Water expansion performance standard for cement stabilized phosphogypsum soil

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Abstract: The application of cement stabilized phosphogypsum soil (CSPS) in highway engineering has great economic and social benefits. There are few corresponding classification standards for water swelling during long-term use and so a test method for water swelling of CSPS is designed. Unconfined compressive strength (UCS), dry shrinkage (DS), and temperature shrinkage (TS) of CSPS at three cement contents and eight phosphogypsum (PG) contents were measured. This paper analyzes the reasons for the poor early mechanical strength of CSPS. Based on the water expansion tests, ultimate expansion without load ($\delta_e$) is proposed as the evaluation index for water expansion. Combined with the relationship between the shear strength of the CSPS after the termination of water expansion, $\delta_e$, and PG content, a water swelling classification standard for CSPS is established. The results show that $\delta_e$ is negatively correlated with cement content, and positively correlated with PG content. In combination with direct shear test results, CSPS is divided into mild water expansion ($0 < \delta_e \leq 15$), moderate water expansion ($15 < \delta_e \leq 80$), and severe water expansion ($\delta_e > 80$).

1. Introduction
Phosphogypsum (PG), a solid waste, is a by-product of phosphoric acid production by reaction of phospholime and sulfuric acid in a chemical plant[1]. According to statistics, 4.5 to 5.5 tons of dry base PG will be produced from 1 ton of wet process phosphoric acid production[2][3]. The annual production of PG is about 75 million tons, but its use rate is low, and storage occupies significant land resources. Phosphogypsum is often combined with inorganic binder to treat subgrade soil in engineering practice[4]. PG stabilized materials have more research on strength[5][6]. However, PG stabilized materials will expand under the action of external water, causing uneven arching and other flaws in road structures.

The potential economic and social benefits of PG use in engineering can not only solve storage problems, but also reduce the amount of inorganic binder needed. A lot of research has occurred on the properties of PG stabilized materials. Parreira et al. studied the influence of cement type, content, and compaction on unconfined compressive strength (UCS) and expansion of cement stabilized PG and showed that the main factors affecting strength are cement content and compaction[7]. Silva et al. applied a mixture of hemihydrate PG, cement, and fine-grained laterite to an asphalt surface mixture to
evaluate the influence of hemihydrate PG content on strength and durability of the mixture[8]. Mashifana used citric acid, oxalic acid, sodium carbonate, sodium bicarbonate, and four other chemical reagents to treat PG, mixed the treated PG with lime and fly ash, and studied the influence of curing temperature and PG content on the UCS of the mixture. UCS was the highest under the conditions of higher curing temperature and 30% PG content[9]. Dong et al. studied the mechanical strength of two PG stabilized ash materials. The pozzolanic reaction was considered the basis for strength formation of the two PG stabilized materials. The hydration product formed ettringite and filled gaps in the materials. Lime fly ash PG stabilized materials have higher strength, but the strength will be reduced if the PG content is too high[10].

Liu et al. added two kinds of modified PG to persulfate cement and found that UCS of the persulfate cement improved, and the addition of modified PG makes the ettringite hydration product more stable[11]. Xu et al. used fly ash, PG, and calcium carbide slag as curing agents to study curing agent effects on soil, and obtained optimal proportions of curing agents corresponding to each stage in curing process. When the proportions of fly ash, PG, and calcium carbide slag are 21.6:26.8:51.6, the solidified soil has both early strength and late strength, and the proportion is suitable for the hydrate reaction process[12]. Li et al. used sodium silicate to improve cement stabilized PG as a pavement base, and found that when the amount of sodium silicate dissolved in water is 2%–4%, the UCS, water stability, and DS of the cement stabilized PG mixture improved[13].

