Using statistical analysis to create a new database of Nanofluids' specific heat capacity

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Abstract

Nowadays, heat transfer fluids (HTFs) with high thermal properties are needed to develop more efficient and compact energy systems to achieve sustainable development goals. Nanofluids (NFs), through the incorporation of nanoparticles in conventional HTFs, become one of the most suitable techniques to improve their thermophysical properties. However, despite its potential industrial applications, there is not only a lack of a theoretical framework but also a clear trend about its behavior. Therefore, this work aims to perform a critical review and statistical analysis to understand the NFs heat capacity (Cp). To this end, a wide variety of NFs from the literature was processed using Principal Component Analysis (PCA) and Response Surface Methodology (RSM). Finally, a database with Ansys Granta Constructor 2021 software was created and an analysis with Ansys Granta Selector 2021 was performed. As a result, the key parameters that impact the Cp of several nanofluids are obtained as well as: their high-temperature dependence, the nature of the liquid medium, and the type of nanoparticles. In addition, the results allow to identify and design nanofluids with specific properties for specific working conditions.

Keywords: Nanofluids; Concentrated Solar Power (CSP); Thermal Energy Storage (TES); nanoparticles; Principal Component Analysis (PCA); Response Surface Methodology (RSM).

1. Introduction

Energy is an essential resource for humanity, and its storage is one of the fundamental points for industrial development. Energy storage is considered a key factor for enabling technology for fully implementing renewable energies (RE), solving their main drawback, and facilitating sustainable development goals. Therefore, incorporating a storage system allows to implement more efficient, safe, and flexible systems [1,2]. In Pparticular, concentrated solar power (CSP) plants that incorporate thermal energy storage (TES) facilities have become one of the most promising renewable solar technologies due to their cost-efficiency relation [3,4]. Several parameters of working fluids must be considered to design more efficient systems. Usually, these working fluids are simultaneously used as TES materials and/or as heat transfer fluids (HTF). Therefore, the design, development, and improvement of TES and HTF fluids are essential for developing high-efficiency and optimal systems [5].

In the last years, researchers in diverse fields have been interested in the improvement of thermophysical properties observed in fluids when across the addition of nanoparticles (NPs) were added into fluids (*i.e.*, oils [6], polyethylene glycol [7], molten salts [8], water [9]). This fact encourage researchers to develop a new class of fluids known as nanofluids (NFs), studied for the first time in 1995 by Choi S. *et al.* [10]. NFs are engineered by suspending nanoparticles (NPs) (that are between 1 nm and -100 nm) in diameter in a base fluid. The NPs materials can be metallic, non-metallic, oxides, carbides, ceramics, carbon based, a mixture of different NPs them known as (hybrid NFs), and even nanoscale liquid droplets [11–14]. The NFs show improved thermophysical properties such as heat and mass transfer, thermal conductivity (κ), or specific heat capacity (*Cp*) compared to the base fluid [15–17]. Furthermore, a large variety of new potential applications of NFs have emerged over time, such as: lubricants [18], desalination [19], CO₂ absorption/capture [20], batteries [21], industrial cooling [22], or heat exchangers [23], among others.

Specifically, NFs attracted the scientific community's attention due to their anomalous *Cp* improvement, reaching enhancements up to 30-40 % with by the addition in low concentrations of low NPs concentrations (*i.e.*, 1 wt.%) [24]. Several studies have been carried out in recent years to investigate the NFs *Cp* enhancement, and great advances have been made to understand the phenomenaon behind [25]. Moreover, the main parameters and properties that influence the *Cp* variation have been identified in the last years: temperature and pH along with NPs parameters (*i.e.*, the concentration, size, shape, or nature of the NPs), temperature, and pH mainly, among others [16,26]. Nevertheless, to achieve its scalability and understand its full potential, further investigations are required in order to develop a strong theoretical framework. Despite this, no studies have been found in which a addressed to clarify the trend observed in *Cp* values is showed [16]. In addition, there is a high dispersion on the experimental-measured data

reported in the literature : Thus, there are experiments that under the same experimental conditions show report different Cp values and performance. Furthermore, the results are contradictory in many cases, with unclear trends on their behavior [27–32]. Therefore, there are not clear tendencies on how the incorporation of NPs affects Cp. Another difficulty seen in the literature is the few specifications and non-accurate descriptions of the measurements and calculation methods. For example, there is no information that indicate at which temperature thermal values were obtained, or which methodology was followed for preparing the NF. As a result, there does not seem to be a clear specification or protocol for properly measuring NFs. These facts make the comparison of results and the comprehension of their thermal performance under real conditions a challenging task.

This study aims to perform a critical review and analysis to understand the NFs Cp experimental reported data. For this purpose, several parameters from NFs employed in energy storage applications were collected from the literature: such as base fluid and NPs material, size, and concentration of the NPs, temperature, Cp enhancement, sampling, and experimental procedure (*i.e.*, related to the equipment and methodology as well). Furthermore, a principal component analysis (PCA), ANOVA test, and response surface methodology (RSM) were employed to determine the main parameters affecting the Cp and their behavior and to develop a mathematical model. The Ansys Granta Constructor 2021 tool was used to create a database which was analyzed using Ansys Granta Selector 2021 to select NFs with specific properties and to observe their behavior. The main goal of this study is to unify Cp values and to identify clear trends to design NFs for applications with specific thermal requirements.

