

Comparative study on different methods of activation of recycled powder grouts

Research Article

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Received 18 September 2024; Accepted 21 November 2024

Abstract: The high-value utilization of recycled powder (RP), primarily derived from construction and demolition waste, has been limited due to its low reactivity. In this study, the effect of RP subjected to three types of inorganic alkalis (sodium hydroxide, sodium carbonate, and calcium hydroxide [CH]), two alkanolamines (diethanolisopropanolamine [DEIPA] and triisopropanolamine [TIPA]), elevated temperatures, and their combined activation on the technical properties of RP grouts was analyzed. X-ray diffraction (XRD) and scanning electron microscopy (SEM) were employed to investigate the development of the mineral composition and micromorphology of the grout pastes. The results indicated that alkali and thermal activation of RP had negative effects, while combined activation improved the fluidity of the grout pastes. The compressive strength of alkali-activated groups was slightly enhanced at 1 day but significantly decreased at 28 days. In contrast, the compressive strength of grouts activated with CH, alkanolamines, and thermal treatment was found to be improved at all ages. The compressive strength of the grout paste containing 40% combined-activated RP was measured at 43.1, 73.3, and 95.7 MPa at 1, 3, and 28 days, respectively, which represented increases of 20.4, 19.6, and 17.7%, respectively, compared to the non-activated grout. Combined activation demonstrated the most improvement in the microstructural density of the grouts when compared to the single-activation mode.

Keywords: Grout • Recycled powder • Activation mode • Technical properties

1. Introduction

Currently, many countries around the world are grappling with the issue of construction and demolition waste (CDW) due to infrastructure upgrades and the construction and demolition of buildings. It is estimated that more than 10 billion tons of CDW are generated globally each year [1]. Significant amounts of CDW have been produced as a result of rapid urban construction, demolition and renovation of old buildings, and development and construction of new city districts in China over the past decade. However, CDW is usually disposed of in landfills, which not only wastes resources but also occupies a substantial amount of land, degrades the soil quality, disrupts the urban landscape, and poses a threat to the ecological

environment [2–6]. Furthermore, the average recycling rate of CDW in China has remained below 10% [7].

Generally, waste concrete and clay bricks constitute approximately 70–80% of CDW [8–12]. Utilizing waste concrete and clay bricks as recycled aggregates is a relatively straightforward process [10–12]. During the preparation of recycled aggregates from CDW through crushing, screening, and shaping [13], a significant quantity of recycled powder (RP) is generated, accounting for 20–30% of the volume of CDW, with a particle size <75 µm [14]. Some researchers have also suggested that the fine powder collected during the preparation of recycled aggregates may be used as supplemental cementitious materials (SCMs) because it contains unhydrated cement grains, calcite, quartz, ettringite (AFt), mono-sulfoaluminate hydrate (AFm), portlandite, and the calcium silicate hydrate (C–S–H) gel [15–18]. However, several studies have found that the RP obtained directly from recycled aggregate production adversely affects the workability, compressive

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and flexural strength, durability, and other engineering properties of cement-based materials due to its large mean particle size and low reactivity [19–21]. Therefore, RP requires further activation before it can be effectively utilized in cement-based materials.

Over the past decade, various activation methods, including mechanical, chemical, and thermal activation, have been employed to enhance the activity of RP [22–24]. Mechanical activation is a process that improves the reactivity of RP by reducing its particle size and increasing the surface area [25]. Chemical activation can disrupt the Si–O or Al–O bonds present in RP, leading to the formation of new gel products through polycondensation reactions [24,26]. When an alkanolamine is utilized as a chemical additive in cement, it can alter the hydration and hardening properties of the cement bond [27,28].

Although RP has been utilized as a supplementary cementitious material in various cement-based applications, limited research has focused on its use in cement-based grout [29]. In the present work, three types of inorganic alkalis (sodium hydroxide, sodium carbonate, and calcium hydroxide [CH]), two types of alkanolamines (diethanolisopropanolamine [DEIPA] and triisopropanolamine [TIPA]), elevated temperatures, and two combined activation modes were investigated to enhance the technical properties of RP grouts. The research evaluates the compressive strength and fluidity of grouts containing varying levels of non-activated RP. Additionally, X-ray diffraction (XRD) and scanning electron microscopy (SEM) were employed to further analyze the mineral composition and microstructure of grouts with activated RP. The objective of this study is to promote the high-value utilization of RP in the field of cement-based materials.