Expansion of PG stabilized materials has also been studied. Sun et al. studied the influence of blast furnace slag, fly ash, and lime on impurities and expansion of PG based cementitious materials. Addition of these materials reduces the expansion of mortar, and the optimal content of the three materials is about 4% of the weight of PG[14]. Zhang used lime and cement to stabilize DM-PG. The effects of stabilizer type, content, and curing time on CBR and linear expansion of DM-PG were studied. A stable DM-PG blend has high CBR and medium linear expansion[15]. De Rezende et al. studied the application of dihydrate PG and hemihydrate PG mixed with cement and lime in road engineering. Compared with dihydrate PG, hemihydrate PG in cement and lime mixture can reduce swelling and has better mechanical properties[16]. Zhou et al. used PG in road base. Compared with cement stabilized macadam, the UCS and splitting strength of cement stabilized PG macadam at each age are higher, and the resilient modulus increases slowly. At the same time, cement stabilized PG macadam exhibits micro expansion, which can offset deformation caused by internal shrinkage stress, and has good crack resistance[17].

In summary, existing research focuses on mechanical and roadway performance of PG stabilized materials, with less research on water expansion performance. In view of this, the main objectives of this study are to evaluate the water expansion performance of CSPS, and recommend evaluation criteria. Therefore, the mechanical, shrinkage, and water expansion properties of CSPS with three cement contents and eight PG contents were tested. Based on the results of water expansion testing, the evaluation index $\delta$ of CSPS is proposed, and direct shear tests are carried out on specimens after water expansion ceases. The water expansion evaluation standard of CSPS is established according to the relationship curve between CSPS shear strength after water expansion ceases and $\delta$. The results have positive significance for the popularization and application of PG in road construction.
2. Materials and Methods

2.1. Materials
Composite Portland cement, graded 42.5R, was used as the stabilizing material for PG soil. The loess soil is taken from typical local areas. The main technical indexes are shown in Table 1. PG was obtained from Weinan City, Shaanxi Province, China, in gray powder form. Its composition is tested according to specification GB/T 23456—2018[21], as tabulated in Table 2.

| Type | Natural moisture content(%) | Proportion (g/cm³) | Plastic limit (%) | Liquid limit (%) | Optimum water content(%) | Maximum dry density(%) |
|------|-----------------------------|-------------------|------------------|-----------------|------------------------|-----------------------|
| Result | 8.8 | 2.68 | 22.97 | 26.98 | 15.1 | 1.76 |

Table 2. Phosphogypsum chemical properties.

| Type | CaSO₄•2H₂O | SO₃ | Al₂O₃ | SiO₂ | P₂O₅ | F⁻ | Fe₂O₃ | MgO |
|------|-------------|-----|-------|------|------|----|-------|-----|
| Mass percentage(%) | 63.35 | 32.11 | 0.11 | 0.72 | 2.35 | 0.053 | 0.085 | 0.02 |

2.2. Test Method

2.2.1. Compaction test
Based on previous experience of the research group, the cement contents were set at 3.0%, 4.0%, and 5.0%; and PG contents at 0%, 2%, 4%, 6%, 8%, 10%, 12%, and 15%. The compaction and UCS tests were conducted according to the current Chinese specification JTG E51-2009[22]. The maximum dry density (MDD) and optimum water content (OWC) were determined by the heavy compaction test. Cement and water were added in turn to the PG. The loose CSPS was poured into a cylindrical mold with inner diameter of 152 mm and height of 170 mm. The compaction process was conducted in three layers and the compaction number of each layer was 98 times. After several tests on five different water contents, the MDD and OWC at a given cement content were calculated based on the quadratic parabolic curve of dry density versus water content.

2.2.2. UCS test
The UCS test was performed on cylindrical specimens with diameter of 50 mm and height of 50 mm. Specimens were compacted at the determined OWC. Before testing they were first cured for six days at 20±2°C and relative humidity above 95%. Then, they were soaked in water for one day. The UCS was tested by a press, and the loading rate of the instrument was 1 mm/min.

