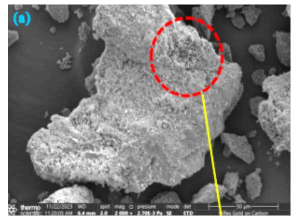
Experimental research on mechanical properties of magnesium oxychloride-based titanium gypsum concrete



Investigación experimental sobre las propiedades mecánicas del hormigón de yeso titanio a base de oxicloruro de magnesio

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RESUMEN

- Para paliar la contaminación ambiental, el despilfarro de recursos del yeso de titanio (TG) y el residuo industrial de la producción de dióxido de titanio por el método del ácido sulfúrico, y para desarrollar nuevos materiales de construcción, se estudiaron los efectos de diferentes dosificaciones sobre la resistencia y las propiedades microscópicas del cemento de oxicloruro de magnesio (MOC) mediante métodos como la resistencia a la compresión ilimitada, el microscopio electrónico de barrido (SEM) y el difractómetro de rayos X (XRD). Los resultados muestran que la resistencia a la compresión de las muestras aumenta primero y disminuye después con el aumento de la cantidad de yeso de titanio. En el grupo de control, cuando la dosificación era del 0%, la resistencia a la compresión de las muestras era de 40,37 MPa. La resistencia a la compresión es de 34,29 MPa con una dosificación del 50%, que es inferior a la resistencia a la compresión del grupo de control en un 15,06%. La estructura interna cambia y se produce una reacción incompleta del SiO_2. La adición de yeso de titanio aumenta la ductilidad del hormigón, pero tiene poco efecto en su módulo elástico. Por lo tanto, la inclusión adecuada de TG en el material gelificante TG-MOC puede mejorar su capacidad de deformación y mantener cierta resistencia. Esto proporciona una referencia útil y una guía para la aplicación del yeso de titanio a base de oxicloruro de magnesio en la práctica de la ingeniería.
- **Palabras clave:** Cemento de oxicloruro de magnesio, Yeso de titanio, Propiedades mecánicas, Análisis microscópico, Resistencia a la compresión.

ABSTRACT

To alleviate the environmental pollution, waste of resources of titanium gypsum (TG) and the industrial residue from the production of titanium dioxide by sulfuric acid method, and to develop new building materials, the effects of different dosage on the strength and microscopic properties of magnesium oxychloride cement (MOC) were studied by methods such as unlimited compressive strength, scanning electron microscope (SEM) and X-ray diffractometer (XRD). Results show that the compressive strength of the samples increases first and then decreases with the increase of the amount of titanium gypsum. In the control group, when the dosage was 0%, the compressive strength of the samples was 40.37 MPa. The compressive strength is 34.29 MPa at 50% dosage, which is lower than the compressive strength of the control group by 15.06%. The internal structure changes, and there is an incomplete reaction of SiO_2. The addition of titanium gypsum increases the ductility of concrete, but has little effect on its elastic modulus. Therefore, the proper inclusion of TG in the TG-MOC gelling material can improve its deformation ability and maintain a certain strength. This provides a useful reference and guidance for the application of magnesium oxychloride-based titanium gypsum in engineering practice.

Keywords: Magnesium oxychloride cement, Titanium gypsum, Mechanical propertie, Microscopic analysis, Compressive strength.

1. - INTRODUCTION

Titanium gypsum (TG) is an industrial waste produced by sulfuric acid method to produce titanium dioxide. Its main component is calcium sulfate dihydrate. For every 1t of TG produced, 5t of TG will be produced. The development of China's titanium dioxide industry has grown at an average annual growth rate of more than 15% in the past 20 years, and the total production of the industry has increased from 181,000 t per year to 2.9543 Mt per year [1]. With the progress of science and technology and the development of human society, industrial development has advanced by leaps and bounds, and the production of industrial solid waste has also increased year by year and accumulated in piles. The treatment of industrial solid waste has become a major problem that bothers people. We must pay attention to it and protect the environment on which we depend for our survival [2, 3].