2. Experimental section

2.1 Raw materials

RP was supplied by Yangzhou Huimin Renewable Resources Co., Ltd (Yangzhou, P. R. China). The SEM image and particle size distribution of RP are shown in Figures 1 and 2, respectively. The D_{50} and specific surface area of RP are $29.01\ \mu\text{m}$ and $378\ \text{m}^2/\text{kg}$, respectively. The RP was placed in a muffle furnace and calcined at 700°C for 120 min, if necessary. P.I. 52.5 Portland cement, which was purchased from Taizhou Yangwan Conch Cement Co., Ltd. (Taizhou, P. R. China), was used in this article. The chemical compositions of RP and Portland cement are listed in Table 1. Analytical-grade

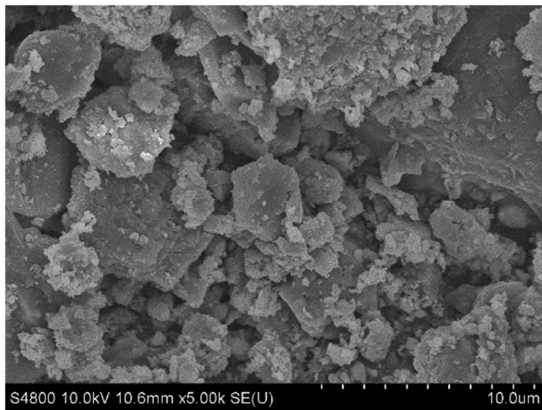


Figure 1. SEM image of RP.

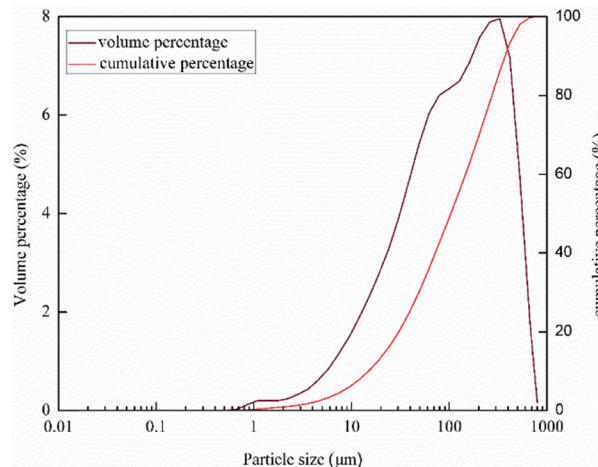


Figure 2. Particle size distribution of RP.

Composition	CaO	Al ₂ O ₃	SiO ₂	MgO	Fe ₂ O ₃	Na ₂ O	TiO ₂	SO ₃	Loss
RP	40.48	7.6	42.56	2.4	4.4	0.38	0.76	—	1.42
cement	63.71	5.0	20.35	1.37	3.3	0.39	—	2.48	2.6

Table 1. Chemical composition of RP and cement (wt%).

sodium hydroxide (NaOH), sodium carbonate (Na₂CO₃), and CH were supplied by Sinopharm Chemical Reagent Co., Ltd (Shanghai, P. R. China). Commercial-grade DEIPA and TIPA were obtained from Sigma-Aldrich Chemical Reagent Co., Ltd (Saint Louis, USA), and the chemical structure and composition of DEIPA and TIPA are illustrated in Scheme 1. Polycarboxylic ether (PCE) superplasticizer (P540) was purchased from Shanghai Chengqi Chemical Technology Company (Shanghai, P. R. China). The defoaming agent (P80) was obtained from MÜNZING Chemie Corporation (Münzingstraße, Germany). The expanding agent (YH-S) was supplied by Tianjin Weihe Technology Admixture Company (Tianjin, P. R. China).

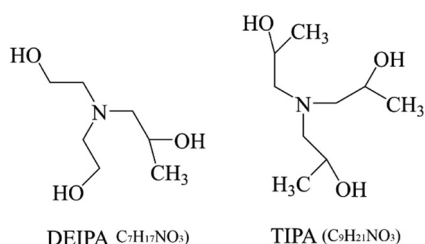
2.2 Sample preparation

Both grout pastes were prepared with a water-to-binder (w/b) ratio of 0.22. The replacement ratios of RP were set at

0, 10, 20, 30, and 40% by mass. The dosages of P540, P80, and YH-S were 0.7, 0.1, and 0.05%, respectively. Cement and RP were mixed in a V-shaped blender according to the predetermined mix ratios to achieve a homogeneous consistency. Subsequently, water and chemical additives were added successively into the NJ-160 cement paste mixer, immediately followed by the addition of the homogeneous mixture. The paste was mixed according to the specified procedure and placed into a test mold measuring 40 mm × 40 mm × 160 mm. The specimens were removed from the mold after 24 h and placed in a standard curing box with a relative humidity of 98% and a temperature of 20 ± 2°C. Compressive strength was tested in accordance with the Chinese Standards GB/T 17671-2021 [30] and was measured at 1, 3, and 28 days, with average results obtained from at least three samples. The mix design and group numbers of grouts are listed in Table 2.