2. Methodology

Data acquisition: The data source for this study groups covers scientific journals and conference proceedings from Scopus database that contain the keywords . The search was effectuated under the following keywords: "Nanofluids" and "Energy Storage-". There were Up to 2020, a total of 301 publications fulfil these requirements associated with this research topic at the end of 2020, and 111 of them articles included reported *Cp* values. Exactly, a total of 884 *Cp* values and their measurement specifications were extracted from these studies.

Principal component analysis (PCA): the database was standardized by the Standard<mark>S</mark>caler Sklearn function to make an equal comparison between among all each values [33]. Afterwards, the database was transformed by a principal component analysis (PCA) defined by Sklearn in Ref. [33]. PCA is a statistical method used for dimensionality reduction, features extraction, or data visualization by an orthogonal transformation of the dataset into a new subspace, with new axes called principal components (PCs) [34–36].

Statistical analysis-response surface methodology (RSM): The data were evaluated by the ANOVA statistical analysis (analysis of the variance) to fit the data to a mathematical model and predict values. To this end, the dependent variable (response) was the specific heat capacity, and the independent variables, the nanoparticles' size, concentration, and the fluid temperature. In this manner, with the help of Stat-Ease code, an experiment designer software, the design of experiments software (Stat-Ease) ADD REFERENCE! and the obtained equations, a response surface was ganalyzed or obtained?? As a result, the developed models using the experimental responses exhibited p-values below 0.0001, implying that the proposed models are relevant considering the factor relations presented for each response or equation.

Ansys Granta 2021 Constructor/Selector database: The database was created with the Constructor software from Ansys (ADD REFERENCE!), employing the data obtained from the literature. The general organization of the database was divided into inorganic, organic, and mixtures and then arranged depending on the base fluid or the type of nanoparticles. The base fluids considered were hydrocarbons, substituted hydrocarbons, ionic liquids, molten salts, oils, organic acids, water, and mixtures, whereas And the nanoparticles treated were those formed by metal oxides, metals, nitrides, and carbon-based materials nanoparticles. Figure 1 depicts the arrangement of nanofluids; and the nanoparticles used in for each base fluid.



Figure 1. Arrangement of nanofluids in the nanofluids database.

3. Results

NFs were studied for diverse applications as HTS or TES materials. although each application needs different thermal requirements. According to this fact, these applications ean be have been classified as high, medium, or low temperature [37]. Therefore, each application needs different thermal requirements. For this reason, a great variety of base fluid materials were

found in the literature quest research. More in detail, **Table 1** summarizes the obtained 36 base fluids, with their corresponding references. It is important noting Point out that solar salt (*i.e.*, NaNO₃ – KNO₃ (60:40)) and water were the most studied systems and from which , so reporting the highest amount of more Cp values were obtained.

Distilled Water (DW) [45][46] Methoxyperfluorobutane (HFE 7100) [70] [70][71][72][73][74][75][76][77] [70][71][72][73][74][75][76][77] Water [78][79][80][81][47][82][48][83] [84] [85] [84] [85] Propylene glycol (PG) [53] Capric acid (CA) [86] Myristic acid-capric acid eutectic (MA-CA) [86] Paraffin [87][88][89] n-octadecane [49] Polyalphaolefein (PAO) [90][91] Engine oil [92][93][46][94] Heat transfer oil [95] Mineral oil [90] Therminol-55 [96] Oleic acid-(DL-menthol) [97] ethylene glycol (EG) [51][50][98][94][46][84][90][99] Glycerol [99] EG/Water [100][101][102][103][72][90] [104][105][106][45][107][108] [112][113][114][28][115][30][11 NaNO ₃ [4][25][59] NaNO ₃ -KNO ₃ (60:40) [122][123][124][125][126][127]	Base material	References		
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	D CL U O	[61] [110][136][62]		
$BaCl_2 - H_2O \qquad [137]$	$BaCl_2 - H_2O$	[137]		
NaCl-CaCl ₂ [138]	NaCl-CaCl ₂	[138]		
BaCl ₂ -NaCl-CaCl ₂ -L1Cl [139]	BaCl ₂ -NaCl-CaCl ₂ -LICl	[139]		
$Ca(NO_3) \cdot 4H_2O \cdot NaNO_3 \cdot KNO_3 \cdot LINO_3$ [30]	$Ca(NO_3)$ ·4H ₂ O-NaNO ₃ -KNO ₃ -LINO ₃	[30]		
$Ca(NO_3)_2 - KNO_3 - NaNO_3 - L1NO_3 \qquad [140][141][32]$	$Ca(NO_3)_2$ -KNO_3-NaNO_3-L1NO_3	[140][141][32]		
$Na_2CO_3 \cdot 10H_2O \cdot Na_2HPO_4 \cdot 12H_2O EHS (Eutectic [142])$	$Na_2CO_3 \cdot 10H_2O \cdot Na_2HPO_4 \cdot 12H_2O EHS$ (Eutectic	[142]		
$\frac{1}{1}$	$\frac{1}{1000} \frac{1}{1000} \frac{1}{1000$	[142]		
$\frac{\text{K}_{\text{NO}3} - \text{Na}_{\text{NO}2} - \text{Na}_{\text{NO}3} (53:40:7 \text{ mol. \%}) (\text{MHS}) \qquad [143]}{\text{K}_{\text{NO}3} - \text{L}_{\text{CO}3} - \text{N}_{\text{CO}3} - N$	$K_{\rm NO_3-NaNO_2-NaNO_3}$ (35:40:7 mol. %) (MHS)	[143]		
K ₂ CO ₃ -LICO ₃ -INd ₂ CO ₃ [144]	K ₂ CO ₃ -LICO ₃ -Na ₂ CO ₃			
$Li_2CO_3-K_2CO_3$	Li ₂ CO ₃ -K ₂ CO ₃	[145][146][147][148][65][64]		
LiNO, NaNO, KNO, [20][05][05][149]	LINO, NONO, KNO.			
Aviation turbine fuel	Aviation turbine fuel	[50][150][151]		
Ionic liquid [Camim][PEa] [152]		[152]		
Ionic liquid [04mm][F16] [155]	Ionic liquid ([Hmim]]EF.)	[155]		
Ionic liquid ([finini]]D147 [134]	Ionic liquid (Camim)[NTfa]	[15]		
Ionic liquid [Campyrr][NTfa] [155]	Ionic liquid [Campyrr][NTf_]	[155]		

 Table 1. Summary of nanofluids base materials collected from the literature and their corresponding references.

