

Article

Expanded Polystyrene for Building Insulation: Effect of Graphite and Moisture on Thermophysical Properties

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Abstract

Improving the energy efficiency of the building envelope is critical for global decarbonization, yet a gap remains in the comprehensive thermophysical characterization of carbon-enhanced Expanded Polystyrene (EPS). This study evaluates the impact of expansion ratios and moisture content on the thermal behavior of two commercial EPS grades, EPS-A ($12.7 \pm 0.5 \text{ kg/m}^3$) and EPS-B ($16.0 \pm 1.1 \text{ kg/m}^3$), investigating the counterintuitive role of graphite (1.4–1.8 wt.%) in enhancing the thermal insulation properties. Thermal conductivity and diffusivity were independently determined via Transient Plane Source (TPS) and Heat Flow Meter (HFM) methods across a 10–50 °C range, while specific heat capacity (c_p) was analyzed using HFM and Differential Scanning Calorimetry (DSC) through the sapphire comparison method and Temperature-Modulated DSC (TOPEM®). Methodologically, it was found that standard HFM protocols are unsuitable for c_p determination in low-density foams, yielding an average relative error of $\pm 29\%$; conversely, the sapphire comparison method provided the most reliable results in agreement with theoretical expectations. Results indicate that the efficacy of graphite as a radiative shield is closely coupled with cellular morphology, proving significantly more effective in the higher expansion grade (EPS-A, 70 wt.% open porosity) than in the denser EPS-B. Furthermore, 30-day water immersion tests revealed that the higher open porosity of EPS-A facilitates increased water uptake of $144 \pm 17 \text{ wt.}\%$ (compared to $97 \pm 7 \text{ wt.}\%$ for EPS-B), causing the geometric densities of the two grades to converge and fundamentally altering thermal transport mechanisms. The study concludes that accurate thermal modeling of carbon-enhanced insulation requires careful selection of testing parameters, particularly when accounting for moisture-induced degradation in high-porosity systems.

Keywords: EPS; graphite; building insulation; thermal conductivity; thermophysical properties

1. Introduction

The building sector remains one of the primary contributors to energy consumption in Europe, accounting for approximately 27% of the EU's total energy use [1,2]. Significantly, 85% of the EU building stock was constructed prior to 2001, with the majority exhibiting suboptimal energy performance [3,4]. Enhancing building energy efficiency is therefore critical for mitigating energy waste, reducing utility costs for consumers, and achieving a zero-emission building stock by 2050, in alignment with the European Green Deal [2]. Furthermore, improving thermal performance is a key lever for alleviating energy poverty and

strengthening Europe's energy independence. Meeting these strategic objectives requires a dual approach: reducing energy demand through the thermal retrofitting of inefficient structures [5], and developing advanced building envelopes that integrate technologies for thermal energy transfer and storage to attenuate peak loads [6–8].

Historically, thermal insulation in the building sector has relied on a variety of materials, ranging from mineral wools to synthetic polymers, each selected for their ability to minimize heat transfer through the building envelope [9,10]. Among these, Expanded Polystyrene (EPS) has emerged as a predominant choice due to its exceptional balance of performance, durability, and cost-effectiveness. The widespread adoption of EPS lies in its closed-cell structure, which effectively traps air—a natural insulator—to provide consistently low thermal conductivity. Furthermore, EPS is valued for its lightweight nature, moisture resistance, and high compressive strength, ensuring long-term stability in diverse climatic conditions. Its versatility makes it a cornerstone material in the pursuit of sustainable architecture [11–16].

However, the widespread use of EPS is not without environmental challenges. Concerns have been raised regarding the fragmentation of EPS as a potential source of microplastics [17], documented not only in marine waters, primarily due to fishery activity [18], but also in freshwater, a phenomenon increasingly attributed to the construction industry [19].

Beyond these ecological considerations, the industry is focused on enhancing thermal performance; in this context, carbon-enhanced materials represent a significant evolution of traditional EPS technology. While standard EPS primarily limits heat transfer through its cellular structure, it remains partially transparent to infrared radiation [20]. The integration of graphite overcomes this limitation by acting as an infrared absorber and reflector within the polymer matrix, selectively attenuating the radiative component of heat transfer [21]. Consequently, graphite-enhanced EPS is increasingly supplanting traditional neat EPS; although adding conductive carbon might seem counterintuitive, it provides superior insulation by significantly reducing radiative heat transfer.

However, this shift in material preference necessitates a more rigorous scientific foundation, as accurate thermal property data are essential for assessing energy savings in modern applications. Precise databases for thermal diffusivity, specific heat, and thermal conductivity are vital for characterizing these advanced composites, particularly in transient regimes. Measuring thermal diffusivity is a complex inverse problem highly sensitive to sample preparation and calibration [22]. In this work, the commercially established Hot Disk technique and direct Heat Flow Meter (HFM) were utilized, as measurements from different techniques often diverge. For specific heat capacity, Differential Scanning Calorimetry (DSC) remains the standard technique, with modern evolutions like Temperature-Modulated DSC (TMDSC) and TOPEM® allowing for the direct determination of quasi-static heat capacity without external calibration [23].

The implementation of these techniques requires a meticulous selection of testing parameters, which are often inadequately defined in equipment datasheets. The lightweight nature of EPS poses a significant challenge, as low-density samples complicate the precise determination of specific heat capacity. These difficulties are compounded by the carbon fillers, which fundamentally alter the material's thermophysical properties. Furthermore, accurate characterization is additionally complicated by environmental factors, such as moisture, which challenge both the reliability of experimental measurements and real-world insulating behavior. Although inherently hydrophobic, EPS contains an open porosity fraction that facilitates water accumulation. When water infiltrates these interstitial spaces, it replaces stagnant air, causing a sharp increase in thermal conductivity; this degradation is driven by the fact that liquid water is approximately 25 times more conductive than air [24–27].



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In recent years, several studies have extensively investigated the impact of such fillers on the fire resistance of the material and strategies to hinder it [28–30]. In parallel, Blazejczyk et al. demonstrated that a small addition of graphite microparticles, typically below 4.3 wt.%, can significantly reduce thermal conductivity by altering both conductive and radiative heat transfer mechanisms [21]. Other research has focused on the degradative effects of solar radiation [31], environmental aging [27], and the impact of moisture on graphite-enhanced systems [32,33].

Despite these advancements, a significant gap remains in the high-precision determination of thermophysical properties across a broad operational range. While existing literature often concentrates on steady-state conductivity, the temperature dependence of these properties is vital for accurate thermal modeling [34]. Furthermore, although Lakatos et al. [32] investigated the thickness and density dependence of thermal conductivity, the cross-validation of multiple experimental techniques remains largely unexplored.

The novelty of this study lies in its systematic methodological comparison aimed at overcoming the sensitivity limitations inherent in characterizing these expanded polymers. Whereas previous studies have often been restricted to evaluating steady-state thermal conductivity at a single temperature or focusing solely on moisture effects, this work provides a more holistic assessment by integrating an extended temperature range, morphological characterization of different expansion grades, and the quantification of humidity-induced performance changes.

