Enhancement of Molten Nitrate Thermal Properties by Reduced Graphene Oxide and Graphene Quantum Dots

Esraa Hamdy, Laila Saad, Fuad Abulfotuh, Moataz Soliman, and Shaker Ebrahim

ABSTRACT: Eutectic molten salts are the most studied medium-high temperature thermal energy storage material due to their potential use in concentrated solar power plants. The aim of this work is to investigate the effect of using reduced graphene oxide (RGO) and graphene quantum dots (GQDs) on the thermal properties of eutectic molten salts. A binary nitrate eutectic mixture of NaNO$_3$ and KNO$_3$ was selected as a base material (BM) for nitrate/carbon-derivative composites. RGO and GQDs were individually mixed with the BM with different fractions ranged from 0.1 to 1.5 wt %. The results showed that RGO enhanced the thermal conductivity, heat of fusion, and total thermal energy storage capacity by 52.10%, 44.48%, and 10.44%, respectively. GQDs slightly improved the specific heat capacity for both solid and liquid phases by 2.53% and 3.13%, respectively. In addition, GQDs promoted the heat of fusion by 31.72% and raised the total TES capacity by 12.26%.

INTRODUCTION

Thermal energy storage (TES) is deemed a recent technology that can be adapted to store solar thermal energy during solar peak hours. TES is integrated with concentrating solar power to reduce carbon dioxide emission and solve the problem of reduction of fossil energy. TES is temporary thermal energy storage and can be classified according to the change in internal energy to sensible heat storage, latent heat storage, and thermochemical categories of heat storage systems. Sensible heat storage depends on the specific heat capacity of the materials, while the latent heat storage pertains to the phase change enthalpy. On the other hand, the thermochemical heat storage depends on adsorption/desorption or other chemical reactions and is more energetic than latent heat storage.

Thermal energy storage using phase change materials (PCMs) has a high thermal storage density with a moderate temperature variation. PCMs can release or absorb sufficient energy at phase transition to supply useful heat or cooling, respectively. PCMs store heat 5–14 times more than sensible heat storage materials, and the size of storage systems using PCMs can be small compared with that of the storage system based on sensible heat.

Molten salts are one of the PCMs that are commercially used for TES. Generally, the molten salts or ionic liquids are known to be in the solid phase at room temperature and atmospheric pressure and are converted to the liquid phase when thermal energy is transferred to them. The molten salts are maintained as liquid throughout the energy storage process and have suitable melting temperature, low cost, high heat capacity, and energy storage density. The melting temperature of molten salt consisted of sodium nitrate and potassium nitrate is about 220 °C. Despite these advantages and their thermophysical properties, the molten nitrates have low thermal conductivity. To address this problem, particles with high thermal conductivity are dispersed into the PCM. This mixture is called composite phase change material...
The effects of adding different nanoparticles (such as mica, SiO₂, Al₂O₃, TiO₂, SiO₂−Al₂O₃, carbon nanotubes, graphene, and fullerene) to molten salts to explore their influence on the thermophysical properties of PCMs were reported. The special 2D sheet structure of graphene can be considered a promising candidate for improving the thermal conductivity of molten salts. Graphene has good specific heat capacity with six atomic degrees of motion (three vibrational and three translational), which become energized and then store heat when temperature increases. Therefore, reduced graphene oxide (RGO) as a type of graphene structure is selected as an additive to the molten nitrates. Also, graphene quantum dots (GQDs) were chosen to study the effect of quantum confinement and high surface area on the thermal properties of molten nitrates. The main objective of this work is to improve the thermophysical properties of NaNO₃−KNO₃ molten salt by incorporation of RGO or GQDs. The effect of different ratios of RGO and GQDs to this molten nitrate salt on the specific heat capacity, heat of fusion, melting temperature, total TES, and thermal conductivity is investigated.

