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Effect of Chemical Admixtures on the Working Performance and Mechanical Properties of Cement-Based Self-Leveling Mortar

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Abstract: In this work, the effect of cellulose ether (CE), tartaric acid (TA), and polycarboxylate superplasticizer (PCE) on the working performance and mechanical properties of cement-based self-leveling mortar is investigated. According to the orthogonal experiment analysis, TA is identified as the most influential factor affecting the working performance, as indicated by factors such as fluidity, fluidity loss, and viscosity. Upon conducting a comprehensive assessment of the working performance and mechanical properties, the optimal parameters are found to be CE = 0.6 wt.‰, TA = 0.5 wt.‰, and PCE = 2.0 wt.‰. A univariate test highlights that that the working performance improves with the higher TA dosages. Specifically, the exponential reduction of fluidity loss corresponds with an increased TA content. Regarding the mechanical properties of cement-based self-leveling mortar, the compressive and flexural strength exhibit enhancement when the TA dosage remains below 0.4 wt.‰ at the early stage, implying that TA has some influence on the hydration process. Impressively, the 1 d compressive and flexural strengths surpass 7 MPa and 2 MPa, respectively, ensuring the viability of subsequent construction activities. Through an analysis of hydration heat, the effect mechanism of TA on the cement-based self-leveling mortar is derived. The result shows that the addition of TA decelerates the hydration process within the initial 10 h, followed by acceleration in the subsequent 20 h to 30 h. Consequently, this delayed formation of the early hydration product, ettringite, contributes to a more porous structure in the slurry, with low friction leading to a better working performance. A large number of hydration products, such as alumina gel and calcium–silicon–hydrate gel, presented in the hardened paste results in the good mechanical properties at 1 d. This study may lay a foundation for the optimization of the dosage of chemical admixtures in the self-leveling mortar and high-performance cement-based materials, and also impart valuable insights for practical applications extending to the realm of building construction and decoration.

Keywords: self-leveling mortar; chemical admixtures; working performance; mechanical properties; tartaric acid

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1. Introduction

Owing to the excellent mechanical properties, durability, and low cost, cement-based materials are commonly used for various applications, including concrete for building foundations, bridges, and highways, as well as mortar for laying bricks and tiles, and stucco for exterior wall finishes [1–3]. Self-leveling mortar is a type of cement-based material used in building construction and decoration. It is known for its low viscosity and can be applied by hand in thin layers ranging from 1 to 10 mm on various types of floor substrates [4–6]. The objective of utilizing self-leveling mortar is to create a uniformly smooth substrate that serves as a base for the final flooring coverings. Self-leveling mortar is delivered as dry mortars to the construction site, where they have to be mixed with a predefined amount of water to obtain a ready-to-use mortar. Self-leveling mortar requires an excellent workability, which allows it to flow and self-level into a smooth and even substrate [7,8]. To meet this requirement, self-leveling mortars are designed to have low viscosity and high fluidity, allowing for easy application and a smooth finish. Another important requirement for self-leveling mortars is compatibility with the substrate onto which it is applied. This includes ensuring that the substrate is properly prepared and cleaned before application, and that the mortar has appropriate bonding and adhesion properties to prevent delamination or detachment [9,10].

To fulfill the requirements of self-leveling, early setting, and final properties, a self-leveling mortar contains at least ten different components plus water. Among these components, several organic additives are required to provide workability properties [11–13]. The commonly used chemical admixtures consist of water-reducing agents, water-retaining agents, retarders, defoaming agents, and hardening accelerators, along with hydrophobic admixtures, expansive agents, etc. Tartaric acid (TA) is used as a retarder in cement-based multicomponent gelling systems to prevent premature solidification before construction [14,15]. It reduces the hydration heat and rate of cement and gypsum, extending their setting and hardening time. Studies show it forms a film coating on cement particle surfaces during the early stage of hydration, influencing the hydration rate and process. The amount used affects ettringite formation and the early strength of the self-leveling mortar. Cement-based self-leveling mortar requires high fluidity and stability, but excessive water addition can lead to bleeding segregation. Polycarboxylate superplasticizer (PCE) is a widely used water-reducing agent in the building materials industry due to its strong dispersion and cost-effectiveness. It improves fluidity without adding excess water, facilitating drying and forming after construction [16–18]. Flatt [19] found that PCE influences cement hydration in three stages: precipitation with hydration products, coating on the cement surface, and existing as a solute in solution. Yamada [20] found that dispersion is influenced by the PEO chain length, molecular polymerization degree, and sulfonate group content. Suitable water-reducing agents and retarders are essential for preparing mortar with suitable characteristics for normal use. Cellulose ether (CE) is a commonly used water-retaining agent that maintains the fluidity of mortar and increases viscosity and fluidity by adhering to cement particle surfaces [21–23]. Cement-based self-leveling mortar has high fluidity, which can lead to fast drying and water loss during construction, affecting the formation of the material. Adding a suitable water-retaining agent, such as CE, can prevent these issues. CE, made from cellulose and sodium hydroxide, is a low-cost and stable option commonly used in the industry. It helps maintain the necessary water for cement hydration and improves the viscosity and fluidity of the mortar by adhering to cement particles. Research has shown that CE can effectively ensure the fluidity of mortar without water absorption or evaporation.

Given the significant influence of chemical admixtures on cement-based materials, researchers have been devoted to exploring their effects on properties, such as rheology, construction attributes, strength, shrinkage, etc., delving into their impact mechanisms through hydration process analyses [5–7,11–13]. Numerous studies affirm that the effect of chemical admixtures on cement-based materials is intertwined with changes in the hydration process and environment, such as the adsorption of the mineral surface, and the change in the hydration-product-formation rate due to affecting the concentration of ions in the solution. In terms of self-leveling mortar, more attention has been paid to the working performance and mechanical properties. For instance, Gasparo et al. [12] studied the impact of latex, PCE, CE, casein, and polyvinyl alcohol on the physical properties of cement-based self-leveling mortar, and illustrated the interaction between organic additives and the cement phase in the hydration solution. Zhi et al. [13] analyzed the effect of three retarders on the setting time of phosphorus-gypsum-based self-leveling mortar, and obtained the optimal composition and contents based on the mechanical properties of hardened mortar. Although numerous studies have researched the properties of chemical admixtures on cement-based materials, the influence rules and mechanisms of the multicomponent system under the coexistence of various chemical admixtures are still in the process of exploration.