2.3 Experimental method

The fluidity was assessed in accordance with the Chinese Building Industry Standards JG/T 408-2013 [31]. Initially, the mixed grouting paste was poured into a truncated cone mold. Subsequently, the mold was gradually elevated to allow for the unrestricted flow of the paste until it ceased to spread. The maximum diameter of the paste's spread was then measured and recorded as the initial fluidity. After a 30-min interval, the grouting paste was

**Scheme 1.** Chemical structure and composition of DEIPA and TIPA.

Sample	w/c	C/S	Cement (%)	RP (%)	P540 (%)	P80 (%)	YH-S (%)	Activator
S1	0.22	1	60	40	0.7	0.1	0.05	—
S2	0.22	1	60	40	0.7	0.1	0.05	1% NaOH
S3	0.22	1	60	40	0.7	0.1	0.05	1% Na ₂ CO ₃
S4	0.22	1	60	40	0.7	0.1	0.05	1% CH
S5	0.22	1	60	40	0.7	0.1	0.05	0.04% DEIPA
S6	0.22	1	60	40	0.7	0.1	0.05	0.25% TIPA
S7	0.22	1	60	40	0.7	0.1	0.05	700°C
S8	0.22	1	60	40	0.7	0.1	0.05	700°C + 1% CH + 0.04% DEIPA
S9	0.22	1	60	40	0.7	0.1	0.05	700°C + 0.04% DEIPA + 0.25% TIPA

Table 2. Mix design and group number of RP grouts.

re-mixed and measured again to determine the fluidity at the 30-min mark. It is essential that each fluidity assessment was conducted at least three times, with the average value calculated from these measurements.

An ADVANCE Bruker D8 X-ray diffractometer was used to identify the mineral compositions (Cu K α radiation, 2θ from 5° to 65°).

SEM images were recorded using an S-4800 scanning electron microscope (Hitachi, Japan), and the fracture surface was sputter-coated with gold before observation.

3. Results and discussion

3.1 Fluidity and compressive strength of grouts with different contents of RP

The influence of RP content on the fluidity of cement-based grouts is illustrated in Figure 3. It can be observed that fluidity increases with the addition of RP content; however, this increasing trend diminishes beyond a certain threshold. The grout without RP exhibits good fluidity and no time loss. Besides, the initial fluidity of grouts containing up to 10% RP exhibited an increasing trend with minimal time loss. Besides, the initial fluidity of grouts containing up to 10% RP shows an increasing trend with minimal time loss. However, when the RP content exceeds 20%, there is a significant decrease in initial fluidity, accompanied by an increase in time loss as the RP content is increased. On one hand, RP may fill voids [32] resulting from aggregate accumulation, optimize grain composition [33], and enhance grout fluidity. On the other hand, RP

typically exhibits high water absorption [34–36], which can adversely affect the workability of fresh grouts. Furthermore, RP possess a large specific surface area, a rough texture, and an irregular shape. Consequently, when the RP content exceeds 20%, fluidity may decrease significantly. Moreover, the internal absorption of RP by PCE may further diminish the fluidity of grouts containing a high proportion of RP [37].

The compressive strength of grouts with varying RP contents is depicted in Figure 4. It is evident that the compressive strength of the grouts at 1, 3, and 28 days decreases as the RP content increases. Furthermore, the reduction in strength exhibits a linear correlation with increasing RP content. Notably, the strength of grout containing 40% RP at each age falls below the specified value outlined in JG/T 408-2013 (Cementitious grout for coupler of rebar splicing).

3.2 Effect of activation modes on the fluidity and compressive strength of RP grouts

The influence of activation modes on the fluidity of RP grouts is depicted in Figure 5. In the case of single activation mode, the fluidity of samples S2, S3, and S7 decreased, while the fluidity of sample S6 increased compared to the non-activated sample S1. Besides, the fluidity of samples S4 and S5 was close to that of the S1 sample. Specifically, NaOH, Na₂CO₃, and thermal activation resulted in a reduction in the fluidity of RP grouts. Furthermore, CH and DEIPA activation showed no significant effect on the fluidity. Notably, TIPA activation may substantially enhance the fluidity. For example, the initial fluidity of samples S2 and S3 decreased by 27 and 29 mm, respectively, compared to that of sample S1. The

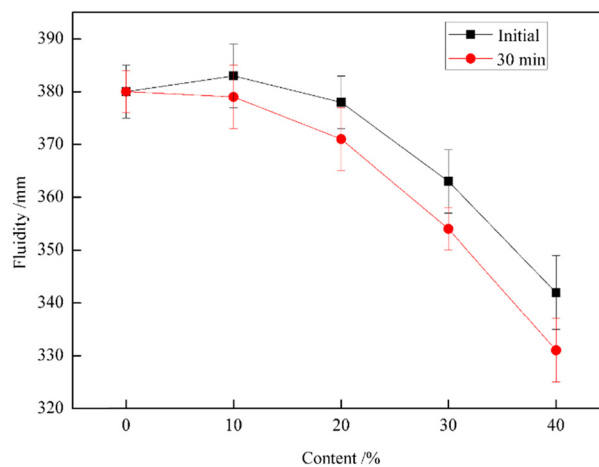


Figure 3. Influence of RP content on the fluidity of cement-based grouts.

