

## Research Article

# Investigation on Microstructure of Potassium Nitrate/Sodium Nitrate Composites for Solar Storage System

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The performance of the material is determined by its structure, i.e., the composition of the material elements of the atomic structure, the molecular structure, and atoms or molecules in space arrangement and the aggregation characteristics. The microstructure of the material has important implications for the material performance. In this paper, five phase change materials, potassium nitrate, sodium nitrate, and the composites of  $\text{KNO}_3\text{-NaNO}_3/\text{graphite}$  (3%, 6%, and 9%), have been studied by the experiment, and the aim is to improve the storage performance of the solar storage system. The results show that potassium nitrate and sodium nitrate are significantly different in microstructure, that is, potassium nitrate is a layered structure and the sodium nitrate is a network structure. And, the graphite as additives can be physically combined with the molten salts, which leads to a different performance and distribution in the mixture. Therefore, it is an effective method in improving the performance of molten salts by using graphite as the supporting material. The results can provide a reference to future choose for solar storage materials.

## 1. Introduction

In the past few decades, with the increasing consumption of fossil fuels and nonrenewable resources, energy problem and environmental issue are becoming more and more important for the worldwide research groups [1–4]. Thermal energy storage, which is an important branch of energy science and technology, has been applied to buildings, solar energy power, environmental projects, and other saving energy fields. However, the solar energy is available only during the day and unavailable for the night. How to store solar energy is an essential topic [5, 6]. In general, the storage system in the energy field has three contributions: sensible heat, latent heat, and chemical storage. Latent heat storage, which uses phase change materials (PCMs) as the heat transfer media or storage media, has more widely application due to their higher latent heat capacity [7, 8]. For example, solid-liquid PCMs and molten salt PCMs have already been utilized in different fields.

It is found that the phase change materials play a crucial role in thermal energy storage due to their storage capacity

and cost efficiency. Therefore, it is very significant and promising direction of the research to study the characteristic of the PCMs. Lv et al. [9] have detailed a study on the thermal properties of paraffin/kaolin PCM. The results showed that the paraffin/kaolin composite with largest particle size of kaolin has the highest thermal conductivity. Madathil et al. [10] have finished the study of preparation and characterization of molten salt. Their results indicated that the nanoparticle addition to molten salts was an efficient method to prepare thermally stable molten salt which can be used in concentrating the solar power plant. Karaipekli et al. [11] have investigated the thermal characteristics of expanded perlite/paraffin composite phase change material using carbon nanotubes. Li et al. [12] have chosen three types of nano- $\text{SiO}_2$  power to modify PX25, and the aim is to prevent paraffin leakage. The microstructure and particle size distribution of PX25 were analyzed using the SEM setup. Song et al. [13] have prepared the niobium carbide power by electrochemical reduction in molten salt. Wang et al. [14] have done the preparation and characterization of mullite whiskers by using a low-temperature molten salt method.

Zhong et al. [15] have synthesized three kinds of porous heterogeneous composite PCMs from expanded graphite and binary molten salts. Fernández and Pérez [16] have also studied the corrosion properties in ternary molten nitrate salts for a CSP plants. Ye et al. [17] have investigated the effect of Fe ion impurity on the corrosion behavior of alloys in molten salts. Reed et al. [18] have studied the synthesis, characterization, and short-term performance of C-Al and C-(Al, Si) systems for high energy density, high conductivity thermal storage materials.

In the above literatures, some researchers focus on the performance of the phase change materials and new materials. However, the investigation of microstructure for the thermal energy storage materials is limited. In the present study, the potassium nitrate, sodium nitrate, and the composites of  $\text{KNO}_3$ - $\text{NaNO}_3$ /graphite have been characterized by scanning electron microscope (SEM). The results can provide a reference to the future design and choice for solar storage materials.

## 2. Materials and Methods

**2.1. Experimental Setup.** The experimental setup is SEM (Figure 1). The main progress is as follows: (1) the SEM works by scanning the sample with a very fine electron beam to excite the secondary electrons on the surface of the sample; (2) the secondary electron is related to the incident angle of electron beam and the surface of the sample structure; (3) the secondary electron is collected by the probe body and shown the synchronization with electron beam scanning image; (4) the image reflects the surface structure of this specimen. And, the surface of specimens will be sprayed on a layer of metal particles after specimen fixation; (5) the heavy metals on the surface will send out a secondary electron signals when the specimen bombarded by the electron beam.