Since the expenditure on construction resources accounts for about half of the world, in order to conform to the trend of protecting the environment and enhancing the people's happiness index, we have to consider the reuse of industrial solid waste to achieve a win-win situation of protecting the environment and saving resources [4, 5]. This has set off a wave of green building materials. In addition, we must also consider the performance of building materials to reduce the impact of energy and environmental issues. Twenty or thirty years ago, in order to protect the environment and save resources, people used recycled concrete aggregates instead of natural aggregate materials as a new type of material to conduct experimental research on concrete with different particle sizes and different admixtures [6]. Considering the use of recycled aggregates, the impact of different qualities on the concrete of recycled aggregates is very different, and the rational and efficient use of recycled aggregates has become a new problem to be solved [7].

As a hot issue in the field of solid waste, TG has a large number of impurities inside, and the accumulation of solid waste is large, which cannot be used as a landfill material. It has become one of the difficult problems plaguing TG production enterprises and environmental protection departments, which has caused a great burden on the environment [8]. In order to solve this problem, many scholars have done lots of tentative researches on TG as a part of cement concrete and other building materials [9-11]. Such as TG, cement, lime according to a certain proportion of the combination were formed TG based stabilizer. A simple physical method was used to change the properties of TG to enhance the stability of TG in expansive soil. A certain proportion of titanium gypsum was used to replace magnesium oxide in magnesium oxychloride cement (MOC), etc. MOC was first discovered by Sorrell in 1867, and the research has shown that MOC is a gaseous gelling material, mainly composed of a mixture of magnesium oxide powder and an aqueous magnesium chloride solution. At room temperature, it mainly exists in two forms: $3Mg(OH)_2 \cdot MgCl_2 \cdot 8H_2O$(3-phase) and $5Mg(OH)_2 \cdot MgCl_2 \cdot 8H_2O$ (5-phase). Adding 5-phase seed crystals from the outside world can effectively increase the generation rate of five-phase crystals in MOC and improve the mechanical properties of MOC [12, 13]. MgO and $MgCl_2$, the raw materials for MOC, can also be obtained from dolomite ore using leaching-carbonation-evaporation-thermal hydrolysis public welfare [14, 15]. The crystalline phase of MOC is needle-shaped crystals with granular surfaces bound in different forms [16, 17]. With the development of the construction industry, the use of magnesium-based building slabs is becoming more and more extensive, and new building materials have attracted everyone's attention. Compared with traditional magnesium-based building materials, the new building materials have more advantages and higher performance in all aspects [18, 19]. Even so, through the experimental research on the mechanical properties of different dosage ratios, it has been found that magnesium-based building materials also have some shortcomings, and their water resistance still needs to be explored [20]. Adding $FeSO_4 \cdot 7H_2O$ and KH_2PO_4 to MOC and carbonizing attack found that FeSO4 and KH_2PO_4 can increase the Mg^{2+} content in the solution, $FeSO_4$ promotes the generation of 5 phases in MOC, and KH_2PO_4 increases the concentration of Cl^-, which has a positive effect on the water resistance of MOC [21-23]. While the microscopic mechanism of TG affect the strength of MOC needs to be further studied.