Liquid argon [1	56]
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For each scientific article, the obtained selected information was the following: base fluid type (BF), density base fluid density (ρ (BF), kg·m³), NPs type, NPs morphology (form factor), NPs concentration (wt. % and/or v. %), NPs density (ρ (NPs), kg·m³), NPs size (S, nm), temperature (T, °C) at was measured the of *Cp* mesurement, *Cp* of BF (J·K⁻¹), *Cp* of NF (J·K⁻¹), NFs *Cp* variation (ΔCp ,%), along with the measurement conditions and procedures. Measurement conditions and procedures that include the measurement methods, measurement techniques, sampling, and measurement conditions (*i.e.*, whether the first measurement was discarded or not). These variables are critical to analyzing the *Cp* measurement methods for characterizing NFs.

3.1. Specific Heat capacity methodology and procedures

Instrumental and methodological analysis: Several techniques were found in the literature review to measure the Cp. These can be techniques are classified as theoretical (10.18) %) and experimental (89.82%) techniques. The experimental techniques include a being the latter transient Double Hot-Wire (DHW), thermal conductivity instrument, simultaneous Thermogravimetry Analysis-Differential Scanning Calorimetry (TGA-DSC), Modulated Differential Scanning Calorimetry (MDSC), handmade setups, and Differential Scanning calorimetry (DSC). Otherwise, theoretical techniques include numerical procedures and simulations (*i.e.*, Molecular Dynamics (MD) simulations (MD). Figure 2 shows a pie chart of the different techniques. The the most used equipment was is DSC, accounting more than 68 % of data measured measurements through this technique. The rest of the techniques represent the 30.1 %: transient DHW (6.4 %), thermal conductivity instrument (0.6 %), simultaneous TGA-DSC (5.4 %), numerical techniques (5.9 %), MDSC (6.9 %), MD simulations (1.2 %) and inhouse devices (3.2 %). However, an important remark is that 1.9 % of the data do not describe the technique used to measure the thermophysical properties. Although this is a small proportion, it creates a gap in the methodological procedure since this parameter is essential has to be described in the papers to reproduce the experiments under the same conditions. In fact, it is not surprising that the DSC was the most employed technique because it is the most precise and suitable instrument to determine thermal properties (*i.e.*, melting temperature, melting enthalpy, or Cp) [38,39]. More in detail, the most used calorimetry instruments (DSC) were manufactured by Mettler Toledo© (26.5 %) and TA Instruments© (22.7 %), representing almost 50 % of the data, followed by Netzsch-Gruppe[©] (15.2), PerkinElmer[®], Inc. (8.6%) and finally, Setaram KEP technologies, Inc. ® (5.2 %).



Figure 2. A pie chart of tTechniques used to determine the specific heat capacity and the percentage of usage: transient DHW, TC (thermal conductivity instrument), simultaneous TGA-DSC, numerical techniques, MDSC (Modulated Differential scanning calorimeter), MD simulations, in-house calorimeters, DSC, and percentage of no data. Those determinations that do not specify the technique used are accounted as *no data*. On the right side is the rate of ratio of experimental and theoretical *Cp* determinations.

Otherwise, the analysis of the measurement methods followed by each instrumental technique is Another important characteristic to considered is the analysis method followed. Thus, six different methods were identified and categorized into three main groups according to the technique. The pie chart shown in Figure 3 presents the ratio (%) of data measured following each method: (1) transient DHW: T-history (1.4 %); (2) Thermal conductivity instrument, numerical, MD simulations and in-house setups equations (13.7 %); (3) TGA-DSC, MDSC and DSC techniques: direct method (11.9 %), ASTM E2716 (6.2 %), ASTM E1269 (46.7 %) and the area method (6.1 %). Finally, a total of 124 *Cp* values (14 %) does not indicate the methodology used. In this case, it is remarkable that the lack of information is higher than the ones for the in the case of the instrumental section. This fact generates uncertainty in the data and an incomplete methodological description because the results cannot be further replicated.



Figure 3. <u>A pie chart of M</u>ethods followed to determine the specific heat capacity: ASTM E1269, No data, equations??, direct and area method, ASTM E2716, Area Method, and T-history. Those determinations that do not specify the technique used are accounted as *no data*.

This fact generates uncertainty in the data and an incomplete methodological description because the results cannot be replicated.

Despite this, the most applied methodology is a standard procedure, "Standard Test Method for Determining Specific Heat Capacity by Differential Scanning Calorimetry" [40] by ASTM International. Therefore, following standardized procedures helps to compare results with each other studies. Otherwise, the second main group corresponds to the Cp determination through equations QUÉ SIGNIFICA!!! (13.7 %), followed by the direct method in DSC (11.9 %). Moreover, the area method (6.1 %) and the standard methodology ASTM E2716 "Standard Test Method for Determining Specific Heat Capacity by Sinusoidal Modulated Temperature Differential Scanning Calorimetry" [41] (6.2 %) shows a similar ratio. Lastly, the less-used method is the T-history (1.4%) which has been used in the transient DHW technique. In addition, Table 2 summarizes the relevant equations used to determine Cp for all the described methods.

