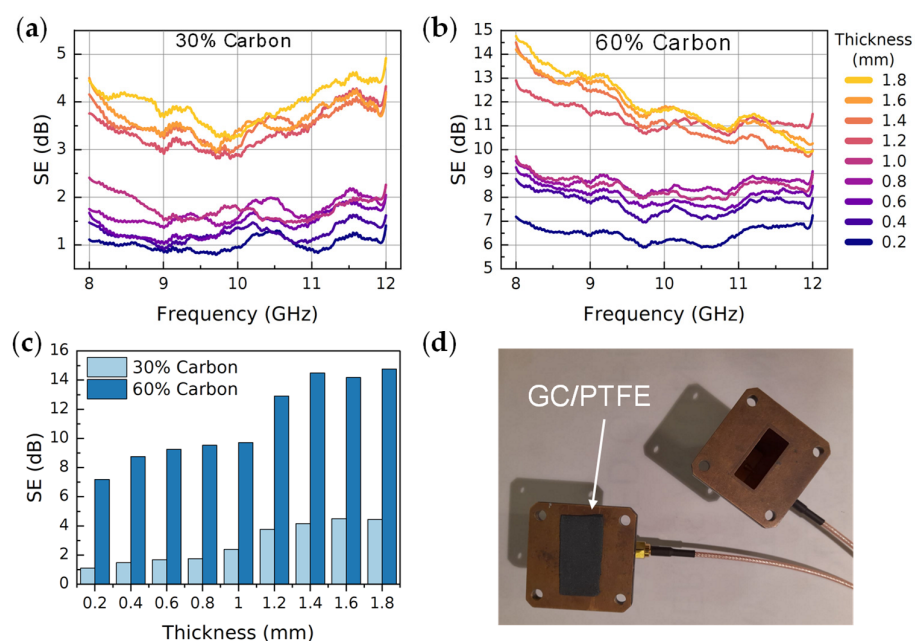


Figure 4. Total shielding effectiveness (SE) for the GC/PTFE composites with (a) 30 wt.% and (b) 60 wt.% GC loading, showing the effect of shielding material thickness (0.2–1.8 mm) across the X-band frequency range (8–12 GHz). (c) SE at 8 GHz of GC/PTFE composites for various compositions and thicknesses. (d) Picture of the Vector network analyzer (VNA) ports used for measuring the S-parameter holding the GC/PTFE sample.

In highly conductive materials, the dominant attenuation mechanism is typically reflection loss, which arises from the strong impedance mismatch [9,24]. According to electromagnetic theory regarding EMI mechanisms, this is generally independent of material thickness, since it depends on the material’s reflectance [6]. Regarding our composite, the clear dependence of SE_{tot} on thickness indicates that absorption loss is also a critical contributing factor for overall performance increase. Indeed, the absorbance increases directly with thickness, as the wave travels a longer path length within the material, providing more opportunities for energy dissipation. This absorption capability is facilitated by the

porous graphitic structure of the GC, which provides internal interfaces for wave scattering, interfacial polarization (due to conductivity mismatch between GC and PTFE), and ohmic losses [9,25].

3. Materials and Methods

Grape seeds, already separated from the exhausted grape marc and dust, were supplied by a local distillery as byproduct of bioethanol production. They were used without drying treatment, with a moisture content of 10% *w/w*, and were mechanically milled to reduce particle size (100–500 μm) for optimal pyrolysis. Methanation catalyst was prepared as reported in reference [14].

GC production by pyrolysis

Grape seeds pyrolysis was carried out in a 100 mL stainless steel autoclave employing a two-step *carbonization/graphitization* thermal process at a pressure of 2.5 bar in H_2 atmosphere. The *Carbonization* process was performed for 6 h after reaching a temperature of 500 $^{\circ}\text{C}$ with a 1 $^{\circ}\text{C}/\text{min}$ heating rate. After cooling, the *graphitization* step was carried out at 800 $^{\circ}\text{C}$ (heating rate of 3 $^{\circ}\text{C}/\text{min}$) for 3 h of graphitization. Thermal treatment times were set to ensure both efficient biomass conversion to GC and catalyst-mediated CO_2 conversion. The biomass and catalyst were spatially segregated within the autoclave by placement in ceramic crucibles. This setup effectively minimized cross-contamination of the solid products and catalyst in the crucibles with the liquid products that condensed on the cooler areas of the autoclave walls.

Preparation of the GC/PTFE composites

Composites were prepared using GC, as the conductive filler, and a 60 wt.% dispersion in H_2O of Polytetrafluoroethylene (PTFE) as the polymer matrix. Composite samples were prepared by mixing the GC powder with the PTFE emulsion to achieve two commonly used filler loadings based on the weight percentage (wt.%): 30 wt.% and 60 wt.% GC relative to the mass of PTFE [9]. The mixtures were mechanically processed and rolled into thin sheets with targeted thicknesses ranging from 0.2 mm to 1.8 mm. The prepared sheets were dried at room temperature for 24 h. The resulting plates were cut to the final dimensions of 40 $\times$ 20 mm.