The composite material formed by adding multi-walled carbon nanotubes to the MOC has high mechanical resistance and reduced water absorption, which provides high-performance and high-quality materials for specific purposes, and has good potential in the construction industry [24, 25]. Adding corn starch/sodium polyacrylate to MOC can effectively improve the compressive strength and water resistance of MOC [26]. Adding ultra-high molecular weight polyethylene (PE) fibers to MOC can produce early high-strength, high-ductility, good durability, and high-potential composite materials [27, 28]. In MOC, if 30% of the MgO mass is replaced with fly ash, slag, partial kaolin and calcined iron-rich kaolin respectively, the water resistance of the MOC gelling material will continue to increase with the continuous increase of the dosage [29, 20]. The molar ratio of different $MgO/MgCl_2/H_2O$ and the maintenance conditions have a large influence on the mechanical properties of MOC, and the compressive strength of the non-lateral line tends to increase first and then decrease as the proportion of the molar ratio increases. With the increase of curing time, MOC water absorption undergoes a hydration reaction, and the compressive strength decreases [31]. The pH value of the immersion solution of the gelling material formed by $MgO/MgCl_2/H_2O$ according to different molar ratios has a significant effect on the phase of the hydrated product. The higher the pH value is, the more stable the phase is. When the pH value exceeds 10.37, the phase stability deteriorates. Among them, the molar ratio of $MgO/MgCl_2$ is 6 as the critical value. When it is less than the critical value, the phase is stable. When it is greater than the critical value, a large number of crystals will form and destroy the internal structure of the gelling material [32]. So there is still a problem for the different dosages of TG affect the strength of MOC that has not been deeply solved.

However, in the practical engineering, how the different dosages of TG affect the strength of MOC and its microscopic mechanism needs to be further studied. The effective combination of TG and MOC can solve the problem of rational utilization of waste gypsum, which will be a trend of large-scale application in future construction.

In this study, through a fixed molar ratio of $MgO/MgCl_2/H_2O$, it was determined that a large amount of TG was added to meet the concrete strength requirements. By using the test methods, such as unconstrained compressive strength test, scanning electron microscope and X-ray diffraction, the effects of different dosages of TG on the strength, product and internal structure of TG-MOC gelling materials were studied. The characteristic of this study explores the reuse of TG and its potential use in non-load-bearing building materials. The rest of this study is organized as follows. Section 2 introduces the test materials and programs used in this experiment. Sections 3 describes the results and analysis, and finally, section 4 summarizes the conclusions.

2. - MATERIAL & METHODS

2.1.- TEST MATERIALS

Due to the different properties of test materials from different places, such differences will have an impact on the test, so the composition of the sample and other properties must be described as follows: in this test, the light-burned magnesium oxide used in the test came from a magnesium manufacturing company in Haicheng City, Liaoning Province, China. Its main chemical composition and content were identified by the X-ray fluorescence spectrometer (XRF) as shown in Table 1. The activity of MgO was determined by hydration method, and the concentration of MgO was guaranteed to be not less than 85%. Magnesium chloride hexahydrate was purchased from a chemical reagent company in Tianjin, China. Its chemical composition and content are shown in Table 2 (See section: supplementary material). TG comes from Henan Baililian Chemical Co., Ltd., Jiaozuo City, Henan Province, China. Its chemical composition is analyzed by XRF identification, XRD and SEM. As shown in Table 3 and Fig. 1

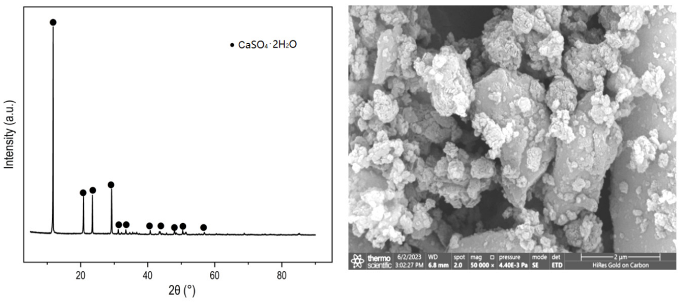
The tests using lightly burned powder, magnesium chloride hexahydrate and PMS data are referenced in [28]. The composition of one of the PMS was detected by XRF, as shown in Table 1.

Ingredients	MgO	MnO	SiO_2	CaO	Al_2O_3	Fe_2O_3	SO_3	MgO activity
Content (%)	85.962	6.706	3.644	2.195	0.648	0.474	0.206	63.5

Table 1. The chemical composition of MgO.