By far, multicomponent cementitious systems are extensively applied in cement-based self-leveling mortar, with common ternary systems encompassing Portland cement, gypsum, calcium aluminate cement, or sulphoaluminate cement. In this work, the effect and mechanism of chemical admixtures (TA, CE, and PCE) on the practicability of self-leveling mortar based on the ternary system (Portland cement, calcium aluminate cement, gypsum) were elaborated. This study investigates the impact of TA, CE, and PCE on the fluidity, fluidity loss, flexural strength, and compressive strength of cement-based self-leveling mortar. An orthogonal test design is employed to explore the influence of these factors on mortar performance, and the results are validated and used to optimize the formulation. The optimized formulation is further utilized to explore the influence of TA on the working and mechanical properties of the mortar by through the single-factor variable adjustment of the mixing compositions. The effects of TA on self-leveling mortar are examined by analyzing early-stage hydration products and gels through characterization analyses of the hydration process. This research reveals the impact and mechanisms of chemical admixtures, especially TA, on the workability and mechanical attributes of cement-based self-leveling mortar, establishing a theoretical and experimental foundation for real-world engineering applications.

2. Materials and Methods

2.1. Orthogonal Experimental Design

An orthogonal experiment was conducted to optimize the preparation of anchoring material, which involved multiple variables and levels [24]. The material in question was a capsule composed of TA, CE, and PCE, and the dosages of each component were considered factors in the experiment. Specifically, the dosages of CE (A), TA (B), and PCE (C) were varied across four levels, each selected based on the typical values of the parameters. Table 1 shows the factors and their corresponding levels. To construct the orthogonal design, a three-factor and four-level orthogonal table was used (Table 2). Based on the L_{16} (4^3) orthogonal experimental design table, a total of 16 mixtures were tested (Table 2).

Table 1. Factors and levels of orthogonal experiment design (wt.‰).

Levels	Factors		
	A (Dosage of CE)	B (Dosage of TA)	C (Dosage of PCE)
1	0.6	0.2	1.4
2	0.7	0.3	1.6
3	0.8	0.4	1.8
4	0.9	0.5	2.0

Table 2. The orthogonal experiment design table L_{16} (4^3).

No.	A	B	C	Dosages of Each Raw Material (wt.‰)		
				A	B	C
1	1	1	1	0.6	0.2	1.4
2	1	2	2	0.6	0.3	1.6
3	1	3	3	0.6	0.4	1.8
4	1	4	4	0.6	0.5	2.0
5	2	1	2	0.7	0.2	1.6
6	2	2	1	0.7	0.3	1.4

Table 2. *Cont.*

No.	A	B	C	Dosages of Each Raw Material (wt.‰)		
				A	B	C
7	2	3	4	0.7	0.4	2.0
8	2	4	3	0.7	0.5	1.8
9	3	1	3	0.8	0.2	1.8
10	3	2	4	0.8	0.3	2.0
11	3	3	1	0.8	0.4	1.4
12	3	4	2	0.8	0.5	1.6
13	4	1	4	0.9	0.2	2.0
14	4	2	3	0.9	0.3	1.8
15	4	3	2	0.9	0.4	1.6
16	4	4	1	0.9	0.5	1.4

2.2. Raw Materials

In this study, two types of cement were used: ordinary Portland cement (OPC) and calcium aluminate cement (CA). The chemical compositions of each cement are listed in Table 3. The median diameter (d_{50}) of the OPC and CA are 15.86 μm and 7.15 μm, respectively. The quartz sand, acquired from the local market, boasts a specific gravity of 2.65 and a mesh size of 70–120. A type of high viscosity CE was selected as the stabilization agent in the self-leveling mortar, which exhibits a molecular weight of 6 W. The redispersible powder, sourced from Wacker Chemicals Co., Ltd., Shanghai, China, is a water-soluble white powder composed of a copolymer of ethylene and vinyl acetate, with polyvinyl alcohol as a protective colloid. Notably, the calcite powder demonstrates a purity level of 99.9%. For further insight, raw materials and the composition of the self-leveling mortar are shown in Table 4.

Table 3. Chemical compositions of OPC and CA used in this study (wt.%).

Materials	CaO	SiO_2	Fe_2O_3	Al_2O_3	MgO	TiO_2	R_2O	SO_3	Loss
OPC	61.25	22.04	3.86	4.21	2.58	/	0.97	2.54	2.55
CA	35.84	7.50	2.42	51.22	0.50	2.10	0.12	0.30	/

Table 4. Raw materials and the composition of self-leveling mortar.

Materials	Composition (wt.%)	Manufacturer	Type
OPC	8	China United Cement Co., Ltd., Jining, China	P.O 42.5
CA	25	Yaobai Cement Co., Ltd., Guizhou, China	CA50
Quartz sand	40	Local market	70–120 mesh
Anhydrite	4	Local market	/
Stabilization	0.03	Basf Chemical Co., Ltd., Beijing, China	Mw = 6W
Defoamer	0.1	Basf Chemical Co., Ltd.	P845
CE	According to orthogonal experiment design	Tianjin Damao Chemical Reagent Co., Ltd., Tianjin, China	AR
TA		Basf Chemical Co., Ltd.	MC400
PCE		Sobute New Materials Co., Ltd., Nanjing, China	PCA-I
Redispersible powder	1	Wacker Chemicals Co., Ltd., Shanghai, China	316N
Calcite powder	Residual	Local market	325 mesh

2.3. Mixing Procedure and Sample Preparation

The mixing procedure for the self-leveling mortar was carried out based on the composition listed in Table 4, and all materials are dry powder. Specifically, 1000 g of the dry mix was mixed with water at a water-to-binder ratio of 0.28. The mixture was subjected to low-speed mixing for 45 s, followed by a 15 s pause. Subsequently, the mortar was mixed at high speed for 60 s.