■ RESULTS AND DISCUSSION

Structural Characterization of CPCMs. Carbon nanostructures were used as additives to the NaNO₃−KNO₃ (60:40) binary eutectic mixture to enhance its thermophysical properties as a heat storage material. Raman spectroscopy was used to analyze and monitor the effects of carbon nanostructures (RGO and GQDs) on the structure of the BM, as shown in Figure 1. Figure 1a depicts that the RGO has an appreciable effect on the vibrational peaks of the BM. It could be observed that RGO reduces the intensity of the NaNO₃ peaks at 1069 and 187 cm⁻¹. At the same time, RGO increases the intensity of KNO₃ peaks at 1052 and 85 cm⁻¹. This phenomenon can be explained based on the formation of chemical bonding between RGO and the BM, which contributes to a higher molar ratio than KNO₃ in the BM. This chemical bonding suppresses the motions of NaNO₃, consequently reducing its vibrational peaks and partially enhances the motions of KNO₃. GQD nanoadditives did not cause a notable change in vibrational spectra positions of the BM, as shown in Figure 1b. This could be due to their small size and the special structure of the GQDs, which do not allow a bond to be formed between the quantum dots and the BM.

The crystalline structures of the prepared different nano-composites are studied by XRD. XRD diffraction patterns of RGO and GQDs dispersed in the BM are shown in Figure 2. The diffraction patterns of the BM indicate the formation of polycrystalline phases with high intensity peak at 28°. The addition of RGO and GQDs to the BM has a slight effect on the crystallinity of the polycrystalline phases of the BM. The diffraction peaks of the additives are covered by main phases of molten nitrate salts as the mass fractions of the additives are very small.

Morphological Investigation of CPCMs. The dispersion degree of carbon nanostructure additives in the BM is a key

![Figure 1. Raman spectra of (a) RGO–CPCM and the base material and (b) GQDs–CPCM and the base material.](https://dx.doi.org/10.1021/acsomega.0c01291)
factor that affects its thermophysical properties. SEM and TEM measurements were carried out to evaluate the degree of dispersion of RGO and GQDs in CPCMs. The TEM image in Figure 3f indicates that the average flake diameter of the RGO nanoparticles dispersed in the BM is $\sim 32$ nm. Images in 3a,b of RGO nanocomposites show higher roughness in the surface compared to the BM. At higher magnification, as shown in 3b,d, it is observed that graphene flakes are attached well to the BM. In addition, non-homogeneous aggregates of RGO can be observed in the form of clusters. From Figure 3a–d, it can be noted that at lower concentrations, RGO is not dispersed well enough in the BM compared to higher concentrations. At a higher concentration of RGO of 1.5 wt %, paths are observed between the RGO and the binary nitrate eutectic mixture. This layer forms continuous percolations paths, as shown in Figure 3e.

Figure 4 displays the dispersion of GQDs in the BM. The TEM image in Figure 4f shows that the average diameter of GQD nanoparticles dispersed in the BM is $\sim 8$ nm. It could be noted that lower concentrations of 0.5 wt % are not enough to disperse in a good manner, as shown in Figure 4a,b. Yellow circles indicate clusters formed by GQDs within the BM. At a higher concentration of 1.5 wt %, GQDs form smooth layers of nanoparticles around the BM, as shown in Figure 4d. At higher magnification as shown in Figure 4e, these layers are not
continuous. They consist of tiny spherical dots with an average size of 50 nm.

**Thermal Analysis of CPCMs.** Thermal analysis was conducted to study the effect of carbon nanostructure additives on thermal properties of the NaNO\textsubscript{3}−KNO\textsubscript{3} binary eutectic mixture. This analysis included determination of the heat of fusion, specific heat, total thermal energy storage, and thermal conductivity of the composites, and the results are presented in the following section.

**Effects of Carbon Nanostructures on Melting Point and Heat of Fusion.** To evaluate the effect of the carbon nanostructures on the phase-change curves, the heat of fusion and the melting point of all samples were estimated and measured from DSC curves and are reported in Table 1. Figure 5 presents the DSC curves of the pristine solar salt and the CPCM with different mass fractions of RGO and GQDs. It is observed that the carbon nanostructures induce a change in the shape of the heat flow curve of the base molten salt. The melting point is estimated by the cross point between the line extended from the baseline at the lower temperature side and the tangent line drawn at the maximum inclination point on the lower temperature side of the fusion peak.

