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Electrospun composite fibers containing organic phase change materials for thermo-regulation: Trends

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ABSTRACT

Thermal management technologies that offer the capability of controlling a system's temperature within a range are needed for a very large set of applications, from electronic circuitry to battery technologies, mechanical systems, and fabrics, to name a few. Phase change materials (PCM), which release/absorb energy during a phase transition, are a potential solution to overcome some thermal management challenges. The encapsulation of PCM necessary for their containment during the phase transformation has been done by multiple techniques. Electrospinning is a very common and effective method to incorporate the PCM into polymeric fibers, however, there is a gap in the current literature regarding reviews that focus solely on electrospun fibers containing PCM. Thus, this review attempts to summarize the trends found regarding the use of electrospinning techniques to generate fiber - organic PCM composites. The most commonly employed organic PCM substances, host polymeric matrices, loadings, and typical variables employed during their manufacturing are presented. Trends regarding material selection, enthalpies of fusion and transformation temperatures of the composites are summarized and discussed. To assist with estimation of the cost-benefit of selecting specific PCM and fiber material combinations, approximate pricing data was gathered from open sources and general comparisons were included.

1. Introduction

Phase change materials (PCM) for thermal energy storage have attracted the attention of scholars and industry since the 1950's due to their ability to absorb and release energy via latent heat as they undergo a phase transformation [1]. The energy crisis in the 70's and interest in using building construction materials to reduce the energy consumption in the 80's increased the number of scholarly activities in the field [2]. A multinational research effort was launched in 1988 by the International Energy Agency to study "Phase-change and Chemical Reaction Energy Storage" [1]. The implementation of solar harvesting systems in the late 90's and early 2000's further propelled the PCM area of research. Six countries in Europe created an energy storage technical framework in 2004 to advance the applications of phase-change energy storage, building on Germany's Innovative PCM-Technology, a consortium of universities (e.g. University Stuttgart), corporations (e.g. BASF), and research institutions (e.g. Bavarian Center for Applied Energy Research) [1]. In recent years, PCM continue to be an alternative for energy

conservation when designing more efficient buildings by integrating them into appropriate building materials such as wallboards, shutters, radiant floors, ceiling boards, and energy storage [3,4]. With the current widespread adoption of sources of energy that offer an alternative to traditional fuels (from solar and nuclear sources to batteries, among others), PCM present a solution to the thermal management challenges implicit in the use of high power and high energy density technologies [5–7] [5–7]. Thus, we can expect continued growth in the PCM field of study.

The chemical makeup of PCM is quite diverse, including organic compounds (e.g., alcohols, esters, fatty acids, paraffins, as well as some polymers, such as polyurethanes, polybutadiene and polyethylene glycol), and inorganic compounds (e.g., hydrated and molten salts, metal hydroxides, metals and alloys), as well as eutectics mixtures of both of those [4].

A comprehensive list of organic and inorganic substances encompasses PCM with transition temperatures in the low, medium, and high temperature ranges (from –20 up to 1000 °C). The low temperature

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PCM are considered to be those with operational range 0 to 65 °C, which are commonly employed for energy-saving buildings [8,9], cooling of electronic circuits [10], battery components [11], thermoregulation of packing and storage units [12–14], and wearable fabrics [15], among others, and are usually based on a solid-liquid transformation. Numerous articles have been published in the last 10 years regarding the storage of latent heat employing organic phase change materials with transition temperatures within the mentioned low interval and this review attempts to review only those within that range.

Organic PCM present advantages such as a high latent heat of fusion, thermal and chemical stability, good nucleation rates, low vapor pressures in the melt form, congruent phase change, ability to be cycled many times, among others. Their disadvantages are associated with their low thermal conductivity, and until certain extent, their low volumetric latent heat storage capacity. Inorganic PSM based on the use of hydrated salts, have higher volumetric latent heat storage capacity, higher latent heat of fusion, higher thermal conductivity and tend to be compatible with polymeric containers. However, most of them suffer incongruent melting and dehydration in the process of thermal cycling, phase segregation and poor nucleating properties. Eutectics present advantageous characteristics such as volumetric thermal storage density slightly above that of individual organic compounds, no segregation and congruent phase change, the downside of their use is that the number of formulations for which thermophysical properties are known is quite limited [4,9].

Many of the published manuscripts focus on the classification of PCM, their thermo-physical properties and the methods employed for their integration into matrices/encapsulation. The later approaches aim to prevent leakage during solid-liquid-solid transformation cycles, avoid contact between the PCM and the environment that contains it, and to increase the heat transfer area. Integration of encapsulated PCM with transformations within the low temperature range mentioned above into diverse systems tends to be done by coating, lamination and spinning [16,17]. Coating is usually performed by dispersing the PCM microcapsules in a solvent containing thickener, antifoaming and dispersant agents, plus polymeric mixtures, then applying such into a surface by pad-dry-cure, dip coating or other transfer methods [18]. Lamination refers to PCM incorporated into layers as thin films of polymeric substances, which allow the integration of higher PCM concentrations per unit area. Spinning, based on the number of articles in the field, is one of the most widely employed approaches to fabricate fibers and incorporate the PCM into fabrics or porous mats. In spinning, melted polymers or polymeric solutions are mixed with PCM and spun using diverse techniques. Solvent spinning is preferred when compared to melt spinning due to the later causing PCM agglomeration. Electrospinning is reported to be the method of choice to produce fine fibers in large quantities.

Electrospinning, a frequently referenced technique that is commonly related to the use of PCM for fabrics/textiles thermoregulation, but which use could be extended to other fields, seems to provide an effective means to contain the organic PCM. However, to the best of our knowledge, there are no review articles that focus solely on electrospun fibers containing PCM. This review attempts to summarize the literature in the PCM field that uses electrospinning techniques to generate fiber-organic PCM composites. The objective of the manuscript is to provide a scope of what materials and experimental variables have been selected in the open literature when electrospinning fabrication was employed. It is our hope that the manuscript will open a window of opportunity to: i) identify which fiber-PCM combinations have been addressed and those that are still to be explored, ii) identify what compositions may present an operational advantage based in thermal properties, and iii) rationalize which variables employed in the fiber fabrication have a significant effect on the characteristics of the product, along price. We also seek to encourage the community in the field to enlarge the PCM composition - fabrication variables - thermal properties - microstructural characteristics data published records, for future endeavors to feed

machine learning algorithms to aid the design and material selection. Reports that employed nanofillers, believed to enhance the performance of the PCM, were not included in the review.