2.4. Test Methods and Characterizations

2.4.1. Fluidity

The fluidity test for the mortar samples was conducted following the guidelines outlined in standard JC/T 985-2017. A testing mold with an internal diameter of 30 mm ± 0.1 mm and a height of 50 mm ± 0.1 mm was horizontally positioned at the center of the testing board. Subsequently, the mold was filled with fresh mortar and lifted vertically within 2 s to enable the mortar to flow unrestrictedly. Following a 4 min interval, the diameters of the mortar in two perpendicular directions were gauged, and the average measurement was recorded as the initial fluidity of the mortar. Similarly, the diameter of the mixture was measured after 20 min and denoted as the fluidity after 20 min. The fluidity loss was calculated using the formula $S = (D_{In} - D_{20})/D_{In}$, where S represents the fluidity loss, D_{In} is the initial fluidity, and D_{20} is the fluidity after 20 min.

2.4.2. Viscosity of Mortar

The viscosity of mortar was determined using a 4 mm orifice ISO flow cup according to GB/T 1723-1993. The flow cup was first sealed and filled with mortar. Then, the outlet of the flow cup was opened and we recorded the flow time of the mortar. The flow time was considered as the viscosity of the mortar.

2.4.3. Flexural and Compressive Strength

The flexural and compressive strength of the mortar specimens were evaluated 1 day after casting. Samples with dimensions of 40 × 40 × 160 mm were prepared for both flexural and compressive strength testing according to the Chinese National Standard GB/T 15780-1995. The samples were first subjected to flexural strength testing, followed by compressive strength testing. The testing equipment and mortar sample are shown in Figure 1.

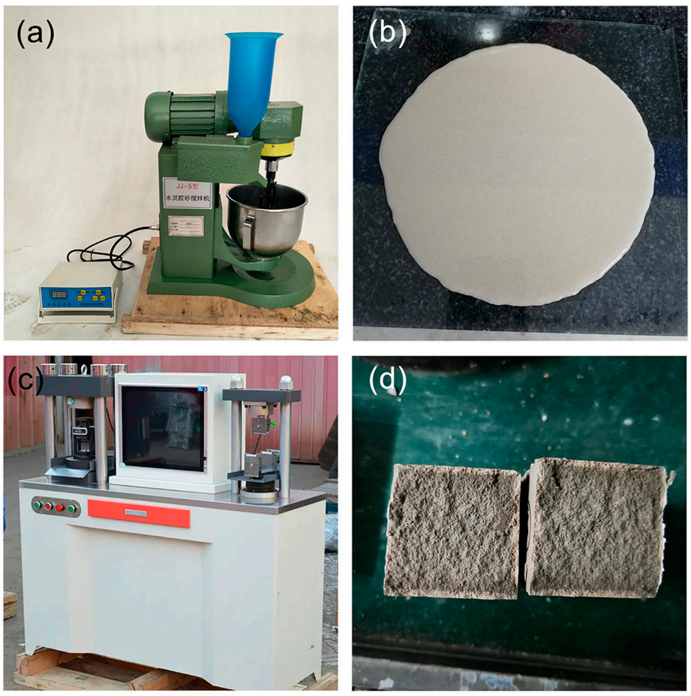

Figure 1. Experimental images: (**a**) Mortar mixer; (**b**) Mortar in the fluidity test; (**c**) Press pressing machine; (**d**) Mortar after flexural strength test.

2.4.4. Hydration Heat Analysis

The hydration heat evolution rate and cumulative hydration heat of the mortar were measured using an isothermal calorimeter (TAM Air) at a test temperature of 20 °C through-out 55 h. The TAM Air is outfitted with eight parallel twin-chamber measurement channels, each held at a consistent temperature. In each channel, one chamber accommodates the sample while the other holds the reference. The mortar samples were rapidly mixed to ensure homogeneity and then placed into the chamber. The hydration heat evolution rate and total hydration heat of the sample were continuously monitored as a function of time.

3. Results and Discussion

3.1. Impact of Chemical Admixtures on the Working Performance of the Self-Leveling Mortar

The comprehensive influence of three chemical admixtures on the working performance of cement-based self-leveling mortar was investigated by an orthogonal experiment. The indicators of the working performance, including initial fluidity, fluidity after 20 min, fluidity loss, viscosity, are shown in Table 5. Also, the experimental results were analyzed by range and variance statistical analysis. The results of the range analysis of the working performance are shown in Table 6.

Table 5. Orthogonal experiment results of working performance.

No.	Initial Fluidity (mm)	Fluidity after 20 min (mm)	Fluidity Loss	Viscosity (s)
1	145	110	0.241	66
2	160	140	0.125	85
3	165	148	0.103	73
4	164	148	0.097	74
5	158	122	0.227	85
6	162	145	0.104	89
7	155	145	0.064	75
8	165	140	0.151	74
9	150	125	0.167	107
10	159	134	0.157	100
11	148	133	0.101	91
12	166	147	0.114	85
13	158	133	0.158	80
14	162	136	0.160	101
15	166	134	0.192	105
16	168	150	0.107	90

Table 6. Range analysis of working performance.