Table 1 shows that the addition of carbon nanoadditives to the BM increases the heat of fusion of the composite. RGO shows an increase in the heat of fusion by 23.33, 24.98, 31.74, 36.96, and 44.48% at weight fractions of 0.1, 0.5, 0.75, 1.0, and 1.5 wt %, respectively. Graphene, which is a two-dimensional sheet structure, achieves the highest increase in the enthalpy of fusion, which could be attributed to the highest binding of RGO flakes to the BM, as shown in the SEM images. When the dispersion and surface area of carbon nanostructures increase, the intermolecular attraction and the surface energy of these binary molten nitrate molecules also enhance\textsuperscript{24} and these intermolecular forces within the CPCMs raise the latent heat of fusion.\textsuperscript{25} The dispersion of carbon nanostructures and the presence and size of agglomerates also affect the heat of fusion of the composites by changing the entropy of the medium.\textsuperscript{9} Consequently, the notable increase in the enthalpy of fusion means that carbon nanostructure additives participate in the phase change, and this increase is directly proportional to the percentage of RGO and GQDs. The optimum value of the heat of fusion obtained at 1.5 wt % RGO is 154.09 kJ/kg.

GQDs show an enhancement of the heat of fusion of the nanocomposites by 6.88, 11.77, 22.21, 29.28, and 31.72% for...
0.1, 0.5, 0.75, 1.0, and 1.5 wt %, respectively, and this behavior is similar to that obtained for RGO with the BM.

Despite the significant change in the heat of fusion of the nanocomposites, the melting temperature is almost constant, as most of the energy is used to break the molecular or atomic bonds of CPCMs by latent heat during phase transition. After the molecular bonds in the composites are broken, the molecules are moving (vibrating) at the same average speed as before, so their average kinetic energy remains the same, and thus, their Celsius temperature remains constant.

**Effects of Carbon Nanostructures on Specific Heat and Total TES Capacity.** Specific heat is another important parameter for qualifying materials for TES application. Different specific heat values are calculated ranging from 150 to 220 °C (where 220 °C is about the onset temperature) in the solid region and from 230 to 300 °C (where 230 °C is about the endset temperature) in the liquid region. Figure 6 illustrates the percentage of change in \( C_p \) with different concentrations of RGO and GQDs in solid and liquid regions, respectively. \( C_p,\text{eff} \) is the specific heat of the CPCM, and \( C_p,\text{o} \) is the specific heat of the BM. Detailed values of the specific heat of CPCM with different carbon nanostructures are shown in Table 2. It was noted that RGO reduces the specific heat of the molten salt down to 1.90, 2.53, 4.43, and 8.23% for the solid region at 0.5, 0.75, 1.0, and 1.5 wt %, respectively. In addition, RGO decreases the specific heat in the liquid region to 1.25,

| mass fractions (wt %) | RGO–CPCM | GQDs–CPCM |
|-----------------------|-----------|-----------|
|                       | heat of fusion (kJ/kg) | melting temperature (°C) | heat of fusion (kJ/kg) | melting temperature (°C) |
| 0.0                   | 106.65    | 223.78    | 106.65    | 223.78    |
| 0.1                   | 131.53    | 223.57    | 113.95    | 223.46    |
| 0.5                   | 133.29    | 223.46    | 119.2     | 223.95    |
| 0.75                  | 140.50    | 223.28    | 130.34    | 222.98    |
| 1.0                   | 148.69    | 222.98    | 137.88    | 222.80    |
| 1.5                   | 154.09    | 222.80    | 140.70    | 222.53    |

Figure 4. SEM images of GQDs dispersed in the binary eutectic mixture: (a and b) 0.5 wt % concentration and (c and d) 1.5 wt % concentration. (e) High-magnification image of the nanocomposites with a non-continuous layer. (f) TEM image of RGO.
1.88, 2.50, and 7.50% at 0.5, 0.75, 1.0, and 1.5 wt %, respectively.

