**Understanding the Heat Capacity Enhancement in Ionic Liquid-Based Nanofluids (Ionanofluids)**

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Nanofluids, systems consisting of a base fluid and dispersed nanoparticles, have attained a large scientific interest recently, mainly due to unusual enhancement in their thermal properties like the thermal conductivity and heat capacity. Whilst both molecular solvents and ionic liquids have been examined, ionic liquids are known for the specific properties caused by the interactions occurring in these systems. Physical properties of very mixtures containing an ionic liquid and nanoparticles still remain not fully investigated and analyzed in the literature, to date. One of the possible issues which limits the fundamental understanding of such systems is related to the determination of the mechanism explaining the unusual enhancement of the thermal properties, in particular the heat capacity. The aim of this work is to discuss the mechanism of heat capacity enhancement of several ionic liquid-based nanofluids doped with carbon nanotubes, boron nitride and graphite, based on the experimental data of density and heat capacity determined as the function of the nanofluid composition and temperature from (298.15 to 363.15) K at 0.1 MPa.

**Keywords:** Ionic Liquids, IoNanofluids, Isobaric Heat Capacity, Carbon Nanotubes, Boron Nitride, Graphite, Nanoparticles

1. **Introduction**

Nanofluids are systems consisting of base fluids (*i.e.* water, ethylene glycol or oils) and nanoparticles (*i.e.* carbon nanotubes, oxides or metals) dispersed, therein. The effort undertaken in preparing such materials, is usually driven by the fact that these solutions exhibit specific properties, which are unusual when compared to other individual components, for example the loading of nanoparticles causes thermal conductivity to increase, as well as the specific heat capacity.[1] This group of systems is particularly of interest due to a large range of possible combinations, driven by the selection of the types of base fluids,[2–4] through a wide variety of nanoparticles,[5–8] to be dispersed and ending on more sophisticated eutectic mixtures.[9–11] In particular, the thermal conductivity and the heat capacity of nanofluids have attained an extraordinary scientific attention because of the unusual enhancement of these properties when a given nanoparticle is dispersed in a specific base fluid. While thermal conductivity was widely investigated in the literature and many theories were proposed,[12–17] heat capacity remained abandoned for a long time, to date. The main point was focused about unusual enhancement caused by the addition of nanoparticles which have usually lower heat capacity than liquids.[18] Following the first approach to describe this behavior based on mixing theory for ideal gas mixtures, the heat capacity should be a decreasing function of nanoparticles loading, which was proved to be wrong.[19] A further derivation leads to the assumption of thermal equilibrium between the particles and the surrounding fluid by including the density of liquid and nanoparticles. Still the reproducibility was very limited.[19] A meaningful theory was recommended by Shin and Banerjee (2011).[10] These authors discussed the specific surface properties of nano-sized materials, the properties of the liquid around nanoparticles and, finally, the layering, caused by liquid molecule adsorption, onto the nanoparticles. The most recent work, reported by Hentschke (2016), introduced the full mathematical fundamentals of the enhancement.[20] This includes the interfacial layering, and some type of overlapping mesolayers.

Ionic liquids are a group of compounds composed solely of ions with melting point below 373.15 K.[21] This is caused by specific interactions occurring between cations and anions, and their cohesive energy is driven by a combination of Coulombic interactions, van der Waals interactions and hydrogen bonding in solution.[22] Their reminiscent properties include, generally, low flammability, wide liquid range, low vapor pressure, high thermal stability and ionic conductivity.[23] All these advantages have led many researchers to consider ionic liquids in many applications, for example*,* catalysis, extraction or as lubricants.[24–27] Ionic liquids have also been used to prepare nanofluids which result in ionanofluids.[28] It has been reported that these particular mixtures can be used as potential materials for many applications (particularly those using heat transfer fluids) driven by the specific properties of ionic liquids.[29] The first report of ionanofluids was described by Nieto de Castro *et al.* (2010) in which the thermal conductivity enhancement was investigated.[30] Nevertheless, the mechanism of this heat capacity enhancement was not discussed in case of ionanofluids.

The aim of this work is the experimental determination of density and specific heat capacity of ionanofluids based on 1-butyl-3-methylimidazolium dicyanamide, 1-butyl-3-methylimidazolium bis(trifluoromethylsulfonyl)imide, 1-butyl-1-methylpyrrolidinium bis(trifluoromethylsulfonyl)imide, 1-hexyl-3-methylimidazolium hexafluorophosphate, and 1-ethyl-3-methylimidazolium ethylsulfate, mixed with different loadings of nanoparticles, such as carbon nanotubes, boron nitride and graphite, up to a nanoparticle weight fraction close to 0.03 (*i.e.* 3% wt%). A further significant analysis of experimental work in terms of possible mechanisms explaining the unusual enhancement of their thermal properties by following those already proposed for simple nanofluids has been performed.

1. **Experimental**

**Materials**

1-Butyl-3-methylimidazolium dicyanamide (CAS: 448245-52-1), [C4C1Im][Dca], and 1-butyl-3-methylimidazolium bis(trifluoromethylsulfonyl)imide (CAS: 174899-83-3), [C4C1Im][NTf2], were bought from Merck (purity ≥98%, ≤0.01% halides, catalogue number: 4900150500) and Sigma-Aldrich (purity ≥98%, catalogue number: 711713), respectively. 1-Butyl-1-methylpyrrolidinium bis(trifluoromethylsulfonyl)imide (CAS: 223437-11-4), [C4C1Pyrr][NTf2], 1-hexyl-3-methylimidazolium hexafluorophosphate (CAS: 304680-35-1), [C6C1Im][PF6], and 1-ethyl-3-methylimidazolium ethylsulfate (CAS: 342573-75-5), [C2C1Im][C2SO4], were synthesized in-house by following the procedures available in the literature.[31–33] All ILs were washed 3-5 times with ultrapure water (18 MΩ cm), and extracted with dichloromethane. Thereafter, the ILs were dried under high vacuum (10-4 Pa) and elevated temperature (333.15 K) for at least 72 h. The final purity of each IL was then checked with 1H and 13C NMR (**Figure S1, SI**), and found to be ≥ 98 % in each case. The water content was assessed by Karl-Fisher titration (Metrohm 899 Coulometer with 803 Ti Stand and Hydranal Coulomat AG), before and after each measurement. All values were below 2 ∙ 10-4 in water mass fraction unit. Moreover, the water content values after each measurement were not deviated from those measured before, showing a non-contamination of water during each measurement. The specification of all the ILs used in this work are reported in **Table 1**, while the structures of investigated ionic liquids are presented in **Figure 1**.

**Table 1** Chemicals description.*a*

|  |  |  |  |  |  |
| --- | --- | --- | --- | --- | --- |
| Chemical name  [CAS] | Supplier | Mass fraction purity | Purification method | Analysis method | Halide content ppm*b* |
| 1-Butyl-3-methylimidazolium dicyanamide  [448245-52-1] | Merck | ≥ 0.98 | Washing-extraction  Vacuum | 1H and 13C NMR  Karl Fisher titration | < 5 |
| 1-Butyl-3-methylimidazolium bis(trifluoromethylsulfonyl)imide  [174899-83-3] | Merck | ≥ 0.98 | Washing-extraction  Vacuum | 1H and 13C NMR  Karl Fisher titration | < 5 |
| 1-Butyl-1-methylpyrrolidinium bis(trifluoromethylsulfonyl)imide  [223437-11-4] | In house | ≥ 0.98 | Washing-extraction  Vacuum | 1H and 13C NMR  Karl Fisher titration | < 5 |
| 1-Hexyl-3-methylimidazolium hexafluorophosphate  [304680-35-1] | In house | ≥ 0.98 | Washing-extraction  Vacuum | 1H and 13C NMR  Karl Fisher titration | < 5 |
| 1-Ethyl-3-methylimidazolium ethylsulfate  [342573-75-5] | In house | ≥ 0.98 | Washing-extraction  Vacuum | 1H and 13C NMR  Karl Fisher titration | < 5 |
| Multi-walled carbon nanotubes  [308068-56-6] | Bayer Material Science | ≥ 0.99 | - | Ashing,*c* TEM, *c* SEM,*d* EN ISO 60,*c* XRD, laser diffraction technique | - |
| Boron nitride  [10043-11-5] | US Research Nanomaterials, Inc. | ≥ 0.998 | - | SEM,*d* XRD*d* | - |
| Graphite  [7782-42-5] | US Research Nanomaterials, Inc. | ≥ 0.999 | - | SEM,*d* XRD*d* | - |

*a* 1H and 13C NMR refer to proton, carbon and nuclear magnetic resonance spectroscopy, respectively; *b* determined by AgNO3 titration; *c* as reported by the supplier; *d* as reported by supplier and confirmed by measurement.



**Figure 1** Structures of investigated ionic liquids.

The carbon nanotubes used were Multiwalled Carbon Nanotubes Baytubes C150 HP from Bayer Material Science (MWCNT, CAS: 308068-56-6, purity ≥ 99 %, 3-15 number of walls, 13-16 nm outer mean diameter, 5-20 nm outer diameter distribution, 4 nm inner mean diameter, 2-6 nm inner diameter distribution, 1-10 mm length, 140-230 kg m-3 bulk density; according to the specifications provided by the supplier). Hexagonal boron nitride and graphite nanoparticles were bought from US Research Nanomaterials, Inc. (BN, CAS: 10043-11-5, purity ≥ 99.8 %, 70-80 nm size, 2290 kg m-3 theoretical true density, < 0.03 % Fe2O3, < 0.002 % CaO, < 0.04 % MgO, < 0.1 % B2O3 for boron nitride, and G, CAS: 7782-42-5, ≥ 99.9 % purity, 400-1200 nm size, < 0.1 % impurities, ~0.2 % water for graphite, respectively). Boron nitride and graphite were pre-processed by ball milling (Retsch Mixer Mill MM 400, frequency 20 Hz, 3 times for 1800 s). The purity of nanomaterials was repeated with X-ray diffractometry (X’Pert Powder PANalytical, 15-90o scan range, 0.0084o scan step size, **Figure S2**, **SI3**). The particle size was checked with scanning electron microscope (650 FEI Quanta FEG, 15-20, < 90, < 100 nm for carbon nanotubes, boron nitride and graphite, respectively, **Figure S3**, **SI3**), and with light scattering (Mastersizer Malvern, dispersion with water, stabilized with sodium laureth sulfate as anionic surfactant, refractive index of 1.330, 1.650 and 2.420 for water, boron nitride and both carbon-based nanomaterials, respectively; 26.60 ± 0.15 nm, 154.5 ± 1.7 nm, and 159.1 ± 3.1 nm for carbon nanotubes, boron nitride and graphite, respectively, **Figure S4**, **SI3**).

Based on the literature review, the most reliable thermophysical properties were presented in the works published by França *et al.* (**2014**),[34] França *et al.* (**2013**),[35] and Nieto de Castro *et al.* (**2010**).[30] Thus, systems reported in these works were selected to compare with the data measured in this study. Nevertheless, only ionanofluids based on carbon nanotubes doped ionic liquids are reported, to date. In fact, to the best of our knowledge, the physical properties of ionanofluids containing boron nitride or graphite dispersed in ILs have not been reported, to date. Manufacture of ionanofluids is a very delicate process, and it requires a careful treatment and the same protocols used previously to produce reproducible nanofluids have been applied, herein. This was also applied in this work: after the nanoparticles were added to ILs, 30 min of magnetic stirring was applied prior to the 1h of sonication required to the formation of nanofluids instead of so-called bucky gels, and the nanoparticles agglomeration breakage. The summary of all investigated ionanofluids are reported in **Table 2**.

**Table 2** Summary of investigated ionanofluids, including the mass fraction *wm,NP*.

|  |  |  |  |
| --- | --- | --- | --- |
|  | 100 × *wm,NP* (%) | | |
|  | Carbon nanotubes | Boron nitride | Graphite |
| [C4C1Im][Dca] | 0.5, 1.0, 3.0 | 0.5, 1.0, 3.0 | 0.5, 1.0, 3.0 |
| [C4C1Im][NTf2] | 0.5, 1.0, 3.0 | - | - |
| [C2C1Im][C2SO4] | 0.5, 1.0, 3.0 | - | - |
| [C4C1Pyrr][NTf2] | 1.0, 3.0 | 1.0, 3.0 | 1.0, 3.0 |
| [C6C1Im][PF6] | 0.5, 1.0, 3.0 | 0.5, 1.0, 3.0 | 0.5, 1.0, 3.0 |
|  |  |  |  |

**Methods**

Density, *ρ*, was measured as a function of the temperature, *T*, using an Anton Paar DMA 4500M densitometer (calibration on ultrapure degassed water and dry air, relative standard uncertainty of density, *ur*(*ρ*) = 0.1 %, relative standard uncertainty of temperature, *ur*(*T*) = 0.01 K, viscosity-induced errors reduced). This equipment works on the principle of oscillating U-tube where approximately 1.5 cm3 sample volume is used within 3 independent repeats of measurement. The temperature range of each measurement was set from (298.15 to 363.15) K.