The aim of the SEM is to gain the physical and chemical properties of tested samples, i.e., composition distribution, crystal structure, electronic structure, internal electric field, and magnetic field. With a different information detector, it can achieve the selection test. If you need to get information from the material microstructure, you can choose the secondary electron, scattering electronic collection; if you want to get information on the chemical composition of substances, you can choose the X-ray collection.

**2.2. Experimental Materials.** In this study, the molten salt composites shown in Figure 2 have been prepared and tested in order to know the mechanism of the materials. Five kinds of materials are potassium nitrate, sodium nitrate, and the composites of  $\text{KNO}_3$ - $\text{NaNO}_3$ /graphite, which has a mass fraction of  $\text{KNO}_3$  (44.62%)/ $\text{NaNO}_3$  (52.38%)/graphite (3.00%),  $\text{KNO}_3$  (43.24%)/ $\text{NaNO}_3$  (50.76%)/graphite (6.00%), and  $\text{KNO}_3$  (41.86%)/ $\text{NaNO}_3$  (49.14%)/graphite (9.00%), respectively.

**2.3. Experimental Process.** This experimental process of the potassium nitrate, sodium nitrate, and their composites are similar. The detailed process is as follows: (1) getting in



FIGURE 1: Photo of SEM.



FIGURE 2: Photo of PCMs.

samples of the materials; (2) preparing SEM instruments; (3) getting water of the materials; (4) putting in the sample after preparing well, then start testing; (5) setting up parameters to obtain the required ratio of SEM images and saving the result; (6) changing the parameters to get another magnification SEM images and saving the result again; (7) repeating step 6 for the other required magnification SEM images.

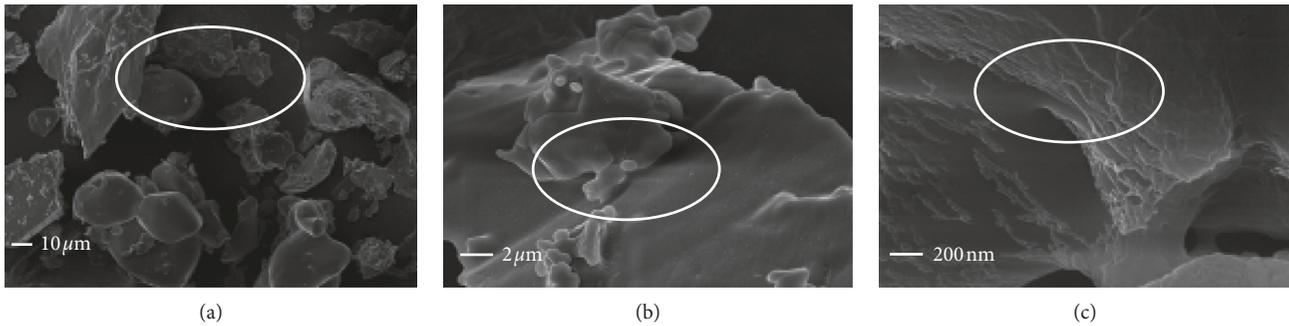
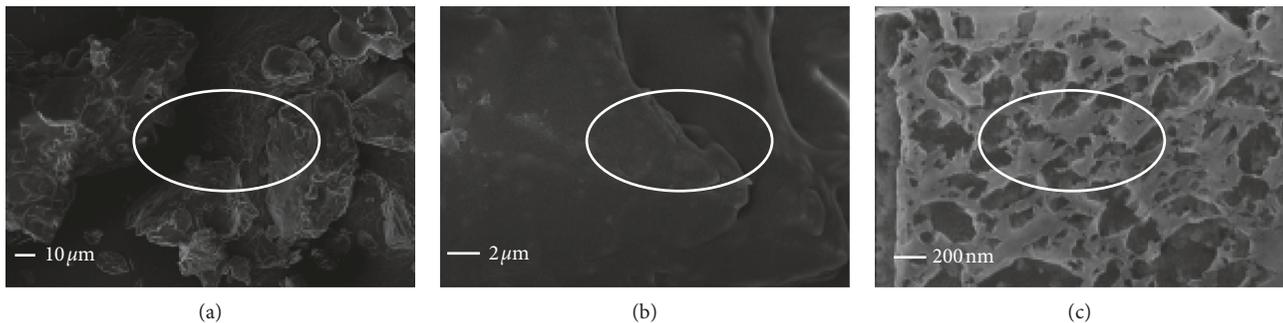
According to the above steps, the images of potassium nitrate, sodium nitrate, and their composites can be observed for different ratios of the SEM setup.