Ingredients	SO_3	CaO	Fe_2O_3	SiO_2	Al_2O_3	TiO_2	MgO	Na_2O	MnO	Others
Content (%)	38.52	30.14	13.64	3.32	3.6	2.9	1.72	0.97	0.66	4.53

Table 3. Chemical composition of TG.



(a) (b)

Fig. 1. Microscopic analysis of TG. (a) TG XRD analysis; (b) TG SEM.

2.2.- EXPERIMENTAL EQUIPMENT

The main equipment and appliances used in the test and the flow chart of sample preparation process are shown in Figs. 2 (See section: supplementary material) and 3, respectively. The process from sample making to testing was as follows: First of all, the TG (raw material) was used for drying, crushing, sieving, and then different dosage of TG with magnesium chloride aqueous solution and magnesium oxide fully were stirred into the mold. After the final condensation of the mold for the removal of the mold, the uniaxial compressive strength was tested after 28 d maintenance. Finally, after crushing, milling, and sieving of the sample, the test was conducted in the XRD and SEM detection.

2.3.- EXPERIMENTAL PROCESS

In order to determine the effect of different TG dosage on the compressive strength of magnesium chloride cement, in this test, the molar ratio of MOC ($MgO:MgCl_2{\cdot}6H_2O:H_2O$) was determined to be a fixed value of 7:1:15 [32]. The dosage of TG is determined according to the percentage of the mass of lightly burned magnesium oxide. To study the effects of different dosages of TG and MOC, 10%, 20%, 30%, 40%, and 50%, respectively. Before preparing the cement mortar, first prepare a certain concentration of $MgCl_2$ aqueous solution and let it stands for 24 h and wait for backup. The dehydrated large pieces of TG are processed into small pieces through an overhead crusher and placed in a ball mill for grinding. Before allowing sealed use, a 200 mesh square hole sieve was used to filter the ground powder.

When preparing the sample, first dry mix the lightly burned magnesium oxide powder and TG powder in a cement mortar mixer for 5 min to make the two materials fully mixed evenly. Then add the supernatant of magnesium chloride solution for 5 min, and finally pour the mortar into a cylindrical mold with a size of 50 mm × 100 mm. The sample preparation is completed within 1 h, and the mold is released indoors (temperature 20±3°C, humidity 60±5%) after 24 h, and it is cured at 23°C and 50% relative humidity for 28 d.

Using a universal testing machine, the loading speed is set to 0.5 kN/s to test the compressive strength of the MOC for 28 d. The strength test is tested according to China standard GB/T 17671-1999 (Cement Mortar Strength Test Method). After the strength test of the specimen is over, take the flakes and blocks in the crushing center of the specimen, soak in anhydrous alcohol for 48 h, and air-dry for 48 h to remove moisture to terminate the reaction. These lumps are ground into powder through a mortar and passed through a 200-purpose square sieve.

Rigaku Smart Lab XRD (Cu target) was used for phase analysis, with a scanning range of 2θ=3°-90° and a scanning rate of 10°/min. Use Jade 6.5 to perform phase analysis of the XRD diagram. The German Merlin Compact SEM was used to scan the gold-sprayed broken sheet sample to observe the microscopic morphology. Then use the Image J software to process the resulting SEM image to obtain the particle size distribution range of the crystal. Use an energy dispersive spectrometer (EDS) to study the distribution of elements.

3.- RESULT ANALYSIS & DISCUSSION

3.1.- INFLUENCE OF DIFFERENT TG DOSAGE ON STRENGTH OF TG-MOC GELLING MATERIALS

The influence of different dosages of TG on the compressive strength of TG-MOC gelling material is shown in Fig. 4 and Table 4 (See section: supplementary material). Compared with the TG dosage of 0%, as the TG dosage continues to increase, the compressive strength of the gelled material increases first and then decreases [22, 28].

The uniaxial compressive strength calculation is described in Eq. (1).

$$P = F / A \tag{1}$$

where, P is the uniaxial compressive strength, MPa. F is the force applied to the sample, N. and A is the cross-sectional area of the sample, m^2.