To correlate the density with temperature, the following linear equation was then used:

 (1)

where *ai* is the regression parameter determined by the least-square method, along with the standard uncertainty, δ*ai*. The coefficient of determination, *R2*, was used to describe the fit. The nanoparticles density was calculated using the following empirical formulae:[36]

 (2)

where *w* is mass fraction, *IL* and *NP* refer to ionic liquids and nanoparticles, respectively. The excess molar volume, *VE*, was also calculated using the following formulae:

 (3)

where *M* and *x* are the molar mass and the mole fraction of each component in selected solutions.

Specific heat capacity, *cp*, was measured as a function of the temperature, *T*, by using differential scanning calorimeter equipment, Q100 TA instruments (modulated differential scanning calorimetry technique, calibration on sapphire with further checking on 1-hexyl-3-methylimidazolium bis(trifluoromethylsulfonyl)imide as a NIST standard.[37] Relative standard uncertainty of measurement *ur*(*cp*) = 3 %, while the temperature relative standard uncertainty, *ur*(*T*) = 0.01 K, nitrogen gas flow rate of 50 cm3 min-1, heating rate d*T*/d*t* = 2 K min-1, amplitude ± 0.5 K, modulation period 60 s, 3 independent repeats of the measurement with 5% repeatability). The specific heat capacity was described as a function of the temperature by the second-order equation:

 (4)

To represent the specific heat capacity as a function of the nanoparticles loading, the mass fraction, *wm,NP* was recalculated to volume fraction, *ϕNP* with the following equation, as it reflects in proper concentration of multiple phase systems description:

 (5)

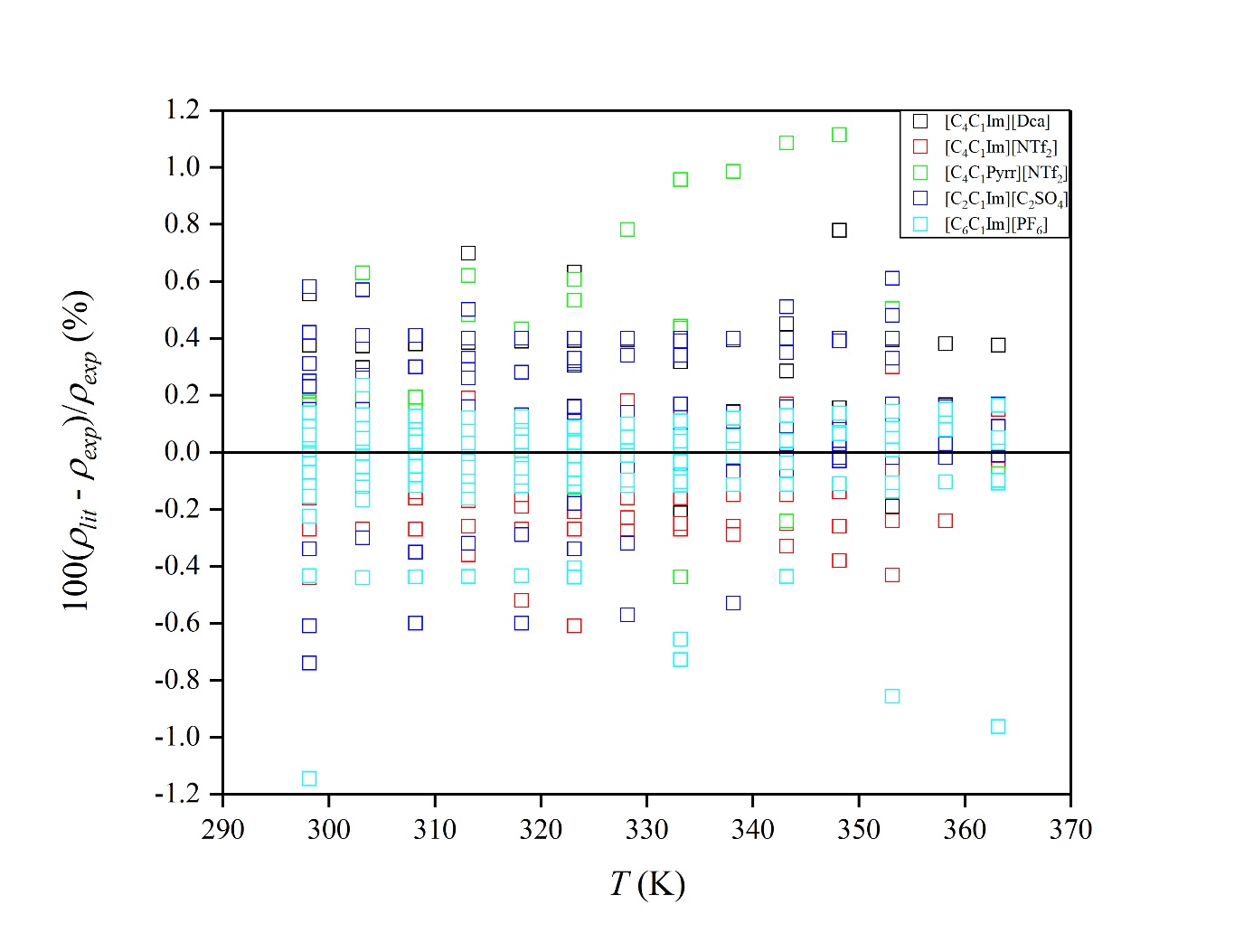
where *m* is a mass, *V* is a volume, density of nanoparticles, *ρNP*, was calculated using equation (2), and density of ionic liquids, *ρIL*, was determined experimentally.

1. **Results and discussion**

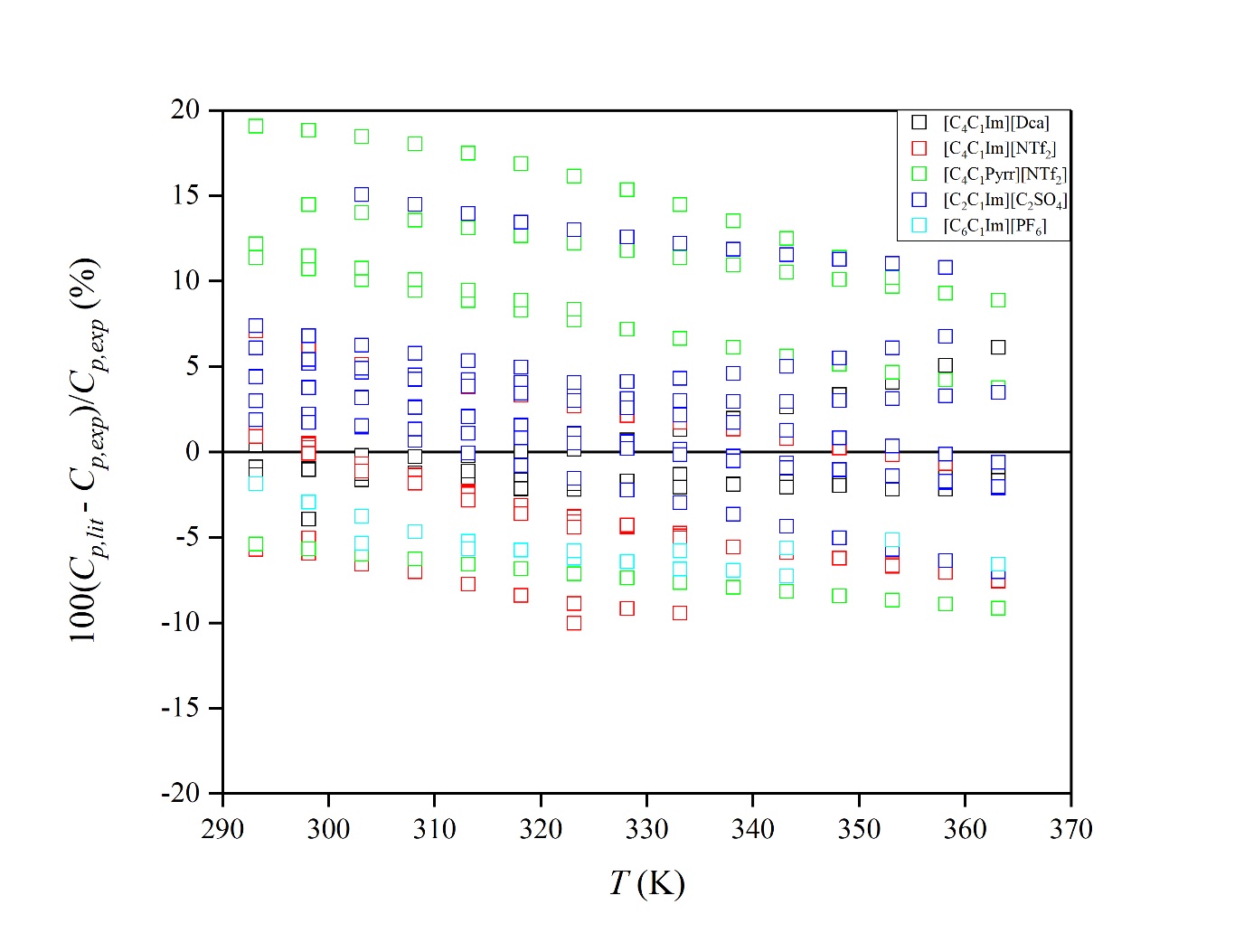
**Properties of pure ionic liquids**

The density and heat capacity were measured for pure ionic liquids and the purity before and after each measurement was studied, in particular the water content which may increase during/after the data collection. Thus, the water content after heat capacity assessments was checked by weighing the mass of the pan, and directly by using Karl Fischer titration after density measurement. No changes were recorded which means that the pans containing studied samples were sealed from the environment and water absorption. It was previously reported that water may influence the physical properties.[38] In the case of heat capacity, 1% of water shifts the heat capacity for about 1.6% (1-butyl-3-methylimidazolium tetrafluoroborate).[39] Nevertheless, density is much less dependent on the water content – 20% of water content changes the density of 1-butyl-3-methylimidazolium bis(trifluoromethylsulfonyl)imide for about 1%.[40]

The experimental data for the density and the heat capacity of the pure ILs are reported in supporting information files (SI1 and SI2). Their values were also compared to those previously reported. An extensive comparison to NIST ILs database was performed by taking into account all available literature positions, the relative deviations are presented in **Figure 2** and **Figure 3** for pure IL density and heat capacity, respectively. As can be seen, the density is a very extensively investigated property of ILs. The deviations observed were approximately (-1.2 – 1.2) % while the main concentration of them is between (-0.2 – 0.2) % as it could be expected regarding solely the standard uncertainty of each equipment used, with maximum of -1.15 %.[41] In the case of heat capacity, the deviations were about (-10 – 20) %, with maximum of 19.06 %.[42] Whereas the standard uncertainty of density measurement is 0.1% and heat capacity of 3%. Such high deviations may be related to different impurity levels and measurement methodology (technique, heating rate, gas flow rate, crucibles used, samples preparation and treatment, calibration). Moreover, such deviations (1% and 20% for density and heat capacity, respectively, or even 40% for heat capacity) are commonly observed in the literature for ionic liquids.[39,43] Diedrichs and Gmehling (2006) critically reviewed the heat capacity measurement approaches, including different techniques and parameters.[44] To the best of our knowledge, and following the above and others’ recommendations, the heat capacity determined in this work are highly accurate.[39,44,42,45] The deviations of pure ILs properties can be observed. Further considerations according to the physical properties and more complicated systems – liquid-liquid (*i.e.* water-saturated equilibrium),[46] solid-liquid (*i.e.* nanofluids),[47] liquid-gas (*i.e.* gas solubility in ILs).[48]



**Figure 2** Density deviations between values in this work (SI) and literature data for [C4C1Im][Dca],[34,49–69] [C4C1Im][NTf2],[40,52,55,58,68,70–111,43,112–123] [C2C1Im][C2SO4],[40,52,58,90,91,95,99,103,118,124–171] [C4C1Pyrr][NTf2],[58,61,94,96,113,137,172–189] [C6C1Im][PF6].[41,81,86,88,99,43,190–211]



**Figure 3** Isobaric heat capacity deviations between values in this work (SI) and literature data for [C4C1Im][Dca],[39,52,55,56,212,213] [C4C1Im][NTf2],[42,52,55,85,114,214–220] [C2C1Im][C2SO4],[39,42,52,134,138,221–226] [C4C1Pyrr][NTf2],[39,42,218,227,228] [C6C1Im][PF6].[193,220]