To these purposes, this work characterizes two commercial graphite-enhanced EPS grades with different expansion ratios to quantify the insulation benefits provided by carbon fillers. Following a morphological and physical characterization, a systematic investigation of thermophysical properties was conducted by comparing multiple techniques and varying testing parameters to highlight the critical challenges inherent in measuring low-density materials. The analysis covers the standard reference temperature for insulation (10 °C) and the broader 0–50 °C range. Finally, the study examines the impact of humidity on specific heat capacity and thermal conductivity, demonstrating how inadequate material conditioning can detrimentally affect performance and how experimental techniques—specifically HFM measurements—are influenced by the presence of condensed water.

2. Materials and Methods

2.1. Materials

Two grades of graphite-enhanced expanded polystyrene (EPS), designated as Type A (EPS-A) and Type B (EPS-B), were investigated. Their physical characteristics, based on the manufacturer's technical data sheets, are summarized in Table 1, while photographs of the panels and representative cut samples are provided in Figure 1. Both materials were sourced from Sto SE & Co. KGaA (Stühlingen, Germany) with the commercial names Sto GK8 300 Plus and Sto GK800 A+, respectively. These two specific EPS grades were selected as they represent common densities utilized in current building insulation practices for energy retrofitting. By comparing two different expansion ratios, the study aims to investigate whether the thermal benefits of graphite remain consistent across the density range typically found in commercial products. The exact graphite content is not specified in the manufacturer's technical documentation; therefore, TGA was performed to quantify the filler loading for both commercial grades. The samples were cut from the commercial panels with a hot resistance wire.

Table 1. Thermophysical properties of the considered EPS sample at 10 °C according to their technical datasheet.

Sample	Bulk Density (kg/m ³)	Thermal Conductivity ¹ (W/m·K)	Specific Heat Capacity ² (J/g·K)
EPS-A	14–16	0.031	1.45
EPS-B	15–22	0.030	1.45
EPS-REF	26	0.033	N/A

¹ EN 12667 [35]/EN 13163 [36]. ² UNI EN 10456 [37].

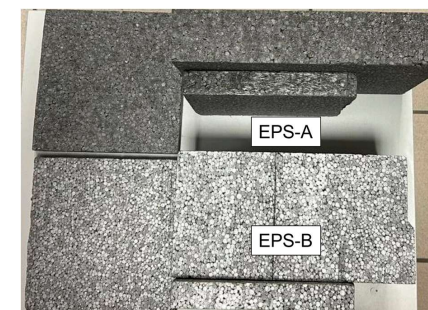


Figure 1. Panels and thermo-cut samples (200 × 200 × 25 mm³) of EPS-A (top) and EPS-B (bottom).

Additionally, Table 1 includes the properties of a reference EPS specimen (EPS-REF) with a thickness 20 mm, provided by Netzsch Analyzing & Testing (Selb, Germany). This calibrated standard, typically used for HFM calibration, serves as a comparative baseline for specific heat capacity and thermal conductivity tests.

2.2. Experimental Methodologies

The morphological features of the specimens were examined via optical microscopy using a Nikon MSZ25 microscope (Nikon Instruments, Tokyo, Japan). To evaluate the internal cellular structure and phase distribution at higher magnifications, Scanning Electron Microscopy (SEM) was performed using a Zeiss Supra field emission scanning electron microscope (Carl Zeiss, Oberkochen, Germany). Imaging was conducted using a secondary electron (SE) detector at an acceleration voltage of 10 kV; prior to analysis, the specimen surfaces were sputter-coated with a thin layer of Pt-Pd to prevent charging and enhance resolution.

Carbon residues were isolated by dissolving the EPS in toluene (1% *w/v* concentration), followed by centrifugation for 5 h at 4400 rpm using a Centrifuge 5702 (Eppendorf, Hamburg, Germany). Following the evaporation of the solvent, the resulting residues were characterized via SEM, utilizing the same operating parameters as the EPS panel analysis.

The bulk (geometrical) density (ρ_{bulk}) was determined as the ratio of mass to geometric volume. This volume was derived from dimensions measured with a digital caliper (resolution ± 0.01 mm), while the mass of each specimen was recorded using a Mettler AM 100 analytical balance (Mettler-Toledo, Greifensee, Switzerland, resolution ± 0.1 mg). The tested specimens consisted of square samples (approximately $20 \times 20 \times 3$ cm³), which had been extracted from larger commercial panels using a hot-wire cutter.

The apparent (skeletal) density (ρ_{app}) was determined using an Ultrapyc 5000 helium pycnometer (Anton Paar, Graz, Austria). Prior to each measurement session, the instrument was calibrated using two stainless steel spheres of known volume. Specimens were

prepared by extracting small beads of expanded material from the EPS panels. The 4.5 cm³ sample chamber was filled to approximately two-thirds capacity to ensure measurement accuracy. Testing was conducted at a controlled temperature of 20 °C and a pressure of 0.21 bar, utilizing a continuous flow mode as recommended by the manufacturer. Twenty measurement cycles were performed for each EPS type. Using the mass values determined previously, the skeletal density was calculated. By correlating the bulk and apparent densities, the open (P_{open}), closed (P_{closed}), and total porosities (P_{tot}) were derived using Equations (1)–(3).

$$P_{open} = \left(1 - \frac{\rho_{bulk}}{\rho_{app}}\right) \cdot 100 \quad (1)$$

$$P_{closed} = \rho_{bulk} \left(\frac{1}{\rho_{app}} - \frac{1}{\rho_{th}} \right) \cdot 100 \quad (2)$$

$$P_{tot} = \left(1 - \frac{\rho_{app}}{\rho_{th}}\right) \cdot 100 = P_{open} + P_{closed} \quad (3)$$

where ρ_{th} represents the theoretical density of the material. For these calculations, the theoretical density of solid polystyrene was taken as 1050 kg/m³ [38].

The water absorption capacity was quantified through a 30-day immersion period to ensure complete saturation of the panels. Throughout this duration, mass gain was monitored daily to track the evolution of the impregnated density (ρ_{imp}).

Regarding the thermal properties, Differential Scanning Calorimetry (DSC) was performed to evaluate whether the integration of graphite influences the glass transition temperature (T_g) of the carbon-enhanced EPS. Thermal analysis was performed using a Mettler DSC 30 (Mettler-Toledo, Greifensee, Switzerland) over a temperature range of 0 to 200 °C. The experimental protocol consisted of a heating scan, a cooling step, and a second heating ramp, all conducted at a constant rate of 10 °C/min. Measurements were carried out under a nitrogen flow of 100 mL/min using 160 µL aluminum crucibles.

Complementarily, thermogravimetric analysis (TGA) was performed to determine the precise graphite content within both EPS grades. The analysis was conducted using a TGA Q5000 IR (TA Instruments, New Castle, DE, USA). For each measurement, a 2 mg specimen was placed in the reaction chamber and heated from 30 °C to 700 °C at a constant nitrogen flow rate of 10 mL/min. A single representative measurement was performed for each EPS grade, from which the following characteristic parameters were identified: the temperature of maximum degradation rate (T_{peak}), the residual mass at 150 °C (m_{150}), and the final residual mass at 700 °C (m_{700}).