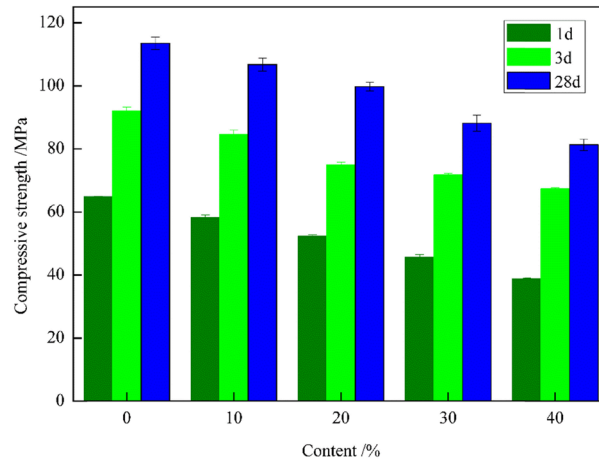


Figure 4. Compressive strength of grouts with different contents of RP.

inclusion of the thermally activated RP adversely affects the fluidity of the grouts, which decreased to 305 and 254 mm for the initial and 30-min fluidity measurements, respectively. In contrast, the initial and 30-min fluidity values of sample S6 were 370 and 356 mm, respectively. Among the two combined activation groups, the fluidity of sample S9 was higher than that of sample S8, attributed to the presence of TIPA.

The dissolution of strong inorganic alkalis, such as NaOH, Na_2CO_3 , and $\text{Ca}(\text{OH})_2$, can increase the concentration of hydroxide ions (OH^-) while decreasing the concentration of calcium ions (Ca^{2+}) in the pore solution [38]. This process may facilitate early hydration, accelerate the growth and recrystallization of ettringite (AFt), and subsequently disrupt the thin AFt film surrounding the surface of cement grain. The dissolution of the AFt thin film may further enhance the hydration rate, assist in the setting, and

ultimately lead to a significant reduction in grout fluidity [39]. Following thermal treatment, the quantity of dehydration products increased, and the hydration rate of calcium oxide (CaO) was elevated, which resulted in the consumption of a substantial amount of mixing water [40]. Conversely, the fluidity of TIPA-activated RP grout was noticeably improved. The inclusion of TIPA, which contains a lipophilic methyl in its molecular chain, may introduce a certain amount of air, provide a ball-bearing effect, and consequently enhance the fluidity [39]. Furthermore, the combined activation of DEIPA, TIPA, and thermal treatment for reactive RP can further improve the grout fluidity.

The influence of activation modes on the compressive strength of grouts is illustrated in Figure 6. The strength of NaOH- and Na_2CO_3 -activated RP grouts increased at 1 day but decreased at 3 and 28 days. The strength of the CH-

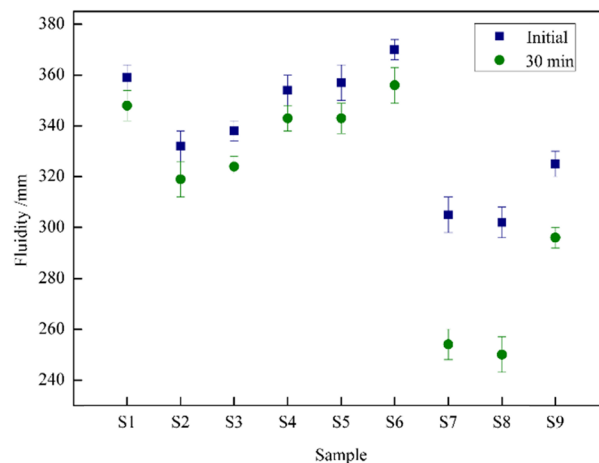


Figure 5. Influence of activation modes on the fluidity of RP grouts.

activated group (S4) increased at each age, particularly at 28 days. Additionally, the strength of DEIPA-activated samples at 1, 3, and 28 days was all improved, although the 28th day strength is lower than the specified value of 85 MPa. In contrast, the strength of the TIPA-activated sample increased and exceeded the specified values. The strength of grout containing thermally treated RP also increased, with the performance at 3 and 28 days surpassing that of single chemical additives. Furthermore, the strength of grouts with combined-activated RP at each age was higher than that of grouts with single-activated RP. Notably, the strength of grouts with CH, DEIPA, and thermally treated combined-activated RP at 3 and 28 days reached the highest values of 73.3 and 95.7 MPa, respectively, representing increases of 19.5 and 17.7%, respectively. Meanwhile, the 1-, 3-, and 28-day compressive strength of the S9 sample was 43.1, 71.6, and 92.2 MPa, which were 20.4, 17, and 13.4% increases compared to the S1 sample, respectively. The addition of inorganic alkalis may disrupt Si–O–Si bonds and enhance the dissolution of quartz [41], thereby promoting the early hydration process of RP grouts. Alkanolamines, such as DEIPA and TIPA, can accelerate the hydration of C_3S , resulting in the formation more additional C–S–H gels. Elevated temperature treatment of RP may produce more reactive components due to the decomposition of the existing hydration products and calcite, leading to the formation of new hydration products and a denser microstructure [42].