2. Electrospinning fundamentals

Electrospinning refers to a method of fabricating fine fibers in which an electric force is employed to draw a continuous jet/stream of charged polymeric solutions or melts. The application of high voltages (up to 30 kV) to liquid droplets produces charges in the liquid, the repulsive electrostatic forces generated counteract the surface tension of the fluid and promote the stretching of the droplet. At a critical point, a stream of liquid gets drawn from the surface (known as a Taylor cone). As the stream of liquid gets drawn and dries/solidifies in midair, the charges migrate to the surface, forming a fiber, which later gets captured by a grounded collector. If the liquid has low levels of molecular cohesion, a stream is formed, enabling electrospinning. If the molecular cohesion in the liquid is high, then a spray (rather than a stream) of droplets is produced, permitting electro spraying.

The basic configuration of an electrospinner includes: a syringe that serves as reservoir to the liquid, a needle/nozzle, a collector plate/rod, and a high voltage source that provides the force to extract the charged fluid and electrically connects the needle to the collector. Fibers can be produced with a single nozzle or a coaxial nozzle. When integrating PCM into a polymeric matrix, the single nozzle approach results in fibers that contain PCM in the interior and in the surface of the fibers. The fibers produced need post-processing to remove the PCM located in the surface and to prevent leakage during the phase transformation. Coaxial nozzles result in core-shell fibers, with PCM located mainly in the core regions. Fig. 1 presents a schematic representation of a coaxial electrospinner.

Electrospinning is one of the most commonly used techniques for the integration of PCM into fibers, although it has also been used to apply coatings [19–24]. One of the advantages of this process when concerned with temperature regulation, is that PCM-spun fibers typically do not require any further modification or treatment. To prevent exposing the PCM to the environment and potential leakage during the phase transformation, the PCM might be either dispersed within the matrix with the PCM loading limitations that such imply, or fully contained, which opens a wider window of PCM mass that can be included in the fiber. This review did not distinguish between those two cases and the data analysis presented includes a combination of those.

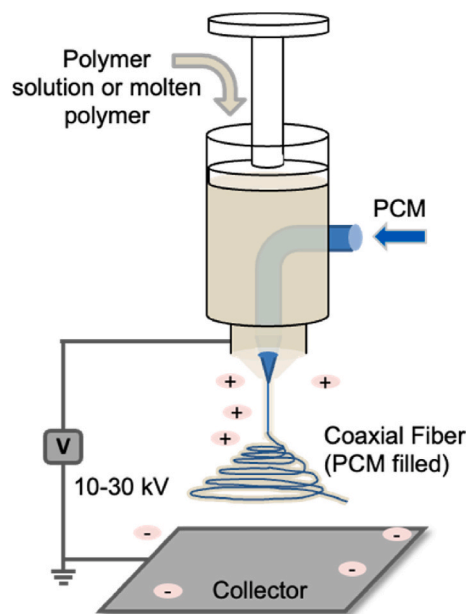


Fig. 1. Schematic representation of electrospinning setup.

3. Methodology for data retrieval, selection and analysis

The journal manuscripts were retrieved from open sources by conducting searches using diverse search engines and databases employing keywords such as electrospinning, electrospun, fibers, PCM, thermoregulation, phase change materials, organic PCM, or a combination of such. Literature included only journal manuscripts published from 2008 to date. The data extracted from the individual articles included transition temperatures, enthalpy values, PCM and polymer identities, percentages of components and experimental conditions employed. In some cases, the latter were extracted directly from tables included in the manuscript or the text, and in some instances were calculated based on the details mentioned in the experimental condition sections of the write up. The total number of cases analyzed was 134. Most data were analyzed using the Pandas data analysis library and the figures presented in the sections that follow, were made with Seaborn Statistical Data Visualization One. Both are veteran libraries of the Python language universe.

4. PCM and polymeric fiber components

Table 1 below defines the acronyms for the diverse PCM found in the literature and later employed in the figures that correlate trends in the field.

The solvents employed in the literature sources reviewed are mentioned in Table 5 thermal performance presented in the next sections, given that those tend to be partially or completely removed during the electrospinning fiber manufacturing process and are not expected to form part of the final structures.

Table 1
Polymer Fiber-PCM combinations.

Polymer Fiber	PCM	Literature
CAc (cellulose acetate)	PEG (polyethylene glycol)	[25,26]
	LA (lauric acid)	[27]
PA6 (polyamide 6)	ES (ethyl stearate)	[28]
	EP (ethyl palmitate)	[28]
PAN (polyacrylonitrile)	BS (butyl stearate)	[28]
	MP (methyl palmitate)	[28]
	PEG	[29]
	EL (ethyl laurate)	[28]
	LA-SA (stearic acid)	[30]
	CA-LA	[31]
	CA-MA (myristic acid)	[31]
	CA-PA (palmitic acid)	[31]
	CA-SA	[31]
	LA	[32,33]
	LA-MA	[34]
	LA-PA	[34]
	MA	[33]
	MA-PA	[34]
	MA-SA	[34]
PET (polyethylene terephthalate)	PA	[33]
	PA-SA	[34]
	SA	[33]
	SS (stearyl stearate)	[35]
	SS/PET (polyethylene terephthalate)	[35]
PMIA (poly (<i>meta</i> -phenylene isophthalamide)	HPCME (Hydroxypropyl cellulose)	[36]
PVA (poly(vinyl alcohol))	NOC (n-octadecane)	[37]
	PEG (polyethylene glycol)	[26,38]
PVB (polyvinyl butyral)	OD (octadecane)	[19,39,40]
	PEG	[41,42]
PVDF (polyvinylidene fluoride)	HX (hexadecane)	[43]
PVP (polyvinylpyrrolidone)	NOC	[43,44]
	PA	[45]
SAN (styrene acrylonitrile)	PA	[45]

5. Findings

5.1. Material selection trends

Fig. 2 presents an analysis of the number of unique/independent records of published articles by Digital Object Identifier (DOI) regarding fibers that include organic PCM generated by electrospinning techniques that were analyzed in this study. That is, a limited number of references included a very large number of individual cases. In contrast, Fig. 3 analyzes the data by the number of unique material data points for which enthalpies and transition temperatures are reported (each data point has a unique material combination by weight percentage (wt%). The first part of the analysis refers to the 17 cases without the addition of thermal, optical, or mechanical enhancers. Those with additives, considered special cases, and as previously mentioned, were not discussed in this paper.