The thermophysical properties—thermal conductivity (λ), thermal diffusivity (α) and specific heat capacity at constant pressure (c_p)—are interrelated according to Equation (4). These parameters were determined using a combination of experimental techniques for both direct measurement and indirect derivation.

$$\lambda = \rho_{bulk} \cdot \alpha \cdot c_p \quad (4)$$

The specific heat capacity was initially evaluated using a Netzsch HFM 446 Lambda Small (Netzsch Analyzing & Testing, Selb, Germany). This approach was selected to preserve the structural integrity of the expanded polystyrene (EPS) by utilizing large-scale specimens (approximately 200 × 200 × 25 mm³). Following preliminary testing, the experimental parameters were refined to optimize accuracy, as detailed in the Appendix A (Tables A1 and A2). The refined tests were conducted at 40, 30, 20, and 10 °C, progressing from higher to lower temperatures to isolate the effects of humidity condensation. During this procedure, specimens were placed in the chamber under a contact pressure of 2 kPa and the upper and lower plates were cooled simultaneously in 10 °C increments under static air

conditions. Calibration of the heat flux sensors was performed using the EPS-REF standard, applying a default temperature gradient of 20 °C between the plates. Additionally, the air resistance contribution was accounted for through an empty-stack calibration with a plate separation of 2.6 mm.

As an alternative technique for specific heat capacity evaluation, DSC measurements were conducted using a Mettler Toledo DSC 5+ (Mettler-Toledo, Greifensee, Switzerland). For all measurements, 40 µL open aluminum crucibles were used under a constant nitrogen flow of 100 mL/min. Two distinct approaches were employed:

- Sapphire Method: performed according to the ASTM-E1269-11 standard [39] at heating rates of 1, 5, and 10 °C/min. This method required three distinct runs per sample: a baseline (empty crucibles), a reference run (sapphire standard), and the specimen measurement;
- Temperature-modulated Order-based Parameter Estimation Method (TOPEM®): conducted at heating rates of 0.1 and 1 °C/min, with pulse heights of 0.05 and 0.1 °C, respectively. For this stochastic modulation technique, only the baseline and specimen were measured, as the software directly calculates the quasi-static c_p from the reversing heat flow.

To obtain a sufficient mass within the limited crucible volume, the low-density EPS required a ‘backforming’ thermal treatment. This was performed at 120 °C in a press to increase the material density, acknowledging that this modification may alter the original thermal characteristics, even though it is well below the onset degradation temperature of the material (see Section 3.2). For each test, three specimens with masses ranging between 6 and 8 mg were analyzed.

The thermal conductivity and diffusivity were independently determined via the Transient Plane Source (TPS) method and a Heat Flow Meter (HFM). For the TPS measurements, a Hot Disk® TPS 2500 S (Hot Disk AB, Gothenburg, Sweden) was used in accordance with ISO 22007-2 [40]. These tests were conducted within a Memmert HCP climatic chamber (Mettmert GmbH + Co. KG, Schwabach, Germany) to maintain a controlled environment at 30% relative humidity across three investigation temperatures: 10, 30, and 50 °C. A Kapton-insulated sensor (Model 4922, Hot Disk AB, Gothenburg, Sweden, radius 14.61 mm) was employed, applying a heating power of 15 mW for 80 s. To minimize the influence of surface porosity and localized imperfections, the sensor size was selected to cover the maximum available sample surface. For each measurement, a temperature variation curve of 200 points was recorded, with only the final 150 points analyzed—as recommended by the manufacturer—to ensure a fully developed transient regime. The TPS software (Hot Disk Desktop App v. 7.7) facilitated the simultaneous determination of thermal conductivity and diffusivity; the volumetric heat capacity ($\rho_{bulk} \cdot c_p$) was both input as a known parameter and calculated by the instrument to verify internal consistency.

In parallel, steady-state thermal conductivity was measured using a Netzsch HFM 446 Lambda Small (Netzsch Analyzing & Testing, Selb, Germany). Three specimens of both EPS-A and EPS-B were tested to validate the TPS results and evaluate the impact of moisture; measurements were conducted in both the dry state and after a 30-day water immersion period, allowing 30 min for surface desorption prior to testing. Prior to the experimental campaign, the HFM was calibrated using the EPS-REF standard. Specimens were placed in the chamber under a contact pressure of 2 kPa, with a temperature gradient of 20 °C established between the plates. Measurements were recorded at five mean temperatures ranging from 10 to 50 °C. To prevent environmental interference, a nitrogen purge (5–15 mL/min) was maintained throughout the testing. Finally, the theoretical thermal conductivity for neat EPS was calculated as a function of geometric density (ρ_{bulk}) and temperature (T , °C) using Equation (5) (tolerance 2%). Derived from the HFM technical manual’s calibration

data, this equation establishes a baseline for neat EPS, enabling a quantitative assessment of the thermal performance enhancement provided by the graphite fillers.

$$\lambda = 5.78 \times 10^{-4} - 1.25 \times 10^{-4} (\rho_{\text{bulk}} - 26) + 1.134 \times 10^{-4} \times (T + 273.15) \quad (5)$$

3. Results and Discussion

This section highlights the fundamental differences between the two investigated EPS grades. While both materials are produced through an industrial process integrating graphite into the polystyrene matrix, EPS-A underwent a higher degree of expansion than EPS-B, resulting in distinct density profiles. Following detailed physical and microstructural characterization, the analysis shifts to the material's thermal behavior. The core of this study evaluates thermophysical properties using multiple experimental techniques to quantify the specific impact of graphite integration and moisture content. Furthermore, a systematic investigation of testing parameters and a comparative analysis were conducted to ensure result convergence and to validate the findings against established literature values.

3.1. Physical and Microstructural Properties of Graphite-Enhanced EPS

The morphology of carbon-enhanced EPS was characterized via optical microscopy; representative images are provided in Figure 2, showing an overview of bead dimensions (Figure 2a,b) and higher-magnification views of the internal microstructure (Figure 2c,d).

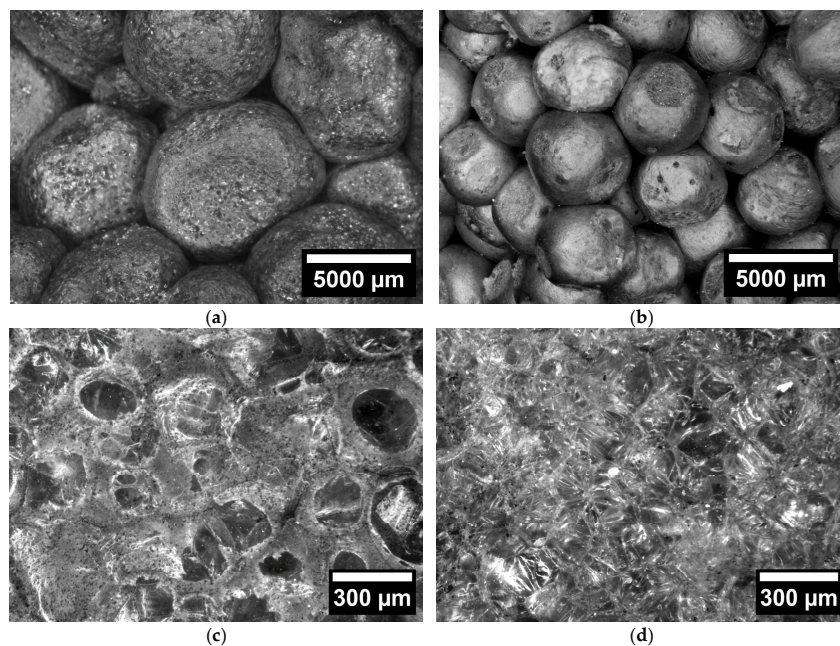


Figure 2. Optical observations of morphology and microstructure: (a) overview of EPS-A; overview of EPS-B (b); details of a flat cut surface of EPS-A (c); details of a fracture surface of EPS-B (d).

