Experimental Study on Mechanical Properties and Structural Characteristics of Modified Sludge in Foundation Pit

Shuren Wang^{*}, Jialin Wei, Xinyu Wang, Zhengsheng Zou

School of Civil Engineering, Henan Polytechnic University, Jiaozuo, 454003, China *Corresponding Author.

Abstract

To explore the efficient method of sludge modification, Ultra-fine Portland cement (UPC) was introduced as a sludge modifier regarding Ordinary Portland Cement (OPC) modified sludge as a reference. The mechanical properties and microstructural changes of UPC-modified sludge with different curing time and cement content were carried out by unconfined compressive strength (UCS), X-ray diffraction (XRD), mercury intrusion porosimetry (MIP), and scanning electron microscopy (SEM) tests. Results show that the UCS of UPC-modified sludge varies with curing time and cement content in the same way as that of OPCmodified sludge. However, compared with OPC-modified sludge, UPC has a higher sludge modification efficiency, and the UPC-modified sludge has greater compressive strength, significantly early-strength, and stronger resistance to deformation. The stress-strain curves of UPC-modified sludge present significant peak stresses, and which show a brittle failure mode. The combination of the hydration products calcium silicate hydrate (C-S-H) gels and ettringite (Aft) crystals are the essential reason for the improvement of the macroscopic strength of the modified sludge. In contrast to OPC, the UPC hydrates faster and more fully. The UPC-modified sludge can generate more hydration products under the same conditions, this is why that has high efficiency and early-strength. The conclusions obtained in this study can provide a reference for the similar engineering application of ultra-fine cement in modified sludge.

Keywords: Sludge; modification; ultra-fine Portland cement; pore structure; microscopic morphology

I. Introduction

There are deeply sludge layers widely distributed in the Yangtze River basin and coastal areas of China, which show many adverse effects on foundation support and urban underground space construction. Therefore, it is usually necessary to carry out the corresponding reinforcement treatment in the actual foundation engineering.

As an effective method to improve the engineering properties of soil, the curing agent modified soil has received extensive attention in the word [1]. Ordinary Portland Cement (OPC) is the most popular soil curing agent, it has many advantages such as excellent mechanical properties, low cost, and easy availability [2]. Nonetheless, related studies have shown that the high organic matter content in the sludge are very unfavorable to the strength formation of OPC-modified sludge, which leads to the less than ideal effect of OPC-modified sludge [3]. For improving the sludge modification effect, Wang et al. adopted alkaline admixture to dissolve the organic matter to enhance the sludge modification effect [4]. Although admixtures can effectively improve the effect of OPC modified sludge, there is usually an optimal limitation of the admixture content to achieve the desired effect of sludge modification. And admixtures also have some disadvantages, so it is difficult to apply the method of improving the effect of OPC-modified sludge by adding admixtures.

As well as OPC, UPC (Ultra-fine Portland Cement) is also an industrialized product, but compared with OPC, UPC has a larger specific surface area, faster hydration rate, higher strength, and better stability [5]. Reinhardt was the first to introduce the functions and applications of ultrafine cement [6]. UPC was often used as a grouting material for sand layer grouting to reinforcement [7]. Currently, a few scholars have broadened the application range of ultra-fine cement to the study of soil modification. Mollamahmutoglu and Avc studied the effect of slag-

based superfine cement (SSC) on the modification of clayey soil [8]. Zheng et al. used UPC to replace part of OPC as a composite curing agent to study the modification of mucky soil [9].

The existing studies have focused on looking for admixtures that are more adaptable to the properties of OPC and sludge itself based on the modification mechanism. However, there are few studies based on the influence of cement fineness on the effect of sludge modification. For this reason, this study investigates the mechanical properties and microscopic mechanism of UPC-modified sludge through a series of tests such as unconstrained compressive strength (UCS), X-ray diffraction (XRD), mercury intrusion porosimetry (MIP), and scanning electron microscopy (SEM) with OPC as the reference. The research results can lay a certain foundation for further research and engineering application of the ultra-fine cement in modified sludge.

