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Study on Reclamation of CO_2-hardened Sodium Silicate Used Sands by CaO Powder

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Abstract

Aiming at the problems of wet reclamation consuming a lot of water, dry(mechanical) reclamation having wear and power consumption, this paper to find suitable reclamation reagents to reduce the influence of harmful substances in used sodium silicate sands. By comparing the reclamation effect of CaO, $Ca(OH)_2$ and $Ba(OH)_2$ reclamation powder reagents, it was concluded that CaO had the best reclamation effect. Through the single factor experiment, the influence of CaO on the reclamation effect was explored: 1. addition amount of CaO;2. the additional amount of water ;3. reclamation time. The orthogonal results showed that the CaO reclamation effect was the best when the amount of CaO was 1.5%, the amount of sodium silicate was 4.0%, the amount of water added was 6.0%, and the reclamation time was 12.0h. In this experiment, 82.2% carbonate and 75.0 % silicate in used sands can be removed. The microscopic analysis of the reclamation sands was carried out by scanning electron microscope (SEM); The surface was relatively smooth, without large area cracks and powder accumulation. Compared with the used sands, the instant, 24h ultimate, and residual strengths of the reclaimed sands were increased by 536.5%, 458.1%, and 89.8%, respectively, which was beneficial to the reclamation of the CO_2 sodium silicate used sands.

Keywords: CO_2 hardened sodium silicate used sands, CaO, Reclamation, Orthogonal experiment

1. Introduction

CO_2 sodium silicate sands have the advantages of low cost, good fluidity, fast hardening speed and low gas generation and are widely used in foundry production [1-3]. The reclamation of CO_2 sodium silicate bonded sands is a difficult problem restricting the development of used sodium silicate sands [4,5]. A large number of experiments and research results showed that sodium silicate remains on the surface of the reclaimed sands grains were complex and difficult to decompose and act as a nuclear for new sodium silicate leading to premature hardening of the sands [6,7]. Its bond film is mainly made of particle silica gel and carbonate, silicate, etc., which can be roughly divided into three types: soluble, slightly soluble, and insoluble [8]. The soluble part accounts for about 30~35%, and its components are sodium carbonate and sodium silicate. The slightly soluble part is high modulus sodium silicate for dehydration, about 55~60%. The insoluble region accounts for about 5~10%, and it exists in the glass state [9]. The used sands were reclaimed by reducing the harmful part soluble in water and restoring the bonding ability of high modulus sodium silicate existing in water loss. The reclaimed sands were equivalent to new sand in physical, grain size distribution, casting finish and quality [10]. Therefore, it is of great significance for the reclamation of CO_2 hardened sodium silicate used sands to improve the properties of used sands to meet the standards by adding reagents to reduce harmful substances in used sands [11].

Our team study on sewage harmless treatment in the wet reclamation process of used water glass sands through CaO powder, $Ca(OH)_2$ powder and $Mg(OH)_2$ powder. The results show that the best treatment effects come from treatment using lime milk which was obtained by mixing H_2O and CaO with a ratio of 6 [12]. On this basis, On this basis, this study CO_2 sodium silicate sands were reclaimed by alkaline earth metals CaO, $Ca(OH)_2$ and

$Ba(OH)_2$ powder reagents; the optimum reagent was selected by comparing the properties and morphology of reclaimed sands. The influence of the optimal reagent addition amount, water addition amount and reclamation time on the reclaimed CO_2 hardened sodium silicate sands were analysed, and the reclamation effect was evaluated from the macroscopic strength of reclaimed sands and the microscopic SEM method [13, 14].

In this work, a study of reclamation of CO_2 hardened sodium silicate used sands was presented. The influence of the four factors (reagent amount, water amount, reclamation time, and sodium silicate amount) on properties were analysed by changing four parameters. This work aims to find out the influence of different combinations of four parameters on the properties of reclamation sands. To find out the impact of these parameters and the optimal scheme parameter, all factor and level combinations needed to be analysed and investigated. Orthogonal experimental design is a widely used multi-factor experimental method based on an orthogonal array. According to related factors and levels, $L_9(3^4)$ orthogonal arrays [18] were used to study optimal parameters. Orthogonal arrays of the nine cases were selected with different geometry settings. The same results for the 81 conditions can be obtained by the investigations of the nine cases in the Orthogonal array. Then the optimum reclamation scheme was obtained under different combinations of parameters.