As shown, there is a clear preference for the use of Polyethylene Glycol (PEG) as PCM, followed by the organic PCM fatty acid lauric acid (LA), and the alkane octadecane (OD). The wide use of PEG may be derived from its biocompatibility and the fact that it is used in the medical industry and chemical sectors. It is a hydrophilic polyether compound which use extends from surfactant, emulsifier, humectant and has been considered safe for dermal exposure [46]. The use of LA is associated with its thermal properties, highlighted in the next section. When the data is analyzed by chemical group, the use of fatty acids and their eutectics constitutes the larger number of cases, those are followed by alkanes. With regard to the matrix, the most commonly used polymer, by a very wide margin, is the thermoplastic polyethylene terephthalate (PET), which belongs to the polyester family, followed by polyacrylonitrile (PAN). The wide selection of PET is believed to derive from being one of the most abundant thermoplastic resins, to the point that some [47] consider it to be the only fiber and film forming saturated polymer with commercial importance. PET is the most common polymer employed in recent years in this field with nearly 50% of the experiments studied in this paper used polymeric fibers containing PCM employed PET [27], [35,48]. These resins are known for their stiffness (flexural modulus 2.8–3.5 GPa), strength (UTS 150 Mpa) [49,50], and ability to be recycled (e.g. Polystarusa). PET is often the material of choice for plastic bottles, food packaging and fibers for clothing. The second most used polymer (10.47% of experiments in the articles found) was polyacrylonitrile (PAN), a synthetic thermoplastic [28,30]. Since PAN decomposes and does not melt under normal conditions, it is used in fibers for textiles, ultra-filtration membranes, and is a precursor for the generation of carbon fibers for light-weight structural materials [51]. Other polymeric matrices employed to produce fibers with PCM employing an electrospinning method include polyvinylpyrrolidone (PVP) [43,44], polyamide 6 (PA6) [34], polyvinylidene fluoride (PVDF) [41,42], styrene acrylonitrile (SAN) [45], polyvinyl alcohol (PVA) [37], polyvinyl butyral (PVB) [19] and poly (*meta*-phenylene isophthalamide) (PMIA) [36].

5.2. Enthalpies and transition temperature trends

The body of literature regarding electrospun fibers containing PCM has many different PCM compositions and diverse polymers. Figs. 4 and 5 associate the most commonly used PCM with the polymeric matrix employed and graphed by either enthalpy of fusion (Fig. 4) or temperature of phase transition (Fig. 5). The enthalpies and transition temperatures tend to be the main factors considered for PCM and polymeric fiber materials selection, since those are directly associated to the performance of the systems where the fibers are employed. The larger the enthalpy of the PCM-fiber combination, the more effective is the temperature regulation ability of the fabric. The use of phase change materials that absorb energy during heating stages and release it during cooling periods needs to be targeted to the temperatures of the environments where those will be used. Some of the literature in the field of

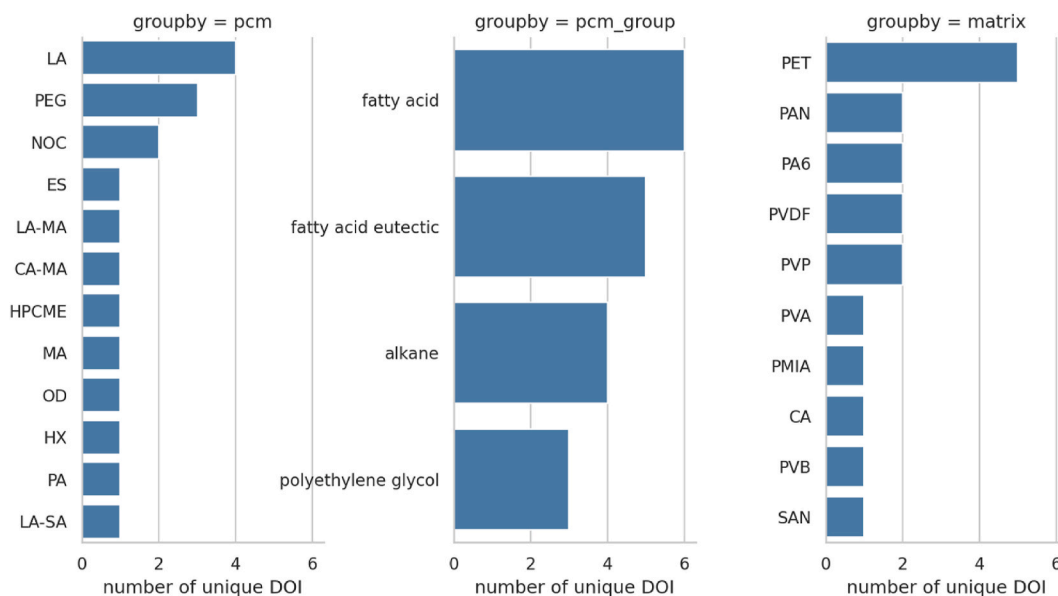


Fig. 2. Analysis of the number of unique/independent records Digital Object Identifier (DOI). Left: By PCM, middle: by group of PCM, and right: by polymer employed.