Graphene has six atomic degrees of motion that become energized and then store heat when temperature increases. However, it is possible that these modes could be partly suppressed or their dispersion could be altered when graphene is in strong contact with a substrate (thus lowering the specific heat). The same behavior is observed with RGO–CPCMs as RGO binds well with the BM and forms a special layer, which limits the lattice vibrations (phonons) of the binary eutectic salt. At very low concentration of RGO (0.1 wt %), the specific heats in the solid region and liquid region are slightly enhanced by 1.90 and 1.88%, respectively. This could be attributed to the weak binding of the RGO compared to the higher concentrations.

GQDs show a different behavior, as the specific heat decreases for 0.1 and 0.5 wt % by 4.43 and 3.16% in the solid region and 4.38 and 3.75% for the liquid region, respectively. This could be attributed to the poor dispersion and cluster formation of GQDs at low concentrations. These clusters reduce the motion of the BM, subsequently lowering the specific heat. With increasing GQD concentration, the specific heat values start to increase. Specific heat values in the solid region are slightly enhanced by 1.27, 1.90, and 2.53% at 0.75, 1.0, and 1.5 wt %, respectively. Also, specific heat values in the liquid region are improved by 1.25, 1.88, and 3.13% at 0.75, 1.0, and 1.5 wt %, respectively. It has been reported that the specific heat capacity of a nanoparticle is enhanced when the size of the nanoparticle is decreased. As GQDs have a very small particle size, this leads to less constrained surface atoms of the lattice of the nanoparticle due to the less number of bonds. Since the bonds can be visualized to act like springs, the surface atoms vibrate at a lower natural frequency and higher amplitudes, resulting in higher surface energy and higher specific heat values.

The total TES capacity of the CPCMs with different carbon nanostructures and concentrations are calculated via the summation of sensible heat storage capacity and latent heat storage capacity, as summarized in Figure 7. It could be observed that GQDs have the best impact on improving the total TES capacity, scoring 12.26% at 1.5 wt %, followed by RGO, which achieves 10.44% at 1.0 wt %.

Effects of Carbon Nanostructures on Thermal Conductivity. One of the main disadvantages of molten nitrate salts is their relatively low thermal conductivity. In this section, thermal conductivity results of the CPCMs are presented. The detailed values of the nanocomposites’ thermal conductivity are summarized in Table 3. Figure 8 presents the effect of RGO and GQDs on the thermal conductivity of binary nitrate molten salt, where $\lambda_s$ is the thermal conductivity of CPCMs and $\lambda_o$ is the thermal conductivity of the BM.

It is observed that RGO has a good impact on thermal conductivity enhancement of the BM. The thermal conductivities are improved by 35.42, 37.50, 43.80, 50.00, and 52.10% at 0.1, 0.5, 0.75, 1.0, and 1.5 wt % RGO, respectively. This could be explained by two factors. First, the good dispersion of RGO in the BM, forming a special layer that acts as a thermally conductive network (also known as thermal percolation networks). Second, the distinct geometry of graphene has a 2D sheet structure material, providing a high heat flow through their sheets. Thus, the good binding of graphene flakes to the BM and the formation of a special layer provide an environment suitable for thermal percolation.

The 0D GQDs obstruct the thermal conductivity by 8.30, 33.30, 41.70, 72.90, and 79.20% at 0.1, 0.5, 0.75, 1.0, and 1.5 wt %, respectively. This reduction could be attributed to the formation of a non-continuous layer formed by the GQDs. These dots act as barriers that restrict energy carriers’ (phonons) movement, hence leading to thermal conductivity reduction. Also, the thermal boundary resistance, which presents between different aggregating 0D GQDs and the 3D BM, plays a role in the heat transfer reduction, in addition to the quantum confinement effect. Finally, it could be concluded that RGO is a promising additive to enhance the thermal conductivity of the BM.