Table 2. Equation ² s description of the specific heat capacity methods: Model I, Model II, in-house
calorimeter, diffusivity, effective volumetric specific heat, effective specific heat, simulations, Debye
theory, ASTM E1269, ASTM E2716.

Method	Formula		
Model I	$C_{p,nf} = \emptyset \cdot C_{p,s} + (1 - \emptyset) \cdot C_{p,bf} \qquad (eq. 1)$ $C_{p,nf} = \text{specific heat capacity of the nanofluid. } C_{p,s} = \text{solid particle specific heat capacity. } \emptyset = \text{particle volumetric}$ $\text{concentration and } C_{p,bf} = \text{base fluid specific heat.}$		
Model II	$C_{p,nf} = \frac{\oint \cdot \rho_s \cdot C_{p,s} + (1 - \oint) \cdot \oint \cdot \rho_{bf} \cdot C_{p,bf}}{\rho_{nf}} \qquad (eq. 2)$ $\rho_s = \text{solid particles density. } \rho_{bf} = \text{base fluid density and } \rho_{nf} = \text{nanofluid density.}$		

Effective	$(\rho C_p)_{nf} = \left[\phi_p \cdot \rho_p \cdot C_{p-p} + (1 - \phi_p) \cdot \rho_f \cdot C_{p-f} \right] \qquad (eq.3)$				
volumetric	Ø = particle volumetric concentration and subscript <i>nf</i> indicate the nanofluid, <i>p</i> the nanoparticle, and <i>f</i> the base				
specific heat	fluid.				
Effective specific heat	$C_p{nf} = \frac{\left[\phi_p \cdot \rho_p \cdot C_{p-p} + \left(1 - \phi_p\right) \cdot \rho_f \cdot C_{p-f} \right]}{\phi_p \cdot \rho_p + \left(1 - \phi_p\right) \cdot \rho_f} \qquad (eq.4)$ ϕ = particle volumetric concentration and subscript <i>nf</i> indicate the nanofluid, <i>p</i> the nanoparticle, and <i>f</i> the base fluid.				
In-house calorimeter	$C_{p,nf} = \frac{\dot{Q} \cdot \Delta t - m_c \cdot C_{pc} \cdot \Delta T_c - m_{co} \cdot C_{p,co} \cdot \Delta T_{co} - m_{IN} \cdot C_{p,IN} \cdot \Delta T_{IN} - \dot{q}_L \cdot \Delta t}{m_{nf} \cdot \Delta T_{nf}} (eq.5)$ $\dot{Q} = \text{heat applied, } \Delta t = \text{interval time (s), } \Delta T = \text{temperature rise (K), } m = \text{mass (kg), } Cp = \text{specific heat capacity, } \dot{q}_L = \text{heat transfer to the environment}_{r}, \text{ and the Subscripts } C \text{ indicates the container, } CO \text{ the heating coil, and } IN \text{ the insulatorion.}$				
Diffusivity	$C_{p-nf} = \frac{k_{nf}}{\alpha_{nf} \cdot \rho_{nf}} \qquad (eq. 6)$ $\rho_{nf} = \emptyset \cdot \rho_p + (1 - \emptyset_p) \cdot \rho_f \qquad (eq. 7)$ $k_{nf} = \text{ thermal conductivity, } \alpha_{nf} = \text{ thermal diffusivity, } \rho_{nf} = \text{ nanofluid density, } \emptyset = \text{ particle volumetric concentration, } \rho_p = \text{ particles density and } \rho_f = \text{ fluid density.}$				
MD <mark>s</mark> imulations	$C_p = \left(\frac{\partial E}{\partial T}\right)_p = \frac{\langle \delta E^2 \rangle_{NPT}}{k_B \cdot T^2} \qquad (eq.8)$ k_B = Boltzmann constant, T= temperature, E= total internal energy and C_p = specific heat capacity.				
Debye theory (Phonon heat capacity)	$C_{v} = \frac{1}{\rho V K_{B} T^{2}} \sum_{\vec{q}, p} \frac{(\hbar \omega_{\vec{q}, p})^{2} e^{\frac{\hbar \omega_{\vec{q}, p}}{K_{B} T}}}{(e^{\frac{\hbar \omega_{\vec{q}, p}}{K_{B} T} - 1})^{2}} \qquad (eq. 9)$ $C_{v} = \text{ specific heat capacity at constant volume, } k_{B} = \text{ Boltzmann constant, } T = \text{ temperature, } \hbar \omega_{\vec{q}, p} = \text{ phonon energy, } \rho = \text{density and } V = \text{volume.}$				
ASTM E1269	$C_p = C_{p,sapphire} \cdot \frac{\Delta q_s \cdot m_{sapphire}}{\Delta q_{sapphire} \cdot m_s} = C_{p,sapphire} \cdot \frac{(q_s - q_o) \cdot m_{sapphire}}{(q_{sapphire} - q_o) \cdot m_s} \qquad (eq. 10)$ $C_p = \text{specific heat capacity, } q = \text{heat flow, } m = \text{mass, and subscript } s \text{ indicates samples (pure mixtures or nanomaterials) and } st \text{ the standard materials.}}$				
ASTM E2716	$\begin{split} C_{p,rev} &= \frac{A_{mhf}}{W_s \cdot A_{mrh}} \qquad (eq.11) \\ T &= T_o + \beta \cdot T + A_{mhr} \cdot \sin(wt) \qquad (eq.12) \\ C_{p,non} &= \frac{\langle P \rangle}{W_s \cdot \beta} - C_{p,rev} \qquad (eq.13) \end{split}$ $C_{p,rev} = \text{reversing component of the apparent specific heat, } A_{mhf} = \text{amplitude of the first harmonic of the heat} \\ \text{flow, } A_{mrh} = \text{amplitude of the applied heating rate, } W_s = \text{mass sample, } \beta = \text{heating rate, } A_{mhr} = \text{amplitude of the} \\ \text{perturbation, } w = \text{frequency of the perturbation, } T = \text{sample temperature, } t = \text{time, } \langle P \rangle = \text{average heat flow, and} \\ C_{p,non} = \text{non-reversing specific heat.} \end{split}$				
Areas Method	$C_{p,m} = \frac{C_{p,s} \cdot A_m}{A_s} \qquad (eq. 14)$ $A_s = \frac{\dot{Q}_s}{m_s} = C_{p,s} \cdot \beta \qquad (eq. 15)$ $A_s = \frac{\dot{Q}_m}{m_m} = C_{p,m} \cdot \beta \qquad (eq. 16)$ $A_s = \text{Integrated peak area for the sapphire curve, } A_m = \text{integrated peak area for the material curve, } C_{p,s} = \text{sapphire sample mass, } m_m = \text{material sample mass, } \dot{Q}_s = \text{the sapphire heat flux signal, } \dot{Q}_m = \text{material heat flux signal, and } \beta = \text{heating rate.}$				