II. Experimental Programs

2.1 Test materials

The sludge used in the test was taken from the foundation pit project of Yue Feng North Station of Fuzhou Metro Line 4, and the basic physical and mechanical properties of the sludge are shown in Table 1. The two types of cement are Panlongshan brand UPC with a specific surface area of 1180 m²/kg and Firm brand P·O42.5 OPC with a specific surface area of 357 m²/kg. The relative fractions of clay, silt and sand particles in the sludge were 22.5%, 76.2%, and 1.3%, respectively. The chemical composition of test materials are shown in Table 2. It can be seen that the main components of the sludge are SiO₂, Al₂O₃, and Fe₂O₃, and the main components of cement are both CaO, SiO₂, and Al₂O₃.

| Natural water content | Density (γ/kN·m ⁻³) | Specific gravity | Liquid limit [*] | Plastic limit | Plasticity index | Liquidity index (I _L) | Natural void | Organic matter ^{**} | pН | UCS (kPa) |
|-------------------------------|--|---------------------|------------------------------|------------------|-------------------------------|--------------------------------------|-----------------|---------------------------------|-----|--------------|
| (W ₀ / 78) 62.5 | 16.02 | 2.62 | (WL) 76) 58.4 | (WP/70) 38.5 | (1 _P / 76) 19.9 | 1.21 | 1.685 | 8.51 | 7.2 | 53.0 |
| note | * 17mm; ** Burning method (burning at 900 °C for 5 hours). | | | | | | | | | |

Table 1 Physical and mechanical properties of sludge

| Test | Chemical composition (%) | | | | | | | | | | | |
|-----------|--------------------------|----------|---|---------|-------|-------------------------------|------------|------|------|------|------|-------|
| | SiO. | A1.O. | Al_2O_3 Fe_2O_3 CaO MgO SO_3 Na_2O K_2O T | CaO | MgO | SO. | Na.O | K.0 | TiO. | MnO | P.O. | Other |
| materials | 510_{2} | A_2O_3 | | 110_2 | WIIIO | 1 ₂ O ₅ | components | | | | | |
| Sludge | 60.47 | 23.66 | 6.06 | 0.65 | 1.41 | 1.95 | 0.61 | 3.81 | 0.94 | 0.1 | 0.13 | 0.21 |
| OPC | 19.41 | 6.49 | 3.4 | 59.49 | 4.05 | 4.33 | 0.61 | 0.96 | 0.5 | 0.4 | 0.18 | 0.18 |
| UPC | 22.04 | 9.05 | 3.87 | 53.96 | 3.52 | 4.87 | 0.36 | 1.18 | 0.47 | 0.17 | 0.18 | 0.33 |

Table 2 Chemical composition of the test materials

2.2 Test scheme

According to the construction design of the cement mixing pile for the foundation pit of the North Yue Feng Station, the experimental designed cement content is 10-30%, the water-cement ratio is 0.8, and the longest curing time is 28 days. The specific test scheme is shown in Table 3, where the cement content is the ratio of the weight of the cement to that of the sludge in the natural water-content state.

Table 3 Test scheme

| Test materials | Cement content (%) | Curing time (days) | | | | |
|----------------|--------------------|--------------------|--|--|--|--|
| UPC, OPC | 10, 20, 30 | 1, 3, 7, 14, 28 | | | | |
| | 15, 25 | 1, 7, 28 | | | | |

2.3 Specimen preparation

Before specimen preparation, the sludge was put in a dark place to air dry and was removed the obvious stones. The air-dried sludge was broken up by a wall-breaking material machine and passed through a 0.5 mm sieve. The sieved sludge powder was placed in a self-sealing plastic bag, sealed, and stored after measuring its water content. The specimens preparation were performed according to the test standard [10]. Firstly, the sludge powder, cement, and water was weighed according to the designed ratio and the sludge powder and cement were mixed by a customized dry powder mixer. Secondly, the weighed water was added to the mixed powder until the mixture was uniformly in a slurry mixer. Thirdly, the uniformly mixed modified sludge was put into the cylindrical steel mold with an inner size of Φ 3.91 cm × 8.00 cm in 5-8 layers. The specimen was manually vibrated and was added to the mold. Forthly, the surface of the specimen was covered with the plastic wrap and the 6 cm × 6 cm glass sheet was covered it for 1 day before demoulding and weighing. Finally, the specimen was wraped and numbered with the moist plastic wrap and then it was put it in the sealed plastic bag at room temperature until the curing time.

