Multifunctional Carbon-Based Hybrid Foams for Shape-Stabilization of Phase Change Materials, Thermal Energy Storage, and Electromagnetic Interference Shielding Functions

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Abstract: Carbon-red mud foam/paraffin hybrid materials were prepared and studied for their thermal energy storage and electromagnetic interference (EMI) shielding properties. The host matrices were prepared utilizing the polymeric foam replication method, with a polyurethane sponge as a template, resin as a carbon source, and red mud as a filler. The paraffins, n-octadecane (OD) and the commercial RT18HC, were used as organic encapsulant phase change materials (PCMs) into the open pore structure of the foams. The foams’ morphological and structural study revealed a highly porous structure (bulk density, apparent porosity $P > 65\%$), which exhibits elliptical and spherical pores, sized from 50 up to 500 $\mu$m, and cell walls composed of partially graphitized carbon and various oxide phases. The hybrid foams showed a remarkable encapsulation efficiency as shape stabilizers for paraffins: 48.8% (OD), 37.8% (RT18HC), while their melting enthalpies ($\Delta H_m$) were found to be 126.9 J/g and 115.5 J/g, respectively. The investigated hybrids showed efficient electromagnetic shielding performance in frequency range of 3.5–9.0 GHz reaching the entry-level value of ~20 dB required for commercial applications, when filled with PCMs. Their excellent thermal and EMI shielding performance places the as-prepared samples as promising candidates for use in thermal management and EMI shielding of electronic devices as well.

Keywords: carbon foams; thermal energy storage; red mud; porosity; EMI shielding; organic PCMs

1. Introduction

The phase change materials (PCMs) have attracted increasing attention over the last decades as the most important factor for the development of Latent Heat Thermal Energy Storage Systems (LHTESS) [1,2]. PCMs are actually substances with high latent heat, capable of storing and releasing large amounts of energy at a certain temperature. These materials are exhibiting great potential in various technological applications such as in textiles [3], electronics [4], and in the building sector [5], where high latent heat is needed.

Currently, one of the main challenges in the construction industry is the buildings’ reduction of cooling and heating requirements, especially when considering the climate change. PCMs could enhance buildings’ thermal performance when introduced into building components (e.g., impregnating concrete, gypsum, ceramic masonry [6]). Their incorporation into these materials could prevent rapid changes on the indoor temperature, reducing the losses of large amounts of energy, throughout the reduction of heating and/or cooling demands, while maintaining the same comfort level.

Generally, PCMs used in building construction components can be organic and inorganic materials, as well as eutectics [7–12]. The organic PCMs are mainly paraffins and
fatty acids, whereas the inorganic ones are usually salt hydrates. The eutectics are mix-
tures of two or more organic and/or inorganic PCMs, whose melting temperature covers
some specific applications [13]. Among these materials, organic PCMs have several assets
when compared to the other ones (inorganic, eutectics), such as the absence of subcooling
effects, non-toxic nature, great stability upon cycling, and transformation without phase
separation [14–16]. However, it should be noted that paraffins are quite flammable and
exhibit low thermal conductivity, which combined with high volume variation during the
phase transition and high leakage in the melting state could cause serious failures when
utilized in thermal systems [17]. The most expedient method to prevent paraffin leakage
during solid–liquid phase transition, and to reduce down the corrosion level of structural
materials, is the use of a shape stabilizer (ss) supporting matrix, which encapsulates the
PCM that allows it to sustain its molten form stationary at elevated temperatures [7,18].

One promising route to enhance the thermal conductivity of the final hybrid product
(building component + PCM + ss) is the dispersion of high thermal conductivity additives
into the PCM, such as (i) expanded graphite, (ii) carbon nanotubes (CNTs) or nanofibers,
(iii) metal, and (iv) ceramic nanoparticles [19–22]. One other way to achieve the desirable
reinforcement of the hybrids is to select a highly conductive ss matrix, such as metal foams
and/or ceramic porous/foam structures [23–25]. In order to find an ideal combination of
materials, a large number of porous matrices have been investigated so far as thermally
conductible as candidates for PCMs, including activated carbons and other carbon-based
porous nanomaterials, zeolites, silicate mesoporous materials, porous/foams ceramics,
polymers, metal-organic frameworks, metallic foams, and other various types of composites,
i.e., the supporting materials are more than one [24,26–28].

Carbon-based porous materials are considered as shape stabilizers for PCMs, due
to their higher thermal conductivities (up to 2.1 W/(m·K)) [29] when compared to the
polymers (<0.05 W/(m·K) or ceramic analogues (~0.1 W/(m·K). On the other hand, even
though the metal foams exhibit high thermal conductivities (>10 W/(m·K), [30] they must
possess an optimal porosity, depending on the PCMs thermal and geometrical specifications,
in order to favor efficient heat transfer. Thus, a large number of research studies have
reported the development of carbon-based foams, aerogels, or graphene PCMs-composites,
synthesized by using biomass, polysaccharides, and synthetic polymers as raw materials
and templates [31–36].

Additionally, lightweight carbon foams have also been considered as the most promis-
ing candidates for providing excellent electromagnetic interference (EMI) shielding prop-
erties, since they possess a three-dimensional (3D) network structure that enhances the
multiple reflections of incident electromagnetic waves and improves the microwave ab-
sorption [37–39]. Generally, metals show good shielding performance, but their composites
suffer from several drawbacks, including high densities, high reflection, easy corrosion, and
processing difficulties. However carbon-based foams have many other advantages such
as low density, good stability, resistivity against chemical corrosion, operational thermal
conductivity, high porosity, and the ability to suppress EMI from electronic devices and
reduce the risk of electromagnetic fields (EMFs) exposure of people in their houses and
workplaces [37,39–41].