When the TG dosage is 0%, the compressive strength is 40.37 MPa. When the TG dosage is 10%, the compressive strength is 63.35 MPa, which is the highest strength at this time, which is 57% higher than the strength of the control group. It is also incorporated with 10% TG, and the strength of cement rubber sand can only reach P.O 42.5 Cement strength index requirements. When the TG dosage was 20%, the compressive strength was 44.97 MPa, which was 11.39% higher than the strength of the control group. When the TG dosage was 30%, the compressive strength was 42.55 MPa, which was 5.35% higher than the strength of the control group. The 30% titanium gypsum mixed with cement and FAC-1 can be formulated into C30 concrete [33]. When the TG dosage was 40%, the compressive strength was 38.34 MPa, which

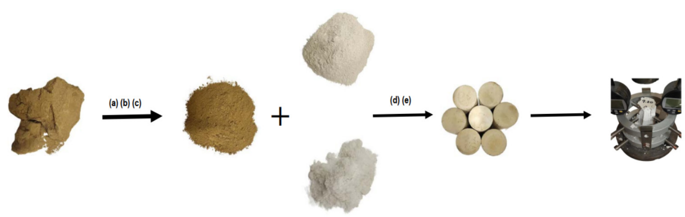


Fig. 3. Flow chart of sample preparation process.

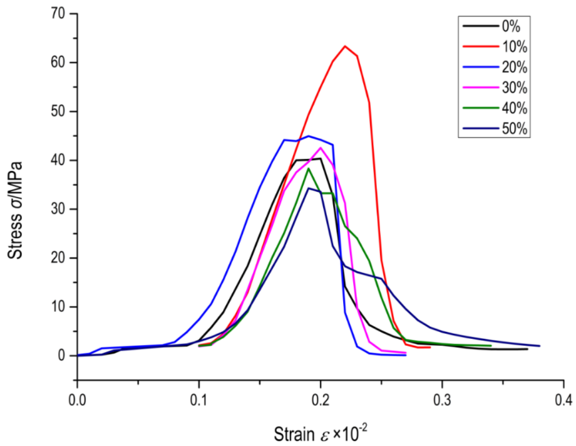


Fig. 5. Stress-strain curves of different dosages of TG.

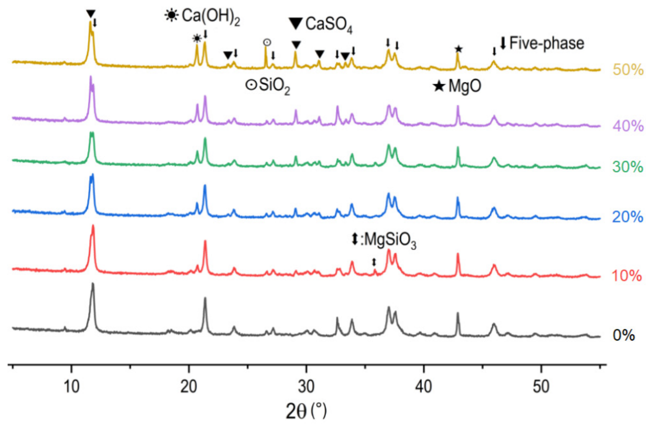


Fig. 6. XRD diagram of TG-MOC gelling material with different dosages of TG.

was 5.03% lower than the strength of the control group. When the TG dosage was 50%, the compressive strength was 34.29 MPa, which was the lowest strength at this time, and the strength was reduced by 15.06% compared to the control group. It is much higher than the 28-day age of 5.32 MPa when TG and fly ash are added to cement in a 5:5 ratio [34]. When the dosage of TG is 42.9% to 50.3%, it is mixed with desulfurized gypsum, titanium slag powder, lime alkali exciter and ordinary silicate cement to prepare a composite gelling material that only reaches the Building gypsum (China GB/T 9776-2008) 2.0 strength grade [35].