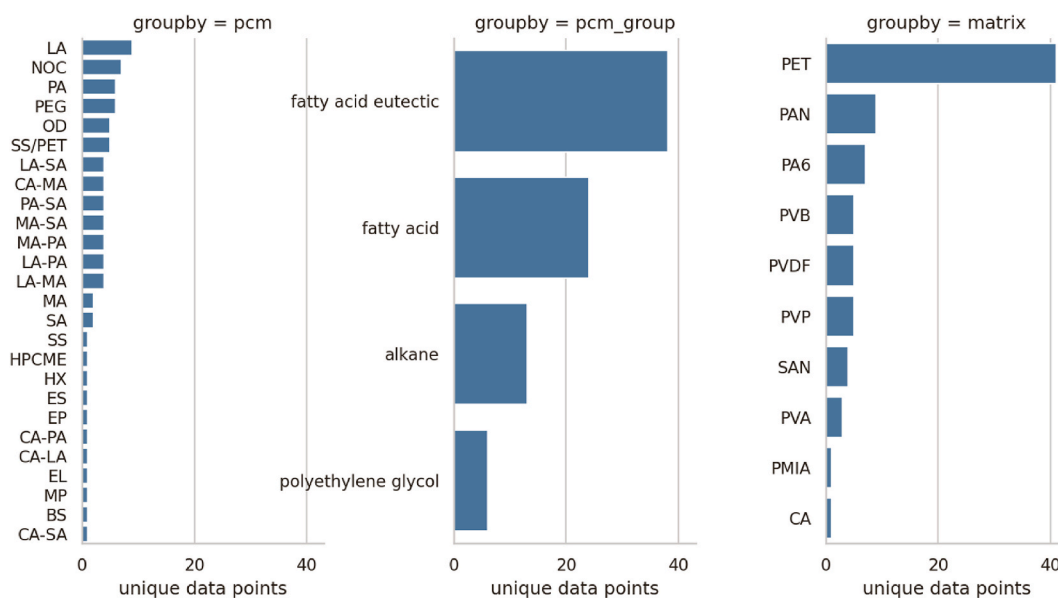


Fig. 3. Number of unique material data points for which enthalpies and transition temperatures are reported (each data point has a unique material combination by wt%). Left: By PCM, middle: by group of PCM, and right: by polymer employed.

smart temperature regulated textiles that expands on this concept is found in the review published in Ref. [52].

The black lines employed in Fig. 4 represent the 95% interval or variability of the heat of fusion reported for samples at diverse PCM wt %, while the actual histograms refer to the average value of ΔH of fusion (J/g) for the composites. It is worth noting that before this study, there has not been a parametric study that reports the comparative thermal properties of composites that use the same PCM with the diverse polymers presented herein. To note, the ΔH of fusion reported above are those of the PCM - polymeric fiber composite, not those of the pure PCM substances reported elsewhere. From Fig. 4, composites that used cellulose acetate (CAC) as the polymer fiber along with PEG as phase change material, show the largest enthalpies of fusion. The composite formed from the inclusion of lauric acid (LA) in PA6 presents a slightly higher heat of fusion than the LA - PET composite, while the

combination of palmitic acid (PA) - PET has a higher enthalpy of fusion than PA - SAN. Other than those two PCM, there is little overlap between PCM identity or correlations between how the different polymers containing PCM have a role in the thermal properties of the composites. Some PCM combinations, such as PEG included in PVDF, have the largest change in enthalpy based on the percentage weight of phase change component employed, although such combination of PCM-polymer is only studied by 2 reports [41,42].

When a PCM - fiber combination is selected for thermal storage application, the melting temperature must fall within the desired operating temperature range, thus, the window of transformation temperatures shown in Fig. 5 could be quite useful to select composites of known properties. The broader body of literature regarding electrospun fibers that employ PCM, as shown in Fig. 5, expand over a range of temperatures, from approximately 22 to 64° C. Stearic acid (SA) composites

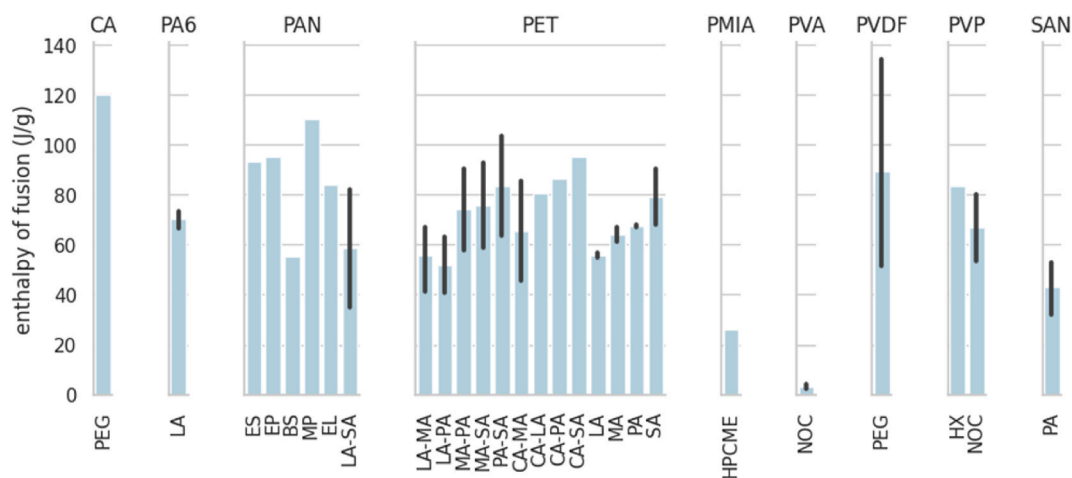


Fig. 4. Enthalpy of fusion. The correlation between most common polymeric matrices and PCM composites found for electrospun fiber-PCM composites literature. The black bar represents the 95% interval using bootstrapping. (Note: mixtures with additives such as thermal enhancers or reinforcement were excluded from this figure).