So far, some studies reported the fabrication of carbon foams utilizing recycled indus-
trial wastes [41,42]. For instance, carbon foam hybrids were synthesized as lightweight EMI
shielding materials, using as raw materials a mixture of dried Red Mud (RM), which is a
solid containing metallic oxide impurities that is a waste product of bauxite refinement [41].
The prepared hybrid foam showed excellent EMI SE in the 8.2–12.4 GHz region (51.4 dB at
8.2 GHz) and excellent fire-resistant performance, due to the thermal insulating nature of
metal oxides present in red mud. In another study, phenolic foam composites reinforced
with red mud microparticles were prepared using a thermal foaming method [43]. The
composite foams reinforced with 15 wt% of modified red mud microparticles showed
improvement on their tensile and impact strength by ~82%, along with enhancement of
their flame retardant properties. However, it should be noted that none of these stud-
ies investigated carbon foams incorporating organic PCMs, and hence did not evaluate the electromagnetic interference shielding effectiveness of these materials in conjunction with their capability for efficient thermal management, including shape stabilization, heat absorption, and thermal conductivity.

In this present work, hybrids derived of shape-stabilized organic PCMs into carbon-red mud hybrids were fabricated and studied for the first time, concerning their thermal and EMI shielding properties. It should be noted that as shape stabilizers, carbon foams were prepared according to already published research works [40,41]. However, our pioneer hybrids, which incorporated organic PCMs into the carbon foam matrices, presented multifunctional properties such as: shape-stabilization of PCMs, latent heat thermal energy storage, good mechanical behavior, light weight, and EMI shielding properties. All these properties mentioned before are of high importance in construction section, where the building components (i.e., gypsum or cement boards) should possess enhanced thermal energy storage and EMI shielding efficiency.

2. Materials and Methods

2.1. Preparation of Carbon-Red Mud Foams (CRMFoams)

Carbon red mud foams (CRMFoams) were synthesized based on experimental routes found in published research [40,41] with some modifications, using commercially available polyurethane (PU) foams (Scotch-Brite™ 2-In-1 Sponge the yellow part). Initially, a liquid phenolic resin black (C₈H₆O₂) by “MATHIOS refractories S.A.” and an acetone (C₃H₆O) mixture was fabricated (C₈H₆O₂:C₃H₆O = 50:50 ratio), which was stirred for 1 h. Subsequently, a second mixture consisting of RM (thermophysical properties are given in [44]) and acetone was prepared and placed into a sonication bath for 1 h to achieve more efficient dispersion of the RM (Ultrasonic cleaner WUC-A 1, 2–22). It should be noted that prior to the dispersion procedure, the RM was pulverized in a planetary mill (Fritsch Planetary mill, Pulverisette 5 05.6020.00) for five hours, resulting in a thin powder. Afterwards, the thin powder was passed through a 70 µm mesh sieve to retain the large agglomerates.

After the dispersion, the thin RM powder was added to the initial phenolic resin mixture (C₈H₆O₂:RM = 5 or 2.5) and stirred for 1 h under continuous magnetic stirring. The resulted polyurethane foam pieces were cut in cylindrical shapes (d = 3 cm, h = 2 cm) and dipped into the homogenous slurry. Then, the impregnated pieces were dried at 80 °C for 24 h, followed by 10 h curing at 220 °C in an air atmosphere, while the carbonization took place in the presence of an inert atmosphere up to 1100 °C for 1 h (hereafter named CRMFoam-x, where x = 5 and 2.5 the ratio C₈H₆O₂:RM).

2.2. Preparation of Hybrid Foams, ss-CRMFoam@PCMs

Prior to the infiltration process, the CRMFoams were dried at 150 °C for at least two hours to remove any remaining water. Then, the CRMFoams were dipped into melted paraffins (pure paraffin wax—industrial RT18HC of Rubitherm Technologies and n-octadecane (OD) of Sigma Aldrich (thermophysical properties are presented in Table 1), heated until 90 °C) for almost five minutes in order to stop the bubbles’ extraction from the porous medium (hereafter named ss-CRMFoam-x@OD and ss-CRMFoam-x@RT18, where x = 5 and 2.5 is the ratio C₈H₆O₂:RM). After the removal of the foams from the melted paraffins, the samples were placed onto filter papers to release the excess paraffin that was not supported from the foam’s pores or from the foam’s surface. The successful encapsulation of the paraffins into the foams was tested by weighting the samples prior to and after the experimental procedure. Additionally, the possible release of the paraffin from the ss-CRMFoams@PCMs was examined via leaching tests, according to published standards [45].
Table 1. Thermophysical properties of the organic paraffins.

| Paraffin     | Melting (°C) | Solidification (°C) | Melting Latent Heat (kJ/kg) | Solidifying Latent Heat (kJ/kg) | Density (g/mL) | Thermal Conductivity (W m⁻¹ K⁻¹) | Ref. |
|--------------|--------------|---------------------|-----------------------------|--------------------------------|----------------|-----------------------------------|------|
| RT18HC       | 17–19        | 19–17               | 260                         | -                              | 0.77 (liquid)  | 0.2                               | [46] |
| n-Octadecane | 28.12        | 25.1                | 236.98                      | 233.27                         | 0.868          | 0.334                             | [47] |

2.3. Leaching Tests

Leaching tests were performed to examine the ability of the foams to entrap the paraffin. First, the weight of the ss-CRMFoams@PCMs was measured, and then they were placed onto filter papers at 30 °C for 8 h and afterwards sequentially at 80 °C for 72 h. Afterwards, the weight was measured once more in both cases.