**Density of ionanofluids**

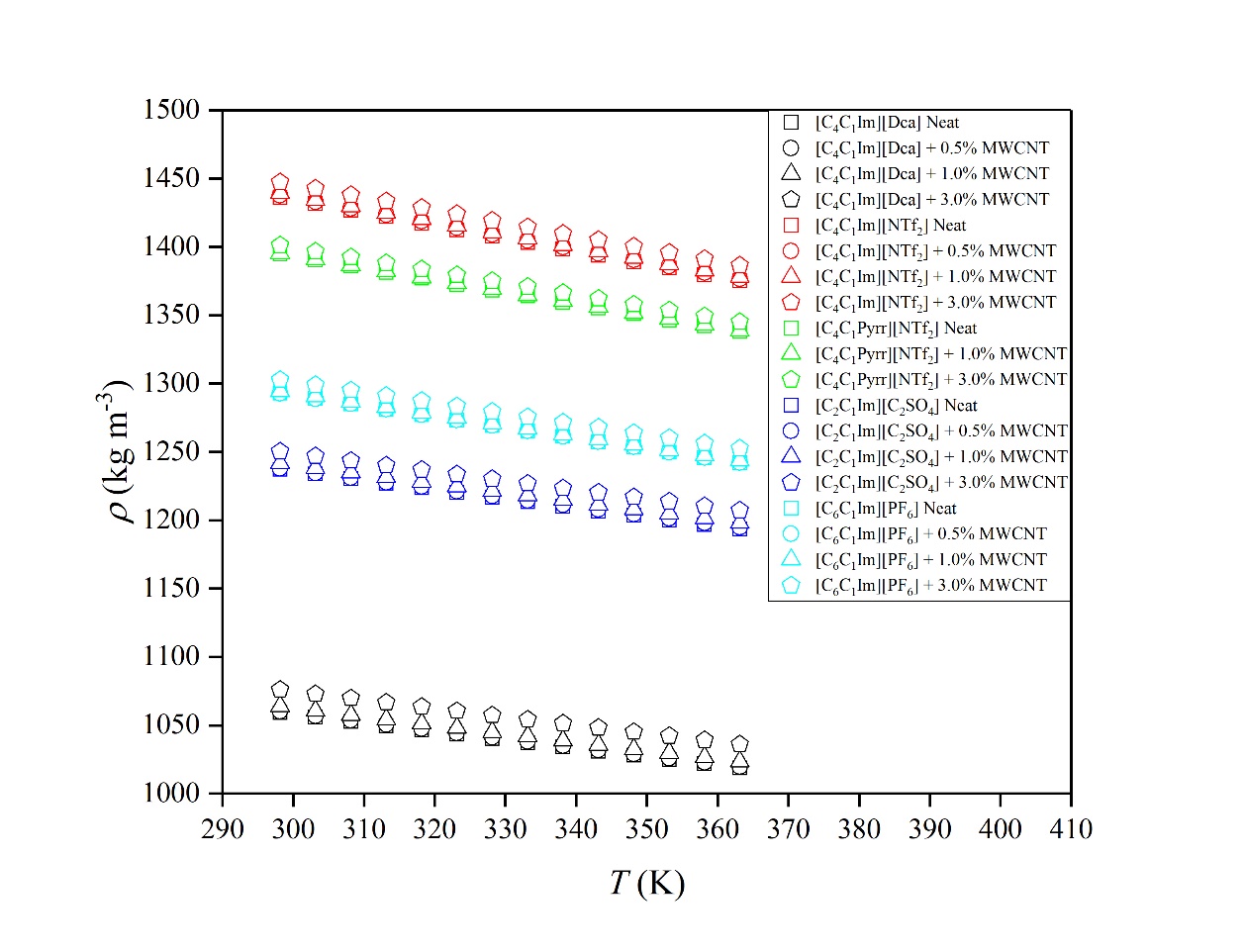
The density of ionanofluids containing different loading of carbon nanotubes, boron nitride or graphite nanoparticles dispersed in selected ILs was measured as a function of temperature. The results are shown in **Figure 4**, **Figure 5** and **Figure 6** for ionanofluids with carbon nanotubes, boron nitride and graphite, respectively. The parameters of equation (1) are summarized in **Table 3**. It can be seen that, for all cases, the density decreased as a function of temperature, whereas the loading of nanoparticles caused the density shift to higher density values, as expected. It indicates that the bulk density of all solid materials exhibits higher values. The experimental data were collected in an Excel file (SI1). It can be observed that the addition of nanoparticles into ionic liquid is the base fluid independent. In the literature, it can be found that the density of nanofluids could be then determined by using the empirical equation (2) originally proposed by Pak and Cho (1998) or by using an ideal excess molar volume-based approach.[36,229] In other words, the assumption made during the calculation of nanoparticles density in the case of the ideal excess molar volume-based approach is that the ideal molar volume is equal to the real molar volume of each nanofluid and investigated temperature. These two methods were applied during this work and the results of both nanoparticles density calculations are summarized in an Excel file available in the supporting information, and shown in **Figure S5** (SI3). Interestingly, the overall AARD for the density prediction of ionanofluids by using the empirical equation (2) model is close to 0.10 % in each case, while larger deviations are observed using the ideal excess molar volume-based approach, close to (0.44, 0.79 and 0.60) % for carbon nanotubes, boron nitride and graphite based ionanofluids, respectively. Herein, deviations observed using the ideal excess molar volume-based approach are presented in Figures S6-S8 of the SI. In fact, this latter approach does not lead to better results of ionanofluids density prediction (higher AARD) than that proposed by Pak and Cho. However, as shown in **Figures S9-S11** of the SI, the hypothetical excess molar volumes calculated using the NPs density values determined from this empirical equation (2) model resulted in larger absolute values (all negatives in the present cases) than those observed using the ideal excess molar volume-based approach, as expected. This finding is also in a good agreement with previous assessments about the scientific meaning of excess properties of systems consisting of more than two phases in solution. From a thermodynamic perspective, in such a case, this excess property is meaningless as an ideal reference state cannot be truly defined.[230] Therefore, the empirical equation (2) model originally proposed by Pak and Cho seems to be more reliable to determine NPs density as a function of NP structure and temperature.[36]

Using this recommended approach, the density values at 298.15 K are (1626.34, 1911.89 and 1711.99) kg m-3 for carbon nanotubes, boron nitride and graphite, respectively, with the standard uncertainty as described in the experimental part for density. It can be seen that carbon nanotubes and graphite have similar density values which indicates similarity in the packing of particles in the IL, as well as, the interactions between the ionic liquids and these nanoparticles (any bulky layering on the surface). These data are necessary for efficient and accurate nanofluids properties prediction while most of these are correlated to volumetric concentration of nanomaterials, for example thermal conductivity.[35]

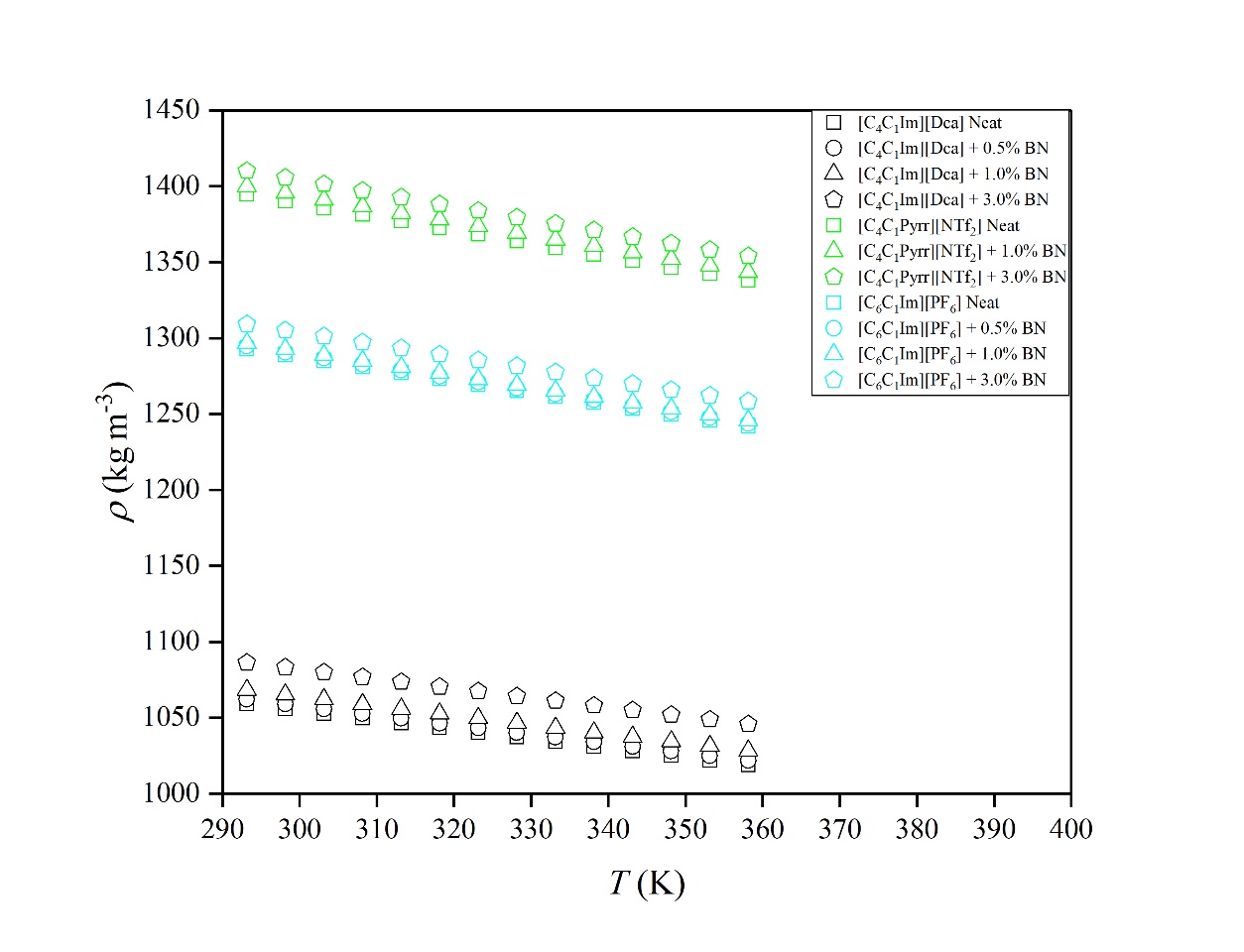
The values of calculated density were further used to calculate the theoretical density with equation (2). The results are summarized in the Excel file (SI1). The representation of calculated density values against the experimental data is shown in **Figure 7**, while the deviations can be found in **Figure 8**, **Figure 9** and **Figure 10** for ionanofluids containing carbon nanotubes, boron nitride and graphite doped, respectively. The AARD values were 0.10 % in each case, which indicated high correlative capability, and the values did not exceed 0.3 % deviation, apart from the system containing [C4C1Im][NTf2] + 3% MWCNT (*w*/*w*) where deviations close to (0.3 – 0.4) % are observed.

