Preparation of Binary Mixed Inorganic Hydrate

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Abstract. In this paper, 54 binary mixed inorganic hydrates composed of Na₂SO₄·10H₂O, Na₂CO₃·10H₂O, CH₃COONa·3H₂O and MgSO₄·7H₂O were prepared, and try to research their reasonable and feasible preparation methods. The results show that the thermogravimetric ratio of binary mixtures varies with the composition and proportion of binary mixtures. There is evaporation of water in the mixtures preparation process, so appropriate deionized water should be added to supplement the evaporated water in the mixtures preparation of phase change materials.

1. Introduction
Phase change thermal storage technology has the great advantage constant temperature and the heat storage density and phase change thermal storage system can be supplied heat or discontinuous supply and demand conditions incompatible energy supply effectively solve problems such as time and space conflicts are one effective way to improve energy efficiency. Phase change material is a phase change key technology, thermal storage is essential to select a suitable material. A number of phase change material, a high temperature inorganic hydrated salts, a small viscosity, high specific heat capacity, low cost, has recently become a research hotspot of scholars.

Zeng et al. [1] prepared the composite heat storage material by molten impregnation method, which had good heat storage performance and the impregnation rate was 83.17%. Xu et al [2] The sodium acetate trihydrate experiment results show that the use of carboxymethyl cellulose as a thickening agent is preferably a phase change material results study, nine water sodium silicate, Na₄P₂O₇·10H₂O as a nucleating agent better. Liu et al. [3] By adding a certain amount of NaCl into Na₂SO₄·10H₂O, the phase transition temperature of Na₂SO₄·10H₂O was reduced to 27°C. The experimental results show that the addition of carboxymethyl cellulose (mass fraction 2%), and borax (mass fraction 3%) in Na₂SO₄·10H₂O can alleviate the problem of supercooling and phase separation. Leng et al [4] were prepared by vacuum suction expandable graphite/amorphous composite octahydrate phase change material, and a thermal property testing and characterization. Zhao et al. [5] prepared by mixing an inorganic hydrated salt systems (CaCl₂-MgCl₂-H₂O), and which can be used to obtain a low temperature phase change material greenhouse at night. Kong et al [6] found that the adsorption amount of ceramic calcium chloride hexahydrate phase change material is significantly larger than the organic n-decanoic acid. Karkri et al. [7] mixed high density polyethylene (HDPE) with microencapsulated paraffin wax to form shaped phase change materials. The results show that increasing paraffin content has obvious effect on enhancing latent heat and sensible heat of phase change material. Zhang et al. [8] prepared a binary fatty acid mixture with high latent heat value of
phase transformation, and its phase transformation temperature was 20-40°C. Bai et al. [9] used the physical adsorption method using a fatty acid had better storage of the phase change as plasterboard wall material. Wu et al. [10] prepared a phase change material by mixing stearic acid with sodium hydrogen phosphate dodecahydrate, which has a supercooling degree of about 3°C. After many cycles of experiments, it is proved that the hybrid phase change material has the characteristics of low undercooling, high latent heat and stable performance. Yin et al. [11] with sodium carbonate and potassium carbonate as raw materials, high latent heat, high melting point as the inorganic additive, static melt applications were prepared by the low transformation temperature, high phase transition latent heat of the molten salt mixture, and using the difference scanning calorimetry study their thermal characteristics thereof.

Of sodium sulfate decahydrate (Na₂SO₄ • 10H₂O), sodium carbonate decahydrate (Na₂CO₃ • 10H₂O), sodium acetate trihydrate (CH₃COONa • 3H₂O), magnesium sulfate heptahydrate (MgSO₄ • 7H₂O). Preparation of a composition of the present 54 different ligands inorganic hydrated salts heat binary mixture, than the comparative study of the relative change in the process of weight loss percentage, which is intended to work out reasonable and feasible manner of formulation.