CONCLUSIONS

The nanostructure of the carbon derivatives of RGO and GQDs had a different effect on the thermophysical properties of the NaNO₃ + KNO₃ (60:40) binary eutectic mixture. RGO, a 2D sheet structure, was the most effective additive to enhance both the thermal conductivity and the heat of fusion. The results indicated an increase by 52.10% and 44.48%, respectively. Specific heat capacity was reduced by 8.23% and 7.50% in solid and liquid regions for RGO–CPCMs, respectively. Hence, RGO enhanced the total TES capacity by only 10.44%. GQDs were the best additive to enhance the specific heat in both solid and liquid regions by 2.53% and 3.13%, respectively. GQDs also showed the highest degree of enhancement in total TES capacity by 12.26%.
EXPERIMENTAL SECTION

Materials. The molten salt used in this study was composed of a binary nitrate eutectic salt, known commercially as "solar salt." This solar salt is a mixture of NaNO₃ and KNO₃ in proportions of 60 and 40 mol %, respectively. The NaNO₃ and KNO₃ were purchased from ACROS Organics and Fisher Scientific, respectively, with a purity of more than 99%. Sodium dodecyl sulfate (SDS) was used as a surface-active agent to improve the dispersion of RGO and GQDs in the BM. SDS was purchased from Fisher Scientific with a purity of more than 99%.

METHODS

Preparation of Carbon Nanostructures. The GQDs were prepared by direct glucose pyrolysis. Two grams of glucose was added into a beaker and heated to 250 °C using a...
hot plate. Then, the glucose was liquefied after approximately 5 min, and the color of the liquid was changed from colorless to yellow and then orange in 20 min. The obtained orange liquid for preparing GQDs was added drop by drop into 100 mL of 12.5% ammonia solution under vigorous stirring. Then, the solution was heated at 70 °C for 3 h until the odor of ammonia disappeared and the solution was neutralized to pH 7.0 \(^{16}\).

**Graphene Nanoscale.** In order to prepare reduced graphene, first, 15 grams of monohydrate glucose was added to 166.6 mL of distilled water and ammonia solution was added dropwise till the pH reaches 10.5. It was then placed in a Teflon-lined autoclave and was heated in an oven to 200 °C for 4 h. Second, it was removed to be washed and filtered by distilled water. The slurry was then taken and dried in the vacuum oven for 8 h. Third, it was placed in a muffle furnace for 1 h under nitrogen gas at 600 °C. \(^{19}\)

**Preparation of Molten Nitrate Salt.** The binary nitrate salt base material (BM) with 60% NaNO\(_3\) and 40% KNO\(_3\) was prepared. Then, this mixture was treated in a vacuum oven at 150 °C for 3 h. The mixing of the salts was carried out in a glovebox under nitrogen, and small quantities were mixed uniformly using a mortar and a pestle. The molten nitrate salt was taken in a crucible, placed on a hot plate under N\(_2\) to avoid oxidation at higher temperatures, and then statically heated to melt at 250 °C for 2 h. The mixture became transparent during the melting process. After that, the molten salt was cooled to room temperature gradually and the solid phase formed was a white mass. Finally, the solid was ground to powder form and sealed inside the glovebox.

