Preparation and Research on Expanded Perlite Composite Phase Change Material

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Abstract: To improve the application of phase change energy storage materials in residential geothermal heating, a composite phase change material with a phase change temperature range of 40℃ to 65℃ is designed. In this paper, two inorganic hydrated salt phase change materials Na2HPO4·12H2O and CH3COONa·3H2O are mixed in different proportions and make eutectic mixture of binary phase change materials, composite phase change material prepared using expanded perlite as carrier. The samples were analyzed by step cooling curve, DSC, SEM, XRD, the results show: the best ratio of Na2HPO4·12H2O to CH3COONa·3H2O is 60:40, it has no phase separation and the subcooling is 3.6℃, its phase transition temperature range is 31.48 ℃ ~ 72.36 ℃, and its phase transition enthalpy is 115.21 J/g. Expanded perlite composite phase change material has no leakage phenomenon, its phase change enthalpy is 109.34 J/g, and the phase change temperature range is 40.84℃ ~ 60.52 ℃, it meets the application standards of geothermal heating.

1. INTRODUCTION
Using phase change latent heat of phase change materials to storage and release energy is an effective way to improve energy efficiency[1]. Compared with organic phase change materials, inorganic phase change materials have a wider range of sources, lower prices, no irritating odors, and higher phase change latent heat and thermal conductivity[2], among them, inorganic hydrated salt phase change materials have attracted more attention due to their suitable phase transition temperature (about 0℃~150℃)[3]. But inorganic hydrated salt phase change materials are prone to leak during the solid-liquid transition process[4], and using porous material adsorption is an effective way to solve the leakage problem[5].

Expanded perlite (EP) is a white or light white porous material that is formed by the instant expansion of acidic volcanic glassy lava after high temperature roasting, and has good adsorption capacity [6]. Expanded perlite composite phase change material has good heat storage performance, construction operability and durability, etc., and plays a vital role in building energy saving [7]. The preparation methods are roughly divided into direct dipping method, microcapsule method and vacuum adsorption method [8]. In this paper, the adsorption of expanded perlite is used to fill the internal pores with phase change materials, the leakage of the phase change material during the phase transition is solved by means of packaging.
2. EXPERIMENT

2.1. Experimental materials
Na₂HPO₄·12H₂O, CH₃COONa·3H₂O, analytically pure; expanded perlite. Maintaining the Integrity of the Specifications.

2.2. Experimental process

2.2.1. Preparation of inorganic hydrated salt phase change materials
Na₂HPO₄·12H₂O and CH₃COONa·3H₂O are mixed according to different ratios to prepare nine groups of samples A-I, as shown in Table 1. The sample is heated in a 80 ℃ water bath, then the sample is allowed to cool naturally at room temperature. Collect the temperature data during the cooling process through the JK-XU multi-channel temperature inspection instrument and draw the step cooling curve, by comparing the size of supercooling and the phenomenon of stratification, the optimal ratio is preferred.

Table 1: Na₂HPO₄·12H₂O/CH₃COONa·3H₂O ratio table of the nine samples

| Sample | Content/ % |
|--------|------------|
| A      | 90/1       |
| B      | 0          |
| C      | 80/20      |
| D      | 70/30      |
| E      | 60/40      |
| F      | 50/50      |
| G      | 40/60      |
| H      | 30/70      |
| I      | 20/80      |
|        | 10/90      |

2.2.2. Preparation of composite phase change materials
The direct impregnation method is used to combine the expanded perlite with the binary phase change material. Weigh the binary phase change material according to the best proportion in the above test results, heat in a 80 ℃ water bath until completely melted, then sonicate in a 60 ℃ constant temperature water bath ultrasonic cleaner for 10 minutes to form a saturated solution. Then add an appropriate amount of expanded perlite to a saturated solution of the phase change material. In order to ensure that the carrier and the phase change material are mixed uniformly, the carrier and the phase change material are placed in a constant temperature electric blast drying oven at 60 ℃ for 8 hours, and then cooled at room temperature to obtain a composite phase change material.

2.3. Analysis and characterization
Temperature data in the cooling process were collected by JK-XU multi-channel temperature inspection instrument, draw a step cold curve; the microstructure of composite phase change materials before and after adsorption was observed by scanning electron microscope (SEM); x-ray diffraction (XRD) was used to compare and analyze the main components and structure of composite phase change materials before and after adsorption; the thermal properties of composite phase change materials before and after adsorption were compared by differential scanning calorimeter (DSC).