3.2.- DEFORMATION EFFECTS OF DIFFERENT TG DOSAGE ON TG-MOC GELLING MATERIALS

The deformation effects of different dosages of TG on the TG-MOC gelling material are shown in Fig. 5 and Table 5. As seen from Fig. 5, the stress-strain curves of different TG admixtures are all complete arc curves, and the concrete has good deformation ability during compression. The strain corresponding to 0% is 0.2×10^{-2}, the strain corresponding to 10% is 0.22×10^{-2}, the strain corresponding to 20% is 0.21×10^{-2}, the strain corresponding to 30% is 0.205×10^{-2}, the strain corresponding to 40% is 0.19×10^{-2}, and the strain corresponding to 50% is 0.185×10^{-2}. The strain amount of TG incorporated was larger than that of the control group, and the addition of TG increased the ductility of the concrete. When the TG dosage is 30%, 40%, and 50%, the elastic modulus of concrete gradually decreases. When the TG dosage is 10% or 20%, the elastic modulus of concrete gradually increases. Although there are changes, the amount of change is not large. The inclusion of TG has a certain impact on the elastic modulus of concrete, but the impact is small.

The elastic modulus calculation is described in Eq. (2).

$$E = (F/A)/(\Delta L/L) \quad (2)$$

where, E is the elastic modulus, MPa. ΔL is the length of the deformation of the material under the action of force, N. L is the initial length of the material, m.

3.3.- XRD ANALYSIS OF TG-MOC GELLING MATERIAL

To illustrate the influence of different dosages of TG on the phase composition of the TG-MOC gelling material, Fig. 6 shows the XRD spectrum of the sample cured for 28 d in the range of 5°-60°. According to the atlas, the main phases of the composite gelling material are gypsum stone ($CaSO_4$), magnesite (MgO), slaked lime ($Ca(OH)_2$), and perovskite ($MgSiO_3$). Among them, the five-phase is the main hydration product of the ternary system ($MgO-MgCl_2-H_2O$). MgO comes from light-burned magnesium oxide raw materials, $CaSO_4$ comes from TG raw materials, and Ca $(OH)_2$ is replaced by Mg $(OH)_2$ and $CaSO_4$. Mg $(OH)_2$ is produced due to incomplete hydration of the five phases. MgSiO3 is produced by the chemical reaction between SiO_2 in TG and MgO [25, 28].