## 3. Results and Discussions

Figures 3 and 4 show the images of potassium nitrate and sodium nitrate under the three ratios of the SEM setup, respectively. In these figures, magnification of images is as follows: (a)  $1 \times 10^3$ , (b)  $1 \times 10^4$ , and (c)  $1 \times 10^5$ .

It can be seen from Figure 3(a) in  $1 \times 10^3$  magnification that  $\text{KNO}_3$  is under the condition of about 20 microns in diameter and tends more block or pie. With the increase in magnification (Figure 3(b)), we can observe smaller aggregates attached on the surface of the  $\text{KNO}_3$  and the surface of  $\text{KNO}_3$  is smooth. When the magnification raises to  $1 \times 10^5$  times (Figure 3(c)), it can be found that the image of  $\text{KNO}_3$  showed obvious stratification.

Also, it can be seen from Figure 4(a) that  $\text{NaNO}_3$  presents the block distribution and gathers together. In Figure 4(b), it is very clear that there are many convex parts

FIGURE 3: The images of  $\text{KNO}_3$  under different ratios of the SEM setup.FIGURE 4: The images of  $\text{NaNO}_3$  under different ratios of the SEM setup.

on  $\text{NaNO}_3$  surface. In Figure 4(c),  $\text{NaNO}_3$  has the largest images of the  $1 \times 10^5$  times and the  $\text{NaNO}_3$  surface has shown a lot of holes and the internal mesh structure with a loose reticular structure.

In Figure 5, it can be known that the particles of the image of  $\text{KNO}_3$  (44.62%)/ $\text{NaNO}_3$  (52.38%)/graphite (3.00%) present block and pie form in  $1 \times 10^3$  magnification. Particle size distribution is different in this figure, where part of the particles stick together and part of the particles disperse from each other. With the increase in multiples, the SEM image has a good agreement with separate image of  $\text{KNO}_3$  and  $\text{NaNO}_3$ . And, we found that, in a relatively complete plane, a layer of flocculent substances is spread, which is a flock net-like structure. And a small amount of graphite makes the surface of the structure of composite smoother. However, the graphite did not affect the structure of  $\text{KNO}_3$  and  $\text{NaNO}_3$  very much.

In Figures 3–5, with the increase in magnification, the size of the materials what we can see ranges from micrometers to nanometers. By comparing Figures 3 and 4, it is very obvious that  $\text{KNO}_3$  shows a more stable layered structure, and the aggregation degree is higher with larger particles. However, the  $\text{NaNO}_3$  shows a loose network structure, and aggregation degree is lower with the smaller particles. In Figure 5, the SEM image meets the common features shown in Figures 3 and 4, and  $\text{NaNO}_3$  is located at the surface of the  $\text{KNO}_3$ . Difference between  $\text{KNO}_3$  and  $\text{NaNO}_3$  in the SEM image is due to their different way of crystal gathering in the process of formation of the crystal. The  $\text{KNO}_3$  crystal aggregation degree is higher than that of

$\text{NaNO}_3$ 's. Also, the particle size is larger and presents a layered structure. So, the  $\text{KNO}_3$  crystals should be gathered in the form of overlap with the face and along the direction of crystal growth. However,  $\text{NaNO}_3$  crystal aggregation degree is relatively lower and shows a more angular shape and loose network structure, which conforms to the crystal accumulation fork model. In the process of  $\text{NaNO}_3$  crystal gathering, multiple  $\text{NaNO}_3$  crystals cross together and contradict each other, which changes  $\text{NaNO}_3$  crystal growth habit and also reduces the rate of the  $\text{NaNO}_3$  crystal growth rate.