2.2.3. DS test
The DS test setup was composed of a static resistance strain surveying instrument and a high-low temperature testing chamber, as shown in figure 1. According to the MDD and OWC of CSPS with different mix proportions in Table 3, a beam specimen 100 mm × 100 mm × 400 mm was formed under static pressure. The mixture of the same mix ratio should be molded into 6 specimens. The pieces are kept in standard conditions for 7 days under the condition of 20°C and humidity above 95%, and soaked in water for 24 hours on the seventh day. The temperature of the stability chamber is controlled at 20°C±1°C and the relative humidity is controlled at 60%±5%. A strain gauge automatically records dry shrinkage strain data until the moisture content of the specimen reaches a constant value.
2.2.4. **TS test**

TS testing is carried out beam samples prepared according to Table 3 in the high-low temperature chamber with an adhesive foil strain gauge, as shown in figure 2. Three specimens of each mix proportion were formed. The specimens cured for 7 days at 20°C and relative humidity above 95%, and soaked in water for 24 hours on the seventh day. After soaking in water for 24 hours, the specimens are put into an oven and baked for 10–12 hours to constant mass. After roughing a side of the specimen with sandpaper, the strain gauge shall be pasted with 502 glue, and a lead wire shall be connected. The other end of the lead wire shall be connected to the static strain gauge. The stability chamber temperature was reduced from 40°C to -20°C at 0.5°C/min. The chamber maintained a constant temperature for three hours. The temperature and strain variations were automatically measured.

2.2.5. **Water expansion test**

Calcium sulfate dihydrate in PG reacts with the main components and hydration products of cement to form ettringite and other new products, resulting in volume expansion under the action of the external water environment[23]. In this paper, the influence of external water expansion on the stability of CSPS is studied. The main equipment includes a press, WZ-2 dilatometer, and dial indicator. The test setup is shown in figure 3. The specific steps are as follows:

1. After mixing eight PG samples with soil and OWC, put them into bags and simmer for 12 h. Mix them evenly with cement contents of 3%, 4% and 5%.

2. Cylindrical specimens with diameter of 100 mm and height of 100 mm, were compacted to 97% by static pressing. A ring cutter with inner diameter of 61.8 mm and height of 20 mm was used for sampling.

3. Put the ring cutter and the specimens into the WZ-2 dilatometer with the blunt mouth downward, place the piston plate with holes on the top surface of the specimens, align the piston center, install the dial indicator, inject an appropriate amount of water into the dilatometer to keep the water level with the bottom surface of the specimens, and start the water expansion test.

4. Keep the water surface level with the bottom of the sample constantly, and record the dial indicator reading every 24 hours until the sample stops expanding.
2.2.6. Direct shear test
Cheng’s research on expansive soil strength parameters showed that shear strength can better reflect the strength characteristics of soil expansion termination based on triaxial or direct shear testing[24]. According to the fast shear test method of clayey soil, a strain controlled direct shear apparatus is adopted, and the shear rate is set at 0.8 mm/min. Specimens are taken out of the dilatometer after water expansion ceased and put into the direct shear apparatus, and the transmission device is installed. Set the vertical axial force P as 100 KPa, 200 KPa, 300 KPa, and 400 KPa, record the dynamometer reading, calculate the shear strength under different vertical axial forces, and draw the curve of vertical axial force versus shear strength.

3. Results and discussion

3.1. Compaction Properties
Taking the 3% cement as an example, the MDD and OWC of CSPS with different PG contents are given in Table 3. Under the same cement content, the OWC linearly increases with the PG content for CSPS. However, MDD is the opposite. With increasing PG content, the proportion of loess decreased. However, the density of PG is lower than that of loess, resulting in decreasing MDD with increasing PG content.

Table 3 Compaction test results.

| Cement content(%) | PG content(%) | MDD(g/cm³) | OWC(%) |
|-------------------|--------------|------------|--------|
| 0                 | 1.885        | 16.3       |
| 2                 | 1.864        | 16.5       |
| 4                 | 1.844        | 16.6       |
| 6                 | 1.828        | 16.8       |
| 8                 | 1.816        | 17.1       |
| 10                | 1.804        | 17.3       |
| 12                | 1.797        | 17.6       |
| 15                | 1.789        | 17.9       |