2.4 Test methods

The test procedure is shown in Figure 1. Specimen preparation is performed first, followed by a series of tests such as UCS, XRD, MIP, and SEM.





The UCS was tested by a WDW-50 kN universal testing machine and the axial loading rate was 0.1 kN/s. Two specimens were prepared for each mixed series to calculate their average values as a representative result. The representative internal fragments of the specimens destroyed by the UCS test were dehydrated with absolute alcohol and then dried at 40 °C for 1 day before the microscopic tests. The XRD tests were performed on pulverized sludge and modified sludge using a D8 Advance X-ray diffractometer. The MIP tests were carried out on cubic modified sludge specimens with a side length of approximately 5 mm using an AutoPore Iv 9510 fully automatic porosimeter. The SEM tests were performed on specimens with fresh cross-sections using a Merlin Compact scanning electron microscope.

III. Results and Discussion

3.1 Mechanical properties analysis of modified sludge

3.1.1 Strength characteristics

The strength ratio is defined as the ratio of the UCS of the UPC-modified sludge to that of the OPC at the same curing time and cement content. Figure 2(a) shows that the UCS of the UPC-modified and OPC-modified sludges both exhibit a rapid and then slow growth with the increase of curing time, and the strength increased rapidly before 7 days and relatively slowly after 7 days. In addition, the strength growth of UPC-modified sludge before 7 days is significantly greater than that of the OPC, which is mainly due to the UPC has a smaller particle size, greater specific surface area, and higher activity than that of the OPC. Figure 2(b) shows that the UCS of modified sludge increases roughly linearly with the increase of cement content. The change in strength growth of UPC-modified sludge is significantly influenced by cement increment. However, the effect of cement increment on the strength growth of the OPC-modified sludge is influenced by curing time. As far as OPC-modified sludge is concerned, when the curing time is short, the increase in strength caused by the increase in cement is small. On the

contrary, when the curing time is longer, the increase in strength caused by the increase in cement is more significant.



Fig 2: The curves of UCS and strength ratio with variation of curing time and cement content, respectively. (a) Curing time; (b) Cement content.

For the modified sludge with 20% and 30% Cement content (%), the strength ratio gradually decreases with the increase of curing time before 7 days and tends to stabilize at about 1.5 after 7 days. For the modified sludge with 10% cement content (%), the strength ratio as a whole gradually decreases with the increase of curing time. Besides, the strength ratio increases as a whole with the increase in cement content, but the increase in the strength ratio was smaller after 7 days and was evident at 1 day. This phenomenon shows that the UPC-modified sludge is not only more efficient and has greater strength than that of the OPC but also has early-strength characteristics. The use of UPC for foundation reinforcement of deep sludge layers has important significance for the projects that have a large impact on the construction period and require early strength.

3.1.2 Stress-strain and failure characteristics

As seen from Figure 3, "U" represents UPC-modified sludge specimens, "O" represents OPC-modified sludge specimens, and the number after the letter represents the corresponding percentage of the cement content.





Fig 3: The stress-strain curves of modified sludge (a) 1 day; (b) 7 days; (b) 14 days; (c) 28 days

(1) For UPC-modified sludge, the stress-strain curves of the modified sludge specimens can be roughly divided into three stages: linear elastic deformation, plastic deformation, and strain-softening stages. The stress-strain relationship curves exhibit the characteristics of strain softening. There is obvious peak stress in the stress-strain relationship curves, and the specimens present a brittle failure mode. With the increase of cement content and curing time, the plastic deformation of the specimens is significantly reduced and the brittleness characteristics are enhanced.