Range Analysis	Initial Fluidity (mm)			Fluidity after 20 min (mm)			Fluidity Loss			Viscosity (s)		
	A	**B**	**C**	**A**	**B**	**C**	**A**	**B**	**C**	**A**	**B**	**C**
K1	634	611	617	546	490	524	0.566	0.794	0.608	298	338	347
K2	640	643	650	552	555	543	0.548	0.547	0.660	323	375	360
K3	623	634	648	539	560	563	0.539	0.461	0.528	383	344	344
K4	654	663	636	553	585	560	0.618	0.470	0.477	376	323	329
k1	158.50	152.75	154.25	136.50	122.50	131.00	0.141	0.198	0.152	74.50	84.50	86.75
k2	160.00	160.75	162.50	138.00	138.75	135.75	0.137	0.136	0.165	80.75	93.75	90.00
k3	155.75	158.50	162.00	134.75	140.00	140.75	0.134	0.115	0.132	95.75	86.00	86.00
k4	163.50	165.75	159.00	138.25	146.25	140.00	0.154	0.117	0.119	94.00	80.75	82.25
R	7.75	13.00	8.25	3.50	23.75	9.75	0.0197	0.0831	0.0456	21.25	13.00	7.75
Optimal		A3B4C2			A4B4C3			A3B3C4			A1B4C4	

In the range analysis, Ki (i = 1~4) represents the sum of the indicator under 1~4 levels, and ki (i = 1~4) is the average value of the indicator under 1~4 levels, indicating the influence of different levels of each factor on the working performance. The range R is the extreme difference, which can be obtained by subtracting the maximum and minimum values of their means, k1, k2, k3, and k4. The range R illustrates the degree of each factor's influence. The larger the R-value, the higher the influence of the factor on working performance, and vice versa.

3.1.1. Analysis of Fluidity

As for the fluidity, the initial fluidity is mainly related to the dispersion of particles in the paste. Chemical admixtures are rich in functional groups, which could absorb on to the surface of the particle and alert interfacial properties [25,26]. As shown in Figure 2, the initial fluidity lays in the range of 150~170 mm, which can meet the engineering requirement. In that condition, slurry could automatically flow to form a flat surface [27,28]. Furthermore, the initial fluidity is basically increased with the content of the chemical admixtures, indicating the chemical admixtures are conductive to the dispersion of particles. By sorting the range of the three influencing factors, the order is B > C > A. The result indicates that the order of importance for the factors affecting the initial fluidity of cement-based self-leveling mortar is TA > CE > PCE. Comparing the impact of the four levels of each factor on the initial fluidity, the combination of A4B4C2 is obtained, indicating that the optimal levels for CE, TA, and PCE are 0.9 wt.‰, 0.5 wt.‰, and 1.6 wt.‰, respectively. Paradoxically, the minimum point on the A curve is attained at level 3. This observation could be explained by the low fluidity value in the No. 11 experiment, where the fluidity is 148 mm. The lower fluidity can be attributed to the relatively higher content of TA and PCE, as well as the lower CE content, all occurring simultaneously in the mixture.

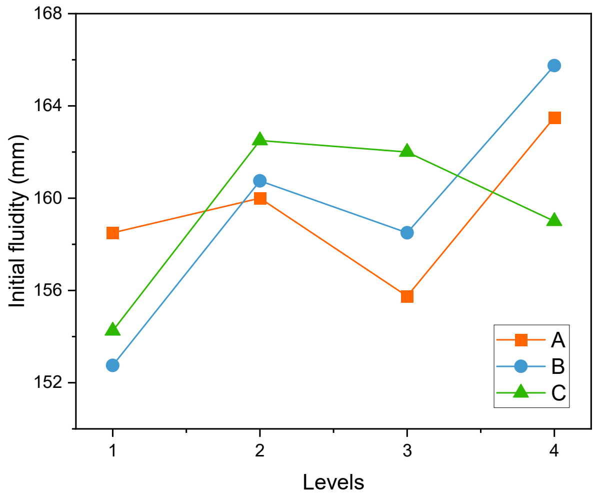

Figure 2. The changing trend of the initial fluidity of the three factors at four levels.

Furthermore, the analysis of variance is used to analyze the results of the orthogonal experiment initial fluidity of the cement-based self-leveling mortar, as shown in Table 7. It can be seen that the F values of B and C are larger than the critical value of $F_{0.05}$ and $F_{0.1}$, respectively, implying TA and PCE are the significant factors of the initial fluidity of the cement-based self-leveling mortar. It can be concluded that the factors that affect the initial fluidity of the cement-based self-leveling mortar are ranked as follows: TA > PCE > CE. This phenomenon may be related to the electronegativity of chemical admixtures. The stronger the electronegativity, the greater the degree of adsorption on the particles [29–31]. It is known that TA is a small molecule with two carboxylic acid and hydroxyl groups, PCE is a long-chain molecule with carboxylic acid functional groups located on the comb branch chain, and CE is a polymer compound with ether structure. Thus, TA has the strongest electronegativity among three chemical admixtures, followed by PCE, and the weakest is CE. According to the above analysis, the significant effect of the TA dosage on the initial fluidity is related to its strong electronegativity.

Table 7. Analysis of variance for the initial fluidity.

No.	SS [1]	df [2]	MS [3]	F [4]	F_α	Significance
A	125.18	3	41.73	4.29	29.457 (α = 0.01)	
B	306.68	3	102.23	10.51	9.28 (α = 0.05)	**
C	172.18	3	57.39	5.89	5.39 (α = 0.1)	*
Error	29.19	3	9.73			

[1] SS, sums of squares; [2] df, degrees of freedom; [3] MS, mean squares; [4] F, test statistic.

Experimental results of the fluidity after 20 min and the range analysis of the factors for the cement-based self-leveling mortars are shown in Table 6. Similar to the range analysis method for the initial fluidity in the orthogonal experiment, the range sizes of the three factors were sorted in the order of B > C > A. That is, the significant orders of the factors affecting the fluidity after 20 min are TA > PCE > CE. At this stage of hydration, the cement and anhydrite are initially hydrated to form hydration products such as ettringite, aluminum glue, calcium hydroxide, etc. Therefore, the fluidity of the paste is related to the microstructure built by the hydration products. As presented in Figure 3, the fluidity after 20 min is increased significantly with the increase in the TA content, while almost steady with the other two chemical admixtures. Due to the limited range of the overall variation, curves A and C exhibit some degree of fluctuation. The significant influence upon TA can be ascribed by the retardant of the hydration effect, especially delaying the formation of ettringite at the early hydration period [32,33]. Therefore, the fluidity after 20 min is significantly more influenced by the TA dosage than PCE and CE. Comparing the influence of each factor's four levels on the fluidity after 20 min, the optimal combination is A4B4C3, with the optimal condition being CE = 0.9 wt.‰, TA = 0.5 wt.‰, and PCE = 1.8 wt.‰.

