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Synthesis and Characterization of Enhanced Binary Carbonate Molten Salts Using Hybrid Nanoparticles (Al_2O_3–ZnO)

Shoumik Joy, Md.Yasir Bhuiyan and A.S.M Sayem*

Department of Mechanical Engineering, Chittagong University of Engineering & Technology, Chattogram-4349, Bangladesh

ABSTRACT

Efficient thermal energy storage (TES) is necessary for the reliability of renewable energy systems, with molten salts recognized as effective high-temperature heat-transfer and storage media. This study examines the improvement of binary carbonate molten salts through the addition of Al_2O_3–ZnO hybrid nanoparticles produced via a one-step synthesis method. Two formulations have been created: a base mixture of Na_2CO_3 and K_2CO_3 (Sample 1) and a modified composition incorporating 0.5 wt% Al_2O_3–ZnO nanoparticles (Sample 2). The samples went through controlled stirring, evaporation, drying, and grinding, followed by comprehensive characterization utilizing Scanning Electron Microscopy (SEM), Fourier Transform Infrared Spectroscopy (FTIR), and Ultraviolet–Visible (UV–Vis) spectroscopy. The SEM analysis of Sample 2 demonstrated the presence of finer, needle-like structures characterized by an increased surface area and uniform dispersion of nanoparticles, which enhances heat conduction efficiency. FTIR confirms retention of the carbonate structure with strengthened metal–oxygen interactions, while Sample 2 shows up to 12–15% higher transmittance and an overall 6–8% increase relative to sample 1. UV–Vis results further reveal enhanced transmittance (up to 10–15%) accompanied by a marked 85–95% reduction in absorbance, indicating reduced agglomeration. The findings indicate that the addition of Al_2O_3–ZnO hybrid nanoparticles significantly improves the thermal stability, heat transfer properties, and energy-storage capacity of binary carbonate molten salts, highlighting their applicability in advanced solar-thermal thermal energy storage systems.

Keywords: Thermal Energy Storage, Binary Carbonate Molten Salts, Hybrid Nanoparticles, Nanofluid, and Characterization

1. Introduction

Effective energy storage is essential to controlling the variability related to renewable energy sources such as solar and wind. Thermal Energy Storage (TES) is recognized as a cost-effective and reliable method among current technologies. Molten salts are highly renowned for their excellent thermal stability and enhanced heat retention in Concentrated Solar Power (CSP) and other high-temperature energy systems [1].

Molten salts such as nitrates, carbonates, chlorides, and mixtures are promising candidates for high-temperature thermal energy storage. Improving their heat capacity enhances storage efficiency by reducing the required material volume and minimizing the size of the heat transfer system [2]. Adding nanoparticles to molten salts can also make them hold more heat. Since the initial reports of heat capacity increases in carbonate salt-based and chloride salt-based nanofluids in 2011, numerous investigations have documented the increased heat capacity of several types of molten salt-based nanofluids, like nitrate salts [3], carbonate salts [4] and chloride salts [5] can store more heat than regular molten salts. Nanofluids have emerged as a promising innovation in thermal engineering, providing opportunities to improve energy efficiency by boosting heat transfer capabilities in applications. Such improvements are vital for overcoming current environmental and energy challenges [6]. Mono nanofluids, while able to modify their thermophysical properties through nanoparticle concentration, often show limited stability and performance. Hybrid nanofluids that combine two or more types of nanoparticles have been developed to overcome these limitations, resulting in improved base fluid properties and enhanced heat transfer efficiency [7].