2.4. Porosity and Density Measurements

Porosity and density measurements were performed using Archimedes’ immersion method according to the ASTM C20–00 standard [48]. The determination procedure of the apparent porosity, water absorption, apparent specific gravity, and bulk density of the CRM-Foams involved: (a) the drying of the specimens to constant weight and the determination of their dry weight (D), (b) the immersion of the specimen into distilled water and its boiling for two hours, followed by cooling to room temperature, (c) determination of the weight, S, of each test specimen after boiling and while suspended in water, (d) Determination of the saturated weight, W (after determination the suspended weight, all drops of water from the surfaces were removed by means of a cotton cloth). Then, the calculation of Exterior Volume (V), Apparent Porosity (P), Water Absorption (A), Apparent Specific Gravity (T) and Bulk Density (B) of the specimens, was obtained by the following equations:

\[ V (\text{cm}^3) = W - S \]  
\[ P(\%) = \frac{[(W - D)/V]}{100} \]  
\[ A(\%) = \frac{[(W - D)/D]}{100} \]  
\[ T = \frac{D}{D - S} \]  
\[ B(\text{cm}^3) = \frac{D}{V} \]

2.5. X-ray Diffraction Measurements

The X-ray powder diffraction patterns of the materials were collected on a D8 Advance Bruker diffractometer using Cu Ka (40 kV, 40 mA, \( \lambda = 1.54178 \text{ Å} \)) radiation and a secondary beam graphite monochromator (measurement conditions: 2θ = 10–70 degrees, in steps of 0.02 degrees and 2 s counting time per step).

2.6. Vibrational Spectroscopy

Raman spectra were recorded with a micro-Raman system RM 1000 Renishaw using a laser excitation line at 532 nm (Nd-YAG) in the range of 500–2000 cm⁻¹. Laser power ~10 mW was used with 2 µm focus spot.

Infrared (FT-IR) spectroscopy was performed on powdered samples dispersed in KBr pellets with a JASCO FT/IR-6000, Fourier transform spectrometer. The spectra were obtained as the average of 32 scans at 4 cm⁻¹ resolution and were measured in the wavenumber range of 400–4000 cm⁻¹.

2.7. Thermogravimetric Analysis (TGA)

TGA measurements were performed using a Setsys Evolution-Setaram TG-DTA analyzer. The experimental procedure involved the introduction of approximately 30 mg of
the sample in a platinum crucible, setting the heating and gas (N$_2$) flow rates, and then performing the test. For all experiments, heating and N$_2$ flow rates were kept constant at 5 °C min$^{-1}$ and 25 mL min$^{-1}$, respectively, in the temperature range from ambient to 700 °C.

2.8. Scanning Electron Microscopy (SEM)

SEM was utilized to observe the morphology of the CRMFoams. Images were taken with a JEOL JSM-5600 system, which is equipped with an energy dispersive spectrometry (EDS) by Oxford Instruments company. The sample was gold sputtered prior to the measurements, to achieve easier high-resolution imaging.

2.9. Thermal Properties

The thermal properties of the fabricated samples were measured by Differential Scanning Calorimeter (DSC) at a heating rate of 10 °K/min under N$_2$ atmosphere. In addition, the thermal conductivity $\lambda$ of the CRMFoams was determined on a thermal conductivity analyzer (TCi Thermal Conductivity Analyzer, C-Therm Technologies), according to ASTM D7984-16 [49], using the Modified Transient Plane Source technique. Cylindrical samples with nominal diameters of 40 mm (thickness) and height of ~30 mm were tested, and the final value of $\lambda$ was calculated using the average of 10 individual measurements.

2.10. Mechanical Properties

The mechanical properties were evaluated by measuring the compressive deformation behavior of foams, by means of a universal testing machine Shimadzu Autograph AG 25 TA, 0.5 mm/min displacement; the results are the average of measurements made on, at least, 5 specimens.

2.11. Electromagnetic Interference (EMI) Shielding Properties

The EMI shielding effectiveness properties were evaluated using a P9372A Keysight Streamline Vector Network Analyzer (Keysight, Santa Rosa, CA, USA) and two sets of microwave standard 15 dB gain waveguides (WR 187 and WR 147, respectively, obtained from Advanced Technical Materials Inc. (ATM), Patchogue, NY, USA) covering a broad C frequency band in the range of 3.5–9.0 GHz, which is typical for wireless communications, such as satellite communications, Wi-Fi devices, cordless telephones, weather radar systems, etc. In particular, every sample was placed in the middle of each set of waveguides, and its scattering parameters (S-parameters; $S_{11}$, $S_{12}$, $S_{22}$, $S_{21}$) were recorded.

3. Results and Discussion

3.1. Characterization and Properties of CRMFoams

3.1.1. Optical and SEM Images

Figure 1a,b shows the optical images of CRMFoam-5 and CRMFoam-2.5 at different magnifications. The images of both carbon foams reveal highly porous bodies with more or less elliptical and spherical pores with sizes ranging from 50 up to 500 μm. It should be noted that the CRMFoam-25 sample, showed smaller wall thickness than that of CRMFoam-5 sample. A possible reason for this last fact is the higher employed temperature for the pyrolysis of CRMFoam-2.5 sample (1100 °C) than that for CRMFoam-5 (1000 °C) sample, which favors greater sintering of red-mud constituted oxides. The SEM images of the same carbon foams are depicted in Figure 2 as well the elemental color mapping at the microstructural level using EDS analysis. As shown in this figure, the carbon foams exhibit submicro-sized porous and a dense wall structure on which the various elements of red-mud are uniformly dispersed.
Figure 1. Optical images of CRMFoam-5 (a) and CRMFoam-2.5 (b) samples at different magnifications.