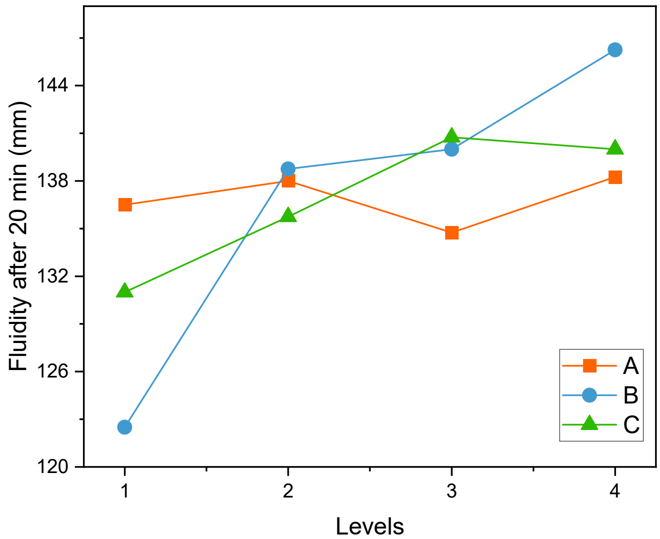

Figure 3. The changing trend of fluidity after 20 min of the three factors at four levels.

Using the variance analysis method, an orthogonal test was conducted to analyze the fluidity after 20 min of the cement-based self-leveling mortar. The significance of the three factors was determined, as shown in Table 8. It can be seen that, at a confidence level of 99%, the F value for B is larger than the critical value of $F_{0.01}$. Moreover, the F value of C is larger than the critical value of $F_{0.05}$. Therefore, the effects of TA and PCE on the fluidity after 20 min in the cement-based multicomponent system are both significant. From the analysis of the two types of fluidity results, TA has a relatively significantly influence the fluidity at both hydration periods, while PCE has no significant impact at the beginning of hydration and becomes a significant factor at 20 min. This indicates that the effect of chemical admixtures on the mortar requires an activation time, and the activation time of PCE ranges from 5 min to 20 min.

Table 8. Analysis of variance for fluidity after 20 min.

No.	SS [1]	df [2]	MS [3]	F [4]	F_α	Significance
A	31.25	3	10.42	1.26	29.457 (α = 0.01)	
B	1119.25	3	373.10	45.22	9.28 (α = 0.05)	***
C	242.25	3	80.75	9.79	5.39 (α = 0.1)	**
Error	24.75	3	8.25			

[1] SS, sums of squares; [2] df, degrees of freedom; [3] MS, mean squares; [4] F, test statistic.

Next, the changing trend of fluidity loss is shown in Table 6 and Figure 4. By sorting the range sizes of the three influencing factors, the order is B > C > A. According to the requirements for the use of cement-based self-leveling mortar, the material needs to have a high flowability to fully demonstrate its characteristics. Therefore, the sequence of factors affecting the flow loss of the cement-based multicomponent binder system is TA > PCE > CE. As shown in Figure 4, the fluidity loss is basically decreased with the content of TA and PCE. This trend is attributed to the fact that TA and PCE can retard hydration by adsorbing onto mineral phases and inhibiting the precipitate of hydration products [34]. On the contrary, CE brings a slight increase in fluidity loss when the content is 0.9 wt.‰. This may be due to the effective interaction between CE molecular chains and hydration products after hydration, resulting in a faster growth rate of frictional force in the paste and a greater loss of fluidity [35–37]. Comparing the effects of the four levels of each factor on the flow loss, the optimal combination is A3B3C4, which means the optimal condition is CE = 0.8 wt.‰, TA = 0.4 wt.‰, and PCE = 2.0 wt.‰.

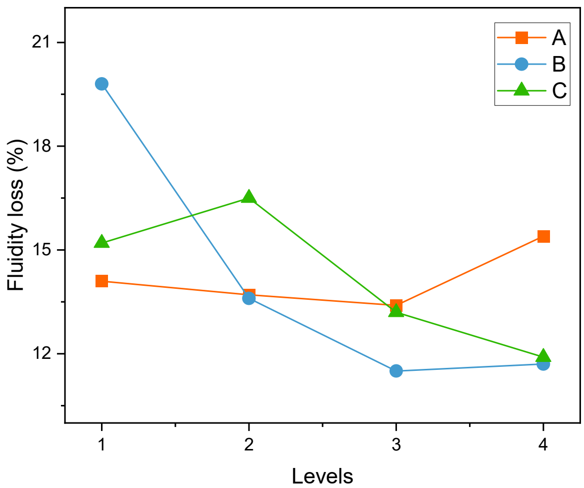

Figure 4. The changing trend of fluidity loss of the three factors at four levels.

3.1.2. Analysis of Viscosity

Viscosity is also an important indicator of the working performance. The results of the viscosity test and its range analysis are shown in Table 6 and Figure 5. By ranking the range sizes of the three influencing factors, it can be determined that the order of importance of the factors affecting the viscosity of the cement-based self-leveling mortar is: CE > TA > PCE. In this case, CE is the most significant factor affecting the viscosity of the paste, owed to the combined effects of the high molecular weight, hydrogen bonding, spatial interaction, and ion effect of CE [21,38,39]. However, upon reaching a CE dosage of 1.8, the viscosity value ceases to increase, implying that the dosage has already reached its peak value. By comparing the effects of the four levels of each factor on the 5 min viscosity, the combination of A1B4C4 was obtained, with the optimal level of CE at 0.6 wt.‰, TA at 0.5 wt.‰, and PCE at 2.0 wt.‰.