Debjyoti's research focused on adding 1 wt.% silica (SiO_2) nanoparticles into a eutectic mixture of lithium carbonate (Li_2CO_3) and potassium carbonate (K_2CO_3) at a 62:38 ratio to develop high-temperature nanofluids. The results indicated that the addition of silica nanoparticles increased the specific heat capacity by approximately 19% to 24% [8]. Binjian investigated the thermal characteristics of molten salt nanofluids and observed that the specific heat capacity showed a nonlinear increase with nanoparticle concentrations of 0.5%, 1.0%, and 1.5%, achieving a maximum enhancement of 38.7% at 1.0% concentration. This enhancement is connected with the formation of secondary nanostructures through the concentrated aggregation of alumina nanoparticles within the molten salt layer [9]. Mao Shuai synthesized a ternary carbonate base salt composed of 31.5% Na_2CO_3, 31.5% Li_2CO_3, and 37% K_2CO_3. Nanofluids were prepared by dispersing SiO_2 and Al_2O_3 nanoparticles of different sizes and concentrations via a two-step solution method. The addition of 1.0 wt% 30 nm SiO_2 nanoparticles resulted in an 83.5% enhancement in specific heat capacity in the solid phase, a 159.4% increase in the liquid phase, and a 20.8% increase in thermal conductivity relative to the pure salt mixture [10]. Tiznobaik examined molten salt eutectics (62:38 Li_2CO_3–K_2CO_3) enhanced with silicon dioxide nanoparticles of various sizes (5–60 nm). Modified differential scanning calorimetry identified a consistent 25% increase in specific heat capacity in nanoparticle-enhanced fluids compared to pure molten salt [11]. Patricia Andreu explored the effect of doping solar salt (60% $NaNO_3$ + 40% KNO_3) used in CSP plants with silica nanoparticles at different concentrations (0.5% to 2%). The study reported a maximum specific heat increase of 25.03% at an optimal nanoparticle concentration of 1 wt.% [12]. Sreeram and Dileep showed that the addition of about 10 vol% of organo-phosphorus-coated–coated zinc particles to an alkali chloride salt eutectic enhanced its volumetric thermal storage capacity by 15–34%, while also ensuring strong chemical stability and resistance to degradation during repeated thermal cycles [13]. Lixia found that the low specific heat capacity and thermal conductivity of molten salts limit their energy storage efficiency; however, the addition of small quantities of nanoparticles can enhance their thermal properties by more than 25%, thereby significantly improving the efficiency of CSP systems [14]. Wole-Osho and Eric C. carried out an experimental study on Al_2O_3–ZnO hybrid nanofluids, revealing that the mixing ratios of particles significantly impact their thermophysical properties, with viscosity increasing by up to as 96.37% [15].

The research aims to enhance the thermal properties of binary carbonate molten salts through the incorporation of Al_2O_3–ZnO hybrid nanoparticles, to overcome their low specific heat and stability limitations. The study analyzes morphology and nanoparticle dispersion to enhance heat transfer efficiency. This study investigates the lack of research on hybrid nanoparticle systems in carbonate salts and introduces a new approach for the development of high-performance materials designed for advanced thermal energy storage applications.

2. Methodology

2.1 Materials and Equipment

The materials and equipment utilized for the preparation and characterization of the binary carbonate molten salt samples are summarized in Table 1.

Table 1 Materials and Equipment

Sl	Chemical Components	Equipment Components
1	Na_2CO_3 (Sodium Carbonate)	Magnetic Stirrer
2	K_2CO_3 (Potassium Carbonate	Hot Plate
3	Al_2O_3 (Aluminum oxide Nanoparticles)	Oven
4	ZnO (Zinc Oxide Nanoparticles)	Mortar and Pestle
5	Distilled Water	Thermometer, Vial Tube

2.2 Sample Preparation

Two binary carbonate molten salt samples were synthesized for nanofluid production using the one-step synthesis method. Sample 1 comprised 6.2 g Na_2CO_3 and 3.8 g K_2CO_3 dissolved in 250 mL of distilled water, while sample 2 contained 90% of the salt masses with 0.5 wt% Al_2O_3 and 0.5 wt% ZnO nanoparticles. Both mixtures were stirred, evaporated at 160°C for 4 hours, dried at 110°C for 6 hours, and then ground into a fine powder for characterization. The schematic diagram illustrates the sequential steps of the one-step synthesis process.