**Preparation of Composite Phase Change Materials.** As it has been explained previously, the material composed by nanoparticles dispersed within the BM is usually presented as a nanocomposite when the BM is in the solid state. Nano-composites can be obtained from two different synthesis methods: the two-step solution method (direct synthesis method) or liquid solution method and the stirring method, which requires melting the BM and dispersing nanoparticles in the liquid solution. \(^{28}\) Due to the relatively high melting temperature of the BM, the direct synthesis method was used. \(^{27}\) First, an amount of binary nitrate eutectic salt (original PCM) was inserted to a vacuum oven and dried at 150 °C to remove moisture for about 1.5 h. Second, SDS was added to the carbon nanostructure with a mass ratio of 1:1 to enhance the dispersion process. RGO or GQDs were added with different mass ratios of 0.1, 0.5, 0.75, 1.0, and 1.5% of the molten nitrate salt. Third, SDS was put into a crucible with deionized water and ultrasonically vibrated for about 30 min. Then, RGO or GQDs were added to SDS solution and ultrasonically vibrated for 1.5 h. These RGO or GQDs in the SDS solution were poured into the BM and ultrasonically vibrated for 3 h to form a uniform CPCM solution. The nanocomposites formed were dried by evaporating the water onto a hot plate. Finally, the solid powder nanocomposites were triturated in the crucible to obtain the fine powder of CPCM.

**Characterization Techniques.** A field emission scanning electron microscope (SEM, Joel JSM 5300, Japan) was used to analyze the morphology and the dispersion of RGO or GQDs in the BM. The morphology was carried out for each nanocomposite after DSC measurement. The nanocomposites were sputtered and coated with a thin layer of Au using a sputter-coating unit (JFC-110E). In addition, the micro-structures of RGO and GQDs/molten nitrate salt specimens were investigated using a high-resolution transmission electron microscope (HR-TEM). The sample was suspended by dispersing 10 mg of sample in 5 mL of ethanol and sonicated.

Binary nitrate salt, RGO/molten nitrate salt, and GQDs/molten nitrate salt nanocomposites were characterized by Raman spectra, which were recorded using a microscope equipped with triple monochromatic combined with a peltier cooler charge coupled device detector system. The spectra were obtained in back-scattering geometry with an argon laser of 514.5 nm line that was focused on the sample for an excitation of 2 mW. The phonon frequencies were acquired by fitting Lorentzian line shapes to the experimental peaks after background subtraction.

The XRD patterns of binary nitrate salt, RGO/molten nitrate salt, and GQDs/molten nitrate salt were obtained using an X-ray 7000 Shimadzu-Japan at room temperature.

| Ratios of RGO and GQDs | Thermal Conductivity of RGO (W m\(^{-1}\) K\(^{-1}\)) | Thermal Conductivity of GQDs (W m\(^{-1}\) K\(^{-1}\)) |
|-----------------------|-----------------------------------------------|-----------------------------------------------|
| 1.0 0.72 0.28         |                                               |                                               |
| 1.5 0.73 0.10         |                                               |                                               |

**Figure 8.** Thermal conductivity of RGO–CPCMs and GQDs–CPCMs.
The dry nanocomposites were tested using a differential scanning calorimeter (Perkin Elmer DSC6). These nanocomposites were inserted to the standard aluminum pans and heated in the range from 35 to 300 °C at 20° per minute under N2 at a flow rate of 20 mL/min and the temperature scale was calibrated by a reference indium. From the DSC thermogram analysis, the heat of fusion and melting temperature were calculated using the Pyris-6DSC software. In addition, the calculated by a reference indium. From the DSC thermogram the DSC curve, and (d

\[ \frac{\partial Q}{\partial T} = \frac{m \cdot \Delta T}{m \cdot \frac{\partial T}{\partial r}} \]  

where \( m \) is the sample mass, \( \frac{\partial Q}{\partial T} \) is the heat flux given by the DSC curve, and \( \frac{\partial T}{\partial r} \) is the heating rate.

Thermal conductivity was tested and measured using a hot disk analyzer with hot disk thermal constants analyzer software version 6.2.1. The tested prepared nanocomposites were compressed as compact disks with thicknesses not smaller than their radius.

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**Notes**

The authors declare no competing financial interest.

### ACKNOWLEDGMENTS

The research leading to these results have received funding from the European Union Seventh Framework Program (FP7/2007-2013) under grant agreement no. 608593.

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10. Acknowledgments

The authors declare no competing financial interest.

### ACKNOWLEDGMENTS

The research leading to these results have received funding from the European Union Seventh Framework Program (FP7/2007-2013) under grant agreement no. 608593.

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