A comparison of Figures 2a and 2b reveals a distinct difference in expansion grades; the average bead diameters for EPS-A and EPS-B were measured at 6.3 ± 1.3 mm and

4.7 ± 0.3 mm, respectively. Figure 2c highlights microvoids ranging from 0.1 to 0.3 mm in EPS-A, along with visible graphite microplatelets with dimensions < 10 μm ; these features are also faintly detectable in EPS-B (Figure 2d) and are absent in EPS-REF (Figure A1). Notably, there is no evidence of percolation among the carbon planes, a critical factor in maintaining optimal thermal insulation properties.

Complementarily, Figure 3 shows the cellular structure detectable with SEM observations, while Figure A2 details the morphology of the carbon residues isolated after PS dissolution and centrifugation.

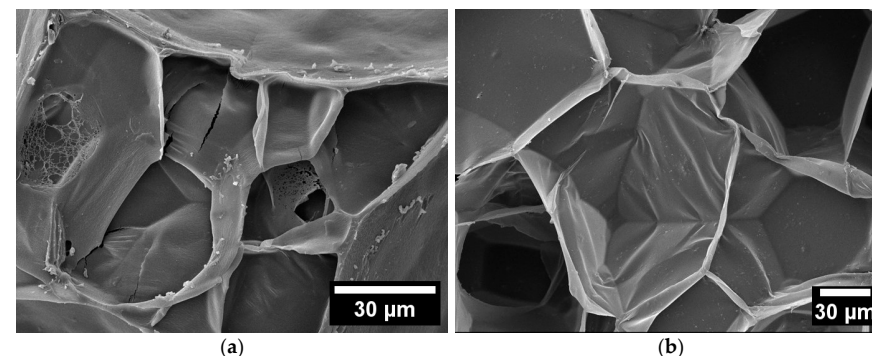


Figure 3. Cellular structure evidenced in SEM observations: (a) EPS-A; (b) EPS-B.

The bulk density was measured using a caliper and a precision balance, while the apparent density was determined via helium pycnometry. For the purpose of this study, density was assumed to remain constant across the investigated temperature range. The resulting values for density and porosity are summarized in Table 2, as derived from Equations (1)–(3).

Table 2. Density and porosity values.

Sample	ρ_{bulk} (kg/m^3)	ρ_{app} (kg/m^3)	P_{open} (vol.%)	P_{closed} (vol.%)	P_{tot} (vol.%)	ρ_{imp} (kg/m^3)
EPS-A	12.7 ± 0.5	42.7 ± 1.2	70.2	28.6	98.8	31.0 ± 1.4
EPS-B	16.0 ± 1.1	43.1 ± 1.9	62.9	35.5	98.5	31.0 ± 1.9

ρ_{bulk} = bulk/geometrical density; ρ_{app} = apparent/skeletal density; P_{open} = open porosity; P_{closed} = closed porosity; P_{tot} = total porosity; ρ_{imp} = bulk density after water impregnation.

The data confirm that the specific batch of EPS-A utilized in this study underwent a more extensive expansion process, as evidenced by its lower experimental geometric density (12.7 ± 0.5 kg/m^3) compared to EPS-B (16.0 ± 1.1 kg/m^3). Although the manufacturer's technical data sheets (Table 1) indicate potentially overlapping density ranges for these commercial grades, our characterization demonstrates that these specific samples represent two distinct morphologies. This difference in expansion is further corroborated by the significantly larger average bead diameter of EPS-A (6.3 ± 1.3 mm) compared to EPS-B (4.7 ± 0.3 mm). The primary source of uncertainty in the geometric density measurements stems from the irregular surface morphology of the foam specimens. Furthermore, mechanical interaction errors between the caliper and the compliant EPS surface can lead to localized volume reduction if excessive pressure is applied during measurement.

The water uptake stabilizes after a week, and at the end of the 30-day period, the mass increment was quantified as 144 ± 17 wt.% for EPS-A and 97 ± 7 wt.% for EPS-B. Interestingly, following water immersion, the geometric densities of the two EPS grades converge; this is attributed to the larger open porosity of EPS-A, which facilitates a higher degree of water uptake relative to EPS-B.

3.2. Thermal Characterization

The thermal properties of the investigated EPS grades were characterized via DSC (Figure A3) and TGA (Figure 4), which primarily serves to quantify the carbon filler content. The key findings from both thermal analysis techniques are reported in Table 3.

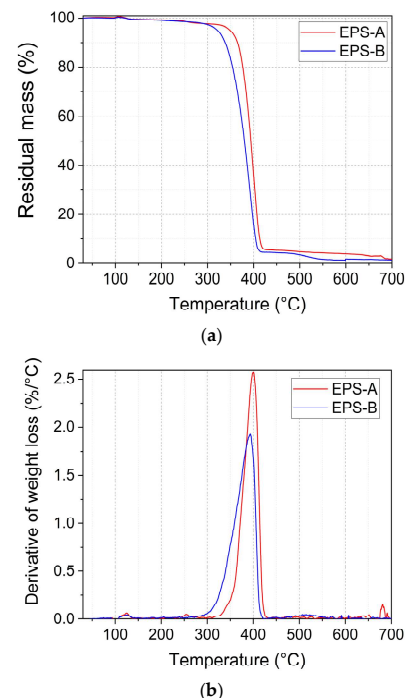


Figure 4. Thermogravimetric analysis (TGA) results: (a) residual mass; (b) derivative of weight loss.

Table 3. Thermal properties derived from DSC and TGA.

Sample	$T_{g,h1}$ (°C)	$T_{g,c}$ (°C)	$T_{g,h2}$ (°C)	T_{onset} (°C)	T_{peak} (°C)	m_{150} (wt.%)	m_{700} (wt.%)
EPS-A	100.5	93.0	102.6	313.2	399.1	99.5	1.4
EPS-B	102.0	87.2	97.0	274.3	393.4	99.5	1.8

$T_{g,h1}$ = glass transition temperature in the first heating; $T_{g,c}$ = glass transition temperature in cooling; $T_{g,h2}$ = glass transition temperature in the second heating; T_{onset} = temperature corresponding to the onset of thermal degradation; T_{peak} = temperature corresponding to the maximum degradation rate; m_{150} = residual mass at 150 °C; m_{700} = residual mass at 700 °C.

The glass transition of PS was observed near 100 °C, with no substantial modifications induced by the presence of the filler [41]. As expected, in cooling, the glass transition temperature is lower due to thermal inertia and kinetic reasons.