After mixing  $\text{KNO}_3$ ,  $\text{NaNO}_3$ , and graphite with a certain proportion, the image of potassium nitrate/sodium nitrate composite is formed without a normal distribution. Sodium nitrate crystals are attached to the surface of the potassium nitrate crystal, which should be related to the structure of the two materials. In fact, the  $\text{NaNO}_3$  crystal structure is the loose network structure and the  $\text{KNO}_3$  crystal layer structure is stable. The  $\text{NaNO}_3$  crystal is easy to disperse into smaller crystal aggregations, and its structure, related to  $\text{KNO}_3$ , is not easy to disperse when in combination. So, it repairs the phenomenon that  $\text{NaNO}_3$  adhere to the surface of  $\text{KNO}_3$ .

Figures 6 and 7 show the images of  $\text{KNO}_3$  (43.24%)/ $\text{NaNO}_3$  (50.76%)/graphite (6.00%), and  $\text{KNO}_3$  (41.86%)/ $\text{NaNO}_3$  (49.14%)/graphite (9.00%) under three ratios of the SEM setup, respectively. In these figures, magnification of images is as follows: (a)  $1 \times 10^3$ , (b)  $1 \times 10^4$ , and (c)  $1 \times 10^5$ .

It can be seen from Figures 6 and 7(a) in  $1 \times 10^3$  magnification that the composite of  $\text{KNO}_3$ - $\text{NaNO}_3$ /graphite

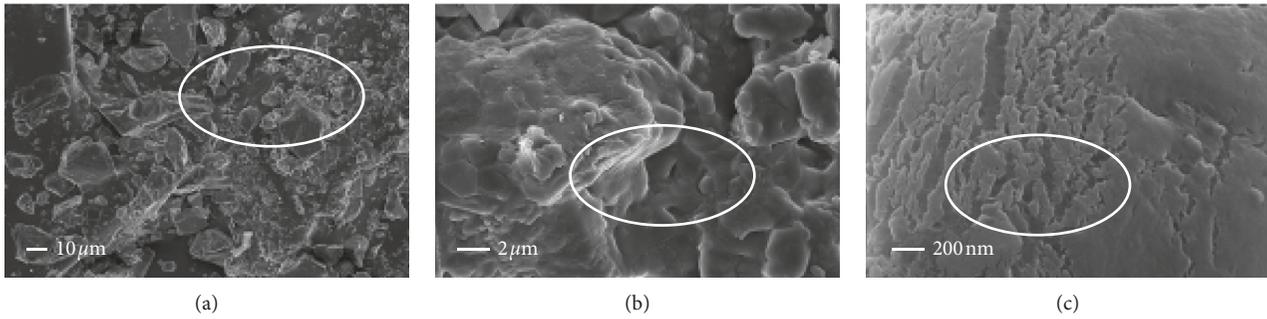


FIGURE 5: The images of  $\text{KNO}_3$  (44.62%)/ $\text{NaNO}_3$  (52.38%)/graphite (3.00%).

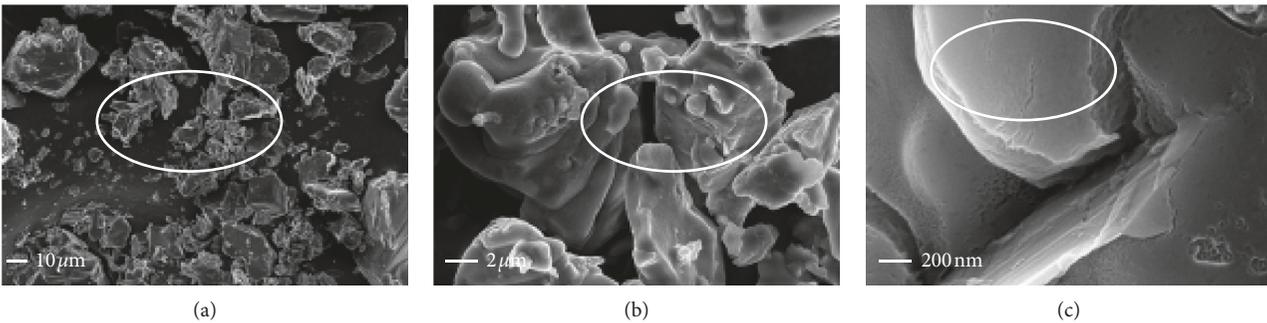


FIGURE 6: The images of  $\text{KNO}_3$  (43.24%)/ $\text{NaNO}_3$  (50.76%)/graphite (6.00%).