França *et al.* (**2014**) investigated the thermophysical properties of dicyanamide-based ionic liquids, including the density. Based on these density values of ionanofluids containing the carbon nanotubes, one can see that is also possible to accurately calculate ionanofluids density at 298.15 K using equation (2), within RAD values close to (0.11, 0.09, 0.17, 0.25, 0.03, 0.17) % for [C2C1Im][Dca] + 0.5% MWCNT (*w*/*w*), [C2C1Im][Dca] + 1.0% MWCNT (*w*/*w*), [C4C1Im][Dca] + 0.5% MWCNT (*w*/*w*), [C4C1Im][Dca] + 1.0% MWCNT (*w*/*w*), [C4C1Pyrr][Dca] + 0.5% MWCNT (*w*/*w*), [C4C1Pyrr][Dca] + 1.0% MWCNT (*w*/*w*), respectively.[34]

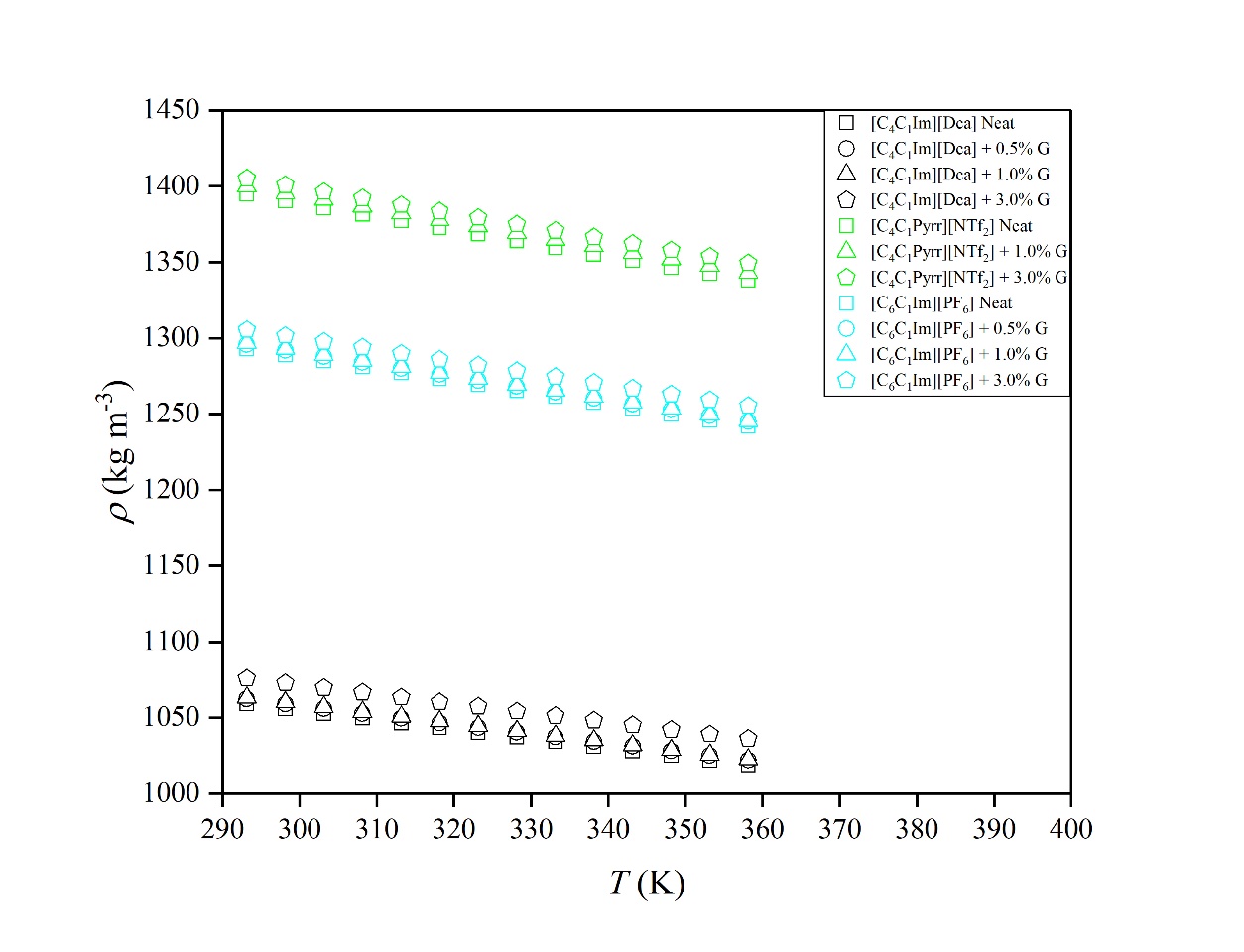
Density is a highly accurate property which may give a useful information about interfacial structure of nanoparticles. Shin and Banarjee (2011) showed that there is a specific interfacial organization occurring between fluid and nanoparticles.[10] Hentschke (2016) gave a full theoretical interpretation of the interfacial layer on nanoparticles with including the density and mass fraction of interfacial layer.[20] However, the assumption made in his work was that the density of interfacial layer is similar to this of bulk liquid. Based on the results reported in this work (for example the empirical equation (2)), it can be seen that the density of interfacial layer is similar to that of the bulk liquid because no excessive deviations are observed. Finally, the most recent theoretical and experimental work of França *et al.* (2017) proved the existence of interfacial layer.[231]



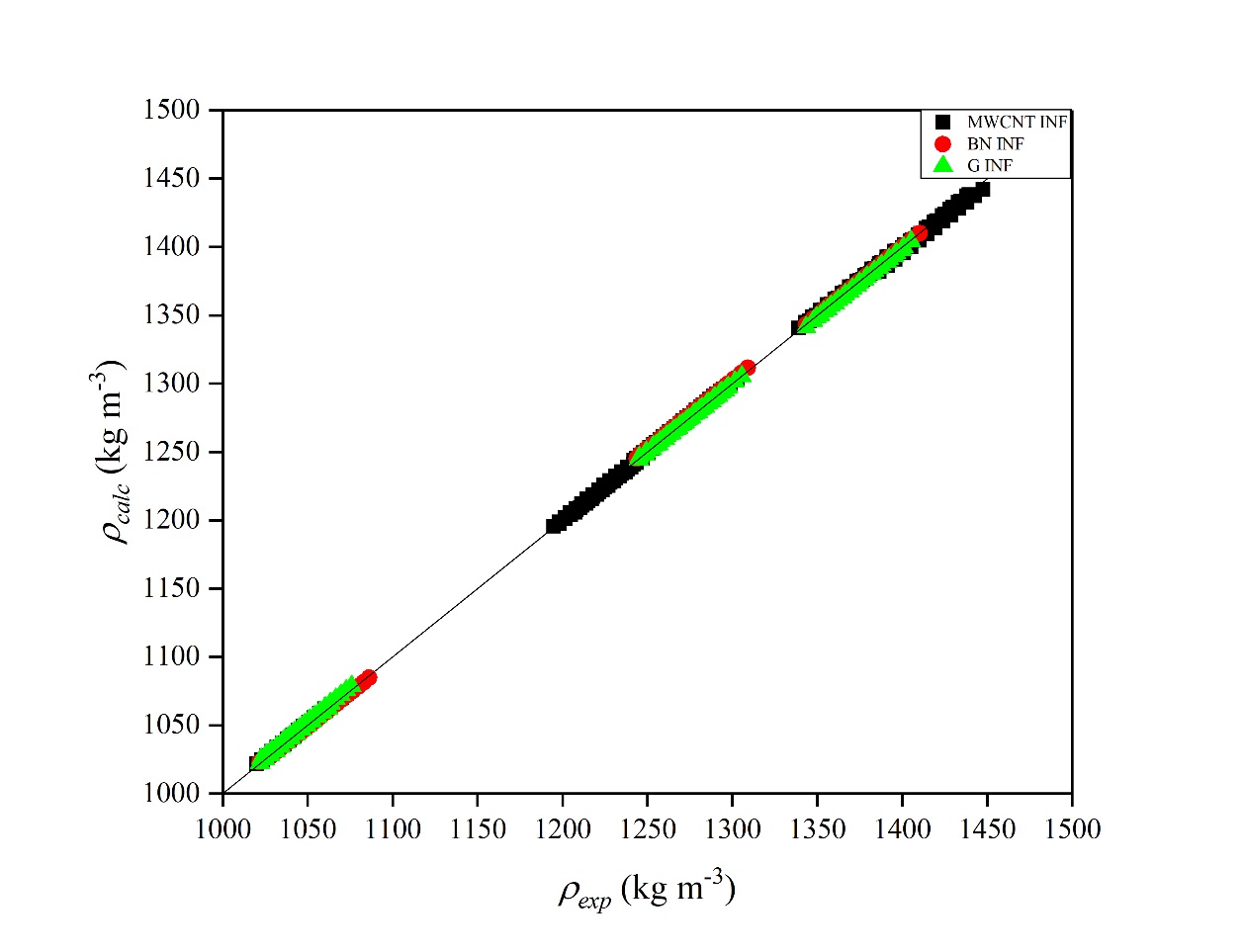
**Figure 4** Temperature dependence of the density of investigated multiwalled carbon nanotubes-doped (MWCNT) ionanofluids.



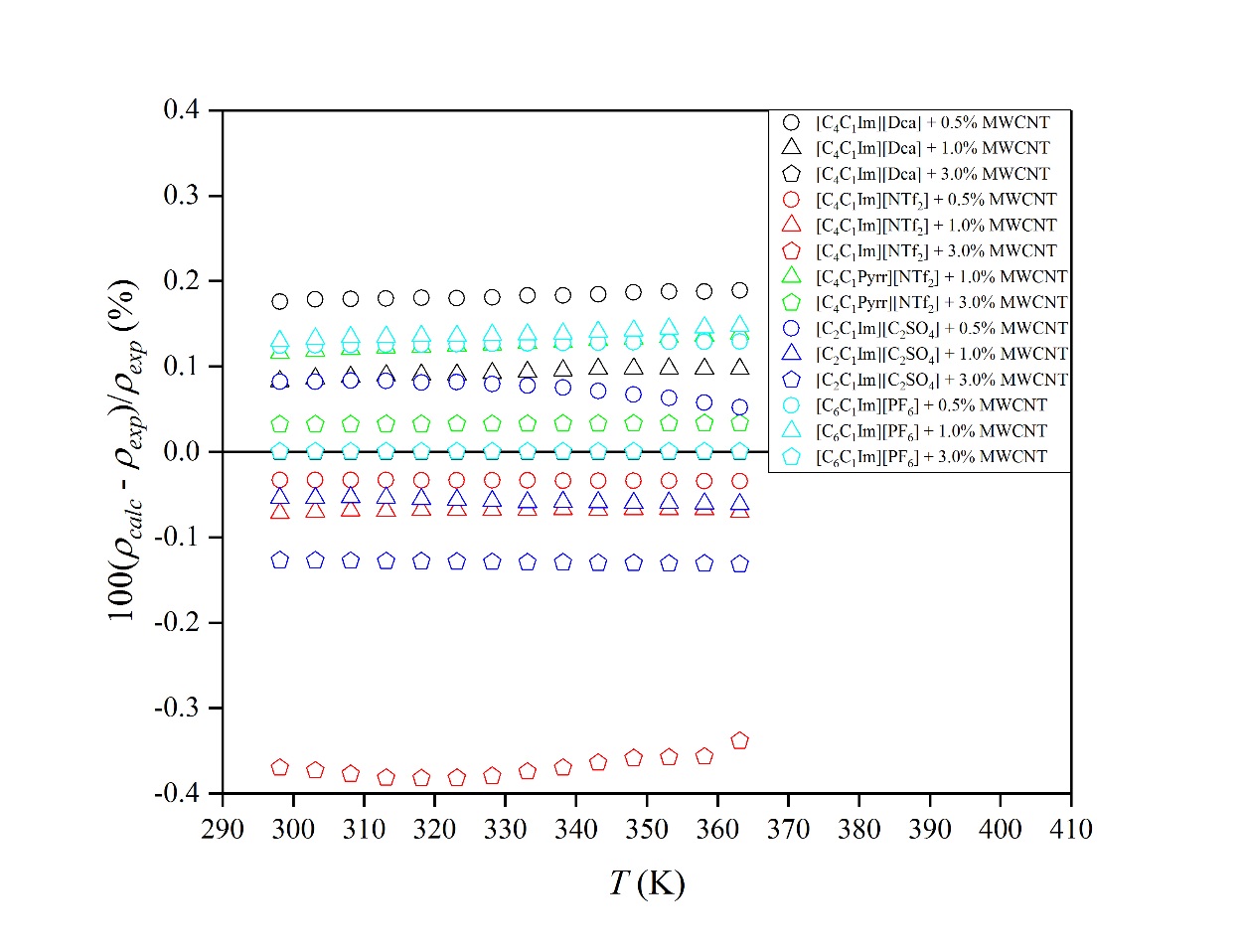
**Figure 5** Temperature dependence on the density of investigated boron nitride-doped (BN) ionanofluids.