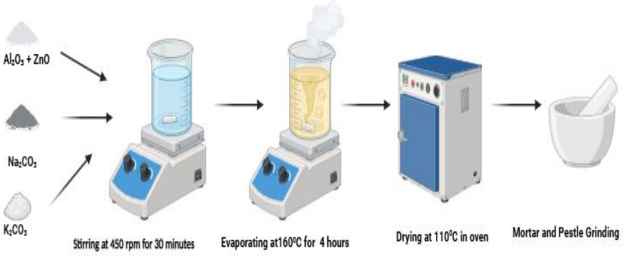

Fig. 1 Schematic diagram of one-step synthesis nano fluid preparation method

2.3 Preparation of Binary Carbonate Molten Salts

The experiment followed an eight-step procedure for preparing two binary carbonate molten salt samples.

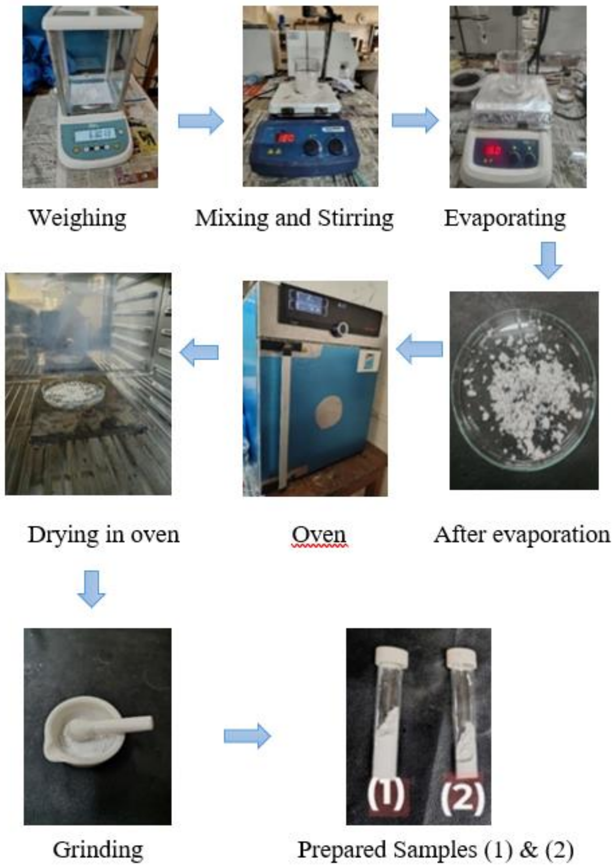

Fig. 2 Preparation process of binary carbonate molten salts

2.4 Characterization Techniques

2.4.1 Scanning Electron Microscopy (SEM)

SEM analysis using a JSM-7610FPlus microscope was used to analyze the surface morphology, particle distribution, and nanoparticle dispersion in samples 1 and 2, showing enhanced needle microstructures that increase surface area and allow for improved thermal performance.

Fig. 3 Scanning Electron Microscopy (SEM)

2.4.2 Fourier Transform Infrared Spectroscopy (FTIR)

The FTIR spectra for samples 1 and 2, obtained with a PerkinElmer spectrometer, exhibited distinct absorption bands indicative of functional groups and bonding structures. Differences in transmittance and peak intensity indicate enhanced molecular interactions, reflecting improved thermal stability and heat transfer efficiency in the hybrid composites.

Fig. 4 Fourier Transform Infrared Spectroscopy (FTIR)

2.4.3 UV-Vis Spectroscopy

UV–Vis spectroscopy was carried out with a PerkinElmer Lambda 365+ spectrometer to assess the optical transmittance and absorbance of Sample 1 and Sample 2, focusing on nanoparticle dispersion and optical stability.

Fig. 5 UV-Vis Spectroscopy

3. Results and Discussion

3.1 Morphology and Microstructure

To examine the microstructural features responsible for the enhanced thermal performance of binary carbonate nanofluids, SEM analysis was performed on both prepared samples. Figures 1(a) and 1(b) present the micrographs of nanofluids without hybrid nanoparticles at magnifications of 5 µm and 10 µm, whereas figures 2(a) and 2(b) display the corresponding images of samples containing Al_2O_3–ZnO hybrid nanoparticles, captured at 3000× and 4000× magnifications.