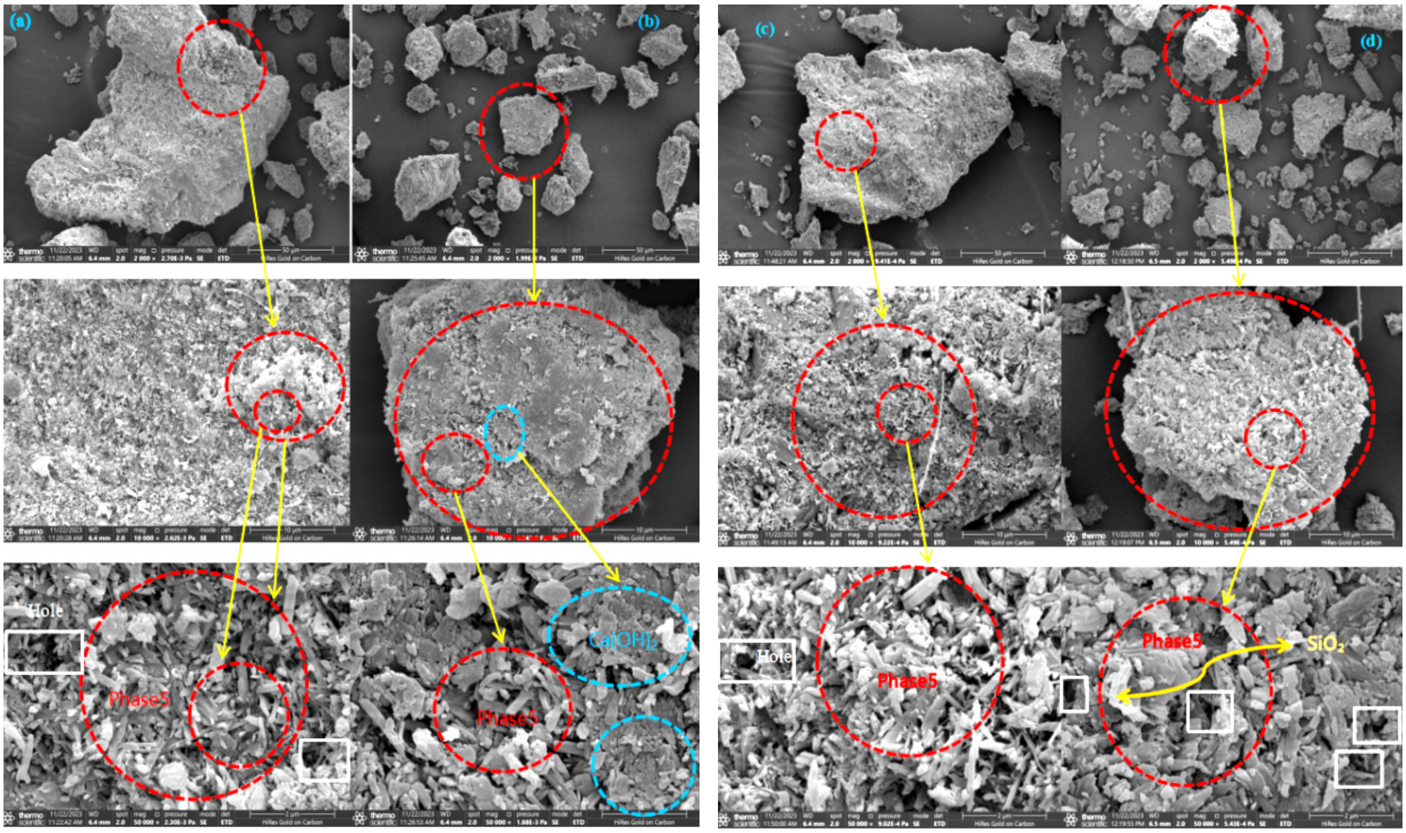
TG Doping (%)	0	10	20	30	40	50
Compressive strength (MPa)	40.37	63.35	44.97	42.55	38.34	34.29
Strain $\times10^{-2}$	0.200	0.220	0.210	0.205	0.190	0.185

Table 5. The compressive strength and strain values for different doses of TG.

At 2θ=11.76°, as the TG dosage continues to increase, the peak of the five-phase diffraction peak gradually decreases, from the initial higher than the $CaSO_4$ diffraction peak to the later lower than $CaSO_4$. At 2θ=20.68°, with the increase of the TG dosage, the diffraction peak of the product $Ca(OH)_2$ appears, and the peak value increases with the increase of the TG dosage. After TG was incorporated at 2θ=42.9°, the peak intensity of MgO's diffraction was slightly weakened compared with that of the control group. At 2θ=26.52°, when the TG dosage is 50%, the peak value suddenly increases because SiO_2 cannot fully react with MgO. Since the SiO_2 crystal is flaky, this leads to a change in the internal structure of the TG-MOC gelling material, which is consistent with the strength change trend and SEM results.