(2) For the modified sludge specimens with a cement content of 10%, which shows a desirable elastoplastic characteristic at the curing time. The stress-strain relationship curves present a strain hardening type, and the specimens exhibit a plastic failure mode. For the modified sludge specimens with a cement content greater than 10%, the stress-strain curve gradually changes from strain hardening to strain-softening types with obvious peak stresses as cement content and curing time increase, and its failure mode gradually changes from plastic failure to brittle failure. This is similar to the UPC-modified sludge specimens.

The strength of cement-modified sludge mainly comes from the hydration products of cement, which cement soil particles and form soil skeleton, and the strength of soil skeleton represent the overall structural quality of soil body, the greater the strength of soil skeleton, the better the overall structure of soil body [11]. The hydration reaction of UPC is rapidly when it meets with water, and a large amount of hydration product is generated soon, which makes the modified sludge resistant to external forces as a whole even under the condition of relatively short curing time. Therefore, the failure of UPC-modified sludge exhibits greater brittleness. Compared to UPC, OPC has a smaller specific surface area and lower activity, and the hydration rate is slower when it meets with water. The hydration products generated in OPC-modified sludge are not sufficient to form a good overall soil structure when cement content is low and curing time is short. Therefore, the failure of OPC-modified sludge exhibits greater plasticity.

There are three typical failure modes of the specimens. (a) The plastic shear failure: the specimen failure is characterized by multiple cross-cracking, which is the main form of plastic failure. (b) The brittle shear failure: the specimen is destroyed with an inclined crack at a certain angle to the axial direction, which is the failure of part of brittle failure. (c) The brittle tension failure: where cracks develop along the axial direction until penetration, which is the mainly brittle failure of specimens.

3.1.3 Deformation characteristics of modified sludge

The failure strain ε_f is the strain corresponding to the peak stress in the stress-strain curve, and the higher ε_f , the stronger tenacity of the material, otherwise the stronger brittleness. E_{50} refers to the secant modulus corresponding to the compressive strain of $0.5\varepsilon_f$ in the stress-strain curve, which reflects the material's resistance to deformation (Figure 4).

It can be seen from Figure 4(a), the failure strain ε_f of UPC-modified sludge is relatively concentrated, which is mainly distributed in the range of 0.8-1.3%. In contrast, the OPC-modified sludge failure strain ε_f is mainly distributed in the range of 1-3%, with a relatively large distribution, and shows a decreasing trend with increasing strength as a power function. It can be observed from Figure 4(b) that the deformation modulus E_{50} of both UPC-modified and OPC-modified sludges increases with increasing strength and shows a linear correlation. The slope of the fit function of the UPC-modified sludge is greater than that of the OPC, and the UPC-modified sludge performes stronger deformation resistance.



Fig 4: The curves of ε_f and E_{50} with valation of UCS, respectively (a) ε_{f} ; (b) E_{50}

3.2 X-ray diffraction analysis

As seen from Figure 5, the crystals chemical products of UPC-modified sludge are basically the same as those of OPC-modified sludge, which is mainly cement hydration products ettringitr (Aft). The difference is that the portlandite $(Ca(OH)_2)$ diffraction peaks are obviously present in the X-ray diffraction patterns of the OPC-modified sludge at 1 and 7 days.



Fig 5: X-ray diffraction patterns (a) UPC-modified sludge; (b) OPC-modified sludge

In addition, the related studies mentioned that the hydration products of cement-modified soil are mainly hydrated calcium silicate (C-S-H), Aft and Ca(OH)₂ [12]. Because C-S-H is a gel, it is difficult to be detected by XRD test. Among the three cement hydration products mentioned above, Aft has a greater contribution to the early strength; the formation of C-S-H gel is the main reason for the increase in strength of modified sludge; while Ca(OH)₂ can dissolve in water to maintain the alkaline environment of the soil, meanwhile, it reacts with the active SiO₂ in the cement to form more gel products C-S-H. An obvious phenomenon is that the diffraction peak intensity of Ca(OH)₂ gradually decreases with the curing time, which may be due to the increasing CSH gel encapsulating it,

and this is also why there is no obvious $Ca(OH)_2$ diffraction peak in the higher-activity UPC-modified sludge and the 28 days OPC-modified sludge.