Direct	$\dot{Q} = C_p(T) \cdot \beta \qquad (eq. 17)$		
method	\dot{Q} = Heat flux. $C_p(T)$ = specific heat capacity in function of the temperature. β = heating rate.		
T- <mark>h</mark> istory	$T'_{S} = \frac{dT_{S}}{dt} = \frac{h_{air} \cdot A_{surface} \cdot (T_{air} - T_{S})}{m_{S} \cdot C_{p,s}} \qquad (eq. 18)$		
	T_{air} =temperature of furnace air, T_s =sample temperature, m_s =sample mass, $C_{p,s}$ = specific heat capacity sample		

The first eight equations of in Table 2 have been included in the equation methods groupfirst, the equations formulated for the NFs; Model I, eq. 1, was developed by Pak and Cho *et al.* [42] and is based on the liquid-particles mixture theory and the dependency of the particle concentration. However, this model is approximately valid for dilute suspensions when small density differences exist between base fluid and nanoparticles. Xuan and Roetzel *et al.*, [43] modified this correlation by assuming thermal equilibrium between the nanoscale solid particles and the liquid phase, including the density (Model II), eq.2. Nevertheless, the literature showed a large gap between the expected values and the values experimentally obtained. Therefore, the values obtained through these models tend to present exhibit a high standard deviation. Derived from these two models, eq. 3 and eq. 4 and from the definition of the nanofluids density [44] were defined the effective volumetric specific heat and the effective specific heat of NFs, respectively. On the other hand, eq. 3 describes the equation mainly used in the in-house calorimeters, as a function of the temperature, time, mass, and the applied heat flux [45]. Another way to determine de the *Cp* was related to its determination through thermal conductivity and thermal diffusivity values, eqs. 4-5, eq. 5 [42,46,47].

Alternatively, thermophysical properties can be determined theoretically from MD simulations (*i.e.*, Molecular dynamics simulations, MD). These numerical techniques that use internal energy terms to predict the Cp, such as the eq. 8 [48,49]. Finally, the Cp at constant volume can be determined through theoretical models as eq. 9 describes (Debye theory) [50]. In the same way, eq.10-17 describe all the methods derived from a calorimetric instrument (*i.e.*, Differential scanning calorimetry technique): direct method, ASTM E2716, ASTM E1269, MDSC, and the areas method. The ASTM E1269 is a standard three-step procedure: the baseline heat flux (q_o) was obtained with two empty-pans as the first step analysis. In the second step, the heat flux of a reference sample with a known Cp (*i.e.*, sapphire sample) was measured ($q_{ref/sapphire}$). Finally, in the third step, the sample heat was determined (q_{sample}), and by eq. 10, the Cp is obtained [39]. On the other hand, can be used other two methods to determine Cp with DSC. First, the area method that consists of two consecutive isothermal segments without a heating stage and a temperature difference of 1 °C between the isotherms. The signal peak due to the isotherm's temperature differences, see eq. 15, is compared with the area of the known

reference material, eq. 16 (*i.e.*, sapphire), to obtain the *Cp* by the through eq. 14 [39]. Otherwise, the conventional method involves the evaluation of the enthalpy curve, measured within a certain temperature range, being the *Cp* the area under the enthalpy curve represents the *Cp*. This is based on the relation between the heat flow rate, the *Cp* at a constant pressure of the sample inside the sample cell, and the scanning rate β , eq. 17 [39]. Finally, the MDSC technique, a variation of the DSC, (which is a DSC variation technique) applies a sinusoidal heat signal (eq. 12), separating the *Cp* into reversing, eq. 11, and non-reversing component, eq.13. The reversing component of the heat flow is obtained from the amplitude of the first harmonic of the heat flow A_{mhf}, through a Fourier transform of the data. Then, the reversing component of the apparent *Cp*, is obtained by dividing the heat flow A_{mhf} by the amplitude of the applied heat rate, A_{mth5} [51,52]. This technique is useful to separate the kinetic contribution (time-dependent factors). Lastly, **a** the **T**-history technique determines the *Cp* according to the eq. 18 [53,54]. This method was first developed by Yinping *et al.*, in 1999 [55] and enables to obtain properties like melting point, fusion heat, degree of sub-cooling, thermal conductivity, and specific heat of several samples simultaneously.