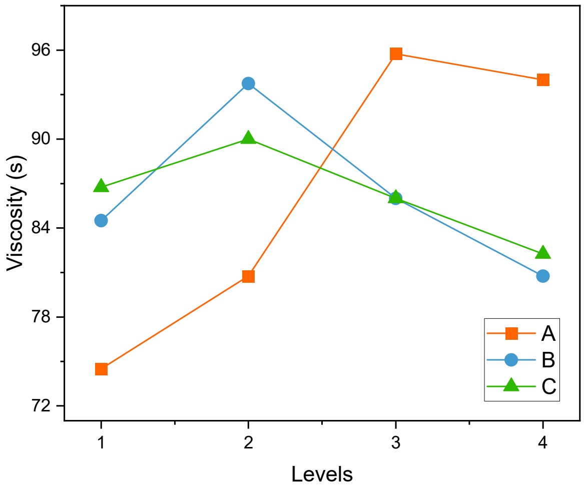

Figure 5. The changing trend of viscosity of the three factors at four levels.

According to the variance analysis of the orthogonal experiment on the viscosity of the cement-based adhesive materials, the significance of the three factors was determined, as shown in Table 9. It can be seen that the F value of A is larger than the critical value of $F_{0.1}$, thus CE is the significant factor of viscosity of the cement-based self-leveling mortar. The F values of B and C are much lower than $F_{0.1}$, indicating the TA and PCE are not significant factors, and the relevant variations can be negligible.

Table 9. Analysis of variance for viscosity.

No.	SS [1]	df [2]	MS [3]	F [4]	F_α	Significance
A	1274.51	3	415.83	5.81	29.457 (α = 0.01)	*
B	133.01	3	44.34	0.68	9.28 (α = 0.05)	
C	121.52	3	40.51	0.55	5.39 (α = 0.1)	
Error	219.49	3	73.16			

[1] SS, sums of squares; [2] df, degrees of freedom; [3] MS, mean squares; [4] F, test statistic.

3.2. Determination of the Optimal Mixture Ratio

Three optimal conditions were obtained from the orthogonal experimental design of each working performance indicator, which is No. 16 of the initial fluidity and fluidity after 20 min experiments, No. 7 of the fluidity loss experiment, and No. 1 of the viscosity experiment, respectively. Therefore, the verification experiment of the orthogonal design had six tests, and the test schemes and results are shown in Table 10. By analyzing the results of the verification experiment, it was found that the mechanical performance of the No. 1, No. 3, and No. 4 was poor, and the compressive and flexural strengths could not meet the application conditions. The mechanical properties of the mortar of No. 5 are improved, where the flow loss was large, making it unsuitable in the condition of long-term transportation. Therefore, the levels of each group in the verification experiment were only the optimal levels obtained from the theoretical analysis and were not the best ratio in practical applications. Therefore, by synthesizing the results of the orthogonal experimental design and the verification experiment, No. 5 in the orthogonal experimental design was determined as the optimal formulation with the level of CE = 0.6 wt.‰, TA = 0.5 wt.‰, and PCE = 2.0 wt.‰.

Table 10. Verification experiment of the orthogonal experiment.

No.	A	B	C	Working Performance				Mechanical Properties	
				Initial Fluidity (mm)	Fluidity after 20 min (mm)	Fluidity Loss	Viscosity (s)	Compressive Strength (MPa)	Flexural Strength (MPa)
1	4	4	1	168	151	0.101	91	5.9	1.98
2	2	3	4	157	146	0.070	74	6.4	2.14
3	1	1	1	144	112	0.222	66	5.5	1.73
4	4	4	2	147	112	0.238	70	5.8	1.82
5	4	4	3	150	118	0.271	65	6.2	2.06
6	1	4	4	162	142	0.123	58	6.7	2.20

3.3. Analysis of the Influence of TA on the Hydration Process of the Self-Leveling Mortar

3.3.1. Analysis of Working Performance

Due to the noteworthy impact of TA on the working performance, an in-depth exploration of its effects on both the working performance and mechanical properties of the mortar was undertaken. The mixing ratio followed the No. 5 formulation, wherein the dosages of CE and PCE were 0.6 wt.‰ and 2.0 wt.‰, respectively. The fluidity of the mortar is shown in Figure 6. It can be observed that, as the amount of TA increased, both initial fluidity and fluidity after 20 min show a steadily increasing trend. Furthermore, the fluidity loss is decreased from 0.36 (TA = 0.2 wt.‰) to 0.11 (TA = 0.6 wt.‰) and decreases exponentially with the increase in the TA content. The mathematical fit formula of the fluidity loss (y) with respect to the TA content (x) is as follows:

$$y = 0.33245 \times \exp\left(-\frac{x - 0.26072}{0.37529}\right) - 0.02038$$

The mortar demonstrated improved fluidity with increasing TA content. This phenomenon is consistent with the previous reports [40–42]. After adding retarders such as TA, the TA in the cement slurry reacts with calcium ions to form complex salts, which are insoluble in water and form a complex salt film on the surface of the particles [43–46]. The appearance of the complex salt film also prevents the hydration products from dissolving in water and even temporarily suspends the hydration reaction when the film is too thick. Therefore, as the amount of TA increases, the hydration reaction process slows down, the setting time is prolonged, and the flowability of the self-leveling mortar within the same time range increases.

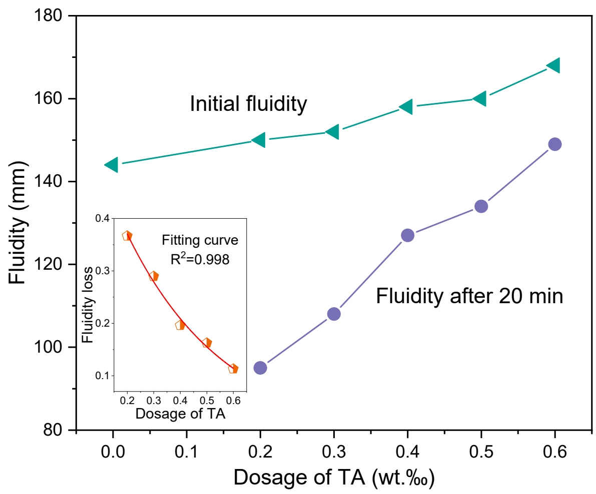

Figure 6. The initial fluidity and fluidity after 20 min of mortar with different TA dosages.