3.3 Pore structure changes of modified sludge

As can be seen in Figures 6 and 7, the cumulative pore volume is smaller than the sludge specimens for all modified sludge specimens, which indicating that the hydration products of cement in the process of modified sludge not only act as cementing soil particles but also fill the pores of the soil.



Fig 6: The cumulative pore volume of different sludges with the cement content of 20% (a) UPC-modified sludge; (b) OPC-modified sludge



Fig 7: The cumulative pore volume of different sludges with curing time of 7 days (a) UPC-modified sludge; (b) OPC-modified sludge

As seen from Figure 6, the cumulative pore volume is smaller than the sludge specimens for all modified sludge specimens, which indicating that the hydration products of cement in the process of modified sludge not only act as cementing soil particles but also fill the pores of the soil.

The cumulative pore volume distribution curve is shifted towards the left axis with the increase of curing time and cement content, demonstrating that the cumulative pore volume and pore diameter inside the specimens gradually decrease. The reason for this phenomenon is that with the extension of the curing time, the continuous hydration reaction of cement causes more cement hydration products to be generated inside the modified sludge specimens with a large curing time, which leads to more pores of the soil being filled. Consequently, the pore structure of the modified sludge exhibits the characteristics that the cumulative pore volume and pore diameter decrease with the increase of the curing time. Furthermore, the hydration reaction of cement in modified sludge is influenced by the cement content. Under the same curing time, the larger cement content, the larger contact area between the cement and the pore water in the specimens, the faster the hydration reaction rate, the more hydration products are generated and the more fully the pores of the soil are filled.

It can be found that except for individual specimen (UPC-modified sludge specimen with the curing time of 7 days and the cement content of 30%), the total intruded pore volume of the UPC-modified sludge is smaller than that of the OPC-modified sludge, indicating that more hydration products are generated in UPC-modified sludge than that of the OPC. As seen from Figures 6 and 7, the pore size distribution curves of all specimens show a single-peak feature with a relatively concentrated pore size in the distribution range. Besides, the most available pore size of modified sludge specimens varied significantly with the increase of the curing time and the cement content. Although the variation of the most available pore size of OPC-modified sludge with different cement content at the curing time of 7 days shown in Figure 7 is not very obvious, it can be clearly observed from the other three figures that the most available pore sizes of modified sludge specimens decrease significantly with the increase of the curing time and the cement content. Furthermore, comparing Figures 6(a) with 6(b) and Figures 7(a) with 7(b), the most available pore size of UPC-modified sludge is smaller than that of OPC-modified sludge under the same curing time and cement content.

It can be noticed that the porosity of the sludge is the largest (Figure 8(a)), which is 61.47%. Besides, the porosity of modified sludge specimens decreases with the increase of curing time and cement content. Except for the UPC-modified sludge specimen with the curing time of 7 days and the cement content of 30%, the porosity of UPC-modified sludge under the same conditions was lower than that of OPC-modified sludge, and this gap becomes larger with the increase of the curing time and the cement content. Figure 8(b) shows the average pore diameter of the sludge and modified sludge. It can be found that the variation of the average pore diameter of modified sludge specimens is consistent with the variation of the most available pore size and the variation of the porosity.



Fig 8: Porosity and average pore diameter of sludges (a) Porosity; (b) Average pore diameter

It is easy to find from Figures 9 and 10 that the pore diameter of all specimens is mainly distributed between 0.01-1 μ m, among which the pore diameter of the sludge is mainly concentrated between 0.1-1 μ m and accounts for 73.83%. As seen from Figure 11, with the increase of the curing time, more pores with the pore diameter of between 0.1-1 μ m in the modified sludge are converted to between 0.01-0.1 μ m, while the percentage of pores in the other pore diameter interval does not change much.