2. Experimental methods

2.1 Raw materials and equipment

The phase change material used in the experiment include: sodium sulfate decahydrate (AR), Sinopharm Chemical Reagent Co., Ltd.; sodium carbonate decahydrate (AR), Sinopharm Chemical Reagent Co., Ltd.; sodium acetate trihydrate (AR), Sinopharm chemical reagent Co., Ltd.; magnesium sulfate heptahydrate (AR), Sinopharm chemical reagent Co., Ltd. The four kinds of inorganic salt hydrates are all analytical grade purity specification.

Laboratory equipment including: electronic balance (Sartorius-BSA2201), Beijing Sartorius Instrument Systems, Inc.; beaker (50 mL), Sinopharm Chemical Reagent Co., Ltd.; alcohol lamp (150 mL), Sinopharm Chemical Reagent Co., Ltd.; Asbestos Network (14 × 14 cm), Sinopharm chemical reagent Co., Ltd.; glass rod (6 × 300 mm), Sinopharm chemical reagent Co., Ltd. and so on.

2.2. Sample Preparation

By setting a mass ratio table prepared mixed phase change material. The total mass of the beaker and the sample was 20 g.

| No. | Name   | Na₂SO₄ • 10H₂O: Na₂CO₃ • 10H₂O | No. | Name   | Na₂SO₄ • 10H₂O: CH₃COONa • 3H₂O | No. | Name   | Na₂SO₄ • 10H₂O: MgSO₄ • 7H₂O |
|-----|--------|-------------------------------|-----|--------|-------------------------------|-----|--------|-------------------------------|
| 1   | 10% : 90% | 10% : 90%                     | 10  | 10% : 90% | 10% : 90%                   | 19  | 10% : 90% | 10% : 90%                   |
| 2   | 20% : 80% | 11% : 80%                     | 11  | 20% : 80% | 20% : 80%                   | 20  | 20% : 80% | 20% : 80%                   |
| 3   | 30% : 70% | 12% : 70%                     | 12  | 30% : 70% | 30% : 70%                   | 21  | 30% : 70% | 30% : 70%                   |
| 4   | 40% : 60% | 13% : 60%                     | 13  | 40% : 60% | 40% : 60%                   | 22  | 40% : 60% | 40% : 60%                   |
| 5   | 50% : 50% | 14% : 50%                     | 14  | 50% : 50% | 50% : 50%                   | 23  | 50% : 50% | 50% : 50%                   |
| 6   | 60% : 40% | 15% : 40%                     | 15  | 60% : 40% | 60% : 40%                   | 24  | 60% : 40% | 60% : 40%                   |
| 7   | 70% : 30% | 16% : 30%                     | 16  | 70% : 30% | 70% : 30%                   | 25  | 70% : 30% | 70% : 30%                   |
| 8   | 80% : 20% | 17% : 20%                     | 17  | 80% : 20% | 80% : 20%                   | 26  | 80% : 20% | 80% : 20%                   |
| 9   | 90% : 10% | 18% : 10%                     | 18  | 90% : 10% | 90% : 10%                   | 27  | 90% : 10% | 90% : 10%                   |
| 28  | 10% : 90% | 37% : 90%                     | 37  | 10% : 90% | 10% : 90%                   | 46  | 10% : 90% | 10% : 90%                   |
| 29  | 20% : 80% | 38% : 80%                     | 38  | 20% : 80% | 20% : 80%                   | 47  | 20% : 80% | 20% : 80%                   |
| 30  | 30% : 70% | 39% : 70%                     | 39  | 30% : 70% | 30% : 70%                   | 48  | 30% : 70% | 30% : 70%                   |
| 31  | 40% : 60% | 40% : 60%                     | 40  | 40% : 60% | 40% : 60%                   | 49  | 40% : 60% | 40% : 60%                   |
| 32  | 50% : 50% | 41% : 50%                     | 41  | 50% : 50% | 50% : 50%                   | 50  | 50% : 50% | 50% : 50%                   |
2.3 Experimental Procedure
The glass rod was put into a beaker of sample, together with the beaker placed on an electronic weighing scales, the read data in electronic balance to be stable and the number of records; Thereafter, the beaker containing the sample (with a glass rod) alcohol lamp is placed on a set of online asbestos, alcohol lamp lit by heating; during heating, with constant stirring using a glass rod test sample to the heated homogeneous sample, and the sample should be noted that should not be kept in the heating process boiling; binary mixture of the inorganic salt hydrate state observation beaker, heated to a molten state when it is heated to complete mixing, and the beaker was removed off alcohol lamp; the beaker was placed bench naturally cooled to room temperature (this process is approximately for 30 min); after completion of the cooling process, the beaker containing the test sample (with a glass rod) is placed on an electronic balance weighed again, the read electronic balance data to be shown and the number of stable recording.