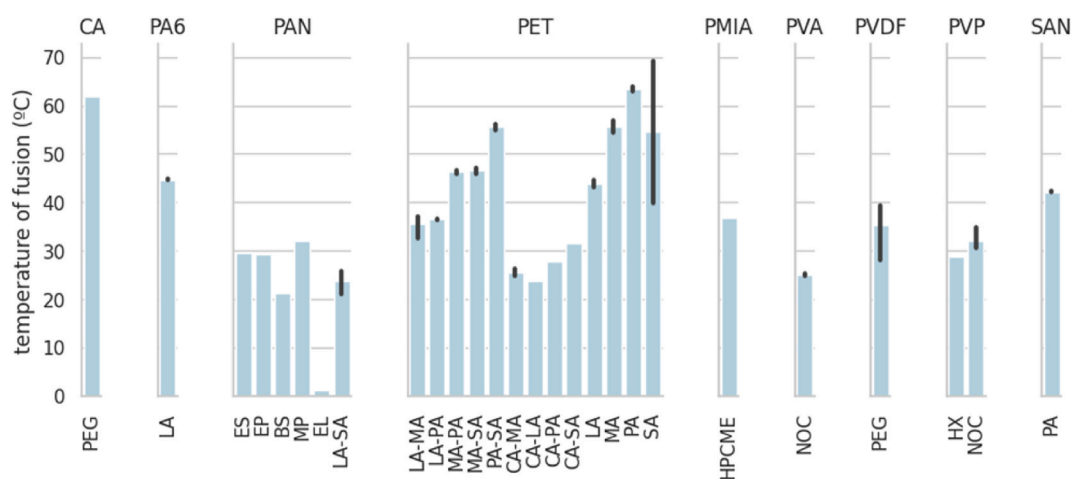


Fig. 5. Temperatures of phase transition of the most common polymeric matrices and PCM types found in the electrospun fiber-PCM composites literature. The black bar represents the 95% interval. (Note: mixtures with additives such as thermal enhancers or reinforcement were excluded from this figure).

present the greater variability in transition temperature when employed at diverse loadings in PET fibers, followed by those that combine PEG with PVDF. For all other composites, the amount of PCM employed, represented by the black bars, does not seem to have an impact larger than a few degrees. Since the enthalpy of fusion value of the PCM as a pure substance is a state function, the variability with loading noted may be explained by differences in the fiber wall thickness, PCM distribution within fiber and/or the thermal conductivity of the components. None of the polymeric fibers were expected to undergo melting at the temperatures at which the PCM or PCM eutectic mixtures suffered their phase transformation, since those are usually lower of than that of any of the polymers employed as matrices or hosts where the PCM is encapsulated [53]. For example, PET has a melting temperature of over 240 °C [54] and SA 69.8 °C [55].

Regarding the types of PCM as previously reported in other studies [56,24,57], and presented in Fig. 6 below, we observe some general trends: polyethylene glycol presents the highest enthalpy of fusion as a pure substance, thus, the block of studies that use it (represented as a brown rectangle) reach the highest values when combined with PVDF and PA6. Fatty acids are the largest group studied and reach the higher values when combined PAN and PET and are the most common type of PCM found in the literature. Alkanes tend to have the lowest enthalpies of fusion and were commonly fabricated along PVP, PAN and PVA. In

terms of temperature, fatty acids present the highest transformation temperatures and the largest spread of data, reaching values from approximately 15 °C to close to 70 °C. The alkanes employed in the studies included in this analysis have melting temperatures in a narrower region, from 20 to 40 °C.

Being a state property, it is important to analyze the enthalpy of fusion in terms of the weight percentage of PCM in the matrix (Fig. 7). The percentage of PCM, in addition to impacting the enthalpy of fusion, also affects the morphology of the fibers. For example, the average diameter of composite fibers generally increases with increasing weight proportion of PCM in the fibers [29,33]. It is noteworthy that the fiber/PCM composites cannot be obtained when the PCM wt% in composite solutions increase over a certain value [58], which could be attributed to the decline of the combination spinnability because of the addition of the less spinnable PCM [28]. As expected, Fig. 7 reveals that there is a correlation between PCM weight percentage included in the fiber and the enthalpy of fusion of the composites. For the same weight percentage, alkanes show a higher performance, in terms of enthalpy, than the other PCM groups. Also noteworthy is the better performance shown by some of the fiber combinations with the polyethylene glycol PCM group. The maximum percentage by weight of PCM found in the literature (i.e. LA, MA and PA) was 70% [33]. On average, the percentage by weight of PCM used in all the body of research analyzed, was