Fig 9: Percentage of pore diameter interval of different sludges with the cement content of 20% (a) UPCmodified sludge; (b) OPC-modified sludge

As shown in Figure 10(a), for the UPC-modified sludge at the curing time of 7 days, with the increase of the cement content, more pores with the pore diameter between 0.1-1 μ m in the modified sludge are converted to between 0.01-0.1 μ m, while the percentage of pores with the pore diameter <0.01 μ m increases, and the percentage of pores belonging to between 1-10 μ m and >10 μ m does not change significantly. As shown in Figure 10(b), for the OPC-modified sludge at the curing time of 7 d, the pore diameter interval distribution variation is significantly different from that of the UPC-modified sludge. With the increase of the cement content, the percentage of pores with pore diameter between 0.01-0.1 μ m and 0.1-1 μ m does not change significantly, but the percentage of pores with pore diameter <0.01 μ m increases, and the percentage of pores between 1-10 μ m and >10 μ m does not change significantly, but the percentage of pores with pore diameter <0.01 μ m increases, and the percentage of pores between 1-10 μ m and >10 μ m decreases. This is the reason for the difference that the hydration rate of OPC is relatively slow, and the additional hydration products generated due to the increase in cement content are not enough to make more pores with the diameter between 0.1-1 μ m.

As seen from Figures 9 and 10, the percentage of pores in the UPC modified sludge specimens with the pore diameter between 0.1-1 μ m is less than that of the OPC, while the percentage of pores in the UPC modified sludge specimens with the pore diameter between 0.01-0.1 μ m is more than that of the OPC under the same curing time and cement content. Combined with Figure 10(a), it can be shown that the structure of the UPC-modified sludge specimens under the same conditions is denser than that of the OPC.



Fig 10: Percentage of pore diameter interval of different sludges with the curing time of 7 days. (a) UPCmodified sludge; (b) OPC-modified sludge.

With the increase of curing time and cement content, the cement hydration products in the modified sludge specimens increase and fill more pores, which shows the increase of the strength of the modified sludge. Meanwhile, compared with the OPC one, the UPC hydration reaction is rapid and sufficient due to its larger specific surface area and higher activity, which can rapidly produce more hydration products and then fill more microscopic pores, thus it shows that UPC-modified sludge has obvious early-strength and which is much higher than that of the OPC on the macroscopic level.

3.4 Microstructure analysis of the sludge

As shown in Figure 11, the pictures that the chemical products in the modified sludge are mainly flocculent C-S-H gels and needle-rod Aft crystals. The flocculent C-S-H gels and the needle-rod Aft crystals interweave with each other, which being together wrapped and cemented the mineral aggregates of the sludge. The hydration products of cement make modified sludge form a dense overall structure by changing the connection mode of mineral aggregates and the pore structure of the soil, which makes the modified sludge exhibit an increase in strength on a macroscopic scale.

In addition, a small amount of hexagonal plate-like $Ca(OH)_2$ crystals are observed in the OPC-modified sludge, which is consistent with the XRD test results [12]. The C-S-H gels in the UPC-modified sludge specimens are more obvious than that in the OPC, and the UPC-modified sludge structure is more dense under the same curing time and cement content. This result is consistent with the strength of the modified sludge and the MIP test results.



Fig 11: The microstructure of modified sludge with 20% cement content and 7 days curing time. (a) UPCmodified sludge; (b) OPC-modified sludge.

IV. Conclusions

The excellent performance UPC was introduced as a sludge modifier, and the mechanical properties and microstructural changes of UPC-modified sludge were investigated by comparing it with that of the OPC. The main conclusions are as following:

(1) The UCS of UPC-modified sludge varied with curing time and cement content in the same way as that of the OPC. Compared with OPC-modified sludge, the UPC-modified sludge is more efficient and has greater strength, and shows obvious early-strength characteristics. The UCS of UPC-modified sludge at 1 day curing time reaches up to about 4 times of the UCS of OPC-modified sludge.

(2) The stress-strain relationship curves of UPC-modified sludge has significant peak stresses which show a brittle failure mode. The modulus of deformation E_{50} increases linearly with the increase of UCS. Compared with OPC-modified sludge, the UPC-modified sludge has stronger resistance to deformation.

(3) The early-strength and high efficiency of the UPC-modified sludge originate from the larger specific surface area and higher activity of UPC. Compared with the OPC, the UPC has a fast hydration speed and sufficient hydration reaction, which is beneficial to improve the overall properties of the modified sludge.

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