The residual mass at 150 °C serves as a proxy for the moisture content of unconditioned EPS; according to the TGA results, both grades exhibited a water evaporation loss of 0.5 wt.% relative to the total initial mass. Most significantly, the TGA results (Table 3) provide a quantitative estimation of the filler content, revealing a graphite loading of 1.4 wt.% for EPS-A and 1.8 wt.% for EPS-B. While these values should be viewed as first-order approximations, they provide sufficient accuracy for the comparative purposes of this study. These findings confirm that both materials possess a relatively low carbon fraction—below 2 wt.%—with no substantial compositional differences between the two grades despite their differing expansion ratios. In both cases, complete thermal degradation of the polymer matrix occurred at approximately 430 °C. When correlated with microstructural observations, these findings suggest that both EPS types were likely derived from identical precursor beads and subsequently subjected to different expansion protocols.

3.3. Thermophysical Properties

This section presents a comprehensive evaluation of the thermophysical properties, focusing on a rigorous comparison between the experimental techniques employed. Beyond standard characterization, a systematic investigation into the influence of testing parameters—such as temperature increments, atmospheric conditions, and heating rates—was conducted. This approach was adopted to identify the optimal configuration for evaluating low-density materials, ensuring that the results are both reproducible and validated across transient and steady-state regimes.

3.3.1. Specific Heat Capacity

Initial experimental efforts focused on measuring the specific heat capacity (c_p) without altering the physical structure of the EPS. Following preliminary calibration trials (detailed in Appendix A), a descending temperature sequence from 40 °C to 10 °C was adopted. This specific cooling protocol was implemented to mitigate moisture condensation effects, which were found to be most prominent at 10 °C, as illustrated in Figure 5 (punctual value summarized in Table A3).

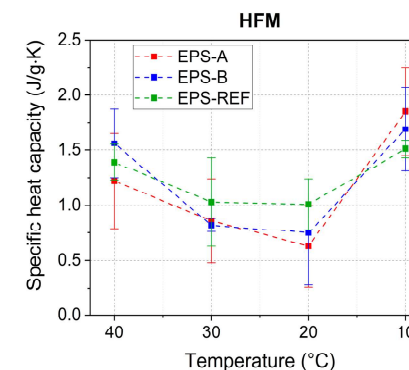


Figure 5. Specific heat capacity as a function of temperature determined via HFM. In addition to the inherent high uncertainty and limited reliability of the HFM for low-density foams, the values at 10 °C illustrate a critical experimental challenge represented by the interference of water condensation artifacts.

Initial measurements of specific heat capacity via HFM yielded unsatisfactory results, characterized by a high average relative error of $\pm 29.1\%$ (Table A6). This is attributed to the extremely low bulk density of the EPS, which results in a total specimen mass within the chamber that is insufficient to generate a heat flux signal above the instrument sensitivity threshold. It is worth noting that the anomalous peak in c_p observed at 10 °C in Figure 5 is attributed to moisture condensation at the sample–sensor interface during the initial stages of the thermal sweep. The latent heat associated with this phase change results in an overestimation of the energy absorbed by the material. This measurement artifact is discussed further in the Appendix A to emphasize the necessity of rigorous environmental control when characterizing low-density foams at temperatures approaching the dew point. Moreover, the discrepancy with expected values can be considered intrinsic to the technique because EPS-REF was used for the calibration procedure.

To resolve these measurement challenges, Differential Scanning Calorimetry (DSC) was employed. To ensure a detectable heat flux signal within the small volume of a DSC crucible, the material's physical state was modified through a back-forming thermal treatment at 120 °C. This process eliminated the air and collapsed the cellular structure, allowing for the concentration of around 10 mg of solid polymer into the crucible. The specific heat capacity of both EPS grades was assumed to be identical, based on the hypothesis that they were produced from the same precursor expandable polystyrene beads. Consequently, measurements were conducted exclusively on EPS-A specimens that had undergone back-forming via thermal press treatment at 120 °C. The temperature dependence of the EPS specific heat capacity, determined via DSC, is illustrated in Figure 6 and Table A4.

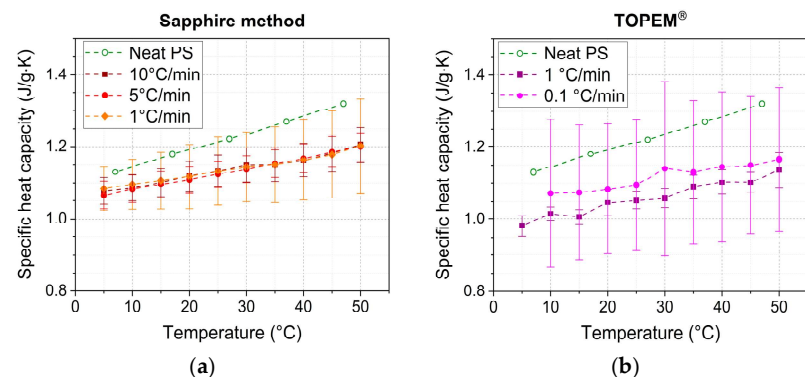


Figure 6. Specific heat capacity determined from DSC measurement on thermally modified EPS-A and neat PS from Polymer Handbook [38]: (a) Sapphire Method; (b) TOPEM®. Data points represent the mean of three independent specimens $\pm$ standard deviation.

The curves obtained using the sapphire method exhibit nearly identical mean values, though measurements at a rate of 1 °C/min show higher uncertainty, a trend that contrasts with observations for denser organic materials [42]. Regarding the TOPEM® measurements, the 0.1 °C/min curve presents unacceptably high uncertainty, whereas the 1 °C/min curve is shifted toward slightly lower values but with improved precision.

Detailed measurement uncertainties are summarized in Table A6. While a clear increase in specific heat capacity with temperature is evident, the measured values remain lower than those reported by Lakatos et al. [43], who found a c_p of approximately 1.3 J/g·K at 25 °C for neat polystyrene. Notably, the value provided in the manufacturer's datasheet

exceeds those of both neat PS and neat graphite. Despite the low weight percentage of graphite (c_p approximately 0.7 J/g·K [44]), its contribution to the overall specific heat of the composite can be considered responsible for the lower values compared with neat PS [38].

All measurements were influenced by the low-density of the EPS, which complicates the accurate determination of specific heat capacity. In the DSC analysis, a high filling factor was required to achieve sufficient mass within the crucible; however, this necessitated compacting the material and compromising its original physical structure. Conversely, while HFM testing preserved the structural integrity of the specimens, the resulting mass remained extremely low and likely insufficient for standard HFM sensitivity thresholds. This stands in contrast to denser construction materials, where the HFM technique is highly accurate due to specimen masses being several orders of magnitude higher [45].

3.3.2. Thermal Diffusivity

There is a notable deficit in the existing literature regarding the thermal diffusivity of both standard and carbon-enhanced EPS. In this study, the Transient Plane Source (TPS) technique, via the Hot Disk apparatus, was the primary method utilized for the direct determination of this property. Alternatively, the thermal diffusivity can be indirectly determined by reversing Equation (5) from the HFM measurement (Section 3.3.1). The temperature dependence of thermal diffusivity for the samples investigated is illustrated in Figure 7. To ensure statistical reliability, three independent specimens of both EPS-A and EPS-B were characterized at each temperature (10, 30, and 50 °C). The data points in Figure 7 represent the mean values derived from these tests, with error bars indicating the standard deviation. The corresponding thermal diffusivity values are summarized in Table A7. Furthermore, Table A8 details the specific heat capacity values adopted for the indirect HFM calculations, as well as the c_p values derived independently from the Hot Disk measurements.