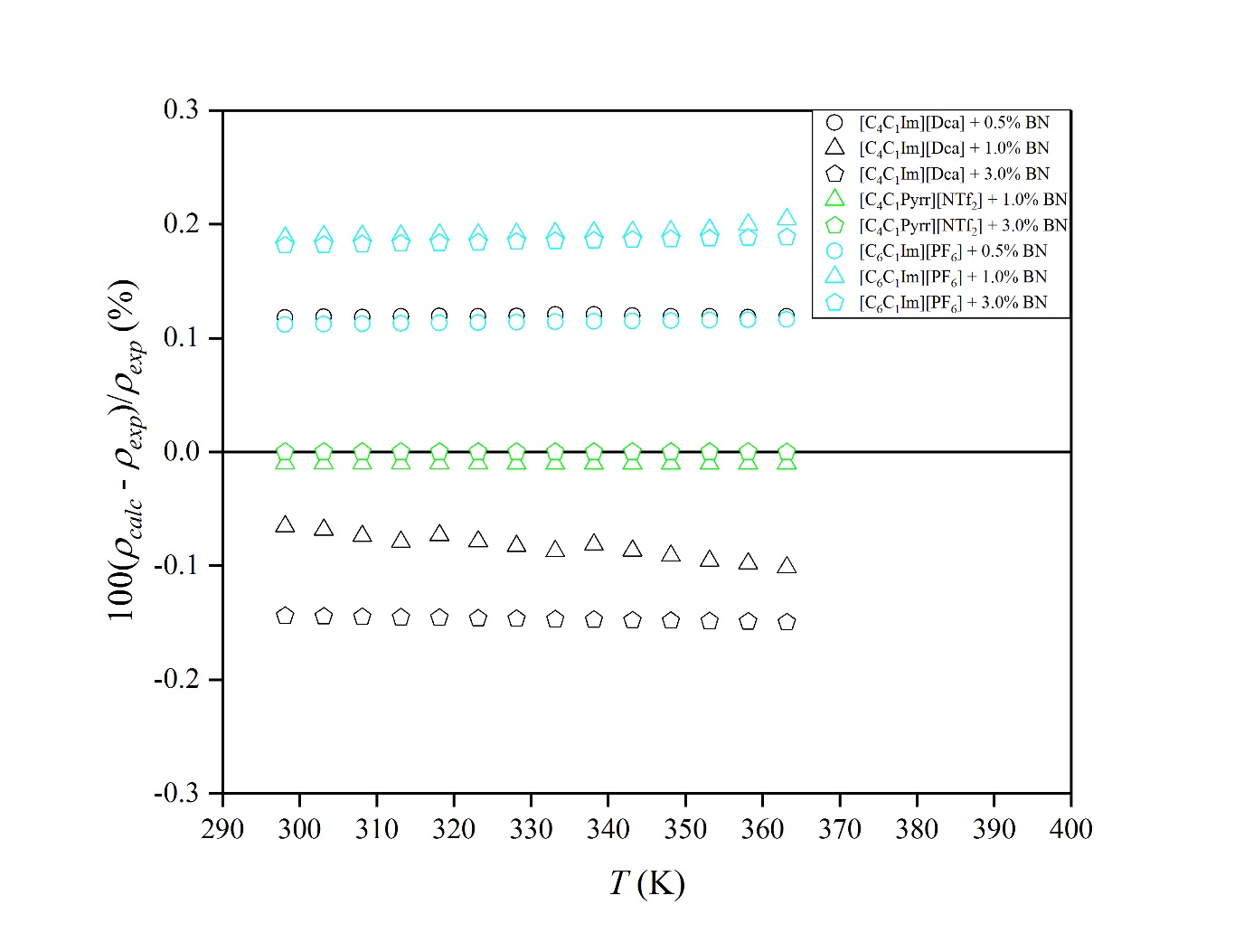
**Figure 6** Temperature dependence on the density of investigated graphite-doped (G) ionanofluids.



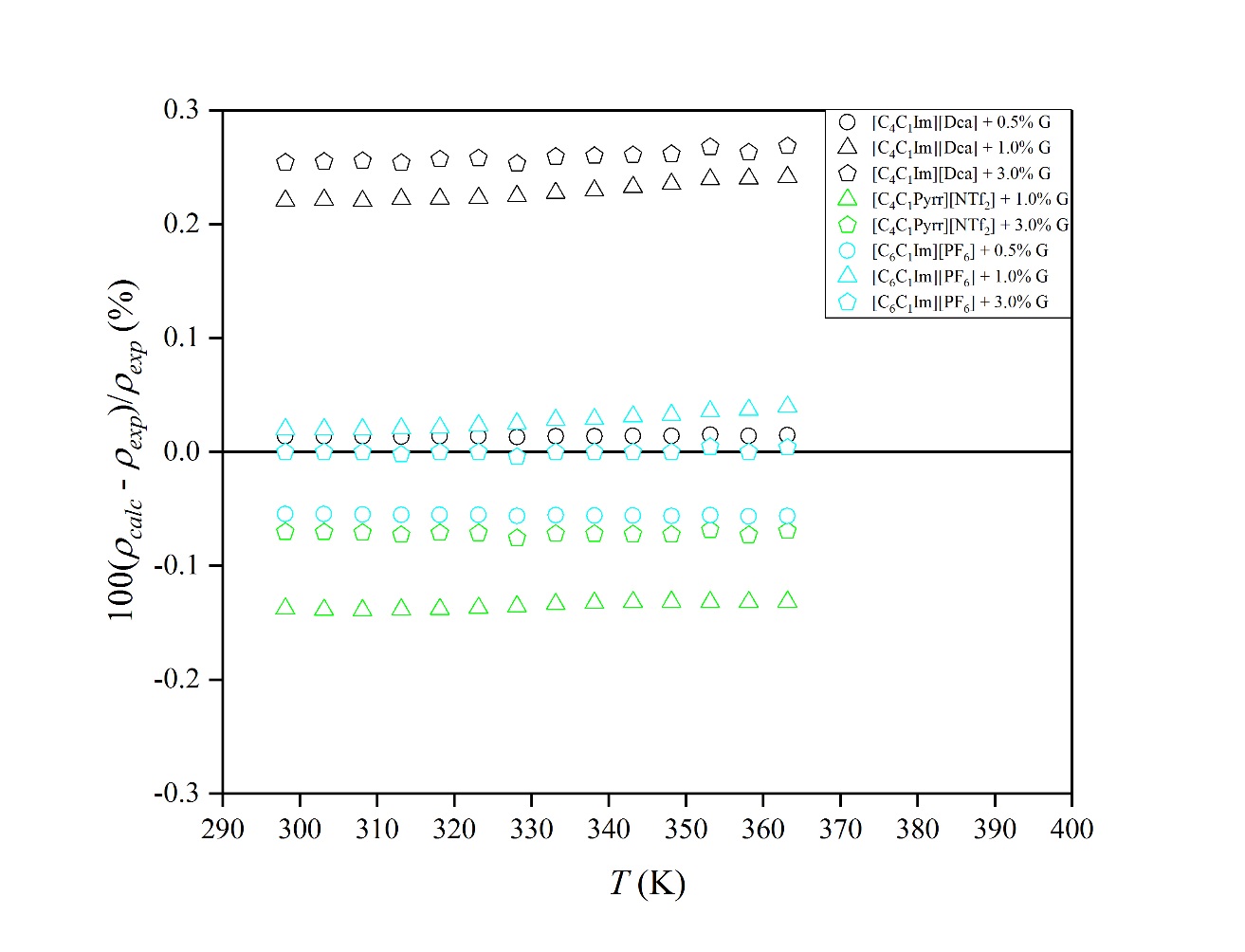
**Figure 7** Calculated density over the experimental values (SI) for ionanofluids (INF) incorporating multi-walled carbon nanotubes (MWCNT), boron nitride (BN), and graphite (G), by empirical equation (2).



**Figure 8** Deviations between calculated density and experimental values (SI) for ionanofluids with multiwalled carbon nanotubes (MWCNT), by empirical equation (2).



**Figure 9** Deviations between calculated density and experimental values (SI) for ionanofluids with boron nitride (BN), by empirical equation (2).



**Figure 10** Deviations between calculated density and experimental values (SI) for ionanofluids with graphite (G), by empirical equation (2).

**Table 3** The parameters, *a0* and *a1*, with their standard uncertainties, δ*a0* and δ*a1*, for *ρ*(*T*).

|  |  |  |  |
| --- | --- | --- | --- |
|  | *a0* ± δ*a0*  kg m-3 | -(*a1* ± δ*a1*) ∙ 10-3  kg m-3 K-1 | *R2* |
| [C4C1Im][Dca] neat | 1243.56 ± 0.39 | 619.1 ± 1.2 | 0.99995 |
| [C4C1Im][Dca] + 0.5% MWCNT | 1244.54 ± 0.38 | 619.2 ± 1.2 | 0.99995 |
| [C4C1Im][Dca] + 0.5% BN | 1243.39 ± 0.43 | 618.9 ± 1.3 | 0.99994 |
| [C4C1Im][Dca] + 0.5% G | 1243.11 ± 0.40 | 617.5 ± 1.2 | 0.99995 |
| [C4C1Im][Dca] + 1.0% MWCNT | 1248.39 ± 0.46 | 619.3 ± 1.4 | 0.99994 |
| [C4C1Im][Dca] + 1.0% BN | 1248.54 ± 0.46 | 615.2 ± 1.4 | 0.99993 |
| [C4C1Im][Dca] + 1.0% G | 1244.44 ± 0.35 | 618.1 ± 1.1 | 0.99996 |
| [C4C1Im][Dca] + 3.0% MWCNT | 1259.22 ± 0.44 | 614.4 ± 1.3 | 0.99994 |
| [C4C1Im][Dca] + 3.0% BN | 1268.34 ± 0.50 | 621.6 ± 1.5 | 0.99992 |
| [C4C1Im][Dca] + 3.0% G | 1254.50 ± 0.48 | 609.7 ± 1.5 | 0.99992 |
| [C4C1Im][NTf2] neat | 1717.43 ± 0.38 | 943.1 ± 1.1 | 0.99998 |
| [C4C1Im][NTf2] + 0.5% MWCNT | 1718.14 ± 0.38 | 940.8 ± 1.2 | 0.99998 |
| [C4C1Im][NTf2] + 1.0% MWCNT | 1719.26 ± 0.45 | 939.5 ± 1.4 | 0.99997 |
| [C4C1Im][NTf2] + 3.0% MWCNT | 1727.40 ± 0.25 | 938.74 ± 0.77 | 0.99999 |
| [C4C1Pyrr][NTf2] neat | 1654.39 ± 0.34 | 871.6 ± 1.0 | 0.99998 |
| [C4C1Pyrr][NTf2] + 1.0% MWCNT | 1654.86 ± 0.34 | 870.8 ± 1.0 | 0.99998 |
| [C4C1Pyrr][NTf2] + 1.0% BN | 1654.85 ± 0.37 | 869.9 ± 1.1 | 0.99998 |
| [C4C1Pyrr][NTf2] + 1.0% G | 1654.32 ± 0.39 | 868.8 ± 1.2 | 0.99998 |
| [C4C1Pyrr][NTf2] + 3.0% MWCNT | 1657.27 ± 0.39 | 859.3 ± 1.2 | 0.99998 |
| [C4C1Pyrr][NTf2] + 3.0% BN | 1664.06 ± 0.45 | 866.5 ± 1.4 | 0.99997 |
| [C4C1Pyrr][NTf2] + 3.0% G | 1655.51 ± 0.44 | 854.6 ± 1.4 | 0.99997 |
| [C2C1Im][C2SO4] neat | 1436.86 ± 0.31 | 670.27 ± 0.93 | 0.99998 |
| [C2C1Im][C2SO4] + 0.5% MWCNT | 1435.65 ± 0.54 | 663.3 ± 1.6 | 0.99992 |
| [C2C1Im][C2SO4] + 1.0% MWCNT | 1440.45 ± 0.31 | 667.02 ± 0.94 | 0.99997 |
| [C2C1Im][C2SO4] + 3.0% MWCNT | 1448.31 ± 0.35 | 664.1 ± 1.1 | 0.99997 |
| [C6C1Im][PF6] neat | 1526.73 ± 0.27 | 784.48 ± 0.82 | 0.99999 |
| [C6C1Im][PF6] + 0.5% MWCNT | 1526.32 ± 0.28 | 782.88 ± 0.85 | 0.99998 |
| [C6C1Im][PF6] + 0.5% BN | 1524.34 ± 0.29 | 784.08 ± 0.88 | 0.99998 |
| [C6C1Im][PF6] + 0.5% G | 1524.90 ± 0.28 | 782.10 ± 0.85 | 0.99998 |
| [C6C1Im][PF6] + 1.0% MWCNT | 1528.01 ± 0.25 | 783.24 ± 0.76 | 0.99999 |
| [C6C1Im][PF6] + 1.0% BN | 1526.98 ± 0.16 | 785.80 ± 0.49 | 0.99999 |
| [C6C1Im][PF6] + 1.0% G | 1530.35 ± 0.25 | 783.40 ± 0.77 | 0.99999 |
| [C6C1Im][PF6] + 3.0% MWCNT | 1533.89 ± 0.35 | 774.9 ± 1.1 | 0.99998 |
| [C6C1Im][PF6] + 3.0% BN | 1538.28 ± 0.40 | 782.1 ± 1.2 | 0.99997 |
| [C6C1Im][PF6] + 3.0% G | 1531.11 ± 0.36 | 770.2 ± 1.1 | 0.99997 |

**Heat capacity of ionanofluids**

The results of the heat capacity measurements for pure ILs and systems with multi-walled carbon nanotubes, boron nitride and graphite are shown in **Figure 11**, **Figure 12** and **Figure 13** for carbon nanotubes-, boron nitride- and graphite-doped ionanofluids, respectively, while the parameters of second-order equation (4) are summarized in **Table 4**. The experimental data are shown in SI2. Unfortunately, very few datasets have been previously reported for similar systems, to date, which limits the comparison that can be made. As can be seen the heat capacity of all data increases with temperature, as expected.