In conclusion, the impact of a single chemical additive activated mode on the activity of RP is limited, and the strength of the grout at various ages can only be marginally improved. Furthermore, NaOH- and Na_2CO_3 -activated RP can significantly influence the later strength while

reducing the fluidity of the grouts. Although the addition of thermally activated RP can enhance the strength of grout at all ages, it also leads to a notable decrease in fluidity. The strength of grout with CH, DEIPA, and thermally treated combined-activated RP exceeded the specified values at all ages; however, the fluidity after 30 min does not meet the required standards. In contrast, the compressive strength and fluidity of grouts with DEIPA, TIPA, and thermally treated combined-activated RP comply with the specified values outlined in the Chinese standards JG/T 408-2013.

3.3 XRD analysis

Figure 7 shows the XRD patterns of the hydration products of the RP grout paste under various activation modes. The primary hydration products in the crystal phase include calcite, portlandite, Aft, quartz (SiO_2), and incompletely hydrated C_3S and C_2S .

On the first day, there was no significant change in the peaks of SiO_2 and portlandite in the hydration products of each of the inorganic alkali-activated samples (S2 and S4) compared to the non-activated group (S1) (Figure 7a). Possibly, the solubility of SiO_2 in the alkali activator was low [43]. Notably, the pozzolanic effect of the RP in the alkali-activated grouts was not apparent, which aligns with the results of the strength testing. In contrast, the peak intensity of SiO_2 in the hydration products of the alkanolamine-activated groups (S5 and S6) decreased. Additionally, the peak intensity of C_3S decreased, while that of Aft increased. On one hand, the dissolution of SiO_2 was facilitated by alkanolamines [44]. On the other hand, the hydration of C_3S may have been accelerated by alkanolamines, resulting in the

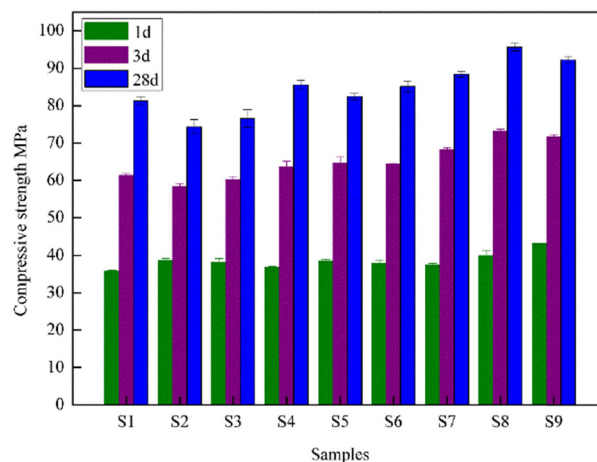


Figure 6. Influence of activation modes on the compressive strength of grouts.

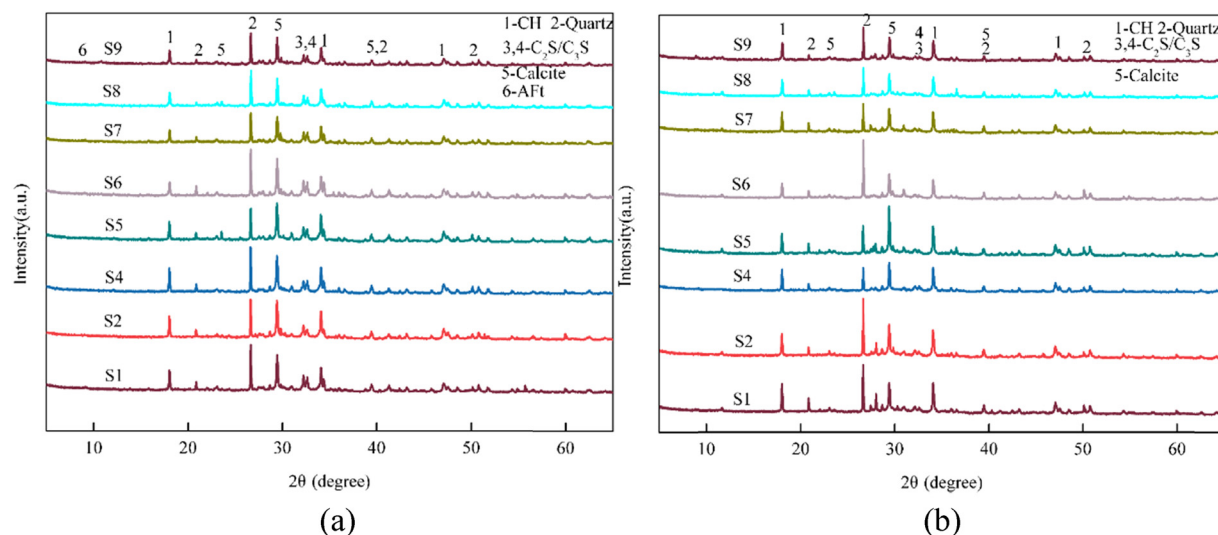


Figure 7. XRD patterns of hardened samples of RP activated by different methods at 1 (a) and 28 (b) days.