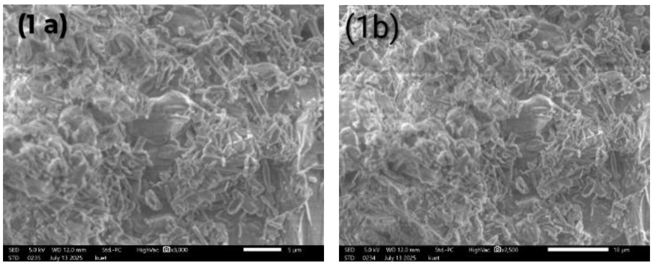

Fig. 6 SEM images of BC nanofluids at 3000× and 4000× magnification without hybrid nanoparticles. Sample (1a) at 5 µm and (1b) 10 µm

The addition of nanoparticles resulted in smaller particle sizes, a uniform distribution, enhanced surface interaction, and increased interfacial bonding within the molten salt matrix. The nanoparticle-enriched samples exhibited distinct needle-shaped formations, which are anticipated to enable directional heat flow and improve the material's thermal energy storage capacity. The morphological enhancements together enhance heat conduction, increasing specific heat capacity.

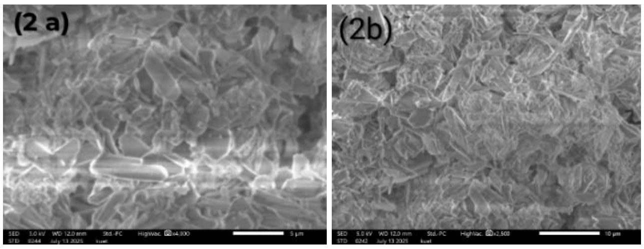

Fig. 7 SEM images of BC nanofluids at 3000× and 4000× magnification with hybrid nanoparticles. Sample (2a) at 5 µm and (2b) at 10 µm

3.2 Functional Group Interactions and Thermal Stability

The FTIR spectra of Sample 1 and Sample 2 (Fig. 8) display the expected carbonate vibration bands along with metal–oxygen bonding features, confirming that the base chemical structure remains intact after the introduction of nanoparticles. Compared with Sample 1, Sample 2 shows a consistently higher transmittance throughout the 500–3500 nm wavelength range. In the mid-infrared region (approximately 1800–2600 nm), the transmittance of Sample 2 increases by about 2–3%. A more pronounced difference appears in the 3000–3500 nm range, where Sample 2 exhibits roughly 12–15% higher transmittance. When

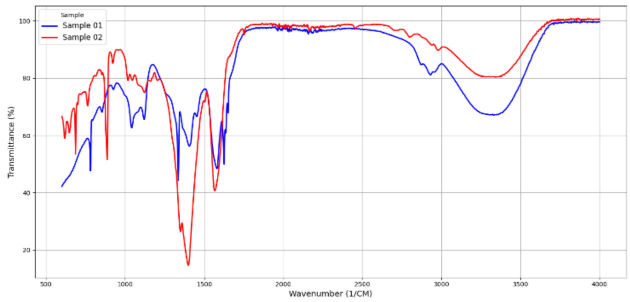

Fig. 8 FTIR spectra comparison of Sample 1 and Sample 2

considered over the full spectral range, Sample 2 demonstrates an overall 6–8% improvement in average transmittance, indicating reduced absorption behavior. This trend reflects improved nanoparticle dispersion and stronger nanoparticle–matrix interactions, leading to enhanced structural stability of the hybrid molten salt system.