3.2. Compressive Strength
The variation of compressive strength with different cement contents is the same. The relationship between compressive strength of CSPS with 5% cement content, PG content, and curing age is shown in figure4. The compressive strength of CSPS is lower than that of cement stabilized soil. However, under the same cement content, the compressive strength of CSPS increases with the increase of PG content and age, and the increasing trend is basically the same. For the CSPS with 5% cement content, when the PG content is 0%, the compressive strength of 14d, 38d, and 90d increases by 9.9%, 17.9% and 48.8% compared with 7d, respectively, when the PG content is 15%, the compressive strength of 14d, 38d and 90d increased by 13.5%, 26.2%, and 58.9%, compared with 7d, respectively. The lower
strength of CPSS can be explained by the retarding effect of PG on cement. This effect leads to incomplete hydration of cement, which makes the cement calcium silicate hydrate (C-S-H) hydration product develop to type I, with low density flocculant and more pore structure, while the crystallization to type II C-S-H with high density solid and less pore structure is not high[10]. In addition, calcium sulfate dihydrate reacts with tricalcium aluminate and its hydration products to produce a series of physical and chemical reactions. Ettringite has expansibility. With the passage of time, ettringite can continue to react with calcium aluminate hydrate to produce single sulfur type calcium sulphoaluminate \( (3\text{CaO} \cdot \text{Al}_2\text{O}_3 \cdot 3\text{CaSO}_4 \cdot 12\text{H}_2\text{O}) \), which is the reason for the later strength improvement of CSPS.

3.3. DS Resistance

The variation of water loss (WLC) with time is consistent for different PG contents. Variations of WLC with time at 4% PG content are shown in figure 5. The WLC of the test specimens increased with time. The WLC is greater at the initial stage. After 10 days, the growth rate stabilizes. It is suggested that wet curing should be carried out within 10 days after construction to prevent excessive water loss and premature shrinkage cracks. WLC of the test specimens increased with increasing PG content. This phenomenon can be explained by the sensitivity of PG to water. Water absorption of samples with high PG content is greater than that of samples with low PG content. Six replications were conducted for each DS test and the average results were used for analysis. Variations of the DS coefficient with PG for different CSPS are shown in figure 6. The DS coefficient of CSPS increases with increasing cement content. Under the same cement content, the DS coefficient of stabilized soil decreases with increasing PG content. This is explained by the way that calcium sulfate dihydrate in the added PG reacts with hydration products of cement to produce ettringite. Ettringite exhibits micro expansion, which can effectively compensate for deformation caused by DS.

![Figure 4. Unconfined compressive strength test results.](image1)

![Figure 5. Variation of WLC with time.](image2)

![Figure 6. Variation of the DS coefficient with PG content.](image3)
3.4. TS Resistance
Variations of the TS coefficient in the CSPS are consistent with the variations in PG content. The TS coefficient of CSPS increases with decreasing temperature. The TS coefficient of CSPS peaks when the test temperature is -10°C. As the temperature continues to decrease, the TS coefficient decreases. The maximum TS coefficients of 3%, 4%, and 5% cement are 18.92, 19.02, and 18.09 με/℃, respectively. With increasing cement content, the hydration reaction of cement intensifies, and the TS stress changes greatly under the action of external temperature.

The variation of average TS coefficient of CSPS with PG content under different temperatures is shown in figure 7. The average TS coefficient decreases with increasing PG content. The maximum average TS coefficients of 0% and 15% PG contents decrease from 20.57 to 15.5 με/℃, a decrease of 24.6%. This is because of the micro expansion characteristics of ettringite formed from the hydration products of PG and cement, and its expansion force effectively offsets the shrinkage stress of stabilized soil caused by temperature changes.

![Figure 7. Variation of the average TS coefficient with PG content.](image)

3.5. Water expansion characteristics

3.5.1. Proposal of ultimate expansion without load
The ultimate expansion without load values for different PG and cement contents are shown in figure 8, indicating that time of final expansion of stabilized soil varies with PG content. The greater PG content, the longer the expansion time of CSPS. For engineering applications, it is more practical to study the final expansion of CSPS. We propose that the ultimate expansion without load, δe, is used as the evaluation index of water expansion of CSPS. The calculation formula is shown in Eq. (1).

\[ \delta_e = R_e - R_o \]  \hspace{1cm} (1)

Where \( \delta_e \) is the ultimate expansion of the specimen without load (0.01 mm), \( R_e \) is the dial indicator reading after the sample is fully expanded (0.01 mm), and \( R_o \) is the initial dial indicator reading (0.01 mm).
δ_ε increases with increasing PG content, which has a significant influence on δ_ε. The influence of cement content on δ_ε is small, and there is a negative correlation between them. The increase of cement content reduces the water expansion of CSPS. Under the action of external water environment, the main swelling product of CSPS is ettringite. When the content of PG is low, an appropriate amount of ettringite can supplement the pores between the CSPS and play a certain role in strength supplementation. As the content of PG continues to increase, δ_ε increases. At this time, excessive ettringite increases the expansibility of CSPS. Therefore, low amounts of PG should be used in CSPS.