Sampling: The high inconsistency about the *Cp* values in the reviewed and reported literature suggest that one of the possible issues that caused the high discrepancy in results was the difficult task of obtaining a good representative sample due to the low concentration of nanoparticles' concentration. Typically, due to the small amount of sample weight that uses standard calorimetric techniques. For this reason, an analysis of the sampling and statistics followed by researchers was performed. Two main aspects were considered, the number of samples and the number of measurements repetitions per each sample. **Figure 4** summarizes the general trends of the sampling performed in the analyzed literature.



Figure 4. A pie chart of the Sampling done in the revised literature to determine specific nanofluids heat capacity values.

Many Cp values were obtained from the analysis of just one sample and by means of more than one measurement repetition (36 %). The choice of this procedure minimizes the effect of non-stability and agglomeration of nanoparticles inside the liquid medium. However, it is not a proper an appropriate measurement procedure from the point of view of sampling and instrumental error because the samples are not there is not a representative sample.

The opposite Another situation consists in analyzing is the cases where more than one sample was analyzed but only performing one repetition for each sample was run. This experimental procedure was used followed for to-obtaining 15.8 % of the total data evaluated.

In addition, the most appropriate procedure is the case where more than one sample and more than one repetition is performed and represents 14.6 % of the analyzed cases. This last sampling procedure shwould minimize the errors associated to the measurement, the nanoparticle concentration, and the stability of the sample, and measurement errors. Hence, this procedure is expected to offer high-quality values regarding measurement uncertainty.

Finally, the least desirable the most unfavorable procedure from a statistical point of view is obtained when corresponds to the analysis of only one sample and with just one measurement repetition are performed. This undesirable condition procedure represents only a 4.4 % of the analyzed data.

Nevertheless, sampling procedure of NFs needs to be carried out in order to reduce measurement errors. Thereby, the first step is to develop a methodology to obtain a good representative sample (*i.e.*, quartering) and determine the minimum number of independent samples to minimize $\frac{1}{4}$ the measurement error due to the inhomogeneity of NFs samples.

As a final fact, a relevant finding is that the 29 % of the data presented in the literature does not report how the Cp result was obtained. The lack of that information about experimental procedure is the base to highlight even more that there is significant uncertainty in the reported Cp values.

Likewise, it is well known that the particle size of the samples influences the thermophysical properties (*i.e.*, melting temperature, specific heat capacity, ...). For this reason, it is crucial first to melt the sample to remove the granulometry effects. In addition, it ensures a better contact of the sample with the bottom of the crucible and removes the air within the sample. As a result, values obtained with a single measurement have low-quality from a statistical point of view. To minimize this effect, it is necessary to perform several measurement repetitions and discard the first analysis (first run) to obtain a more reliable value. From the analyzed data literature, only 21 % of the data considered this factor and whereas the other 79 % of the data do not specify this

information. These analyses reveal another source of error in the reported Cp values, which influences the lack of homogeneity of the results.

3.2. Principal component analysis (PCA) and Response Surface Methodology (RSM)

Finding correlations between the main parameters involved in the Cp enhancement is crucial to understand their behavior and giving a step forward to synthesize and optimize NFs. In this study, the three main parameters were selected have been: the temperature (°C), the NPs concentration (wt.%), and the NPs size (nm). For this purpose, response surface methodology (RSM) was employed to predict the Cp variation (ΔCp , %) and Principal Component Analysis (PCA) was applied to identify the main variables and quantify its their influence on the Cp value.

The analysis of the nanofluids dataset by PCA has shown the values of variances' values of for the three new PCs, representing the full dataset after the transformation (see **Table 3**). One criterion for selecting the number of PCs needed is based on the cumulative percentage of the total variance, which is often considered satisfactory when it lies within the range of 70 - 80 % [56]. All the analyzed data in **Table 2** for PC1 and PC2 presents a cumulative percentage of variance > 70 %, which implies that these PCs can be used to explain the dataset, thus reducing the original dimensionality by one variable. Moreover, PCA transformation has shown the correlation between PCs and the original variables (1 means perfect correlation, -1 inverse correlation, and 0 no correlation). While the initial three parameters (concentration, size, and temperature) show approximately the same values for PC1, in PC2, concentration and size are the predominant features. However, the temperature and size have a higher contribution to PC1 and PC2, respectively.

	PC1	PC2	PC3
Concentration	0.56951	0.627945	0.530418
Size	0.542798	0.771884	0.331007
Temperature	0.617275	0.099399	0.780443
Eigenvalues	1.180865	0.934924	0.892385
Percentage of variance (%)	39.26%	31.08%	29.67%
Cumulative (%)	39.26%	70.33%	100.00%

Table 3. Contribution of the original variables to the PCs, Eigenvalues, percentage of variance, and its accumulative values from the PCA analysis.