In order to ensure and improve the accuracy of experimental data, the present study, the method of averaging several experiments, i.e., the mass ratio of each sample was repeated at least 3 times, and the average of several experiments, a final result of the experiment.

3. Experimental results and analysis

![Figure 1](image1.png)

**Figure 1. Na₂SO₄ • 10H₂O and Na₂CO₃ • 10H₂O mixture mass loss curve**

![Figure 2](image2.png)

**Figure 2. Na₂SO₄ • 10H₂O and CH₃COONa • 3H₂O mixture mass loss curve**
As it can be seen from Figure 1, with the increase of mass fraction of Na$_2$SO$_4$•10H$_2$O, Na$_2$SO$_4$•10H$_2$O and Na$_2$CO$_3$•10H$_2$O mass loss variation of a mixture of an upward trend after the first fall. The lowest and highest mass loss of samples were Sample No. 5 (50%: 50%) and 9 samples (90%: 10%), respectively, 0.139 g and 0.255 g. Sample No. 5 to No. 9 heat the specimen weight loss percentage of 0.7% and 1.28%, respectively (percent weight loss thermal mass equal to the initial mass after completion of the experiment is subtracted and then divided by the initial mass). Na$_2$SO$_4$•10H$_2$O and Na$_2$CO$_3$•10H$_2$O thermal reduction percentage range of the mixture is 0.70% – 1.28%.

As it can be seen from Figure 2, with the increase of Na$_2$SO$_4$•10H$_2$O mass fraction, mass loss Na$_2$SO$_4$•10H$_2$O CH$_3$COONa•3H$_2$O and a mixture of an upward trend after the first fall. Compared with Figure 1, Figure 2 decline curve becomes remarkable. The lowest and highest mass loss of samples were Sample No. 14 (50%: 50%) and Sample No. 10 (10%: 90%), respectively, 0.079 g and 0.193 g. Sample No. 10 heat 14 percent weight loss of the sample was 0.40% and 0.97%, respectively. Na$_2$SO$_4$•10H$_2$O and thermogravimetric CH$_3$COONa•3H$_2$O percentage range of 0.40% to 0.97%. Compared with a mixture of Na$_2$SO$_4$•10H$_2$O and Na$_2$CO$_3$•10H$_2$O is, Na$_2$SO$_4$•10H$_2$O heat and CH$_3$COONa•3H$_2$O mixture of weightlessness small.

As it can be seen from Figure 3, as the quality of Na$_2$SO$_4$•10H$_2$O, Na$_2$SO$_4$•10H$_2$O mass loss MgSO$_4$•7H$_2$O and the mixture continued to rise presentation, the highest and lowest mass loss of samples were Sample No. 19 (10%: 90%) and 27 samples (90%: 10%), respectively, 0.143 g and 0.349 g. Sample No. 19 and No. 27 sample thermal weight loss percentage of 1.75% and 0.72%, respectively. Thermogravimetric Na$_2$SO$_4$•10H$_2$O and MgSO$_4$•7H$_2$O mixture percentage range of 0.72% to 1.75%.