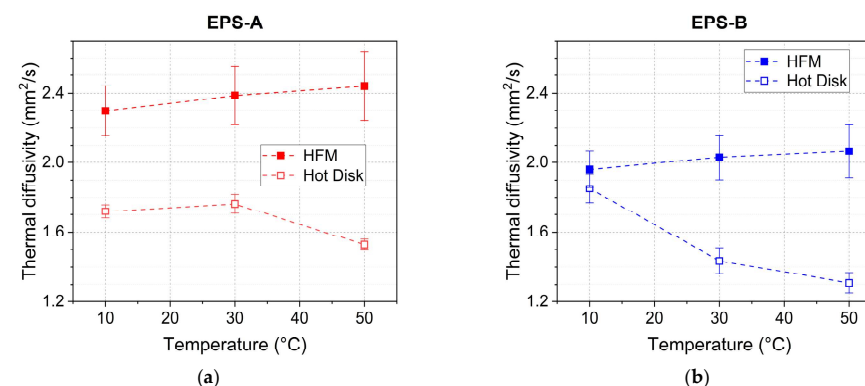


Figure 7. Thermal diffusivity evaluated with Hot disk and indirectly with HFM: (a) EPS-A; (b) EPS-B. Data points represent the mean of three independent specimens $\pm$ standard deviation.

Significant discrepancies were observed between the two measurement techniques, confirming the critical issues in obtaining reliable values for expanded foams. The thermal diffusivity of EPS-A was consistently higher than that of EPS-B, a result attributed to the higher volumetric fraction of air within the more expanded structure. Notably, the diffusivity values reported by Ma et al. [46] for standard EPS are one order of magnitude lower than those measured in this study. This substantial discrepancy is likely attributable

to the presence of the graphite filler, which enhances internal heat transfer mechanisms compared to neat polystyrene foams.

3.3.3. Thermal Conductivity

Thermal conductivity is the most relevant property for thermal insulation applications. Figure 8 illustrates the temperature dependence of thermal conductivity, alongside the theoretical values for neat EPS derived from the calibration in Equation (5). The experimental data and their theoretical counterparts are consolidated in Table A9.

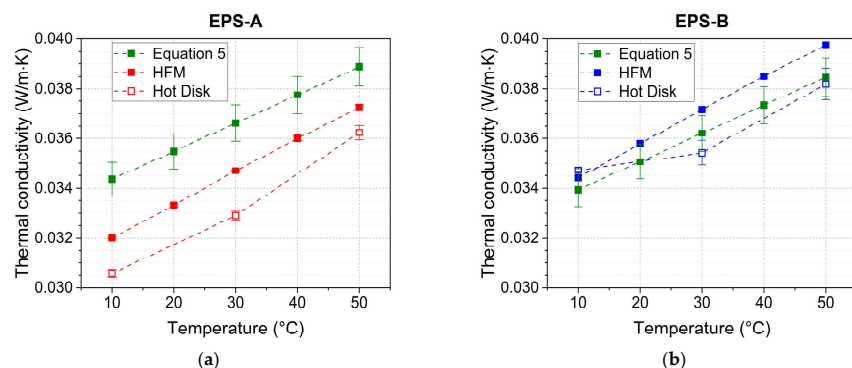


Figure 8. Thermal conductivity evaluated with Hot Disk and HFM compared to neat EPS of the same density from Equation (5): (a) EPS-A; (b) EPS-B. Data points represent the mean of three independent specimens $\pm$ standard deviation.

The thermal conductivity values obtained via HFM were slightly higher than those recorded by the Hot Disk, yet the results remained consistent across both methodologies. All measurement methods and both EPS grades exhibited a clear increasing trend with temperature, aligning with the data reported by Lakatos et al. [32] and the HFM calibration equation (Equation (5)). The dashed lines in Figure 8 are provided solely as a visual aid to highlight this monotonic behavior and do not represent a statistical regression or interpolation of the raw experimental data. Nevertheless, the results are characterized by extraordinarily high repeatability, and the expected increasing trend as a function of temperature is verified [14].

A notable observation is that for EPS-A, the experimental conductivity was lower than the theoretical values derived from the HFM equation for neat EPS, whereas the opposite was true for EPS-B. This indicates that the presence of graphite does not lead to a uniform reduction in thermal conductivity across different expansion grades. Specifically, graphite integration proved effective in reducing thermal conductivity for the higher expansion grade (EPS-A), while for the lower expansion grade (EPS-B), its impact appeared negligible.

These findings align with Akolkar et al. [34], who reported that higher carbon contents (approximately 5 wt.%) effectively reduce EPS thermal conductivity. However, our results indicate that at lower concentrations (below 2 wt.%), efficacy is highly dependent on the foam's physical structure. Specifically, for low filler loadings, advanced expansion seems essential to activate the graphite radiative shielding benefits.

Finally, the impact of water impregnation on thermal performance—determined via HFM—is reported in Figure 9, with the corresponding experimental data consolidated in Table A10.

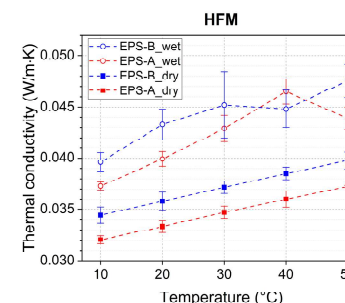


Figure 9. Effect of water on thermal conductivity of EPS. Data points represent the mean of three independent specimens $\pm$ standard deviation.

The presence of water plays a decisive role in increasing the thermal conductivity of the material. At elevated temperatures, although evaporation initiates, the rate of vapor transport is insufficient to significantly reduce the high moisture content localized within the open porosity.

4. Conclusions

The systematic characterization of graphite-enhanced EPS led to the following conclusions:

- Optical and SEM observations reveal a fine distribution of the carbonaceous filler without evidence of a percolative network.
- TGA and microstructural analyses confirmed that both EPS-A and EPS-B share a common chemical origin, with an estimated graphite content of 1–2 wt.%.
- EPS-A achieved a more extensive expansion, yielding a lower geometric density of $12.7 \pm 0.5 \text{ kg/m}^3$ and a higher open porosity fraction of 70% compared to EPS-B, which exhibited a bulk density of $16.0 \pm 1.1 \text{ kg/m}^3$ and an open porosity of 63%.
- HFM was found to be ineffective for measuring the specific heat capacity of low-density expanded polymers, yielding a high average relative error of 29%. However, DSC provided a reliable alternative after thermal densification at 120 °C. Among the protocols tested, the Sapphire comparison method yielded the highest accuracy relative to theoretical values, while the TOPEM technique resulted in a valid alternative adopting a heating rate of 1 °C/min.
- Following 30 days of water immersion, the geometric densities of the two EPS grades converged. This is attributed to the larger open porosity of EPS-A, which allows for a significantly higher moisture uptake of $144 \pm 17 \text{ wt.}\%$ relative to the $97 \pm 7 \text{ wt.}\%$ observed for EPS-B, thereby fundamentally altering the thermal transport of the material.
- The Transient Plane Source (Hot Disk) method proved to be a reliable technique for determining thermal diffusivity and conductivity, with results converging with steady-state HFM measurements.
- The temperature dependence of the thermophysical properties was confirmed, showing consistent shifts relative to the neat PS baseline.
- Comparative analysis against theoretical models for neat EPS successfully quantified the thermal performance enhancement provided by graphite. The findings indicate that graphite integration is not universally effective; its ability to reduce radiative heat transfer is significantly more pronounced in the grade with the higher expansion ratio (EPS-A), whereas the reduction is less substantial at higher densities.