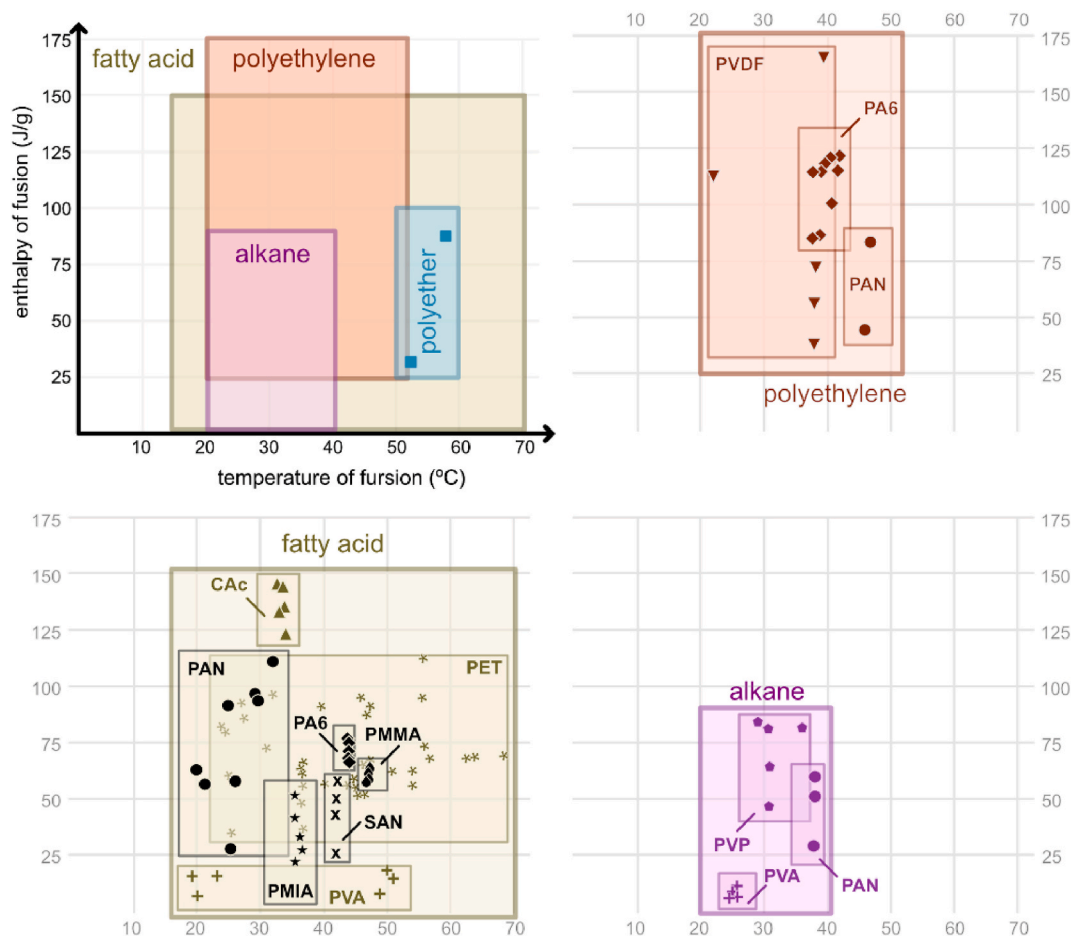


Fig. 6. PCM grouped by type in a temperature vs melting enthalpy coordinate system.

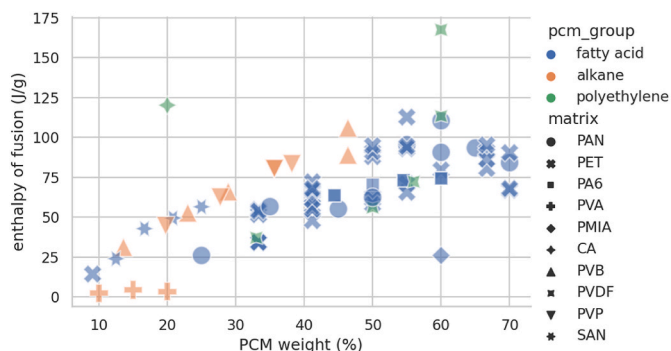


Fig. 7. Enthalpy of fusion of composites vs PCM weight percentage employed for the fiber-PCM combinations analyzed. Note: Polyethylene refers to PEG.

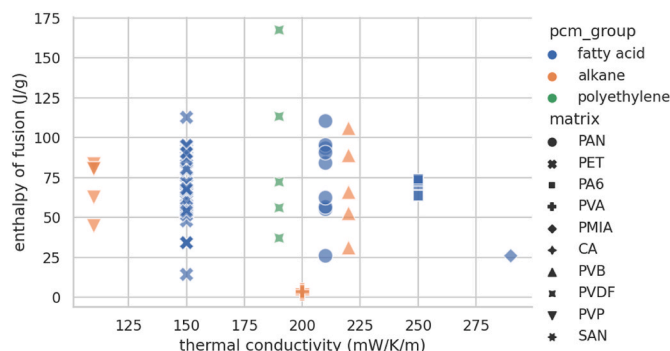


Fig. 8. Heat of fusion of PCM-Polymeric fiber composites vs thermal conductivity of the polymer employed. Note: Polyethylene refers to PEG.

50.7% [26,59].

When plotting all the points found in literature of electrospun fibers with organic PCM composites for enthalpy of fusion and attempting a correlation with the host polymer thermal conductivity, it is not evident that a trend exists. However, when analyzing independent points, such as all the ones represented by the blue symbols (fatty acids), there seems to be a trend that implies that higher enthalpies of fusion are expected for polymers that have higher thermal conductivities (PET vs PAN). See Fig. 8 [60].

5.3. Trends related to experimental variables employed to generate fibers

It is believed that the main effects of changing experimental variables such as voltages, needle sizes, distance of the collector and drum rotating speeds, will affect the composite fiber morphology, however, given that only some reports measured the fiber diameter and that wall thicknesses were normally unreported, such analysis was not performed. It is noted that the values for such variables were within the ranges: from 10V to 30V voltages, from 0.3 mm to 1.49 mm needle sizes, 12 cm–25 cm distances to collector system, and from 80 rpm to 120 rpm in situations where a collector drum was employed. From the statistical analysis conducted looking for trends between all those and fiber

performance, the only variable that showed a correlation was the rotating speed being linked to enthalpy of fusion. Such an unexpected correlation is further described in the next paragraphs. A mean comparison for the rotating speed factor (80, 100 and 120 rpm) MANOVA (dependent variables comprise a theoretical construct, thermal storage, $r = 0.168$, $p < .05$: melting temperature and melting enthalpy) was performed. As multivariate heterogeneity was observed, Box's $M = 33.10$, $p < .001$, multivariate test Pillai-Bartlett trace was employed as it is more robust to heterogeneous variances [61]. Nevertheless, Wilk's λ , Hotelling-Lawley trace and Roy's maximum root provided the same results in terms of the rejection of the null hypothesis. In univariate effects when Levene's test was significant (i.e., heterogeneity of variance), three criteria were applied to validate the correct rejection of the null hypothesis [62]: a) that the empirical F was greater than the theoretical $F(4.01)$; b) that the effect size, d , was significant (95% CI has no 0) and the lower limit magnitude ≥ 0.20); and c) that the ratio between β/α (probability of false acceptance of the null hypothesis/probability of false acceptance of the alternative hypothesis) was ≥ 1 . These criteria were met for significant univariate F . Post hoc analysis were computed with Dunnett's T3 test (conservative test). The effect size was computed as η^2 in multivariate analysis and multiple groups comparisons; and Hedges's g (unbiased) for two groups, computing confidence intervals with Hedges's and Olkin's formula [63]. The magnitude of the g effect size was quantified to interpret the effects as the amount of increase in the dependent measures, using r [64]. The error of empirical models i.e., the probability of a contradictory effect, was evaluated with the Probability of an Inferiority Scores (PIS) [65].