As can be seen from Figure 4, CH$_3$COONa•3H$_2$O and Na$_2$CO$_3$•10H$_2$O mixture mass loss trend seen in Figure 1, Na$_2$SO$_4$•10H$_2$O and Na$_2$CO$_3$•10H$_2$O mixture similar to the mixture with increasing
mass fraction CH₃COONa • 3H₂O, mass loss of the mixture changes tendency to decline slowly after the first rise. The lowest and highest mass loss of samples were Sample No. 32 (50%: 50%) and 36 samples (90%: 10%), respectively, 0.168 g and 0.267 g. Sample No. 32 heat and 36 percent weight loss of the sample were 0.84% and 1.34% CH₃COONa • 3H₂O and Na₂CO₃ • 10H₂O thermal reduction percentage range of the mixture is 0.84% ~ 1.34%.

As can be seen from Figure 5, with the increase of CH₃COONa • 3H₂O content quality score, the trend CH₃COONa • 3H₂O, MgSO₄ • 7H₂O mass loss of the mixture is also decreased and then increased. The lowest and highest mass loss of samples were Sample No. 41 (50%: 50%) and Sample No. 45 (90%: 10%), respectively, 0.206 g and 0.337 g. Heat the sample No. 45 Sample No. 41 is 1.03% and the percent weight loss of 1.69%, respectively. Thermogravimetric percentage ranges CH₃COONa • 3H₂O MgSO₄ • 7H₂O and the mixture was 1.03% to 1.69%.

As can be seen from Figure 6, with the increase in the mass fraction of MgSO₄ • 7H₂O, MgSO₄ • 7H₂O and Na₂CO₃ • 10H₂O mass loss of the mixture exhibit very slow upward trend, the lowest and highest mass loss of samples were Sample No. 46 (10%: 90%) and 54 samples (90%: 10%), respectively, 0.267 g and 0.286 g. Sample No. 46 heat 54 percent weight loss of the sample was 1.34% and 1.43%, respectively. MgSO₄ • 7H₂O and the hot mixture Na₂CO₃ • 10H₂O percent weight loss in the range of 1.34% -1.43%.

Figures 1-6 the mass loss curve 6 mixtures were compared discovery, CH₃COONa • 3H₂O and MgSO₄ • 7H₂O, Na₂SO₄ • 10H₂O CH₃COONa • 3H₂O and Na₂SO₄ • 10H₂O mixture and Na₂CO₃ • 10H₂O, CH₃COONa • 3H₂O and Na₂CO₃ • 10H₂O of law of mass loss curve trends are decreased and then increased, while when the content of the two components, the least loss of quality. Na2SO4 •
10H₂O and MgSO₄ • 7H₂O change the mixture mass loss curve showed a clear upward trend, MgSO₄ • 7H₂O and Na₂CO₃ • 10H₂O trend mixture mass loss curve of a gentle rise. Comparative found mass loss curve trend of the above mixture, in this experiment, MgSO₄ • 7H₂O was mixed with 3 different compound mass loss curve obtained three kinds of mixtures exhibit three different trends. This is because the alcohol lamp heating method in this experiment can not accurately control the heating temperature. With increasing temperature, MgSO₄ • 7H₂O will gradually form a new compound loses water of crystallization, which makes with increasing temperature, the salt hydrate component MgSO₄ • 7H₂O changing combinations, exhibits different quality trends.

4. Conclusion
(1) Thermal binary mixture of an inorganic hydrated salt with a percentage of weight loss and change in proportions of the different components exhibit different trends.
(2) The mixture prepared in this experiment, a mixture of Na₂SO₄ • 10H₂O and CH₃COONa • 3H₂O has the minimum thermogravimetric.
(3) The method of the present experiment the preparation of binary mixed inorganic hydrated salts, should be added to an appropriate amount of deionized water to replenish the water evaporated during heating. The deionized water quality may refer to various thermal analysis the percentage weight loss of the mixture of the above experimental results.

Acknowledgments
The project was supported by Science and Technology Key Program of Beijing Municipal Education Commission (KZ201710016011) and Beijing Scholar Program.

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