Figure 2. SEM images of CRMFoam-2.5 sample at different magnifications (a). Elemental color mapping at the microstructural level (b) and EDS spectrum (c) showing the distribution of elements and the composition of the foam.
3.1.2. X-ray Diffraction Patterns and Raman Spectra

The carbon foams CRM Foam-5 and CRM Foam-2.5 were prepared using red mud as raw material, that was collected from the alumina refining plant Aluminium of Greece MYTILINEOS. Red mud is the main filtered bauxite residue (BR known as ferroalumina), generated during the extraction of alumina from bauxite ore using Bayer process. Chemical analysis of the Greek filtered bauxite residue revealed the following composition for the solid content of BR: 43.59 wt% Fe$_2$O$_3$, 22.78 wt% Al$_2$O$_3$, 8.44 wt% CaO, 6.35 wt% SiO$_2$, 5.88 wt% TiO$_2$, 1.93 wt% Na$_2$O, 0.56 wt% MgO (LOI 9.30 wt%) [50].

The XRD patterns of the red mud and the CRMFoams, CRM Foam-2.5 (40 wt% in RM), and CRM Foam-5 (20 wt% in RM) are presented in Figure 3. The pattern of RM exhibits a number of sharp diffractions peaks, which can be attributed to crystalline phases of hematite (Fe$_2$O$_3$– main peaks at 32.2°, 35.7°, JCPD file no.: 33-0664), gibbsite (Al(OH)$_3$–main peak at 18.2°, JCPD file no.: 70-2038), diaspore (α-AlO(OH)– main peak at 22.3°, JCPD file no.: 79-178138), calcite (CaCO$_3$– main peak at 29.4°, JCPD file no.: 83-0577), sodium aluminum silicate (NaAlSiO$_4$– main peak at 24.1°, JCPD file no.: 02-0339), perovskite (CaTiO$_3$– main peaks at 33.0°, 47.2°, JCPD file no.: 82-0239).

Figure 3. X-ray patterns of red mud (RM) and the CRMFoams, CRM Foam-2.5 (40 wt% in RM) and CRM Foam-5 (20 wt% in RM). No.: 44-1292, austenite (C,Fe-JCPD file no.: 23-0298) and alpha-Fe (Fe, JCPD files no.98-0064). In addition, the phases of gehlenite (Ca$_2$Al[AlSiO$_7$]—main peak at 31.4°, JCPD file no.: 79-2421), iron silicon carbide (C$_{0.17}$ Fe$_{0.81}$ Si$_{0.02}$)—main peak at 39.5°, 44.7°, JCPD file no.: 19-20628), tricalcium aluminate (Ca$_3$Al$_2$O$_6$—main peak at 33.1°, 47.6°, JCPD file no.: 38-1429) and perovskite (CaTiO$_3$—main peaks at 33.0°, 47.2°, JCPD file no.: 82-0239) are easily identified as well.

The pattern of carbon foam CRM Foam-5, is found to be a complex mixture of phases mainly comprised of hematite (Fe$_2$O$_3$–main peaks at 32.2°, 35.7°, JCPD file no.: 33-0664), perovskite (CaTiO$_3$– main peaks at 33.0°, 47.2°, JCPD file no.: 82-0239) and Gehlenite (Ca$_2$Al[AlSiO$_7$]– main peak at 31.4°, JCPD file no.: 79-2421). These phases are formed during thermal treatment at 1000 °C of the preform foams of polyurethane sponge, resin, and RM.
On the other hand, the pattern of carbon foam CRM Foam-2.5, prepared at 1100 °C, is mainly characterized by sharp and intense diffraction peaks at 2θ, 44.8°, 43.6° and 33.5° and several weaker peaks derived from the crystalline phase of martensite (C\text{Fe-JCPD file}). It is clear, from the comparison of X-ray patterns of CRM Foam-5 and CRM Foam-2.5 samples, that the difference in RM content and heating temperature, between the foams leading to the formation of different crystalline phases (i.e., tricalcium aluminate, martensite, austenite, α-Fe, Fe-Si carbide). Most of the above phases in the pattern of CRM Foam-2.5, constitute evidence of reduction of hematite by carbon. Ferrite (α-Fe) is the stable form of Fe at room temperature and may contain traces of C dissolved in interstitial sites of its BCC lattice (<0.022 wt%). Austenite (γ-Fe) is stable at temperatures higher than 727 °C and can dissolve carbon (<2.14 wt%) in interstitial sites of its FCC lattice. Its presence at room temperature can be attributed to rapid cooling that inhibited the transformation of austenite to ferrite. Even, air cooling can suppress the transformation of austenite to ferrite, under the high presence of alloying elements (as in the present case) that inhibit the diffusion of carbon from the austenite lattice to form ferrite and cementite. In the cases where austenite contained high contents of carbon (higher than ~0.4 wt%), rapid cooling led to its transformation to distorted α-Fe, namely martensite, a metastable form of Fe, supersaturated in carbon atoms [51].