The specific heat capacity was also found to increase with nanomaterials loading (with restriction up to 3% in nanoparticles mass fraction units, as investigated), as can be clearly seen in **Figure 11**, **Figure 12** and **Figure 13** for carbon nanotubes-, boron nitride- and graphite-doped ionanofluids, respectively. **Figure 14**, **Figure 15** and **Figure 16** shows the heat capacity enhancement (in comparison to pure ILs) as a function of nanoparticles loading (volume fraction) for carbon nanotubes-, boron nitride- and graphite-doped nanofluids, respectively. The enhancements observed were in the ranges of (5 – 13, 5 – 21 and 12 – 28) % for carbon nanotubes-, boron nitride- and graphite-based ionanofluids. As can be seen, the effect of the addition of carbon nanotubes was the smallest and compared with graphite which showed the largest changes. These dependencies were found to be linear functions:

The mechanism of the heat capacity enhancement has been widely discussed and is based on the mixing theory for ideal gas mixtures.[232] However, it has been shown that this approach is not relevant for nanofluids, where the specific heat capacity of carbon nanotubes is lower than liquids and the enhancement of heat capacity is observed.[20] Another theory proposed is based on the assumption of thermal equilibrium between the fluid and nanoparticles.[232] It was shown that it reproduces better values, however, is still not accurate enough, and the specific mechanism is not explained within this theory. The first attempt to explain the heat capacity increasing was proposed by Shin *et al.* (2011).[10] The authors discussed three different modes: (*i*) higher specific heat capacity of nanoparticles in comparison to their bulk equivalents (based on Al2O3, up to 25%)[233] which is related to the quantization of phonon spectrum with discrete values, which is not limited by the size of nanoparticles (due to resolved surface area); (*ii*) high solid-fluid interaction energy caused by the high surface area per unit mass of nanoparticles which increases the interfacial thermal resistance and acts as additional thermal storage; (*iii*) layering of liquid molecules at the surface of nanoparticles due to the liquid molecules adsorption as semi-solid layer with higher thermal properties than the bulk liquid (usually ~2-5 nm).

The most recent work of Hentschke (2016) discussed the possible mechanism of the heat capacity enhancement, including the mathematical foundation.[20] It was shown that the observed enhancement may be caused by one or both of the following factors: (*i*) nanolayering of nanoparticles with liquid molecules, with the layer of higher heat capacity in comparison to liquids and dependent on the nanoparticles type; (*ii*) the new phenomenological theory based on the attendant mesolayer interacting with other mesolayers with the assumption that the enhancement occurs until some specific loading of nanoparticles, and then becomes a decreasing function of nanoparticles concentration (a few parameters were introduced while the maximum mass fraction concentration was always below 3%). Nevertheless, all of these theories are based on molecular solvents-like nanofluids, and none of them considered ionic liquids as basefluids.

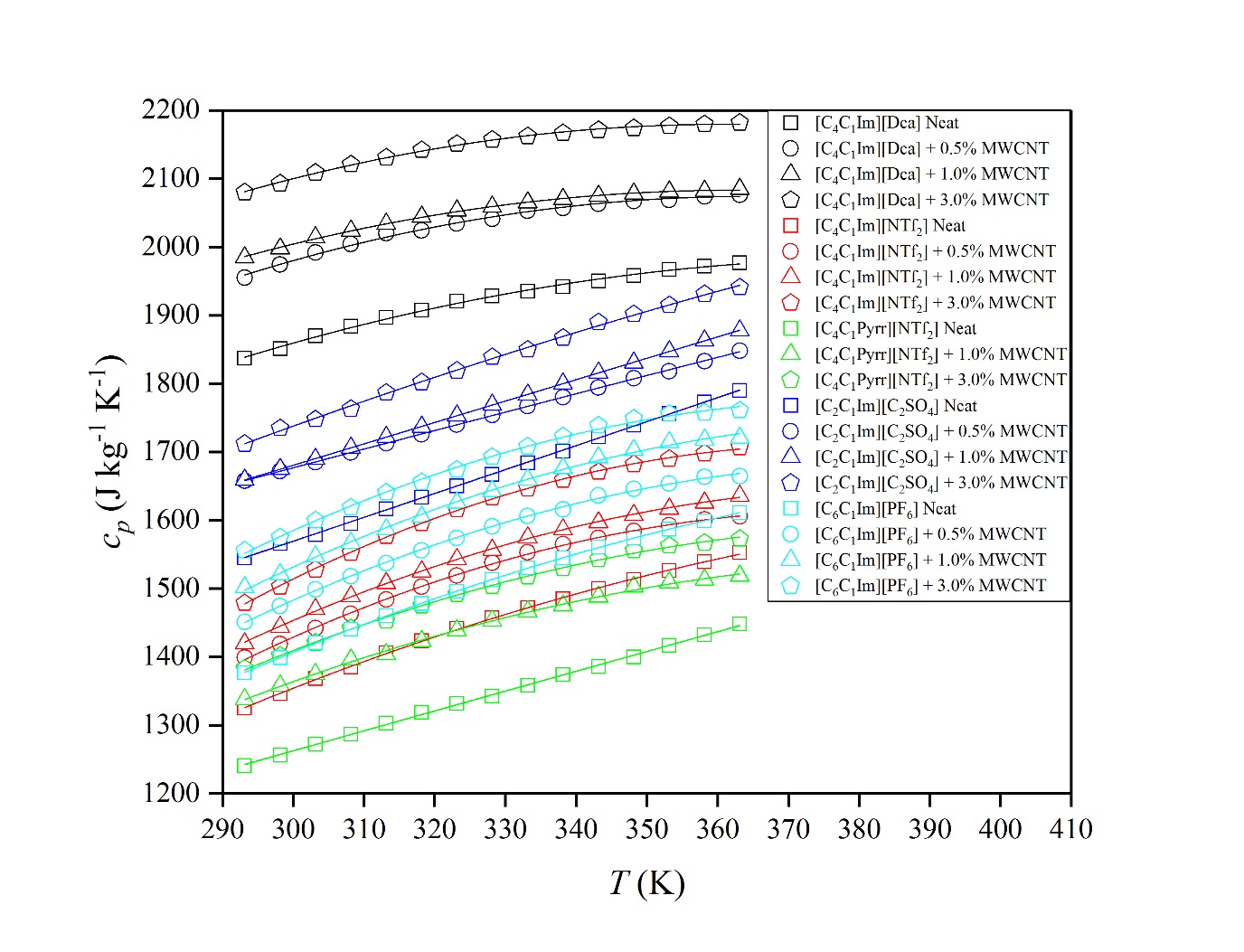
The observed enhancement clearly indicated the existence of interfacial nanolayers. Moreover, as can be seen in **Figure 14**, **Figure 15** and **Figure 16** for carbon nanotubes-, boron nitride- and graphite-doped ionanofluids, respectively, the value of the enhancements observed are different in the case of each type of nanoparticle. This shows that the mechanism is based on the type of nanoparticles instead of type of ionic liquid.

As shown by Hentschke (2016) a maximum enhancement is maintained by the existence of overlapping mesolayers. In this work, this was not recorded (as no significant maximum of the enhancement was observed below 3 wt%) which may indicate a smaller impact of this type of interactions caused by stronger interactions between ionic liquids and nanoparticles, and as a result more compact interfacial layer. The different types of ionic liquids used in this work provide the information about impact of the base fluid on the enhancement. However, ~~due to the linear dependency,~~ it can be assumed that the base fluid type and structure is not an influencing factor (based on the dependency upon the nanoparticles concentration). While different values of enhancements prove the fact that enhancement depends on the type of nanoparticles. Moreover, as pointed out by Hentschke (2016) the heat capacity enhancement depends on the particle size, and thus, the surface area, it can be observed that the enhancement intensity depends in the following sequence: graphite > boron nitride > carbon nanotubes, as well as the nanoparticle size (and reverse order for surface areas), which is in a good agreement with experimental data.

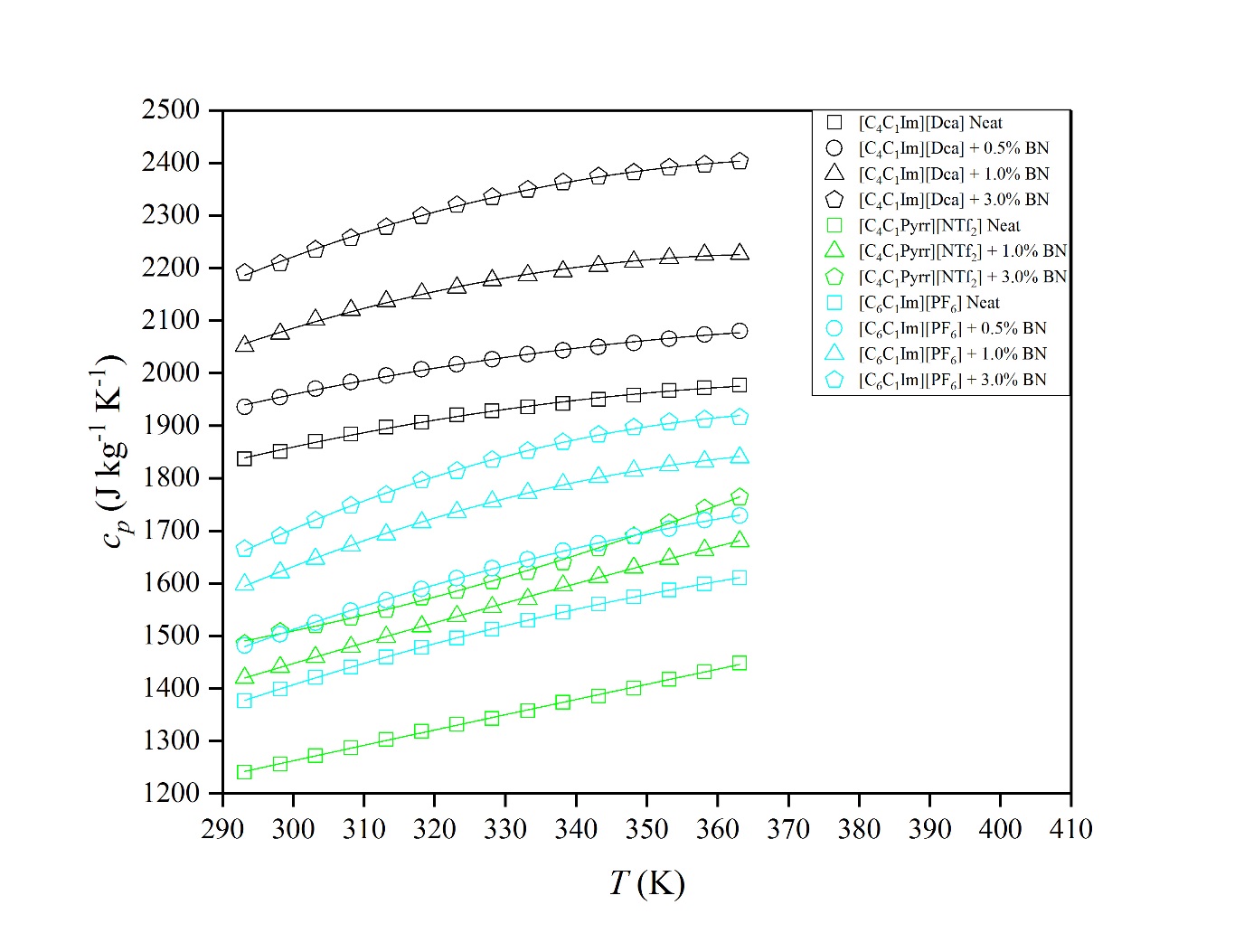
The most likely mechanism for the enhancement in case of ionic liquid-based nanofluids is based on the semi-solid-like liquid ordering on the nanoparticles surface which was described in the work of Shin and Banerjee (2011), with further extension by Hentschke (2016), excluding the interacting mesolayers as no experimental result indicates such a behavior in this work.

Similar work was carried out by França *et al.* (2017) with molecular dynamics simulations of surface nanolayering including the thermal conductivity modelling. The results presented in this work clearly indicate the existence of liquid nanolayers on the nanoparticles surface which is mainly responsible of the enhancement of the thermal properties when a nanofluid is dispersed in a fluid base. As well, no mesolayer interaction was observed. The results presented herein are in a good agreement with those published by França *et al.* (2017).