3. RESULTS AND DISCUSSION

3.1. Step cold curve result analysis
Inorganic hydrated salt phase change materials during cooling, the phase change latent heat is released with the formation of crystals, causing the temperature of the hydrated salt to rise, after maintaining a certain temperature for a period of time, the temperature began to drop again.

Figure 1 is the step cooling curve of the inorganic hydrated salt Na₂HPO₄·12H₂O-CH₃COONa·3H₂O binary phase change, as can be seen, different sizes of subcooling in A-I system. E, F, G, H, I systems have a large degree of subcooling, among them, the undercooling degree of H system reaches 21.9 ℃, and the undercooling degree of A, B, C, and D systems is relatively small. The minimum degree of subcooling in system D is 3.6 ℃.
Figure 2 is a photo of the nine groups of samples A through I after standing for 24 hours. It is obvious that there is no delamination in the D system and the distribution is relatively uniform. In summary, the D system is selected as the best ratio, and a composite phase change material is prepared.

![Figure 2](image-url)

**Fig. 2 Photograph of the binary PCMs of inorganic hydrated salt after standing for 24 hours**

3.2. **SEM result analysis**

(a), (b), and (c) in Figure 3 are scanning electron microscope images of expanded perlite, Na$_2$HPO$_4$·12H$_2$O·CH$_3$COONa·3H$_2$O binary phase change material, and expanded perlite composite phase change material. It can be seen from Figure 3(a), there are abundant pores and gullies in the expanded perlite, which is beneficial to the absorption of phase change materials and allows the phase change materials to fill each pore. The Na$_2$HPO$_4$·12H$_2$O·CH$_3$COONa·3H$_2$O binary phase change material in Figure 3(b) is well mixed, there is no aggregation of a single phase change material, and the dispersion is relatively uniform; In Figure 3(c), the binary phase change material is filled in the pores and gullies of the expanded perlite. It can be clearly seen that the interior of the expanded perlite is filled and the adsorption performance is good.

![Figure 3](image-url)

**Fig. 3 SEM images of expanded perlite, Na$_2$HPO$_4$·12H$_2$O·CH$_3$COONa·3H$_2$O binary phase change material, and expanded perlite composite phase change material.**
Fig. 3 SEM images before and after compounding

3.3. XRD test result analysis
Figure 4 shows the XRD diffraction patterns of CH₃COONa·3H₂O, Na₂HPO₄·12H₂O, expanded perlite and expanded perlite composite phase change materials. Comparing the four XRD diffraction lines of a, b, c, and d, it can be seen that there is an obvious diffraction peak at 56.083º in the a spectrum, but it is not reflected in the d spectrum, which is presumed to be caused by uneven sampling. In addition to this diffraction peak, the diffraction peaks in the three spectra of a, b, and c are all reflected in the d spectrum. It indicates that the expanded perlite and the binary phase change material are physically combined. The addition of the expanded perlite does not affect the crystal structure of the binary phase change material, and there is no chemical action at the same time.

Fig. 4 XRD images of phase change materials and carriers before and after compounding
3.4. Analysis of DSC test results
Figure 5(a) is the DSC curve of the binary phase change material, phase transition temperature range is 31.48 °C ~ 72.36 °C, phase change enthalpy reaches 115.21 J/g, however, the phase transition temperature does not meet the application standards for geothermal heating; Figure 5(b) is DSC curve of expanded perlite composite phase change material, meets the phase transition temperature range for geothermal heating (40 °C ~ 65 °C), phase change enthalpy is 109.34 J/g, this shows that the binary phase change material is well adsorbed in expanded perlite, and no leakage occurs.

![DSC curve before and after binary phase change material compounding](image)

Fig. 5 DSC curve before and after binary phase change material compounding

4. Conclusion
- An inorganic hydrated salt binary phase change material with good temperature regulation performance can be prepared by using inorganic hydrated salts Na₂HPO₄·12H₂O and CH₃COONa·3H₂O at a mass ratio of 60:40, no phase separation, subcooling is 3.6 °C, the phase transition temperature range is 31.48 °C ~ 72.36 °C, and the phase transition enthalpy reaches 115.21 J/g.
- By means of SEM and XRD analysis, the expanded perlite and binary phase change material are physically bonded, the addition of expanded perlite did not destroy the crystal structure of the binary phase change material, no chemical reaction and no new phase formation.
The phase transition temperature range of expanded perlite composite phase change material is 40.84℃~60.52℃ which meets the application standards. Expanded perlite plays a good role in encapsulation, and the phase change material after compounding has no leakage phenomenon, and has a good application prospect in residential building geothermal heating.

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