In order to investigate the graphitization degree of carbon foams, their Raman spectra were recorded and analyzed. Figure 4 shows the Raman spectra of CRM Foam-2.5 and CRM Foam-5 materials. Both samples showed similar spectral features, which can be mainly attributed to carbon vibrations. Specifically, the spectra of CRM Foams exhibit all the main Raman peaks corresponding to the graphitized carbon materials: (a) The graphite band (G-band), centered at ~1580 cm\(^{-1}\), which is due to the in-plane motion of sp\(^2\) carbon atoms in carbonaceous materials with high degree of symmetry and order (i.e., graphene, graphite, SWNTs) [52,53]. It is extremely sensitive to strain effects and is also a good indicator of the number of graphene layers. [54] (b) The defect activated band (D-band), centered at ~1345 cm\(^{-1}\), which is related to lattice motion away from the center of the Brillouin zone and is usually attributed to disordered carbon (sp\(^3\) hybridized carbon), exhibits high sensitivity to the disordered structures. (c) The second order D-band (2D), 2680 cm\(^{-1}\) (exhibits a strong frequency dependence on the excitation laser frequency), which is a second-order two-phonon process Raman signature of sp\(^2\) carbons. It is used for the determination of the number of graphene layers, although its behavior is more complex than the frequency shift observed for the G band. (d) The Raman band D + G, at about 2940 cm\(^{-1}\), which is associated with a D + G combination mode that it is also induced by disorder of carbon structure and the two weak bands at 2458 and 3240 cm\(^{-1}\), which are assigned to the combination D + D' and to the harmonic 2D', respectively (D' corresponds to a phonon belonging to the in-plane longitudinal acoustic branch close to the K point) [52,53]. As it is also observed in Figure 2, the peak positions of the 2D and G bands, together with their intensity ratio (I\(_D")/I_G") \approx 0.84", for both spectra are well correlated with partially graphitized carbon. In addition, the 2D band profile could provide an indication of the graphitic layers’ number [55]. The symmetric shape of the 2D band suggests the presence of graphitized nanosheets on the surface of CRM Foams. On the other hand, in the low frequency range of the spectra (200–800 cm\(^{-1}\)), a number of weak bands are observed, which can be correlated with the vibrations of crystalline oxide and/or iron phases obtained upon pyrolysis of foam preforms, at high temperatures.
3.1.3. Porosity and Thermal Properties

The experimental results of the density, porosity, water absorption, and thermal conductivity measured from the CRMFoams are presented in Table 2. In general, both foams showed low bulk density ($B < 0.55 \, \text{g/cm}^3$) and high porosity ($P > 65\%$) values. Their water absorption ($A$) was calculated at 179.2$\%$ for the CRMFoam-5 sample and 121.7$\%$ for CRMFoam-2.5, whereas the specific gravity ($T$) was 1.76 and 1.55, respectively. These differences can be explained by the fact that the initial resin/RM suspension used for the preparation of CRMFoam-2.5 was more viscous and concentrated due to the higher solid loading of RM (40 wt%) in comparison to that of CRMFoam-5 (20 wt%). Thermal conductivity experiments were carried out using two different pieces from each kind of sample (i.e., four pieces). Five independent measurements were performed to each piece, i.e., ten measurements to each kind of sample. Experimental data were treated using the IBM SPSS ver. 26 software, and the results are reported in Table 2. The equality of mean values was tested via the t-test method and no equality found for a confidence interval of 95$\%$.

Table 2. Properties of CRMFoams.

| Foam Sample | Pyrolizing Conditions | Apparent Porosity $^a$ (%) | Bulk Density $^b$ (g/cm$^3$) | Water Absorption $^c$ (%) | Apparent Specific Gravity $^d$ | Thermal Conductivity (W.m$^{-1}$ K$^{-1}$) | Compressive Strength (MPa) |
|-------------|-----------------------|-----------------------------|-------------------------------|-----------------------------|--------------------------------|--------------------------------------|---------------------------|
| CRMFoam-5   | 1000 °C, N$_2$        | 75.1                        | 0.44                          | 172.9                       | 1.76                           | 0.0400 $\pm$ 0.0041                | 0.22                      |
| CRMFoam-2.5 | 1100 °C, N$_2$        | 65.2                        | 0.54                          | 121.7                       | 1.55                           | 0.0675 $\pm$ 0.0048                | 0.66                      |

$^a$ The apparent porosity ($P$) is the volume of the open pores, into which a liquid can penetrate, as a percentage of the total volume of the specimen.

$^b$ The bulk density ($B$) of a specimen in grams per cubic centimeter is the quotient of its dry weight divided by the exterior volume, including pores.

$^c$ The water absorption ($A$) expresses as a percentage the relationship of the weight of water absorbed to the weight of the dry specimen.

$^d$ The apparent specific gravity ($T$) is the ratio of the mass of a volume of the impermeable portion of a specimen at a stated temperature to the mass of an equal volume of distilled water at a stated temperature.

3.1.4. Mechanical Properties

The mechanical performance of the fabricated carbon-red mud foams (CRMFoam-2.5 and CRMFoam-5) was assessed by performing compression strength tests according
to ASTM C 1424–99 [56]. Stress-strain curves were plotted (Figure 5) to calculate the compressive strength of foams with different red mud contents (CRMFoam-2.5 contains 40 wt% and CRMFoam-5 contains 20 wt% of RM), and the results are shown in Table 2. It should be noted that both samples were tested at 55% compressive strain, and the compressive strength as well as the modulus of elasticity of all samples were calculated from the linear region (red dotted lines) of the profiles. For better understanding of the RM content-density–compressive strength relationship triangle, the values of foams’ bulk density along with their RM content and compressive strength are being quoted in Table 2.