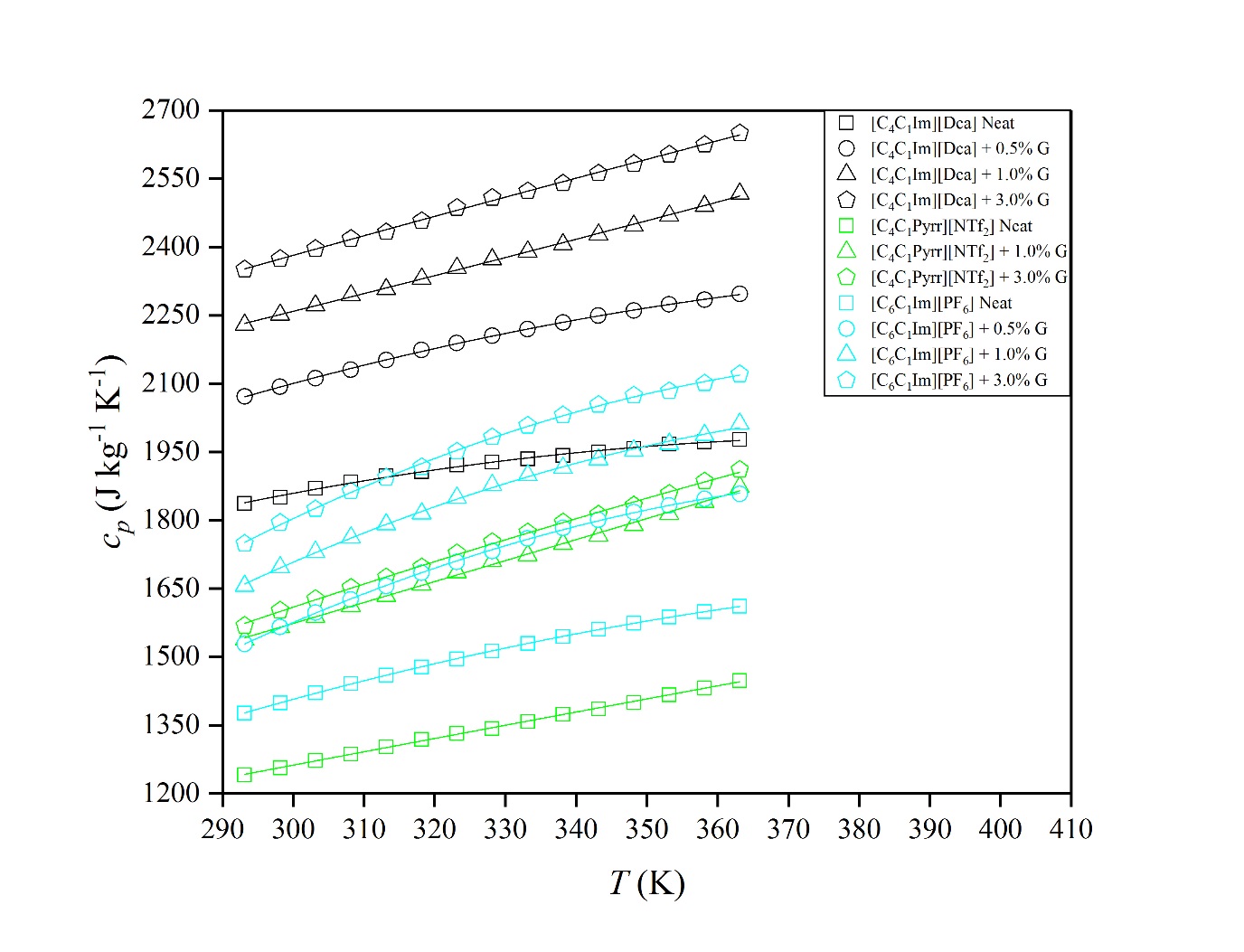
From an industrial perspective, the prior knowledge of the specific heat capacity as a function of temperature is crucial. More specifically, the enhancement in the heat capacity caused by the presence of nanoparticles in base fluids as the function of composition and temperature is of high interest. **Figure** **S12-S14** show that the enhancements obtained for carbon nanotubes-, boron nitride- and graphite-doped ionanofluids (SI3) are ranged from (3.1 to 13.2, 5.3 to 22.0, 10.9 to 34.2) %, respectively. The maximum of enhancement observed were (13.2, 22.0 and 34.2) % for [C4C1Im][Dca] + 3% MWCNT at 293.15 K, [C4C1Im][Dca] + 3% BN at 343.15 K and [C4C1Im][Dca] + 3% G at 363.15 K, respectively. Moreover, the enhancement for carbon nanotubes-doped ionanofluids decreased with temperature whereas is was approximately constant in the case of boron nitride-doped ionanofluids, and increased for the graphite-doped ionanofluids. This also reflects in different type of prospective industrial applications in case of desired temperature profile of heat transfer fluids.



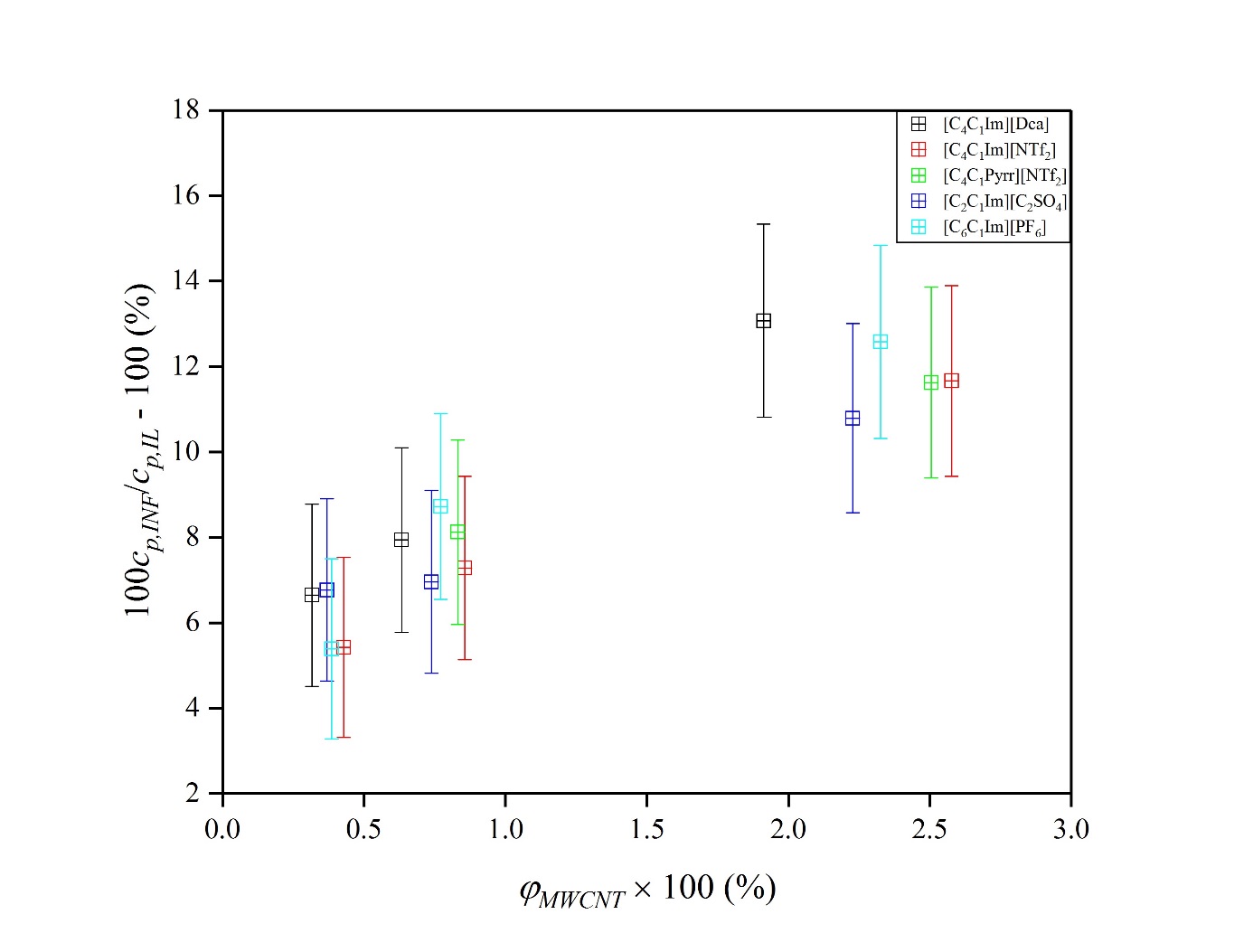
**Figure 11** Temperature dependence on the specific heat capacity of investigated multiwalled carbon nanotubes-doped (MWCNT) ionanofluids.



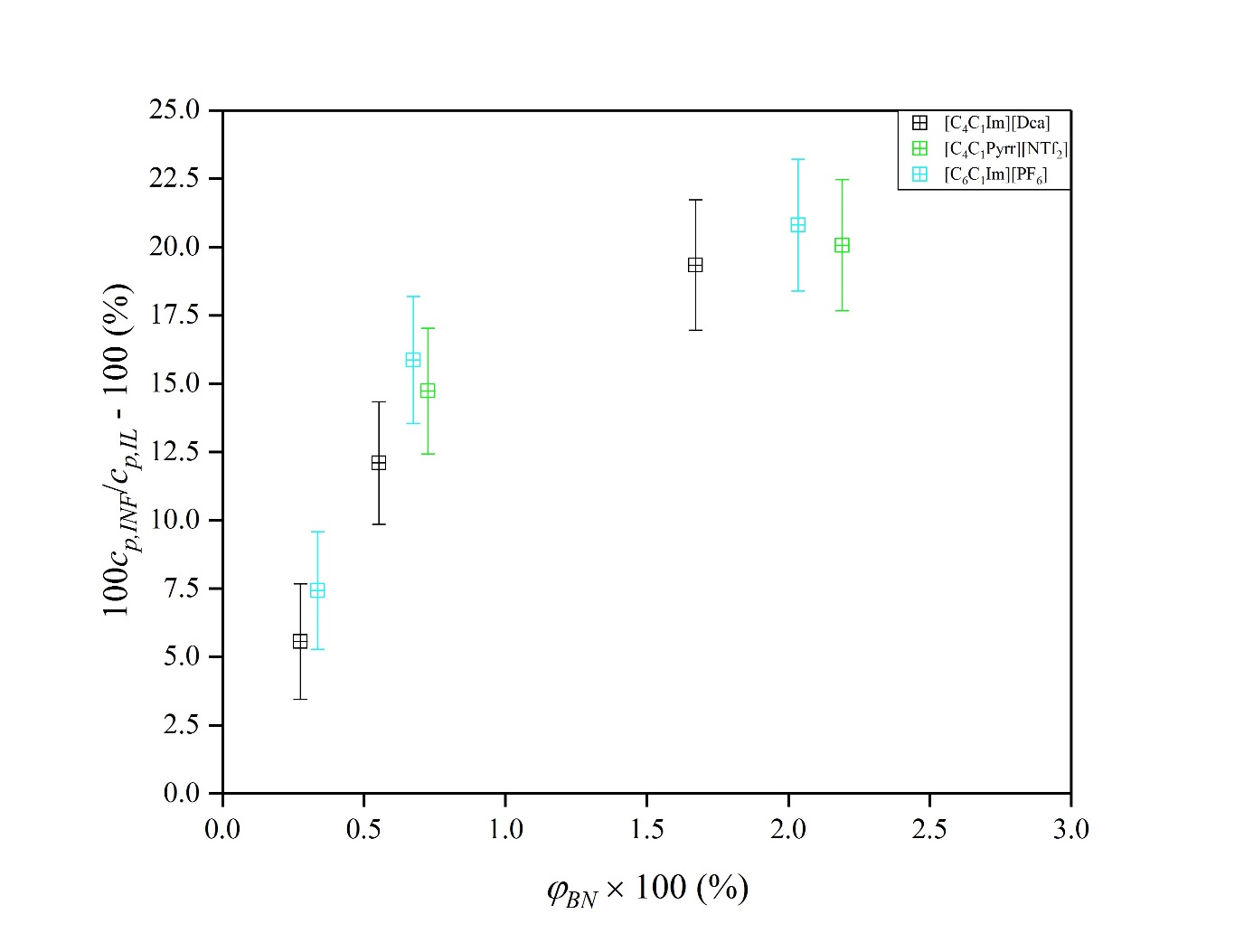
**Figure 12** Temperature dependence on the specific heat capacity of investigated boron nitride-doped (BN) ionanofluids.