3.4.- DIFFERENT DOSAGES OF TG ON MICROSTRUCTURE OF TG-MOC GELLING MATERIALS

As shown in Figs. 7(a), 7(b), 7(c), 7(d) and Table 6 (See section: supplementary material), it is an electron microscope scan of the TG-MOC gelling material, the dosage of TG is 0%, 10%, 40%, and 50%, respectively. When TG doping is at 10%, as observed from Fig. 7(a), it is observed that when TG is not added, microscopic analysis of the MOC cement sample can show that only a small number of holes appear on the surface. Moreover, the micropores in the system are filled with needle-rod-shaped five-phase crystals, and the five-phase crystals are radioactively staggered on the plates. Overall, the internal structure is not very dense. As can be seen from Fig. 7(b), when the TG dosage is 10%, the particle size is much smaller than the commonly used by-product gypsum in industry. This is because TG is macroscopically expressed as a delicate powder. Based on the physical phase composition and microscopic analysis, there has been no significant change in the five-phase existence morphology. TG, as a beneficial filling material, effectively fills the voids within the MOC and enhances the overall compactness of the TG-MOC gelling material. Inside, numerous visible $CaSO_4$ crystals can be observed. With the continuous increase of the TG dosage, when the dosage reaches 40% (Fig. 7(c)), the morphology of the five-phase has undergone significant changes, and the overall distribution is amorphous. When



(a) (b) (c) (d)

Fig. 7. Electron microscope scan of TG-MOC gelling material with different dosages of TG. (a) 0%; (b) 10%; (c) 40%; (d) 50%.

the dosage reaches 50% (Fig. 7(d)), the five-phase is mainly flaky and flaky accumulation. Even though the internal compactness is good, most of it is provided by TG [28].

4.- CONCLUSIONS

To implement the concept of green development and solve the phenomenon of large accumulation, small use, and random discharge caused by imperfect industrial solid waste TG treatment technology, the micro-mechanism analysis and strength test of TG-MOC gelling materials with different TG admixtures were carried out. The main conclusions are obtained as following aspects:

(1) With the continuous increase of TG dosage, the compressive strength of TG-MOC gelling material shows a trend of increasing first and then decreasing. When the dosage is 10%, 20%, and 30% of the mass of MgO, the compressive strength is increased by 57%, 11.39%, and 5.35% compared to the control group, respectively, all of which have a positive phase promotion effect. With the continuous increase of TG dosage, the compressive strength has shown a negative growth trend. When the dosage is 40% and 50% of the mass of MgO, the compressive strength is reduced by 5.03% and 15.06% compared to the control group, respectively, but it still meets the engineering requirements.

(2) The addition of TG can increase the ductility of concrete, but it has little effect on its elastic modulus. Therefore, the proper inclusion of TG in the TG-MOC gelling material can improve its deformation ability and maintain a certain strength.

(3) The XRD spectrum shows the phase composition at different dosage levels. With the increase of TG admixture, the five-phase diffraction peak gradually weakens, and the peak of $Ca(OH)_2$ increases, which indicates that the admixture of TG affects the crystal structure of the gelling material. When 10% TG is incorporated, the SiO_2 in TG and MgO completely react chemically to form $MgSiO_3$. The strength of the composite gelling material increases. When 50% of TG is incorporated, the SiO_2 in TG cannot fully participate in the reaction, resulting in a sharp increase in the peak-to-peak diffraction, indicating that the internal structure of the TG-MOC gelling material has undergone significant changes, which greatly reduces the compressive strength of the composite gelling material.

(4) Electron microscopy scans reveal that when 10% TG is incorporated, it effectively fills the pores within the MOC, enhancing the overall compactness of the gelling material. As the TG dosage increases, there is a noticeable transformation in the five-phase existence morphology, leading to the accumulation of flakes and platelets.

This study shows that it is feasible to prepare concrete by adding TG to MOC. Even if a large amount of admixture reduces the compressive strength of the gelling material and changes the internal phase composition, it still meets the building requirements. The large-scale use of TG not only solves the problem of industrial solid waste treatment, but also makes rational use of magnesite resources, which meets China's green environmental protection and low-carbon development requirements, and is conducive to the development and progress of society. The results of this research provide useful reference and guidance for the application of TG-MOC gelling materials. In the next, we will conduct follow-up experiments in two steps: On the one hand, the properties of TG-MOC gelling materials will be studied under different environmental conditions. On the other hand, the performance of the TG-MOC gelling material is optimized by adding admixtures.

5.- ACKNOWLEDGEMENTS

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