3.3.2. Analysis of Mechanical Properties

Regarding the effect of TA on the mechanical properties, the compressive and flexural strength were collected. From an application perspective, 1 d strengths are used as a representative of the early strength. As shown in Figure 7, the mechanical properties of the mortar are initially increased and then decreased with the addition of TA. The critical value of the TA dosage is 0.4 wt.‰, which is consistent with reported results [47]. The effect of TA on the mechanical properties is related to the hydration process and hydration production. As a commonly used retarder, TA affects the workability and mechanical properties of cement-based self-leveling mortar by acting on the induction period of the hydration process. TA could suppress the hydration process of the aluminate phase and delay the initial formation of hydration products, especially ettringite, thereby influencing the initial structure of the mortar at an early age [48,49]. According to research [50], after hydration for 5 h, the blocking effect of TA on the silicate mineral is weakened, and it helps to increase the precipitation rate of calcium hydroxide and other hydration products. Therefore, the effect of TA on the strength is relatively complex and needs to be inferred based on hydration reactions analysis.

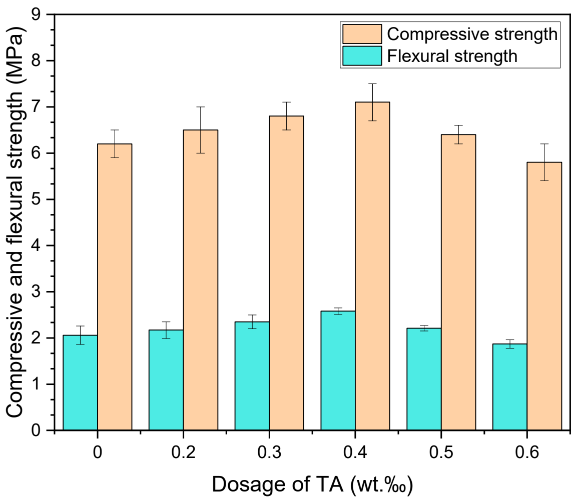

Figure 7. The compressive and flexural strengths (1 d) of mortar with different TA dosages.

3.3.3. Analysis of Hydration Heat

The hydration heat of each sample in the TA single-factor test was measured using an eight-channel calorimeter, and further supported by the analysis of the hydration heat change. The results are shown in Figure 8. As depicted in Figure 8a, with the increase in the TA dosage, the hydration heat of the mortar is delayed. The initial peak of the hydration heat shifts from occurring at 8 h to manifesting at 15 h, while the subsequent peak is postponed from 18 h to 28 h. Moreover, the intensity of the exothermic peak is influenced by the amount of TA, notably with the suppression of the first exothermic peak and the enhancement of the second one. Figure 8b shows that the cumulative hydration heat increases as the TA dosage increases from 0.2 wt.‰ to 0.6 wt.‰. This trend is particularly associated with the second peak of the hydration heat. At a dosage of 0.4 wt.‰ (curve 4), the cumulative hydration heat is about 55 J/g.

The first exothermic peak represents the hydration heat of aluminate minerals (CA, CA_2, C_3A, etc.) and anhydrite, whereas the second exothermic peak represents the hydration heat originating from silicate minerals (C_3S, C_2S, etc.) [51,52]. By scrutinizing the release rate of the hydration heat and the cumulative hydration heat, it can be inferred that the hydration reaction of aluminate minerals and anhydrite is inhibited with the increase in the TA content at the early stage of hydration (before curing). The hydration products of aluminate minerals and anhydrite are basically ettringite and alumina gel. With the increase in the TA content, the formation of ettringite is inhibited, leading to a more porous structure and a better working performance [53]. After approximately 20 h hydration, silicate-mineral hydration is dominant, forming calcium–silicon–hydrate gel to provide strength. According to the hydration heat analysis, when the content of TA increases, the hydration of the silicate minerals is delayed and strengthened. Two main reasons may account for this result.

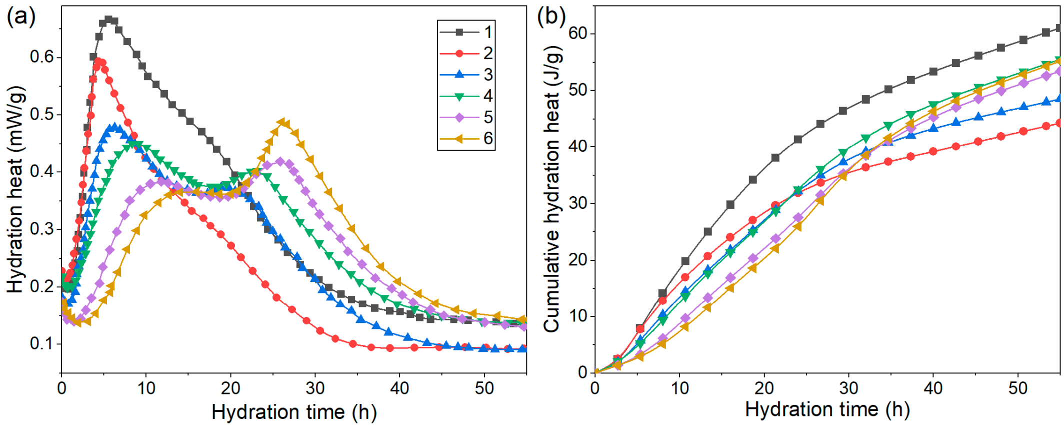

Figure 8. The hydration heat (**a**) and cumulative hydration heat (**b**) of mortar with different TA dosages.