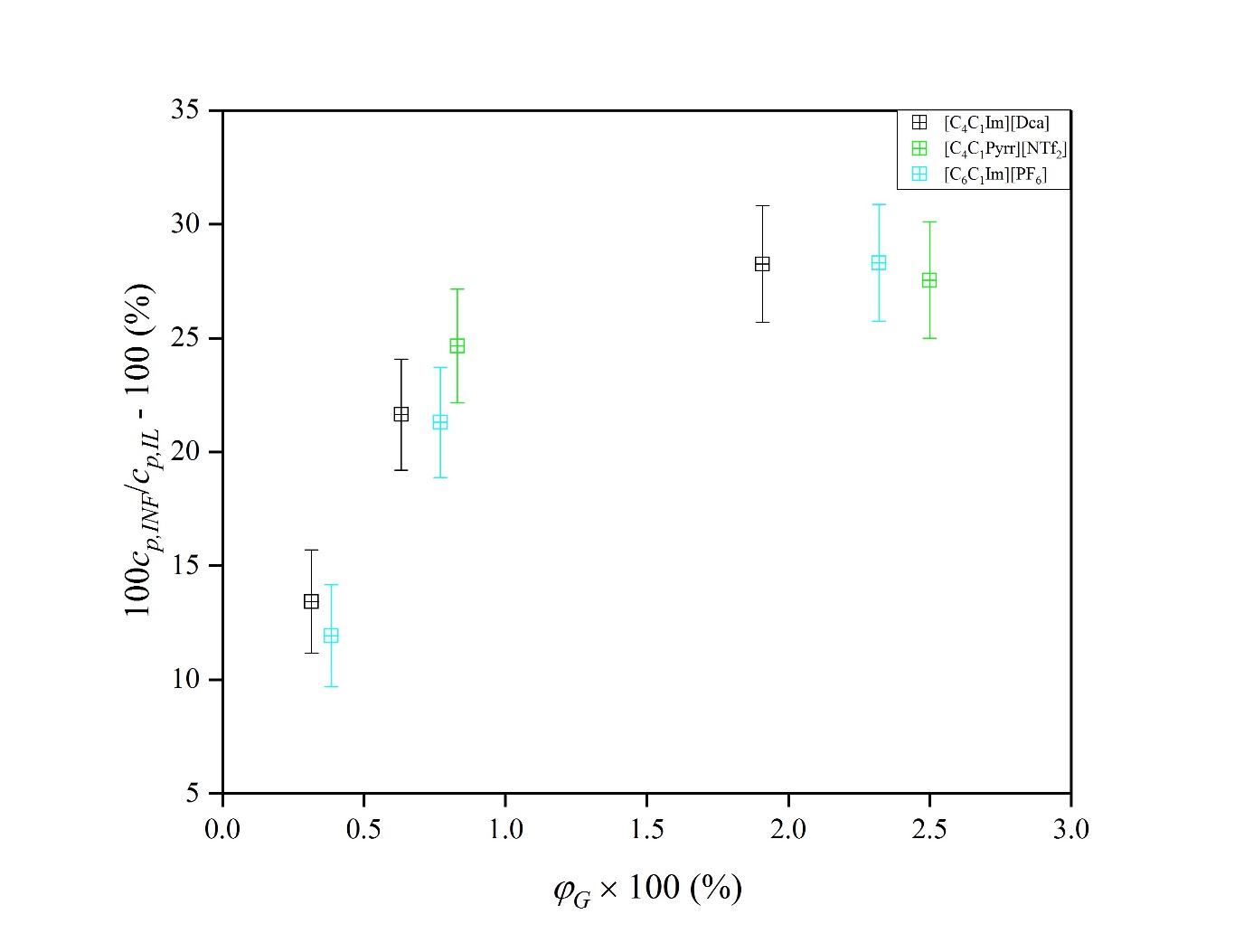
**Figure 13** Temperature dependence on the specific heat capacity of investigated graphite-doped (G) ionanofluids.



**Figure 14** Heat capacity enhancement as a function of the multiwalled carbon nanotubes loading (MWCNT) at 298.15 K.



**Figure 15** Heat capacity enhancement as a function of the boron nitride loading (BN) at 298.15 K.



**Figure 16** Heat capacity enhancement as a function of the graphite loading (G) at 298.15 K.

**Table 4** The parameters, *a0*, *a1* and *a2*, with their standard uncertainties, δ*a0*, δ*a1* and δ*a2*, for *cp*(*T*).

|  |  |  |  |  |
| --- | --- | --- | --- | --- |
|  | -(*a0* ± δ*a0*)  J kg-1 K-1 | *a1* ± δ*a1*  J kg-1 K-2 | -(*a2* ± δ*a2*) ∙ 10-3  J kg-1 K-3 | *R2* |
| [C4C1Im][Dca] neat | 530 ± 160 | 13.0 ± 1.0 | 16.9 ± 1.5 | 0.99693 |
| [C4C1Im][Dca] + 0.5% MWCNT | 920 ± 180 | 16.4 ± 1.1 | 22.5 ± 1.7 | 0.99497 |
| [C4C1Im][Dca] + 0.5% BN | 530 ± 160 | 13.0 ± 1.0 | 16.9 ± 1.5 | 0.99693 |
| [C4C1Im][Dca] + 0.5% G | 690 ± 110 | 14.44 ± 0.65 | 17.12 ± 0.99 | 0.99951 |
| [C4C1Im][Dca] + 1.0% MWCNT | 560 ± 78 | 14.57 ± 0.48 | 20.09 ± 0.73 | 0.99867 |
| [C4C1Im][Dca] + 1.0% BN | 210 ± 140 | 11.65 ± 0.86 | 14.8 ± 1.3 | 0.99775 |
| [C4C1Im][Dca] + 1.0% G | -1060 ± 12 | 3.992 ± 0.038 | - | 0.99874 |
| [C4C1Im][Dca] + 3.0% MWCNT | 610 ± 110 | 15.46 ± 0.69 | 21.4 ± 1.0 | 0.99737 |
| [C4C1Im][Dca] + 3.0% BN | 1800 ± 200 | 21.7 ± 1.2 | 29.4 ± 1.8 | 0.99719 |
| [C4C1Im][Dca] + 3.0% G | -1121 ± 14 | 4.203 ± 0.042 | - | 0.99863 |
| [C4C1Im][NTf2] neat | 1190 ± 100 | 12.93 ± 0.62 | 14.82 ± 0.94 | 0.99956 |
| [C4C1Im][NTf2] + 0.5% MWCNT | 2520 ± 110 | 21.74 ± 0.66 | 28.6 ± 1.0 | 0.99944 |
| [C4C1Im][NTf2] + 1.0% MWCNT | 2150 ± 110 | 19.58 ± 0.67 | 25.2 ± 1.0 | 0.99943 |
| [C4C1Im][NTf2] + 3.0% MWCNT | 2980 ± 120 | 24.89 ± 0.73 | 33.0 ± 1.1 | 0.99942 |
| [C4C1Pyrr][NTf2] neat | -391.2 ± 7.3 | 2.905 ± 0.022 | - | 0.99919 |
| [C4C1Pyrr][NTf2] + 1.0% MWCNT | 1460 ± 180 | 15.1 ± 1.1 | 19.0 ±1.7 | 0.99786 |
| [C4C1Pyrr][NTf2] + 1.0% BN | 328.7 ± 9.9 | 3.734 ± 0.030 | - | 0.99909 |
| [C4C1Pyrr][NTf2] + 1.0% G | -191 ± 18 | 4.608 ± 0.056 | - | 0.99791 |
| [C4C1Pyrr][NTf2] + 3.0% MWCNT | 1760 ± 160 | 17.1 ± 1.0 | 21.9 ± 1.5 | 0.99848 |
| [C4C1Pyrr][NTf2] + 3.0% BN | 2360 ± 230 | -8.5 ± 1.4 | -19.0 ± 2.1 | 0.99850 |
| [C4C1Pyrr][NTf2] + 3.0% G | 570 ± 280 | 9.4 ± 1.7 | 7.1 ± 2.6 | 0.99868 |
| [C2C1Im][C2SO4] neat | -519.1 ± 6.4 | 3.501 ± 0.020 | - | 0.99956 |
| [C2C1Im][C2SO4] + 0.5% MWCNT | 780 ± 58 | 3.24 ± 0.36 | 0.82 ± 0.54 | 0.99979 |
| [C2C1Im][C2SO4] + 1.0% MWCNT | - 740.8 ± 1.1 | 3.1321 ± 0.0035 | - | 0.99998 |
| [C2C1Im][C2SO4] + 3.0% MWCNT | -746 ± 17 | 3.314 ± 0.050 | - | 0.99676 |
| [C6C1Im][PF6] neat | 1284 ± 34 | 13.71 ± 0.21 | 15.81 ± 0.32 | 0.99995 |
| [C6C1Im][PF6] + 0.5% MWCNT | 2210 ± 160 | 20.04 ± 0.99 | 25.8 ± 1.5 | 0.99883 |
| [C6C1Im][PF6] + 0.5% BN | 1517 ± 98 | 15.59 ± 0.60 | 18.32 ± 0.92 | 0.99966 |
| [C6C1Im][PF6] + 0.5% G | 3450 ± 150 | 26.88 ± 0.90 | 33.7 ± 1.4 | 0.99957 |
| [C6C1Im][PF6] + 1.0% MWCNT | 2250 ± 220 | 20.4 ± 1.4 | 26.2 ± 2.1 | 0.99793 |
| [C6C1Im][PF6] + 1.0% BN | 2650 ± 110 | 23.35 ± 0.67 | 30.2 ± 1.0 | 0.99958 |
| [C6C1Im][PF6] + 1.0% G | 3250 ± 310 | 26.3 ± 1.9 | 32.7 ± 2.9 | 0.99820 |
| [C6C1Im][PF6] + 3.0% MWCNT | 2360 ± 250 | 21.7 ± 1.5 | 28.3 ± 2.3 | 0.99720 |
| [C6C1Im][PF6] + 3.0% BN | 3270 ± 150 | 27.44 ± 0.94 | 36.2 ± 1.4 | 0.99925 |
| [C6C1Im][PF6] + 3.0% G | 3760 ± 260 | 29.7 ± 1.6 | 37.3 ± 2.4 | 0.99894 |

1. **Conclusions**

During this work, an extensive study on density and heat capacity of ionic liquids-based nanofluids was carried out. Ionic liquids with different cations and anions combinations were chosen for investigations, moreover, as well as using multiwalled carbon nanotubes, which have been studied previously, other nanoparticles types were selected, namely boron nitride and graphite that have not been investigated in such manners, resulting in the deeper insight into the physical properties, in particular the heat capacity enhancement. The density of nanoparticles was determined based on the density measurements with the original empirical model proposed by Pak and Cho. Further mechanism of the heat capacity enhancement was also studied. Based on the previous reports on the simple molecular solvents-based nanofluids, the mechanism of the heat capacity enhancement of ionanofluids is probably driven by the existing interfacial nanolayering occurring on the surface of nanoparticles. The possibility of mesolayers overlapping, which was also proposed previously, was not found to be present in the present study. Moreover, the heat capacity enhancement is determined by the type of nanoparticles, instead of type of ionic liquid. As a bridge to the industrial application of such results, the heat capacity enhancement as a function of temperature showed a high improvement, even up to about 34% in case of graphite-doped ionanofluids, whereas different type of temperature profile of enhancements were presented.

**Abbreviations and symbols**

|  |  |
| --- | --- |
| IL(s) | Ionic Liquid(s) |
| NP(s) | Nanoparticle(s) |
| [C4C1Im][Dca] | 1-Butyl-3-methylimidazolium dicyanamide |
| [C4C1Im][NTf2] | 1-butyl-3-methylimidazolium bis(trifluoromethylsulfonyl)imide |
| [C4C1Pyrr][NTf2] | 1-Butyl-1-methylpyrrolidinium bis(trifluoromethylsulfonyl)imide |
| [C6C1Im][PF6] | 1-hexyl-3-methylimidazolium hexafluorophosphate (CAS: 304680-35-1) |
| [C2C1Im][C2SO4] | 1-ethyl-3-methylimidazolium ethylsulfate |
| MWCNT | Multiwalled carbon nanotubes |
| BN | Boron nitride |
| G | Graphite |
| *wm* | mass fraction |
| *ρ* | Density (kg m-3) |
| *T* | Temperature (K) |
| *ai* | *i*-regression parameter |
| δ*ai* | *i*-regression parameter error |
| *R*2 | Coefficient of determination |
| *VE* | Excess molar volume (m3 mol-1) |
| *M* | Molar mass (kg mol-1) |
| *x* | Mole fraction |
| *cp* | Specific isobaric heat capacity (J kg-1 mol-1) |
| *ϕNP* | Volume fraction |
| *m* | mass (kg) |
| *V* | Volume (m3) |
| AARD | Average absolute relative deviation (%) |
| RAD | Relative absolute deviation (%) |

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