The practical relevance of this work lies in the multivalence of its findings. Firstly, it establishes critical considerations for thermophysical characterization, showing that characterizing expanded polymers requires a conscious selection of testing parameters to avoid modeling errors. Secondly, the results demonstrate that EPS performance can be severely altered if installed without proper environmental conditioning. Thirdly, we quantify how these properties are affected by small temperature fluctuations, providing a dataset that allows for more accurate dynamic energy simulations. Finally, this study uncovers the counterintuitive efficacy of graphite as a radiative shield; our findings indicate that the additive's ability to reduce thermal conductivity is not universal but is strictly dictated by the material expansion ratio, being more effective in lower-density morphologies where radiative transport would otherwise dominate.

While this study establishes a baseline for commercial graphite-enhanced EPS, future research should explore the impact of varying filler concentrations to evaluate the eventual benefits in further improving thermal insulation. Additionally, long-term environmental aging studies are necessary to evaluate how structural degradation and persistent moisture retention influence the life-cycle energy efficiency of these high-porosity insulation systems.

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Data Availability Statement: The raw data supporting the conclusions of this article will be made available by the authors on request.

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

Appendix A. HFM Specific Heat Capacity

Evaluating specific heat capacity via HFM is an unconventional approach for low-density materials; consequently, the selection of testing parameters is critical and requires iterative adjustments. Initial tests were conducted following the manufacturer's recommendation to use a static air environment instead of nitrogen flow, thereby eliminating convective flows that could interfere with the c_p measurement. An initial temperature increment of 20 °C was adopted over the 10 to 50 °C range. All unspecified parameters remained consistent with the final refined settings. The results for individual specimens from this preliminary series are documented in Table A1.

Table A1. Specific heat capacity obtained with the first set of testing parameters, from 10 to 50 °C, imposing temperature steps of 20 °C and a static air environment.

Temperature (°C)	Specific Heat Capacity (J/g·K)		
	EPS-REF	EPS-A	EPS-B
10	1.004	0.707	0.999
20	0.804	0.553	0.824
30	1.418	1.296	1.157
40	2.438	1.983	1.598
50	1.423	1.724	1.748

The results presented in Table A1 appear inconsistent with literature values and exhibit a non-physical trend relative to temperature increases. Hypothesizing that moisture condensation at lower temperatures interfered with the measurements, a follow-up test was conducted on EPS-REF under a nitrogen flow, while keeping all other parameters constant. These results are detailed in Table A2.

Table A2. Specific heat capacity obtained with the second set of testing parameters, from 10 to 50 °C, imposing temperature steps of 20 °C and an environment with a nitrogen flux.

Temperature (°C)	Specific Heat Capacity (J/g·K)
	EPS-REF
10	0.916
20	1.020
30	1.222
40	1.317
50	1.601

Despite these adjustments, the initial results remained unsatisfactory. Consequently, the final experimental campaign was conducted in a static air environment, employing a descending temperature sequence from 40 °C to 10 °C to isolate the effects of moisture condensation. The upper temperature limit was reduced from 50 °C to 40 °C, as the former exceeds the manufacturer's specified operating range for the material. Furthermore, following the manufacturer's recommendations, the temperature increments for the c_p measurements were reduced to 10 °C to improve the resolution of the thermal data.

Appendix B. Optical Observation of EPS-REF and Scanning Electron Microscopy (SEM) of Carbon Residues

Surface optical observations of EPS-REF (without carbon additives) are shown in Figure A1 to compare bead expansion grades (Figure A1a) and verify the absence of graphite (Figure A1b).

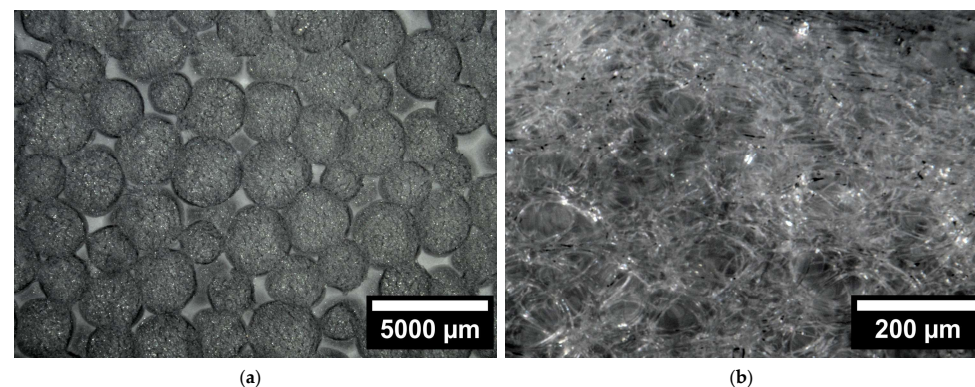


Figure A1. Optical micrographs of reference Expanded Polystyrene (EPS-REF) surfaces: (a) overview showing bead morphology and expansion grades; (b) higher magnification view verifying the absence of carbonaceous additives.

Complementary to the observations of carbon-enhanced EPS, Figure A2 shows SEM micrographs of the carbonaceous residue after toluene dissolution and centrifugation.

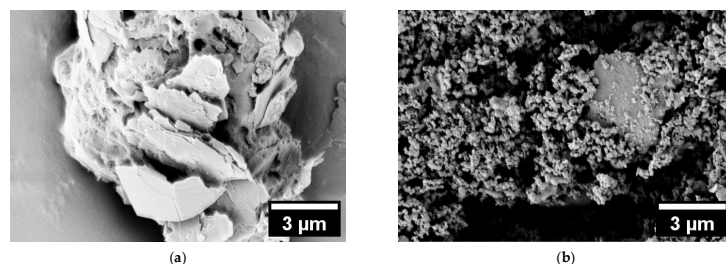


Figure A2. SEM observations of the carbonaceous residues after toluene dissolution and centrifugation: (a) EPS-A; (b) EPS-B.

Appendix C. Differential Scanning Calorimetry (DSC)

Figure A3 shows the DSC thermograms recorded at 10 °C/min, and from which the glass transition temperatures were determined (Table 3).

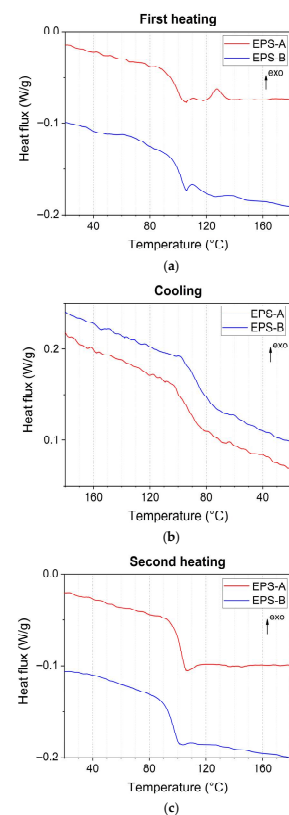


Figure A3. DSC thermograms of EPS-A and EPS-B recorded at 10 °C/min: (a) first heating; (b) cooling; (c) second heating.