3.5.2. Water expansion performance standard of CSPS
The shear strength results are shown in figure 9. Shear strength increases with increasing vertical axial force, and also shows a downward trend with increasing PG content under the same vertical axial force. When the PG content is 0% to 4%, the shear strength of the specimens changes little after water expansion ceases. At this time, the c of CSPS is greater than the expansion force, and the overall stability is high. When the PG content is 4% to 6%, the shear strength of CSPS decreases significantly and quickly. Shear strength tends to be stable when the PG content is 6% to 8%. When the PG content is 4% to 10%, the swelling force of stabilized soil after water expansion is greater than the overall cohesion, but damage is limited, and the stabilized soil still has a certain strength. Therefore, the first cut-off point for the proposed classification appears when the PG content is 4%, and the corresponding δ_ε of 3%, 4% and 5% cement content is 15, 14.8, and 13.2. After rounding, it is considered that the first cut-off point appears at 15. When PG content is more than 10%, the shear strength of stabilized soil decreases significantly and quickly again, and the friction angle and cohesion decrease obviously. At this time, the expansion force causes serious damage to the overall structure, and the corresponding δ_ε is greater than 80. Therefore, it is considered that the second cut-off point for the proposed classification appears when the content of PG is 10% and δ_ε is 80.
Taking $\delta_e$ as the evaluation index for the grading standard of CSPS after water expansion ceases is proposed. The CSPS is classified into mild water expansion, moderate water expansion, and severe water expansion, as tabulated in Table 4.

| PG content/(%) | $\delta_e/(0.01\text{mm})$ | Water expansion classification |
|---------------|-------------------|-----------------------------|
| 0~4           | $\delta_e \leq 15$ | mild water expansion        |
| 4~10          | $15 < \delta_e \leq 80$ | moderate water expansion    |
| 10~15         | $\delta_e > 80$   | severe water expansion      |

4. Conclusions
This paper presents an experimental evaluation of CSPS performance. Water expansion performance is recommended as the standard for CSPS classification. Some important observations and conclusions are as follows:

(1) The UCS test results for CSPS show that the retarding effect of PG makes the hydration reaction of cement incomplete, which leads to the early mechanical properties of CSPS being worse than cement stabilized soil. However, with increasing PG content, ettringite continues to react with calcium aluminate hydrate, and the formation of single sulfur calcium sulfoaluminate increases the later strength of CSPS.

(2) DS and TS test results for CSPS show that addition of PG can effectively improve the shrinkage performance of CSPS. Calcium sulfate dihydrate in PG reacts with hydration products of cement to produce ettringite. Ettringite has micro expansion characteristics, which can effectively compensate for the deformation caused by dry shrinkage.

(3) A test method for water expansion of CSPS is designed, and $\delta_e$ is proposed as the evaluation index of water expansion. The water expansion test results show that $\delta_e$ is negatively correlated with cement content, and positively correlated with PG content. A grading standard of water expansion of CSPS is established based on relationship curves of shear strength, $\delta_e$, and PG content of CSPS after water expansion, and CSPS is classified into mild water expansion ($0 < \delta_e \leq 15$), moderate water expansion ($15 < \delta_e \leq 80$) and severe water expansion ($\delta_e > 80$).

(4) In this paper, the classification index and standard of CSPS are only related to loess. The next step is to study other soils and gravel stabilized with PG, so as to improve the classification standard of PG stabilized materials.

Acknowledgements
This work is funded by the Natural Science Foundation of Shaanxi Province (2019JM-064).
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