formation of additional C–S–H gels. It is noteworthy that a new diffraction peak appears in the XRD pattern of the TIPA-activated group, which may be attributed to hemicarboaluminate [45,46]. In the XRD patterns of the thermally and combinatorially activated groups (S7, S8, and S9), the peak intensity of SiO_2 was lower than that of the other groups. Some SiO_2 may have transformed into amorphous silica under elevated temperature treatment and reacted with alkanolamines, which exhibited weak alkalinity. A certain relationship exists between the peak intensities of portlandite and calcite in the hydration products and the degree of hydration [43]. For the hydration products of S7, S8, and S9 groups, the portlandite peaks were substantially weakened compared to the other activated groups. The peaks of C_3S and C_2S were lower than those of the other samples, indicating that these three groups contained few unhydrated cement particles and had a higher degree of hydration than the other samples. Thus, thermal and alkanolamines activation was beneficial for the early strength development of the system.

On the 28th day, the peak intensity of portlandite in each activated group decreased and was lower than that of the S1 group (Figure 7b). Notably, the SiO_2 peaks of the S2 and S6 groups at $2\theta = 27^\circ$ did not exhibit significant changes compared to the other five activated groups (S4, S5, S7, S8, and S9). This observation may be attributed to the hydration of cement grains and RP. The change in peak intensity suggests that activation may have accelerated the pozzolanic reaction between portlandite and SiO_2 . Furthermore, the content of Aft decreased due to the presence of OH^- , which may facilitate the transformation of Aft to AFm [39]. Similarly, the peak intensity of CH in the S5, S6, S8, and

S9 groups decreased, possibly due to the chelation of alkanolamines with Ca^{2+} . This chelation effect can reduce the solubility of Ca^{2+} and the crystallinity of CH in the pore solution, potentially inhibiting the nucleation and growth of CH [44].

3.4 SEM morphology

The microstructures of the hydration products in various grout pastes on the first day are illustrated in Figure 8. The microstructure of the non-activated group (S1) exhibited a loose structure with several cracks (Figure 8a), resulting in low strength. In addition to the C–S–H gel, small amounts of portlandite and Aft were detected in the pores. The microstructure of the NaOH-activated sample (S2) displayed a significant amount of C–S–H gels, along with numerous pores and several voids (Figure 8b). The SEM image of the S4 sample revealed a large number of hexagonal plate-like CH crystals (Figure 8c), which may be attributed to the nucleation of additional Ca^{2+} and OH^- ions. The morphology of the S5 sample showed a substantial amount of fibriform C–S–H (Figure 8d), which may contribute to a loose microstructure. The microstructure of the S6 sample exhibited several voids (Figure 8e), likely due to the air-entraining effect of DEIPA [45]. Besides, there were several anchor-shaped C–S–H gels and a small number of needle-like Aft crystals, which also contributed to a loose microstructure. The morphology of the S7 sample displayed a large number of hexagonal plate-like CH crystals and C–S–H gels (Figure 8f), which adhered to other hydration products and unhydrated cement grains.