![Figure 5. Representative compressive stress-strain curves of CRMFoam-2.5 and CRMFoam-5.](image)

The CRMFoam-2.5 sample exhibited the best compressive strength with 0.66 MPa, whereas CRMFoam-5 sample showed compressive strength of 0.22 MPa. Regarding the behavior of the elastic modulus, it can be observed that the sample with less apparent porosity (CRMFoam-2.5, 65.2%), exhibited higher value of modulus (12.9 MPa), whereas the CRMFoam-5 (75.1% porosity) a value of 1.56 MPa, confirming the literature reports regarding the relationship between porosity and elastic modulus for ceramic foam materials [57]. Generally, the compressive performance of foam materials varies on several factors such as density, microstructure, and reinforcing agents. The RM presents higher density than the other constituents of the hybrid foams, and therefore by increasing the RM content, the bulk density of the foams increased, leading to enhanced compressive strength of them, and more stiff structure.

3.2. Characterization and Properties of Hybrid Foams ss-CRMFoams@PCMs

3.2.1. Leaching Tests and Thermal Properties

Leaching tests were performed for the CRMFoam-2.5 according to the procedure explained previously. However, only the leaching test results from the CRMFoam-2.5 sample are presented representatively, since CRMFoam-5 showed similar behavior. Table 3 provides a clearer insight about the successful paraffin entrapment, especially after the leaching tests, where no significant quantity of paraffin onto the filter papers was observed. The weight increase constituted a first indication that both RT18HC and octadecane were encapsulated into the porous framework of the foams, while the slight weight loss after leaching tests confirmed the successful paraffin penetration, even at 80 °C, which is much higher than the melting point of both paraffins. Furthermore, the paraffin loading results were in good agreement with the TGA results, indicating that the CRMFoams were able to adsorb and retain large paraffin amounts. In fact, the TGA curves of ss-CRM Foam-2.5@OD and ss-CRM Foam-2.5@RT18 revealed the PCMs’ loading level of paraffins in the stabilizer matrix of CRMFoam-2.5 and how the encapsulation process affected their thermal stability. According to TGA curves of octadecane and RT18HC, these PCMs appeared a one-step
thermal decomposition, with onset and endset decomposition temperatures in the range of 170–190 °C and 200–240 °C, due to the volatilizations of paraffins [58–60].

| Foam Sample | Initial Weight (g) | PCM Paraffin | Final Weight (before Leaching Tests) (g) | Final Weight (after Leaching Tests) (g) | Paraffin Loading Weighing (wt%) | Paraffin Loading-DTA/TG (wt%) |
|-------------|--------------------|--------------|----------------------------------------|--------------------------------------|---------------------------------|-------------------------------|
| CRM Foam-2.5 | 2.9816             | RT18HC       | 9.6545                                 | 8.5260                               | 65.0                            | 61.18                         |
| CRM Foam-2.5 | 2.1430             | Octadecane   | 6.7849                                 | 6.3567                               | 66.3                            | 48.70                         |

Based on these findings, Figure 6 presents the TGA thermograms for the PU template foam and the CRM Foam-2.5, before (CRM Foam-2.5) and after paraffin entrapment with organic PCMs, n-octadecane, and RT18HC (ss-CRM Foam-2.5@OD, ss-CRM Foam-2.5@RT18). As can be seen from the image, the TG curve of the commercial polyurethane foam showed a first thermal degradation at 343 °C and a second at 420 °C, where it lost almost all of its weight. The successful carbonization is obvious by the TG curve of the CRM Foam-2.5, in which no weight loss is present until 800 °C, pinpointing the excellent thermal stability of the sample. With paraffin incorporation, the temperature of the carbon foam rose compared to pure paraffins, to 276 °C for octadecane and 246 °C for RT18HC, respectively, indicating that strong connecting bonds to the matrix were accomplished. Additionally, the temperature shifts demonstrate that the paraffin was successfully encapsulated into the carbon foam’s pores in both samples and thermal stability was substantially improved.

In Figure 7, the results of the leaching tests after 8 h at 30 °C and after 72 h at 80 °C are presented. For the first case (30 °C for 8 h) the ability of the pores to hold the PCMs during the melting process was examined. As seen from the image in Figure 7b, no oily marks are apparent onto the filter paper. For the second one (80 °C for 72 h), the ability of the pores to maintain the encapsulated PCMs in a temperature above the melting point of the
paraffins was determined. The results in this case indicated that the sample immersed in octadecane showed no leakage onto the filter paper (Figure 7d-right), while a small quantity of RT18HC bleached from the porous matrix (Figure 7d-left). Against this background, the paraffin was effectively entrapped into the porous matrices, displaying an efficient material for thermal energy storage and EMI shielding applications.

Figure 7. Leaching tests of the produced hybrids. (a) ss-CRMFoam-2.5@PCMs before the remain at 30 °C for 8 h and (b) after the leaching test, (c) ss-CRMFoam-2.5@PCMs before the remain at 80 °C for 72 h and (d) after the leaching test (OD: Octadecane, RT18: RT18HC).