The results of a MANOVA exhibited a multivariate significant effect for the rotating speed factor, $F(4, 112) = 6.79$, $p < .001$, accounting for the 19.2% of the variance, $\eta_p^2 = 0.192$, of the thermal storage, an extremely powerful result, $1-\beta = 0.992$. At univariate level, the results showed a significant and power, $1-\beta = 0.999$, effect of the speed rotating factor, $F(2, 54) = 14.42$, $p < .001$, explaining 34.2% of the variance, $\eta_p^2 = 0.323$, in melting enthalpy, due to the higher amount of PCM observed in the studies with higher or higher rotational speeds, but a non-significant effect of the speed rotating factor in melting temperature was found $F(2, 54) = 0.87$, $p = .052$, $\eta_p^2 = 0.273$, $1-\beta = 0.108$.

Post hoc analysis (Dunnett's T3-) revealed that a rotating speed of the 120 rpm ($M = 84.51$) generated a significant, $p < .001$, higher enthalpy, due to the higher amount of PCM in the fiber, than a rotating speed of 100 rpm ($M = 78.58$), more than large effect, $g = 1.74[1.24, 2.24]$, and 80 rpm ($M = 27.48$), more than large effect, $g = 6.87[6.73, 7.21]$; and that a rotating speed of 100 rpm yielded a significant, and more than large effect, $g = 1.50[1.31, 1.69]$, amount of melting enthalpy, due to the higher amount of PCM in the fiber, than a rotating speed of 80 rpm. Thus, the speed rotating of 120 rpm appeared to increase the melting enthalpy in a 48.2% ($r = 0.482$) in relation to an 80 rpm rotating speed while the probability of containing lower amount of PCM and, therefore, generating a lower melting enthalpy with a 120 rpm rotating speed than with a 80 rpm rotating speed (error of the model) is lower than 10,000% (PIS = .0000); and 23.3% ($r = 0.232$) in relation to 100 rpm, being the probability of error of 6.1% (PIS = .061); and an increase of 48.0% of melting enthalpy, due to the lower amount of PCM in the fiber, rotating to a speed of 100 rpm over rotating to a speed of 80 rpm, with a probability of error of 5.7% (PIS = .057).

Once that the individual verification of single data points in the raw data was made for rotating speed, it was corroborated that the correlation observed is an artifact, and was a result of the analysis approach not being able to deconvolute the percent of PCM employed in the fiber manufacturing, with the changes in rotating speed. That is, the studies that employed higher loadings of PCM also required higher rotating speeds to collect the samples. Thus, confirming that the amount of PCM included in the fibers is the definitive.

6. Analyses based on cost

Cost data on the price of the chemicals used in experiments is useful when researchers are preparing budgets for project proposals. Industries interested in incorporating some of the composites mentioned above in future products, could consider pricing as well as efficacy of the PCM. To that end the cost data below was prepared as a resource to assist with estimation of the cost-benefit of projects or simulating numerical models [66]. The acquisition cost in dollars per unit (grams) was employed to calculate the cost of each of the chemicals used in the experiments. This methodology allowed for general comparisons between the materials. (Almost all PCM can be ordered in bulk, so larger scale projects would likely have lower per unit costs.) Pricing data for all chemicals is in U.S. dollars and were obtained in May 2022 from the websites of two major providers of chemicals in the US and are provided in the following panels: PCM in Table 3, matrix fibers in Table 4, and solvents in Table 5. Note: Prices may change based on country and market fluctuations.

The average cost of the PCM and matrix fibers used in the study are nearly the same, if excluding outlier stearyl stearate, which is used in only 1 study and is nearly fifty times more expensive than the next costly PCM. Examining the remaining PCM, the most frequently used, lauric acid, is also the least expensive. PEG, the next most frequently used PCM costs nearly three times the average cost of the PCM in the studies. Matrix fiber PET is the most commonly used in the studies and is nearly seven times the cost of PAN, which is the most expensive matrix used in the experiments studied.

Financial costs are not the only concern in the fabrication of PCM in textiles. There are also the costs and benefits of other environmental impacts, such as biodegradability or toxicity, of these materials to consider.

7. Other trends and gaps identified

All the PCM in this study are organic, which are generally nontoxic in nature and widely available [67]. For example, stearic acid (SA) is used in twenty-three of the reviewed studies and has been labeled with low level of concern for dermal sensitization and toxicity in household cleaners by the EPA, and as more environmentally friendly than petroleum-based products [68,69]. The solvents employed to produce the fibers, in the other hand, tend to be toxic and difficult to dispose; however, some research is now oriented to seek for more benign options, such as the work on composite organic PCM [70].

Table 3
PCM prices per mass unit.

PCM Acronym	Phase Change Material	PCM Group	Cost per unit
BS	Butyl stearate	fatty acid	0.111
CA	Capric acid (decanoic acid)	fatty acid	0.090
EI	Eicosane	alkane	0.474
EL	Ethyl laurate	fatty acid	0.075
EP	Ethyl palmitate	fatty acid	0.112
ES	Ethyl stearate	fatty acid	0.104
HPCME	Hydroxypropyl cellulose	fatty acid	0.872
HX	Hexadecane	alkane	0.247
LA	Lauric acid	fatty acid	0.075
MA	Myristic acid	fatty acid	0.206
MP	Methyl palmitate	fatty acid	0.262
NOC	n-Octadecane	alkanes	0.171
OD	Octadecane	alkane	0.426
PA	Palmitic acid	fatty acid	2.492
PEG	Polyethylene glycol	PEG	2.080
PET	Polyethylene terephthalate	fatty acid	0.389
PW	Paraffin wax	alkane	0.079
SA	Stearic acid	fatty acid	4.470
SS	Stearyl stearate	fatty acid	202.000

Note: PCM are sorted alphabetically. Units are in dollars per gram for all materials. Price sources were acquired from websites of two of the largest chemical providers in the US.