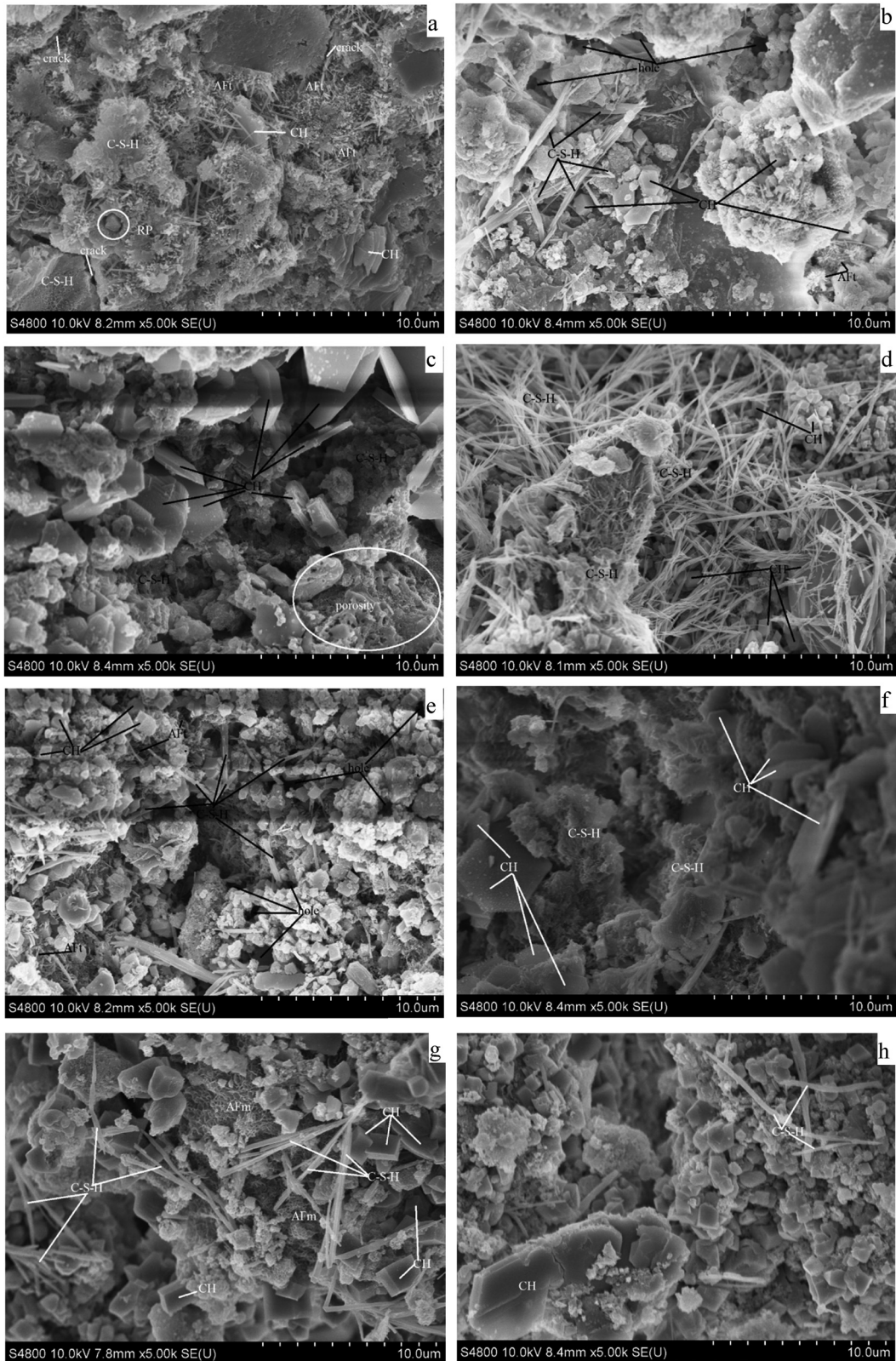


Figure 8. SEM images of samples S1 (a), S2 (b), S4 (c), S5 (d), S6 (e), S7 (f), S8 (g), and S9 (h) at 1 day.