Figure 8 shows the mid-IR spectra of ss-CRMFoam@RT18, ss-CRMFoam@OD, and that of CRMFoam-2.5. The infrared spectrum of CRMFoam-2.5 exhibited several bands, which can be attributed to vibrations of metal-oxygen bonds of crystalline phases of gehlenite, tricalcium aluminate and CaTiO₃ perovskite. Specifically, the absorption band at 560 cm⁻¹ can be assigned to Ti–O stretching and the band at 435 cm⁻¹ to the Ti-O-Ti bending vibrations, respectively [61,62]. Furthermore, absorption bands centered at 1017 cm⁻¹, 975 cm⁻¹ could be assigned to the asymmetric stretching vibrations of Si-O-Si and Si-O-Al groups of gehlenite (Ca₂Al₃[Al₂SiO₇]) [63]. The absorption peaks observed in the frequency range 920–800 cm⁻¹ and below 500 cm⁻¹ can be attributed to AlO₄ tetrahedral and AlO₆ octahedral groups of tricalcium aluminate (3CaO Al₂O₃ or C₃A) [64,65]. On the other hand, the absorption maximum near 650 cm⁻¹ could be assigned to oxidized iron carbide phases, whereas the broad absorption is around at 3450 cm⁻¹ and the weak band at 1630 cm⁻¹, due to vibrations of adsorbed water molecules on KBr or the sample.

Figure 8. FT-IR spectra of CRMFoam-2.5 and ss-CRMFoam-2.5@OD, ss-CRMFoam-2.5@RT18 hybrids.
The incorporation of PCMs into the open cell structure of CRMFoam-2.5 dramatically changed its infrared spectrum. More specifically, the spectra of ss-CRMFoam@RT18, ss-CRMFoam were dominated by the very strong C-H absorption bands of organic PCMs (octadecane and RTH18HC), which absorbs near 2900 cm\(^{-1}\), 1460 cm\(^{-1}\) and 720 cm\(^{-1}\). Peaks observed at 2957 cm\(^{-1}\) and at 2870 cm\(^{-1}\) were attributed to the asymmetric and symmetric stretching vibrations of methyl (CH\(_3\)) functional groups, whereas these at 2926 cm\(^{-1}\) and 2851 cm\(^{-1}\) were attributed to the asymmetric and symmetric stretching vibrations of methylene (CH\(_2\)) groups, respectively [66]. In addition, the absorption peak centered at 1471 cm\(^{-1}\) was attributed to the deformation vibration of methyl and methylene groups, and the peak at 720 cm\(^{-1}\) to rocking vibration of methylene groups as well. The absorption bands of CRMFoam-2.5 were not observed, since they are covered by the strong absorption peaks of PCMs. On the other hand, the non-existence of any features in the infrared spectra of ss-CRMFoam@RT18 and ss-CRMFoam@OD samples, indicated that PCMs are physically stable into the porous of the CRMFoams and no chemical bonds between PCMs and foam matrices were formed.

The phase transition properties of PCMs, n-octadecane, and RT18HC were studied using their DSC thermographs. Figure 9 shows the melting-freezing temperatures (T\(_m\), T\(_s\)) and the corresponding enthalpies (\(\Delta H_m\), \(\Delta H_s\)) for the two PCMs. As shown in that figure, the pure n-octadecane melts at 20.7 °C and freezes at 28.9 °C, while RT18HC melts at 22.2 °C and freezes at 11.5 °C. Both the exothermic and endothermic peaks of two PCMs, were asymmetric, implying the existence of more than one crystalline phase. According to previous studies, the n-octadecane crystals consist of two crystalline phases α- and β, which exhibit very close melting temperatures overlapping each other [67]. The similar asymmetric appearance of DSC peaks of RT18HC, could be explained based on its structure, which seems according to the Rubitherm data sheet [68], to be composed of more than one hydrocarbon (the main phase should be the hexadecane, C\(_{16}\)H\(_{34}\)). The areas of exothermic (melting) and endothermic (solidification) transition peaks of DSC curves were used for the calculation of enthalpies of n-octadecane and RT18HC, which were found to be 261.2 J/g (\(\Delta H_m\)), −254.2 J/g (\(\Delta H_s\)) and 305.0 J/g (\(\Delta H_m\)), −298.0 J/g (\(\Delta H_s\)), respectively.

Moreover, Figure 10 shows the melting and freezing temperatures (T\(_m\), T\(_s\)) as well as the corresponding enthalpies (\(\Delta H_m\), \(\Delta H_s\)) of the two PCMs stabilized into CRMFoam-2.5. As shown in that figure, the melting temperature (T\(_m\)) of octadecane (OD) increases (+3.7 °C, curve of ss-CRMFoam-2.5@OD), while the solidification temperature (T\(_s\)) of RT18HC decreases (−3.5 °C, curve of ss-CRMFoam-2.5@RTH18). The DSC thermograph of CRMFoam-2.5, which is also illustrated in Figure 10, did not show exothermic or endothermic peaks.