Characterization

Solid, liquid and gaseous products were sampled after cooling to room temperature. The solid product yield (wt.%) was estimated from the weight difference between the initial biomass feedstock and the final solid product. Gaseous product yield has been estimated by multiplying the molecular weight of each gaseous species by the respective number of moles produced. The latter were determined by knowing the final pressure (at room temperature) of the gaseous phase, the autoclave volume, and the molar percentages (derived by gas chromatography). As for the moles of H_2 produced or consumed during thermal treatment in a hydrogen ambience, it has been estimated as the difference between the number of H_2 moles estimated before and after the thermal process. The liquid yield was calculated as the difference between the weight of the initial biomass and the combined weights of the solid and gaseous phases.

Raman measurements were performed using a LabRam HR (Horiba JY, HORIBA FRANCE SAS, Palaiseau, France) system with 532 nm laser excitation and by using a long working distance 50 $\times$ objective lens (focusing laser spot of 1.2 μm). The laser power was kept at 10 mW to avoid thermal damage to the biochar and GC samples.

Quantitative analysis on the gaseous mixture was performed using a Thermo-Fisher gas-chromatograph (TCD-GC) equipped with a Supelco Carboxen 1010 Plot capillary column (Thermo Fisher Scientific, Waltham, MA, USA).

The oil phase was recovered with 2×3 mL of hexane in order to solubilize the organics to be analyzed with GC-MS for a semi-quantitative identification of compounds.

Field Emission Scanning Electron Microscopy (FE-SEM) was performed by a Zeiss Sigma microscope (Jena, Germany) operating in the range of 0–10 kV and equipped with both an in-lens secondary electron detector and an INCA Energy Dispersive Spectroscopy (EDS) detector. Samples were mounted onto stainless-steel sample holders using double-sided conductive carbon tape and grounded with silver paste.

TEM analyses were performed by using a JEOL JEM-1011 microscope (Akishima, Japan) operating at 100 kV and equipped with a high-contrast objective lens, a W filament as electron source, with an ultimate point resolution of 0.34 nm. Images were acquired by a Quemesa Olympus CCD 11 Mp Camera (Hachioji, Japan). Samples were prepared by dipping 300 mesh amorphous carbon-coated Cu grids in GC aqueous dispersions.

The experimental characterization of the samples SE was performed using a VNA Keysight FieldFox N9917A (Santa Rosa, CA, USA) connected to two X-band (8.2–12.4 GHz) aluminum coaxial-WR90 waveguide transitions. The samples were mounted between the transitions, carefully aligned, and their S-parameters were recorded over the operating band. All measured S-parameters were first corrected using a full TRL de-embedding procedure [31], employing standard THRU, REFLECT/SHORT, and LINE calibration structures. This step removes the influence of the fixtures and waveguide transitions, yielding the intrinsic transmission and reflection response of the material sample, which forms the basis for the extraction of its shielding efficiency. After de-embedding, the complex S-parameters were regularized by applying a Savitzky–Golay smoothing filter [32] of polynomial order 3 along the frequency axis. The filter performs a local least-squares polynomial fit to each S-parameter component, effectively suppressing high-frequency noise while preserving the underlying spectral features. Finally, passivity was enforced using the built-in passivity-projection routine of the scikit-rf library [33,34], ensuring that each S-parameter matrix satisfies the physical requirement of power conservation. This three-stage pipeline ensures that the resulting S-parameters are both noise-reduced, physically compliant, and representative of the true electromagnetic behavior of the measured material samples.

4. Conclusions

We presented a novel experimental protocol designed for efficient valorization of wine waste using a cascade strategy. Grape seeds from exhausted grape marc, as residual solid byproducts of bioethanol production, have been used as feedstock for GC synthesis by pyrolysis. Hydropyrolysis and CO₂ methanation methodologies have been combined in a one-pot route to valorize pyrolysis byproducts. In particular, the Ni(0)/Ce_xO_y/MK10 catalyst has been demonstrated to enhance both CO₂ footprint mitigation and deoxygenation pathways in bio-oil, effectively reducing the concentration of long-chain carboxylic acids. GC derived by grape seeds has been validated in GC/PTFE composite as an EMI shielding material with a performance comparable to previously reported biochar materials [25]. Moreover, producing graphitic carbon from grape seeds offers economic advantages by valorizing a low-cost, locally available waste stream while simultaneously generating marketable byproducts (such as BTX compounds [18]), bio-oils, and syngas. When integrated into a multiproduct or cascade valorization framework, this approach further improves overall process profitability.

Supplementary Materials: The following supporting information can be downloaded at: <https://www.mdpi.com/article/10.3390/catal16030226/s1>, Figure S1: Fourier Transform Infrared–Attenuated Total Reflectance (FTIR-ATR) spectra of graphitic carbon (GC) obtained after two-step pyrolysis (carbonization at 500 °C, and graphitization 800 °C) of grape seeds. Table S1: Shielding effectiveness of graphitic carbon-filled composites.

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Data Availability Statement: Data are contained within the article and Supplementary Materials.

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Conflicts of Interest: The authors declare no conflicts of interest.

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