On the other hand, RSM is a mathematical and statistical technique useful for modeling and problem analysis, where some variables influence the response. The objective is to optimize this response. In this case, the Cp is a function of $C_p \cong f(T, \emptyset, S) + \epsilon$, where ϵ represents the noise error observed in the response, T the temperature, \emptyset the concentration, and S the NPs size. The expected response can be expressed by $E(C_p) = f(T, \emptyset, S) = \beta$ and the response surface can be represented by: $\beta = f(T, \emptyset, S)$ [57]. To obtain the response surface, it is first necessary to find an appropriate approximation of the functional relationship between Cp and the independent variables. For this purpose, analysis of variance (ANOVA) was performed. ANOVA provides a statistical test of whether two or more population means are equal and therefore generalizes the *t*test beyond two means and is based on the analysis of the p-values to predict an optimal response. Therefore, ANOVA analysis should provide a mathematical function for NFs Cp values. The pvalue represents the smallest level of significance that would lead to the rejection of the null hypothesis, in this case < 0.05 (with a confidence level above 95 %), indicating that the controllable factor does not affect the response [58].

For this propose, several scenarios with different data aggrupation were analyzed:

- (1) all the data together,
- (2) experimental values,
- (3) values obtained under E1269 standard methodology,
- (4) values obtained with statistics (sampling and/or repetitions),
- (5) values obtained with sampling (> 1 sample),
- (6) values obtained with measurement repetitions (> 1 repetition).

In the scenarios (1), (2), (3), (4), and (6), no correlations were found. Therefore, no mathematical model was fitted to the data. Nevertheless, a correlation was found only in the scenario (5) where the obtained Cp values is are an average of more than one independent sample (sampling procedure).

Figure 5 shows the three-dimensional **3D** predicted response surface (right) and the corresponding contour plot (left) from the ANOVA analysis for the scenario 5. The best model to fit the data was is a quadratic model with p-values lower than <0.0001 p-values. Moreover, the Model F-value of 6.72 (este valor de qué propiedad es? Especificar!) indicates that the model is significant, and the lack of fit F-value of 0.95 implies that the lack of fit is not significant relative to the pure error. There is only a 0.01 % change that an F-value this large could occur due to noise. Despite this adjustment, the standard deviation of the model is ± 7 . These deviations agree with Ref. [59]previous work where nanofluids' the thermophysical properties of NFs were statistically analyzed by experimental sampling procedures [59]. The factors terms were *Cp* (response) = 0,3076 -0,0579 · A (temperature) +0,4512 · B (Size) – 1,3651 · C (Concentration) –

 $0.0004 \cdot AB - 0.0073 \cdot AC + 0.023 \cdot BC + 0.00017 \cdot A^2 - 0.0021 \cdot B^2 - 0.2922 \cdot C^2$. Therefore, this model allows identifying NFs trends. In Figure 5, the predicted Cp response as a function of the NPs size and concentration was depicted with the temperature evolution: a-b) at 50°C, c-d) at 200 °C, e-f) at 350 °C and g-h) at 500 °C. In Figure 5 a-b) at 50 °C, it can be identified four main regions delimited by the contour lines; from red (+30 % Cp variation) to dark blue (- 30% Cp variation). Contour lines points to a direction where the Cp is maximized. A curved domain with a maximum Cp improvement of about 20 % was identified between the NPs concentration range 2 to 9 wt. %, and between 65-100 NPs nominal diameter (nm). In contrast, NP concentration increment leads to a Cp variation and NP size decreases, reaching negative values up to -20 %. When the temperature increase (100 - 250 °C), see Figure 5 c-d) and e-f), the contour lines of 10, 0, and -10 shifted towards high NPs size values; disappearing the domain of values higher than 20 % and appearing new domains of negative values (-20 % and -30 %). On the contrary, with the temperature increase, this trend was changed. At higher temperatures (500 °C), see Figure 5 g-h) a domain of maximum Cp variation is shown up to 25 % for NFs with low NPs concentrations and sizes between 0,001 - 1 wt.% and 30 - 90 nm, respectively. Consequently, a strong temperature dependence has been found in the behavior of NFs. This fact is crucial to select the appropriate parameters (i.e., nanoparticles size and concentration) according to their working temperature. These tendencies are consistent with the values obtained from the PCA analysis.



Figure 5. MEJORAR LA CALIDAD DE LA FIGURA! A three-dimensional **3D** response surface (right) and contour plot (left) showing the expected specific heat capacity (y) as a function of the size (x₁) and concentration (x₂), at a fixed temperature: a)-b) at 50 °C, c)-d) at 200 °C, e)-f) at 350 °C and g)-h) at 500 °C.

3.3. Nanofluids selector database representation

The relation between the Cp variation regarding the initial value, the size, and concentration of NPs, help to understand the connection between the Cp change and the NPs addition.

Therefore, creating a database is a helpful way to identify the main trends in NF's behavior. Furthermore, the database makes it possible to define the properties and tolerances of NFs for their in-service engineering modelling/simulation. (i.e., as HTF/TES material in CSP facilities). As seen in the previous sections, the concentration and size of NPs are two of the main significant parameters of NFs thermo-physical properties. Accordingly, a quotient between concentration (wt.%) / size (nm) was defined as an index for NFs: $I^{c/s}$, for better interpretation of the data. In this way, the database makes possible to identify the best parameters for synthetizing NFs with a specific *Cp*. On the other hand, an evidence of the high dispersion of results is elucidated with this database. This fact is observed in the database figures: where the higher the bubble size, the higher the dispersion of the data value.