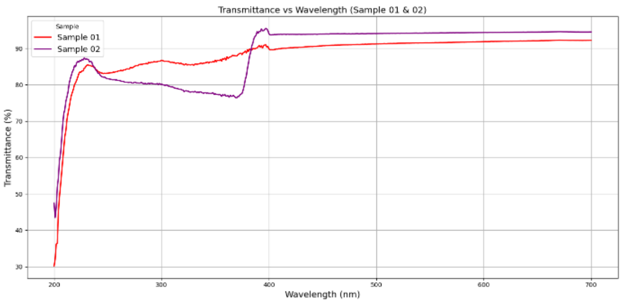

Fig. 9 Comparison of UV-Vis transmittance of sample 1 and sample 2

The optical behavior of the prepared samples was examined using UV–Vis spectroscopy over the 200–700 nm wavelength range (Fig. 9). Throughout most of the spectrum, Sample 2 displays higher transmittance than Sample 1, suggesting lower light absorption and more uniform dispersion of the hybrid nanoparticles within the molten salt. Within the visible region (approximately 400–700 nm), Sample 2 shows an average transmittance increase of about 3–4%. A stronger difference appears at shorter wavelengths (around 200–250 nm), where Sample 2 achieves up to 10–15% higher transmittance. When averaged across the full spectral range, Sample 2 exhibits an overall 4–6% improvement in transmittance, indicating reduced nanoparticle agglomeration. The smoother optical response of Sample 2 reflects improved structural uniformity and supports its enhanced thermal durability compared to the base salt system.

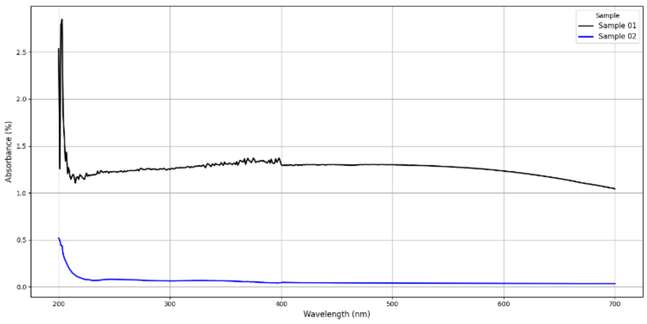

Fig. 10 Comparison of UV-Vis absorbance spectra of sample 1 and sample 2

Figure 10 presents the UV–Vis absorbance behavior of Samples 1 and 2 measured within the 200–700 nm wavelength range. Throughout the spectrum, Sample 1 maintains a noticeably higher absorbance level, while Sample 2 exhibits a much lower absorbance response. Within the visible region (approximately 400–700 nm), the absorbance of Sample 2 is reduced by about 85–95% relative to Sample 1. This marked reduction suggests improved nanoparticle dispersion and a significant decrease in agglomeration within the molten salt matrix. The smoother and consistently lower absorbance profile observed for Sample 2 indicates improved optical clarity and structural uniformity. These features imply enhanced chemical stability and support superior thermal durability of the hybrid nanoparticle-modified molten salt compared to the base system.

3.3 Chemical Stability

The FTIR and UV-Vis analyses indicate significant differences in the chemical stability between the two samples. FTIR spectra indicate that sample 2 displays stronger and more consistent absorption peaks, suggesting enhanced stability in nanoparticle-matrix interactions and reduced chemical alterations. UV-Vis analysis confirms this observation, as sample 2 exhibits increased transmittance and decreased absorbance, indicating reduced nanoparticle agglomeration and improved stability. Meanwhile, sample 1 exhibits heightened absorbance, indicating enhanced nanoparticle interactions and possible chemical changes within the molten salt matrix. The results indicate that sample 2 exhibits greater chemical stability than sample 1.

4. Conclusion

The study revealed a significant enhancement in the thermal characteristics of binary carbonate molten salts upon the incorporation of Al_2O_3–ZnO hybrid nanoparticles produced through a one-step synthesis method. The modified composite (Sample 2) exhibited finer, needle-like structures that increased surface area, consequently improving heat conduction. FTIR analysis showed improved metal–oxygen bonds and stable interactions between nanoparticles and the matrix. Simultaneously, UV–Vis spectra indicated enhanced transmittance and reduced absorbance, signifying improved dispersion and chemical stability. The results demonstrate that Al_2O_3–ZnO hybrid nanoparticles improve the thermal stability, heat transfer, and energy storage capacity of carbonate-based molten salts, making them appropriate for high-performance solar-thermal and thermal energy storage applications.

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