Furthermore, the theoretical melting enthalpies of ss-CRMFoam-2.5@OD and ss-CRMFoam-2.5@RTH18 hybrids were calculated using the following equation [69]:

\[
\Delta_{m}^{\text{Theoretical}} = \Delta_{m}^{\text{PCM}} (1 - \varphi\%)
\]

where \(\Delta_{m}^{\text{Theoretical}}\) and \(\Delta_{m}^{\text{PCM}}\) are the theoretical melting enthalpy of ss-CRMFoam-2.5@PCM hybrids and the measured melting enthalpy of PCM, respectively, and \(\varphi\%\) is the weight fraction of CRMFoam-2.5 sample. Using the above equation, the theoretical melting enthalpies of ss-CRMFoam-2.5@OD and ss-CRMFoam-2.5@RTH18 hybrids were found to be 126.1 J/g and 183.5 J/g, respectively. The \(\Delta_{m}^{\text{Theoretical}}\) value of octadecane (126.1 J/g), seems consistent with the measured value (126.9 J/g) using DSC. In contrast, the \(\Delta_{m}^{\text{Theoretical}}\) value of RT18HC (183.5 J/g) was found to differ significantly from the experimental value (115.5 J/g). A possible explanation for this disagreement could be attributed to the nonhomogenous encapsulation of RT18HC, which in conjunction with the small weight of the sample (30 mg), significantly affected the accuracy of the DSC measurement. In addition, the encapsulation efficiency of n-octadecane and RT18HC in the ss-CRMFoam-2.5...
was calculated based on enthalpies of pure octadecane and RT18HC after leaching tests, utilizing the following equation:

\[
\text{PCM content in ss-CRMFoam-2.5 (wt\%) = } \left( \frac{\Delta H_m}{\Delta H_{\text{PCM}}} \right) \times 100
\]

(7)

where \(\Delta H_m\) is the enthalpy of melting for the PCM encapsulated into ss-CRMFoam-2.5 (J/g) and \(\Delta H_{\text{PCM}}\) is the enthalpy of melting for pure octadecane or RT18HC (J/g). Thus, the latent heat storages of ss-CRMFoam-2.5@OD and ss-CRMFoam-2.5@RTH18 were calculated using Equation (6) and found to be 48.8% and 37.8% for octadecane and RT18HC, respectively.

![DSC curves of pure n-octadecane and RT18HC paraffin.](image-url)

**Figure 9.** DSC curves of pure n-octadecane and RT18HC paraffin.
3.2.2. EMI Shielding Effectiveness

The shielding performance of the developed samples was examined in terms of EMI shielding effectiveness (SE) in the 3.2–9.0 GHz frequency range. $SE_T$ can be expressed as the sum of reflection from the surface ($SE_R$), absorption ($SE_A$) and multiple-reflection ($SE_M$) as follows:

$$SE_T (\text{dB}) = SE_R (\text{dB}) + SE_A (\text{dB}) + SE_M (\text{dB})$$  \hspace{1cm} (8)

Multiple-reflection at internal interfaces inside the material can be excluded if $SE > 10 \text{ dB}$ \cite{70} and thus:

$$SE = SE_T \triangleq 10 \log_{10} \left( \frac{P_{\text{inc}}}{P_{\text{trn}}} \right) = 10 \log_{10} \left( \frac{1}{T} \right) = SE_R + SE_A$$  \hspace{1cm} (9)

where

$$SE_R = 10 \log_{10} \left( \frac{1}{1 - R} \right)$$  \hspace{1cm} (10)

$$SE_A = 10 \log_{10} \left( \frac{1 - R}{T} \right)$$  \hspace{1cm} (11)

$SE_R, SE_A$ refer to the reflection and absorption SE, respectively.
The higher the SE the better the shielding. The SE (also denoted as $SE_T$, with $A$, $T$, $R$ indicating the absorption, transmission, and reflection, respectively) is usually quantified in terms of the logarithm of the incident power $P_{inc}$ over the transmitted power $P_{trn}$ \cite{71-75} and thus expressed in decibels (dB).

Figure 11 shows the reflection (Figure 11a) and the absorption (Figure 11b) spectra of the CRMFoams, in the frequency range 3.5–9.0 GHz. Figure 11a clearly illustrates that the reflection of the CRMFoams is almost negligible indicating that the dominant shielding mechanism is absorption giving a total SE: $SE_T = SE_A$ (see Equation (9)). Moreover, as one can notice from Figure 11d, the absorption levels increase as the porosity increases (sample CRMFoam-2.5 to CRMFoam-5) indicating that indeed enhanced porosity improves the microwave absorption \cite{76-79}.

![Figure 11. Reflection (a) and absorption (b) spectra from 3.5 to 9.0 GHz (C-band) for different CRMFoams. The electromagnetic interference shielding effect (SE) due to reflection (SER; c), and absorption (SEA; d) can also be seen in the frequency range 3.5–9.0 GHz.](image)

It is worth noticing that the encapsulation of the paraffins into the foams increased the EMI efficiency to almost 20 dB reaching the entry-level value required for commercial applications \cite{80} following the literature’s findings that the existence of phase change Materials (PCMs) enhances the electromagnetic interference shielding effectiveness \cite{81}.

4. Conclusions

Carbon red mud foams (CRMFoams) were successfully synthesized, with two different phenolic resin to red mud ratios (5 or 2.5), from commercially available polyurethane foams. The morphological and structural study of the foams revealed a highly porous structure (bulk density < 0.55 g/cm$^3$, apparent porosity $P > 65\%$), which exhibited elliptical and spherical pores, sized from 50 up to 500 μm, while their pore cell walls composed of partially graphitized carbon and various oxide and iron phases. The foams showed remarkable encapsulation efficiency as shape stabilizers for the n-octadecane (OD) and RT18HC. Due to the enhanced porosity and the multiple reflections within the formed micropores, all of the as-prepared CRMFoams showed excellent EMI shielding performance in frequency of 3.5–9.0 GHz, making them promising candidates for use in thermal management and EMI shielding for electronic devices. Finally, the usage of alternate sized red mud and/with additives as well at different loading ratios could further enhance specific properties of the reported materials as for example the formed micropores whose geometry and connectivity determine the EMI and mechanical properties altogether.
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