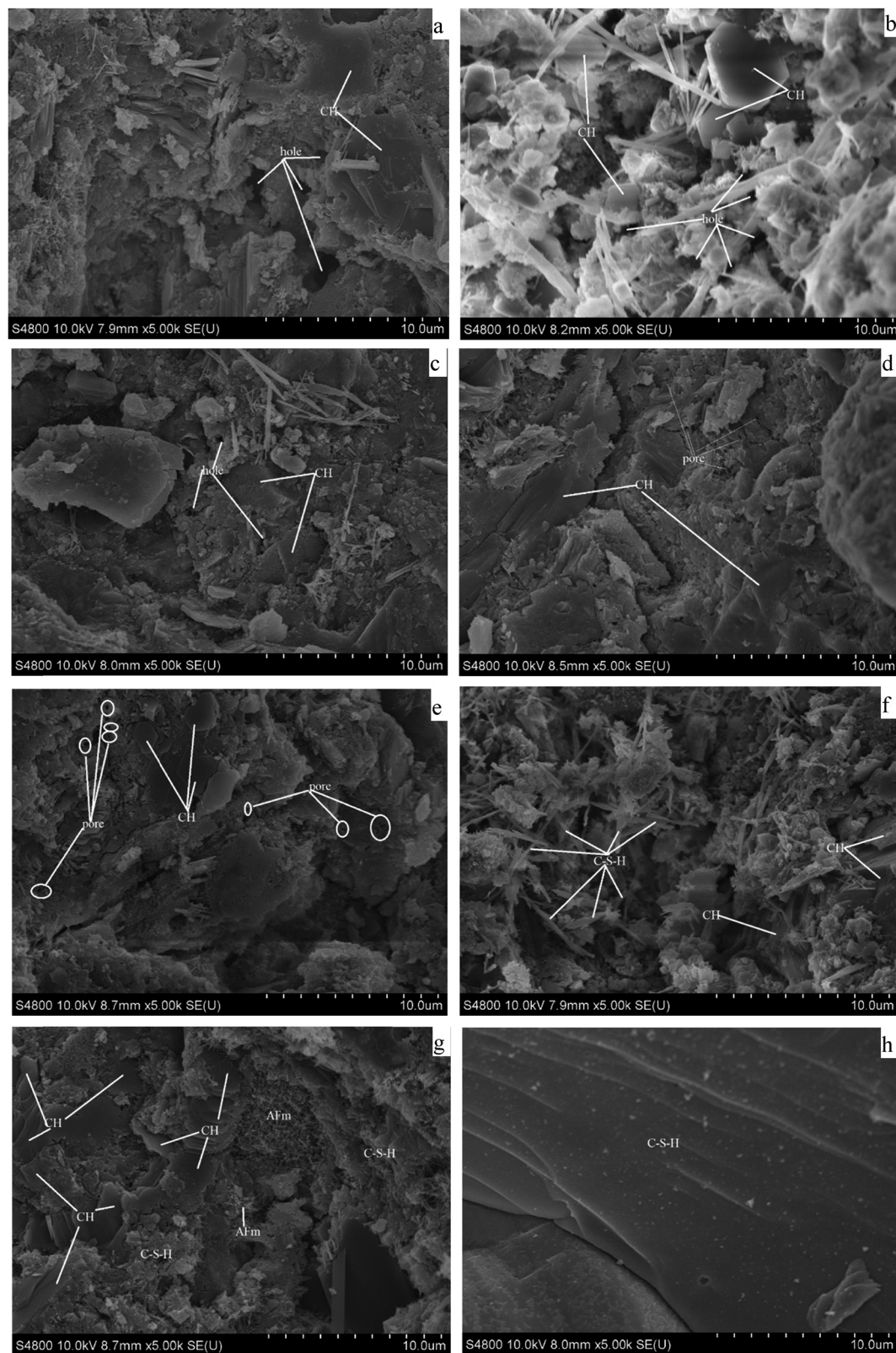


Figure 9. SEM images of samples S1 (a), S2 (b), S4 (c), S5 (d), S6 (e), S7 (f), S8 (g), and S9 (h) at 28 days.

The hydration products consisted of anchor-shaped C–S–H, tetragonal CH, and tufted AFm (Figure 8g).

The microstructure of the S9 sample presented several plate-like and hexagonal CH crystals, along with anchor-shaped and continuous C–S–H gels (Figure 8h). In summary, the internal structural integrity of the C–S–H gel in the activated samples differed from that of the non-activated sample. However, the width of the microcrack, pore size, and density of the microstructure reduced in the combined activated groups compared to the singly activated groups, resulting in increased compressive strength for the former compared to the latter.

Figure 9 illustrates the microstructures of hydration products in various grout pastes on the 28th day. The microstructure of the S1 sample on the 28th day exhibited a loose and porous morphology (Figure 9a). The SEM image of the S2 group revealed an even looser morphology, characterized by voids not filled by the continuously formed C–S–H gels (Figure 9b) when compared to the S1 group. The microstructures of the hydration products in the S4 group exhibited high portlandite content (Figure 9c), resulting in a denser microstructure. In the morphologies of the alkanolamines-activated groups (Figure 9d and e), hydration products such as C–S–H gels, CH, and AFm phases were further formed, interlocked, and created a stable microstructure [47]. Conversely, the morphology of the S7 group (Figure 9f), which was activated by calcination, contained a significant amount of C–S–H gel but exhibited a loose microstructure. Comparing Figure 9g and h, it is evident that more pores and voids were filled with continuous C–S–H gels, resulting in hydration products that interlocked to form a stable microstructure. Additionally, the morphology of S8 displayed a greater presence of the untransformed meta-stable phase AFm, which may contribute to its lower strength compared to S9. Furthermore, when comparing pastes with the non-activated paste and single additive-activated RP, the pastes with combined-activated RP consistently demonstrated a denser morphology.

4. Conclusions

In this investigation, the effect of RP subjected to three types of inorganic alkalis, two alkanolamines, elevated temperature, and combined activation on the technical properties of RP grouts was evaluated. Based on the results obtained, the following conclusions can be drawn.

- (1) The fluidity of grout with TIPA-activated RP is significantly improved, measuring 370 mm initially and 356 mm after 30 min. The fluidity of the S9 sample was

greater than that of the S8 sample, which may be attributed to a ball-bearing effect.

- (2) The 1-, 3-, and 28-day compressive strengths of the S9 sample were 43.1, 71.6, and 92.2 MPa, representing increases of 20.4, 17, and 13.4% compared to the S1 sample, respectively.
- (3) The mechanism of combination activation may be attributed to the formation of additional C–S–H gels and other new hydration products, which are associated with alkanolamines and thermal activation, respectively.

5. Limitations

The properties of cement-based materials can be influenced by various process parameters, including the particle size and distribution, chemical composition, mineral composition, content, and activation mode. Among these factors, the particle size and distribution of supplementary materials, such as fly ash, slag, and RP, play a significant role in the reactivity, physical properties, microstructural development, strength development [48,49], and workability of cement-based materials [50,51]. Generally, the reactivity of RP increases with a reduction in particle size, leading to improved mechanical and microstructural properties of RP grouts. Therefore, further studies should focus on the influence of particle size and distribution of RP on cement-based materials, particularly for those with a high content of RP as supplementary materials.

Acknowledgements

This study was funded by Yangzhou Government-Yangzhou University Cooperative Platform Project for Science and Technology Innovation (YZ2020262).

Author contributions

Design was planned by: LSP, experiments were conducted by: JJC, YCX, YB, and CJ, data analysis was performed by: CJ, YB, YCX and JJC, Manuscript writing was done by JJC, YCX, YB, and CJ, manuscript revision and supervising were conducted by LSP, LQ, and WQS.

Conflict of interest statement

The authors declare that they have no conflict of interest.

Ethical approval

Not applicable.

Data availability statement

All data, models, and code that support the findings of this study are available from the corresponding author upon reasonable request.

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