Firstly, the hydration of the silicate minerals is suppressed by the concurrent hydration of the aluminate minerals [54]. Upon the introduction of TA, the hydration of the aluminate minerals is inhibited, resulting in fewer aluminum ions and less ettringite formation. Consequently, the impact on the hydration of silicate minerals is mitigated, ultimately bolstering their hydration process. Thus, the hydration of silicate minerals is enhanced. Secondly, based on the study upon the influence of TA on C_3S, although the precipitate time of the hydration products is delayed by TA, the precipitate rate can be improved [55]. Furthermore, due to the complexation and adsorption of TA, the crystal growth of calcium hydroxide is enhanced. Thus, the enhancement of the second exothermic peak may be ascribed to the influence of TA on silicate minerals. These two reasons could synergistically bring about alterations in the hydration heat, consequently exerting an impact on the compressive and flexural strengths at the early age. When the TA concentration is below 0.4 wt.‰, owing to the enhanced hydration of silicate minerals, a large number of hydration products of silicate minerals are formed. As hydration proceeds, these hydration products accumulate to engender a dense structure, thereby affording commendable mechanical properties [56]. Moreover, although the formation of ettringite is delayed, the content of alumina gel is increased, resulting in an increase in the mechanical properties [57]. It can be inferred that, when the dosage of TA is 0.4 wt.‰, the hydration process is moderate, and the homogeneously dispersed hydration products foster the most compact structural configuration. Therefore, both the compressive and flexural strengths surpass those of other mortar formulations [58]. However, when the TA concentration exceeds 0.4 wt.‰, due to considerable delays in the hydration of aluminate and silicate minerals, the accumulation of the hydration products remains insufficient, impeding the establishment of an effective structural network. This deficiency culminates in diminished mechanical properties.

3.4. Practicability and Prospect of the Research Results

The widespread application of self-leveling mortar in cementitious materials encompasses a variety of components, including Portland cement, calcium alumina cement, gypsum, and limestone powder. Investigating the effect of self-leveling mortar on the subject of interest holds significant importance. The role of chemical admixtures is crucial in this context. The use of an orthogonal test method, which is a scientifically designed experimental approach, allows for data analysis to identify influential factors. In this study, TA emerged as the most significant factor affecting the fluidity, fluidity loss, and viscosity of cement-based self-leveling mortar among the three types of admixtures investigated. Further exploration was conducted to understand the underlying mechanisms impacted by TA. Single-factor variable experiments demonstrated that an increase in the TA dosage resulted in the increased fluidity of the mortar but decreased flexural and compressive strength. Hydration heat analysis suggested that the addition of TA decelerated the hydration process, consequently delaying the formation of ettringite during the early hydration period. This delay contributed to the formation of a more porous structure within the mortar. The findings of this study contribute to a deeper understanding of the effects of TA on cement-based self-leveling mortar. This understanding serves as a foundation for optimizing the dosage of chemical admixtures in self-leveling mortar and has potential implications for the development of high-performance cement-based materials. By comprehending the influencing patterns and leveraging the action mechanisms, it becomes possible to design materials that are more scientifically rational. This not only provides assurance but also offers a pathway towards achieving sustainable development goals in the field of building construction and decoration. In conclusion, this research provides valuable insights into the effects of TA on the working performance and mechanical properties of cement-based self-leveling mortar. The findings have the potential to contribute to the optimization of chemical admixture dosages in self-leveling mortar and hold relevance for the development of high-performance cement-based materials. Subsequent research related to this topic should focus on several aspects. Firstly, it is necessary to further elucidate the mechanism of TA during each hydration period, distinguishing between chemical and physical adsorption. Then, we can better understand the reaction degree and methods of chemical admixtures in the hydration process. Moreover, investigating the mutual influence of various admixtures under the condition of coexistence, such as whether the effect of chemical admixtures on the cementitious system takes precedence or exhibits a shielding effect.

4. Conclusions

In summary, this study investigated the effect of chemical admixtures, namely tartaric acid (TA), cellulose ether (CE), and polycarboxylate superplasticizer (PCE), on the working performance and mechanical properties of cement-based self-leveling mortar. The study encompassed the following aspects:

(1) An orthogonal experiment was designed to optimize the preparation of the mortar involving multivariate and multilevel factors. This approach facilitated a systematic exploration of the interplay between these factors;

(2) The orthogonal experiment results showed that TA emerged as the most influential factor impacting the fluidity, fluidity loss, and viscosity of the cement-based self-leveling mortar. Its effect outstripped that of CE and PCE. The initial fluidity is basically increased with the content of the chemical admixtures, and followed by the fluidity after 20 min, it increased significantly with the increase in the TA content, but exhibited a steady condition with the other two chemical admixtures. The fluidity loss is basically decreased with the content of TA and PCE, which is related to the retarding of hydration by adsorbing onto the mineral phases and inhibiting the precipitate of the hydration products. As for the viscosity, CE is the most significant factor increasing the viscosity of the paste;

(3) The single-factor variable experiment results showed that, as the dosage of TA increased, the fluidity of the mortar exhibited a gradual enhancement. However, the compressive and flexural strength of the mortar first increased and then decreased with the addition of TA, which is mainly related to the structure in the paste, which is composed of hydration products;

(4) The mechanism for the effect of the TA on the working performance and mechanical properties is elucidated. The hydration heat analysis disclosed that the hydration process of aluminate minerals and silicate minerals is varied by adding TA. The delayed formation of ettringite results in the development of a porous structure of

slurry with reduced friction, leading to improved workability. Moreover, a large number of hydration products from silicate minerals contributes to an increase in the mechanical properties.

In sum, this study provides insights into the effect of chemical admixtures on cement-based self-leveling mortar. These findings could serve as a foundational reference for refining the application of chemical admixtures in self-leveling mortar. Moreover, the implications extend to the realm of developing high-performance cement-based materials. The potential for optimization and informed material design holds promise for advancing the field of construction and decoration in a sustainable manner.

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