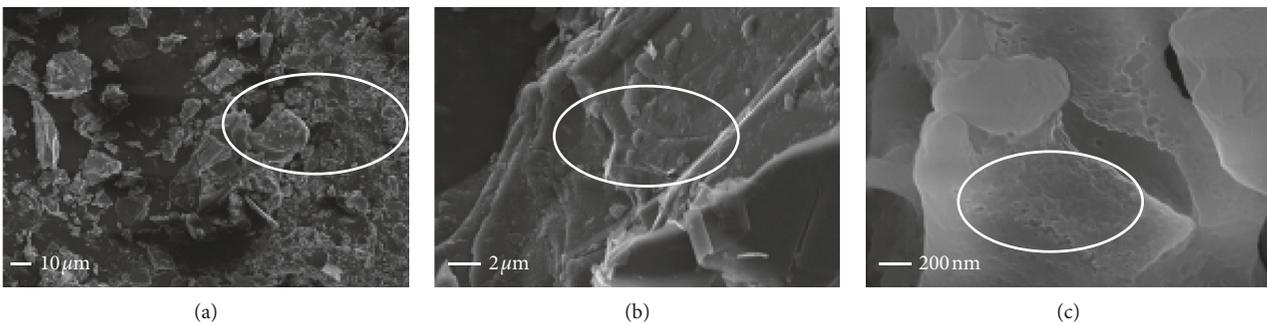


FIGURE 7: The images of  $\text{KNO}_3$  (41.86%)/ $\text{NaNO}_3$  (49.14%)/graphite (9.00%).

presents block and pie form, and with increase in the graphite, the shape of them becomes similar. With the increase in magnification (Figures 6 and 7(b)), it can be observed smaller particles stick together attached on the surface of the composites. And, the surface is relatively smoother in the image of  $\text{KNO}_3$  (41.86%)/ $\text{NaNO}_3$  (49.14%)/graphite (9.00%) in Figure 7(b). When the magnification raises to  $1 \times 10^5$  times shown in Figures 6 and 7(c), the surface of this composite showed an increasingly flat structure with the increasing graphite, and there is a less and less flock net-like structure.

From those figures, it is obviously found that the surface of  $\text{KNO}_3$ - $\text{NaNO}_3$ /graphite composites showed a tighter structure, and the loose network structure gradually disappears with the increase in graphite, which indicates the graphite and molten salt can be tightly integrated physically. Therefore, it is an effective method to improve the properties of molten salt by additive graphite.

#### 4. Conclusions

The structure of the PCMs, especially the microstructure, is of great significance to this material and its application in the all fields. Based on the SEM setup, the microstructure of the  $\text{KNO}_3$ ,  $\text{NaNO}_3$ , and the composites of  $\text{KNO}_3$ - $\text{NaNO}_3$ /graphite has been observed and analyzed. The following conclusions can be obtained:

- (1) The difference in  $\text{KNO}_3$  and  $\text{NaNO}_3$  is caused by their structural differences.  $\text{KNO}_3$  crystals in the process of gathering are collected in the crystal stacking way, and the  $\text{NaNO}_3$  crystals are gathered to fork model. Crystal accumulation mode differences led to the differences in them in shape and internal structure.
- (2) SEM images under different magnifications can be obtained by changing the scanning range of the

electron beam. The results showed that potassium nitrate and sodium nitrate were significantly different in microstructure, that is, potassium nitrate is layered structure and the sodium nitrate is a network structure. Structural differences lead to a difference in the performance and distribution characteristics in the mixture.

- (3) By comparing the microstructure of  $\text{KNO}_3\text{-NaNO}_3$  and the composite of  $\text{KNO}_3\text{-NaNO}_3/\text{graphite}$ , it is found that the graphite can be physically integrated with the molten salts. Therefore, it is an effective method to improve the performance of molten salts by using graphite as the supporting material.

## Data Availability

All data included in this study are available upon request by contacting the corresponding author.

## Conflicts of Interest

The authors declare that there are no conflicts of interest regarding the publication of this paper.

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