Table 4
Matrix Fibers prices per mass unit.

Matrix Fiber Acronym	Chemical name	Cost per unit
CAC	Cellulose acetate	0.2320
Cs0.32WO3	Cesium tungsten oxide	11.0800
PA6	Polyamide 6	0.8440
PAN	Polyacrylonitrile	2.6400
PET	Polyethylene terephthalate	0.3888
PMIA	Poly(<i>meta</i> -phenylene isophthalamide)	1.0100
PU	Polyurethane	0.4020
PVA	Poly(vinyl alcohol)	0.2250
PVB	Polyvinyl butyral	0.2700
PVDF	Polyvinylidene fluoride	1.2500
PVP	Polyvinylpyrrolidone	0.3520
Silk	Silk, fiboin solution	17.7000

Note: Matrix fibers are sorted alphabetically. Units are in dollars per gram for all materials. All the matrix fibers in the reviewed studies are polymers. Of the different polymers used, pricing data was unavailable for two: poly (*meta*-phenylene isophthalamide) and polyvinylidene fluoride. Polyurethane was listed in one experiment, but no molecular weight was given in the study, so we dropped the observation from the previous analyses, but it is included in this panel. Additives such as titanium dioxide and silicon dioxide were excluded. Price sources were acquired from websites of two of the largest chemical providers in the US.

Table 5
Solvent prices per unit.

Solvent Acronym	Chemical Name	Cost per unit
Acetone	Acetone	0.0842
DCM	Dichloromethane	0.0762
DMAC	Dimethylacetamide	0.1580
DMF	Dimethyl formamide	0.1210
Ethanol	Ethanol	0.1280
HFIP	Hexafluoro-2-propanol	2.6200
PVA	Poly(vinyl alcohol)	0.2250
PVP	Polyvinylpyrrolidone	0.3520
TEOS	Tetraethyl orthosilicate	0.1160
TFA	Trifluoroacetic acid	0.5480

Note: Solvents are sorted alphabetically. Units are in dollars per mL for all materials.

The two most frequently used matrix fiber polymers in this review study are non-biodegradable [71,72]: PET (polyethylene terephthalate), which approximately half the cost of polyamide 6 (PA6 or nylon). However, two matrix fibers are biodegradable: polyvinyl alcohol (PVA) [71] and biobased polymer cellulose acetate (CAC) [73], both of which cost less per gram than PET or PA6. The former non-biodegradable examples are polymeric fibers that can be employed for applications that require resistance to environmental factor for long term use, and the biodegradable for applications that could accommodate eco-friendly alternatives with shorter lifetimes. There are few recycling options for acrylic polymers; however there has been some exploration of experimental and industrial recycling techniques for polyacrylonitrile (PAN) and poly(methyl methacrylate) (PMMA) [74].

There seems to be an opportunity to produce other material combinations, for the PCM already studied but with different combinations of polymeric fibers, and for new compositions, such as the ones mentioned in other organic PCM review articles where methods other than electrospinning have been listed [75,76].

Current trends for materials selection design and even discovery, are aided by machine learning algorithms. However, to apply such approach to this field, the number of formulations employed, and the properties and characteristics of the fibers produced, should be enlarged. Adding microstructural detailed information about the fibers produced to the data generated could also render a more complete picture of the potential variables than can be used to tailor organic PCM-polymeric fibers. Other practical aspects of these materials that could be the subject

of a future review include thermal stability, flammability, supercooling, hygroscopicity, environmental impact, and/or resistance to chemical attacks.

8. Conclusions

Electrospinning has been recognized as a well-developed and widely utilized method to generate non-woven fibers. Electrospun fibers containing PCM can be used in many diverse applications and research in this area continues to expand the number of material combinations to generate the composites with optimized thermal properties. The analysis of the literature data aggregated in this study provides researchers with a brief compendium of organic PCM material compositions, identity of polymeric systems employed to contain them, their temperatures of operation, expected enthalpies of fusion and cost for formulation generated by electrospinning techniques.

Some of the trends identified include, for materials selection, a clear preference for the use of Polyethylene Glycol (PEG) as PCM, followed by the organic PCM fatty acid lauric acid (LA), and the alkane octadecane (OD). Regarding the matrix, the most commonly used polymer is the thermoplastic polyethylene terephthalate (PET), followed by polyacrylonitrile (PAN).

Fatty acids are the largest group studied and reach the higher values when combined PAN and PET and are the most common type of PCM found in the literature. Alkanes tend to have the lowest enthalpies of fusion and were commonly fabricated along PVP, PAN and PVA. In terms of temperature, fatty acids present the highest transformation temperatures and the largest spread of data, reaching values from approximately 15 °C to close to 70 °C. The alkanes employed in the studies included in this analysis have melting temperatures in a narrower region, from 20 to 40 °C.

When plotting all the points found in literature of electrospun fibers with organic PCM composites for enthalpy of fusion and attempting a correlation with the host polymer thermal conductivity, it is not evident that a trend exists. However, when analyzing independent points, such as all the ones for fatty acids, there seems to be a trend that implies that higher enthalpies of fusion are expected for polymers that have higher thermal conductivities.

The maximum percentage by weight of PCM included in the polymeric fibers found in the literature was 70%. On average, the percentage by weight of PCM used in all the body of research analyzed, was 50.7%.

The trends and correlations presented herein are expected to expose the reader to a snapshot of the current state of the technology, while the gaps identified point to directions that could be explored in future endeavors.

Author contributions

Conceptualization, ASG and CCL; methodology, EAF, and LA; investigation ASG, CCL, EAF and LA; formal analysis ASG and LA; writing—original draft preparation, review and editing ASG, CCL, EAF and LA; visualization ASG.

Declaration of competing interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

Data availability

Data will be made available on request.

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