Figure 6 shows the nanofluids ΔCp variations as a function of I^{c/s}: **a**) by taking the type of NPs into account and **b**) by base fluids. It can be observed that, generally, metal oxide NPs and carbon base NPs give the most significant variation, particularly, with enhancements up to 150 % when are combined with a molten salt base fluid. These enhancements occur when the I^{c/s} index is between the range of 0.01 - 0.1 wt.% nm⁻¹, (it is important to emphasize that the graphs presented only include spherical shaped nanoparticles when the size is analyzed; nanowires are also included in the database with length x nominal diameter). On the contrary, NFs based on metallic NPs show the worst behavior with variations up to -50 %. Conversely, hydrocarbons, ionic liquids, mixtures, and oils showed a slight positive variation in many cases. However, a severe decrease prevails in many cases with different types of NPs. It is important to highlight that in general, the water base NFs showed a decrease of *Cp*. Therefore, incorporating NPs in water systems does not improve its properties. In the case of substituted hydrocarbons or organic acids are registered enhancements up to 50 %.

Furthermore, in general, the literature's highest ΔCp are obtained with NPs concentrations between 0.1 wt.% and up to 2 wt.%. However, some ΔCp enhancements were reported with at higher NPs concentrations, an example are as the values reported obtained by Y. Huang *et al.* [60].





On the other hand, the size of NPs gives a higher ΔCp enhancement with smaller nominal diameters. For example, from 2 nm to 20 nm of NPs nominal diameter, a significant increase has been observed; whereas, with larger NPs sizes, the increments are less relevant or even negative.

To choose the most suitable NF for a specific application, service temperature is needed to identify the best candidate. **Figure 7** presents the Cp values as a function of temperature. In Figure 7-a) are shown the Cp of the evaluated NFs versus the measurement temperature. As **it** can be noted, most NFs reported data is in the temperature range between 25 °C and 250 °C. However, molten salts can reach up to 600 °C. Water, oils, and some mixtures **exhibit present** the highest Cp of the treated NFs, but molten salts are the most employed fluid despite their relatively low Cp when working at high temperatures.

For this reason, knowing the Cp of this kind of fluids is of great interest. Also, it is essential to consider the Cp and the variation that can be achieved by adding NPs. Thus, Figures **7-b**) and **7-c**) represent the ΔCp as a function of temperature, classified as base fluid type, see **7-b**), and NPs type, see **7-c**).



Figure 7. a) Nanofluids specific heat capacity as a function of the temperature: for a) specific heat capacity (J/kg·°C) of nanofluids families classified as classified by the type of base fluid type, and b)

specific heat capacity variation (%) as a function of temperature classified as the according to the a) base fluid types of family and to the c) Specific heat capacity variation (%) classified as a nanoparticle's family type.

The largest ΔCp are obtained with molten salts at approximately 300 °C. On the other hand, the second group with higher ΔCp enhancements were thermal oils, reaching improvements up to 80 % with presence of metal oxide NPs. Despite this, they do not exceed the maximum Cpvalues of the water in the temperature range between ambient temperature and 90 °C.

Finally, **Figure 8** shows a particular case of molten salts and the type of NPs used, from for sizes in the range 1 nm to 200 nm of the nanoparticle size range. It can be observed that carbonbased and metal oxides nanoparticles give **a** the largest Cp enhancement, especially at concentrations of NPs between the range of 0.1 to 2 wt.% of NPs. Higher variation can be noticed in a particular case of NaNO₃-KNO₃ molten salt with MgO as NPs and a concentration of 5, 10, and 15 wt.%, reaching a maximum of Cp variation of 168 % [60]–[62]. Otherwise, Li₂CO₃-K₂CO₃ molten salt with SiO₂ NPs also shows a remarkable Cp enhancement for concentrations in **a** the range concentration from 0.1 wt.% to 2 wt.%, achieving **a** variations in Cp between 2 % and 124 % in the experimental results [30], [63]–[69].



Figure 8. Specific heat capacity variation (%) as a function of concentration of NPs (wt.%) of for molten salts-based nanofluids is classified by NP as the nanoparticle's family.

4. Conclusions

This paper collected and analyzed 899 values of nanofluids Cp published in the field of thermal energy storage in order to understand their behavior and the effect of incorporating NPs in it. Thanks to the instrumentation analysis, measurement techniques, calculation methods, and the sampling used to determine the Cp, the identification of possible sources of errors that cause the large discrepancy in the results presented in the literature have been done. The first identified

source of error was the few methodology specifications in the *Cp* determination. As a result, in a significant part of the reported results there is a gap for a proper methodological procedure for measuring nanofluids thermal properties. The second main source of error was the lack of adequate sampling and statistical analysis. The main conclusions obtained by PCA, RSM methodology, and the database were summarized as the following:

- Temperature, concentration, and NPs size show a similar influence in the Cp value.
- A mathematical correlation was determined only with a group of values statistically obtained through a sampling procedure.
- It has been verified that metal oxides NPs are the best candidates for *Cp* enhancements and metal NPs the worst ones. Being the MgO NPs, those who provided increments above 100 %.
- Molten salts were the base fluid with larger *Cp* increments and water with the lowest increment values.
- Higher enhancements were obtained with concentrations from 0.1 wt.% to 2 wt.% and nominal diameter from 2 nm to 20 nm.

The database allows identifying and selecting nanofluids for specific applications.

5. Recommendations

To sum up, some recommendations for future work are proposed:

(1) To do a statistical sampling procedure and provide a detailed description of this methodology procedure to present a *Cp* value.

(2) To provide not only the Cp enhancement but also the nanofluid Cp value and the base fluid Cp value separately.

(3) To provide information about the Cp measurement temperature since it has important influence in the thermophysical properties determination.

All these recommendations facilitate the comparison, and even more important, the reproducibility of the Cp values obtained as results and identify NFs behavior patterns and their relationship with the type and concentration of NPs.

Conflict of interest

The authors declare no conflicts of interest.

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