Appendix D. Specific Heat Capacity Results

For completeness, all the specific heat capacity values as a function of temperature are listed in Table A3 (HFM), Table A4 (DSC using the sapphire comparison method), and Table A5 (DSC with TOPEM®) as mean value $\pm$ standard deviation, while Table A6 compares the average errors of the different techniques.

Table A3. Specific heat capacity obtained with refined testing parameters with HFM, from 40 to 10 °C, imposing temperature steps of 10 °C and a static air environment.

Temperature (°C)	Specific Heat Capacity (J/g·K)		
	EPS-REF	EPS-A	EPS-B
10	1.51 $\pm$ 0.08	1.85 $\pm$ 0.40	1.69 $\pm$ 0.38
20	1.01 $\pm$ 0.23	0.63 $\pm$ 0.37	0.75 $\pm$ 0.47
30	1.03 $\pm$ 0.40	0.86 $\pm$ 0.38	0.82 $\pm$ 0.06
40	1.39 $\pm$ 0.16	1.22 $\pm$ 0.43	1.56 $\pm$ 0.31

Table A4. Specific heat capacity obtained with DSC using the sapphire comparison method on thermally treated EPS-A at different rates, from 5 to 50 °C.

Temperature (°C)	Specific Heat Capacity (J/g·K)		
	10 °C/min	5 °C/min	1 °C/min
5	1.08 $\pm$ 0.04	1.07 $\pm$ 0.04	1.08 $\pm$ 0.06
10	1.09 $\pm$ 0.04	1.08 $\pm$ 0.04	1.10 $\pm$ 0.07
15	1.10 $\pm$ 0.04	1.10 $\pm$ 0.04	1.11 $\pm$ 0.08
20	1.12 $\pm$ 0.04	1.11 $\pm$ 0.04	1.12 $\pm$ 0.09
25	1.13 $\pm$ 0.04	1.12 $\pm$ 0.04	1.13 $\pm$ 0.09
30	1.15 $\pm$ 0.05	1.14 $\pm$ 0.04	1.14 $\pm$ 0.11
35	1.15 $\pm$ 0.05	1.15 $\pm$ 0.04	1.15 $\pm$ 0.11
40	1.16 $\pm$ 0.05	1.17 $\pm$ 0.04	1.16 $\pm$ 0.11
45	1.18 $\pm$ 0.05	1.19 $\pm$ 0.04	1.18 $\pm$ 0.12
50	1.20 $\pm$ 0.05	1.20 $\pm$ 0.04	1.20 $\pm$ 0.13

Table A5. Specific heat capacity obtained with DSC using TOPEM on thermally treated EPS-A at different rates, from 5 to 50 °C. N/A means “Not Available”.

Temperature (°C)	Specific Heat Capacity (J/g·K)	
	1 °C/min	0.1 °C/min
5	1.08 $\pm$ 0.04	N/A
10	1.09 $\pm$ 0.04	1.07 $\pm$ 0.20
15	1.10 $\pm$ 0.04	1.07 $\pm$ 0.19
20	1.12 $\pm$ 0.04	1.08 $\pm$ 0.18
25	1.13 $\pm$ 0.04	1.10 $\pm$ 0.18
30	1.15 $\pm$ 0.05	1.14 $\pm$ 0.24
35	1.15 $\pm$ 0.05	1.13 $\pm$ 0.20
40	1.16 $\pm$ 0.05	1.14 $\pm$ 0.21
45	1.18 $\pm$ 0.05	1.15 $\pm$ 0.19
50	1.20 $\pm$ 0.05	1.16 $\pm$ 0.20

Table A6. Comparison of average relative percentage error of the methods for determining specific heat capacity.

Method	Average Relative Percentual Error
HFM	±29.1%
Sapphire 1 °C/min	±8.3%
Sapphire 5 °C/min	±3.4%
Sapphire 10 °C/min	±3.8%
TOPEM 1 °C/min	±2.8%
TOPEM 0.1 °C/min	±17.8%

Appendix E. Thermal Diffusivity and Conductivity

All the punctual values of thermal diffusivity evaluated with the different techniques are summarized in Table A7 as mean value ± standard deviation, while Table A8 reports the volumetric specific heat capacity values adopted for the indirect diffusivity evaluation through HFM and the derived c_p provided by the Hot Disk measurements.

Table A7. Thermal diffusivity values calculated directly with Hot Disk and indirectly with the HFM.

Temperature (°C)	Thermal Diffusivity (mm ² /s)			
	EPS-A		EPS-B	
	Hot Disk	HFM	Hot Disk	HFM
10	1.72 ± 0.04	2.30 ± 0.14	1.85 ± 0.08	1.96 ± 0.11
30	1.76 ± 0.05	2.39 ± 0.17	1.44 ± 0.07	2.03 ± 0.13
50	1.53 ± 0.03	2.44 ± 0.20	1.31 ± 0.06	2.07 ± 0.15

Table A8. Volumetric specific heat capacity values imposed for the indirect calculation of thermal diffusivity and derived from Hot Disk.

Sample—Temperature	Imposed c_p (kJ/m ³ ·K)	Derived c_p (kJ/m ³ ·K)
EPS-A—10 °C	14.0	17.8
EPS-A—30 °C	14.5	18.7
EPS-A—50 °C	15.3	23.7
EPS-B—10 °C	17.5	18.8
EPS-B—30 °C	18.3	24.7
EPS-B—50 °C	19.2	29.2

The thermal conductivity values of the dry EPS are reported in Table A9 as mean value ± standard deviation, while Table A10 lists the values of the water-impregnated EPS sample.

Table A9. Thermal conductivity values calculated directly with Hot Disk and HFM, compared with the theoretical values of neat EPS (without graphite) at the corresponding density. N/A means “Not Available”.

Temperature (°C)	Thermal Conductivity (mW/m·K)					
	EPS-A			EPS-B		
	Hot Disk	HFM	Equation (5)	Hot Disk	HFM	Equation (5)
10	30.58 ± 0.16	32.01 ± 0.03	34.4 ± 0.7	34.69 ± 0.07	34.41 ± 0.08	33.9 ± 0.7
20	N/A	33.31 ± 0.06	35.5 ± 0.7	N/A	35.79 ± 0.09	35.1 ± 0.7
30	32.90 ± 0.18	34.69 ± 0.06	36.6 ± 0.7	35.67 ± 0.43	37.17 ± 0.06	36.2 ± 0.7
40	N/A	35.99 ± 0.08	37.8 ± 0.8	N/A	38.51 ± 0.06	37.3 ± 0.7
50	36.23 ± 0.29	37.25 ± 0.10	38.9 ± 0.8	38.70 ± 0.52	39.75 ± 0.08	38.5 ± 0.8

Table A10. Thermal conductivity values of water-impregnated EPS determined with HFM from 10 to 50 °C.

Temperature (°C)	Thermal Conductivity (mW/m·K)	
	EPS-A	EPS-B
10	37.3 ± 0.04	39.6 ± 0.04
20	39.9 ± 0.07	43.3 ± 0.15
30	42.9 ± 0.13	45.2 ± 0.32
40	46.5 ± 0.12	44.8 ± 0.18
50	43.9 ± 0.11	47.6 ± 0.16

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