2. Materials, measurement methodology and measurement equipment

2.1. Materials and Experiment Equipment

Experiment materials: new and used sands were used from a foundry enterprise in Jiangsu Province. The used granular sands were crushed and sieved from the CO_2 sodium silicate factory with a particle size of 0.425×10^{-3}-0.150×10^{-3}m(the amount of sodium silicate was 8.0%). Reagents: CaO, $Ca(OH)_2$ and $Ba(OH)_2$ powder produced by Sinopharmaceutical Group Co., Ltd., powder purity of more than 98.0%, Industrial sodium silicate with a modulus of 2.3 density of 1.4×10^3 kg/m^3 was produced in Wuhan, China.Experiment equipment: JJ-5 cement mortar mixer, SAC hammer-type sample preparation machine, SWY hydraulic universal strength testing machine, UV5 visible light-ultraviolet spectrophotometer (Mettler-Toledo), SX-1.2-1.0 box-type resistance furnace and JSM-7800 thermal field scanning electron microscope(SEM).

2.2. Experimental methods

2.2.1. Reagent primary selection experiment

The effects of three reagents on used sand reclamation were studied in three steps.

Step 1: Three different reagents CaO, $Ca(OH)_2$ and $Ba(OH)_2$ were added to 500.0 g of used sand to obtain different reclamation sands, respectively. The amount of the three reclamation reagents was 1.0% of the used sands. The amount of reagent was determined according to our team study on sewage harmless treatment in wet reclamation process of used water glass sands[12]. The amount of water was 1.0% of the used sand. At the same time, the used sands without reclamation reagents were a blank comparison test. In the first step, the four kinds of sand are poured into the sand mixer, then the reagents were added, and the mixture was stirred for 3 minutes to obtain four kinds of reclaimed sands, which were then taken out. The reclamation time was 24h.

Step 2: The four kinds of sand and sodium silicate were poured into the sand mixer for 3 minutes to obtain the reclamation sands. the amount of sodium silicate was 6.0% of the mass of the used sands. The reclamation sands were made into samples with a height and diameter of 30mm (30×Φ30mm), and then cured by CO_2 gas blowing for 15s with 15L/min. The compressive strengths of the samples were measured by SWY hydraulic universal strength tester.

Step 3: The compressive strengths of the new sands, used sands, CaO reclaimed sands, $Ca(OH)_2$ reclaimed sands and $Ba(OH)_2$ reclaimed sands were measured by SWY hydraulic universal strength tester and the surface morphology of the five sands samples were observed by a JSM-7800 scanning electron microscope(×1300). The total carbonate was determined by adding HCl to produce the carbon dioxide gas volumetric method and the total silicate was measured using silicon molybdenum yellow spectrophotometry by UV5 visible light-ultraviolet spectrophotometer. according to our preliminary study [15-17].

2.2.2. Single-factor experiment

In this chapter, the influence of CaO amount, water amount, and reclamation time on used sand reclamation was discussed through 1#, 2 #, and 3# experiments.

The 1# sequence was chosen to study the effect of the amount of CaO on the properties of reclamation sands. The additional amount of water was 5.0% of the used sands, and the amount of CaO was 0.5%, 1.0%, 1.5%, 2.0%, and 2.5%. The mixture was mixed for 3 minutes, set for 24h and then the reclamation sands and sodium silicate with the sand mass of 6.0% were made into samples with a height and diameter of 30mm (30×Φ30mm).

The 2# sequence was chosen to study the effect of the amount of water on the properties of reclamation sands. the amount of CaO was 1.0% of the mass of the used sands, and the amount of water was 2.0%, 3.0%, 4.0%, 5.0% and 6.0%. After 24.0h, the mixture was made into the same sample by the above method.

The 3# sequence was chosen to study the effect of reclaimed time on the properties of reclamation sands. the amount of CaO was 1.0% of the mass of the used sands, the additional amount of water was 5.0% of the mass of the used sands, and the reclaimed time was 1.0h, 2.0h, 12.0h, 24.0h, 48.0h, the mixture was made into a sample according to different reclamation times.

Table 1.
Scheme and sequence of Single-factor experiment

	CaO amount	Water amount	Reaction time	Sodium silicate amount
1#	0.5%-2.5%	5.0%	24.0h	6.0%
2#	1.0%	2.0%-6.0%	24.0h	6.0%
3#	1.0%	5.0%	1.0h-48.0h	6.0%

2.2.3. Orthogonal experiment

The main purpose of this paper was to study the effects of four factors, namely CaO addition amount(A), sodium silicate addition amount (B), water addition amount (C)and reclamation time (D), different factors combinations of collocation on the reclamation effects of CO_2 hardened sodium silicate used sands, without considering the interaction between the factors. The evaluation indexes were instant strength, 24.0h ultimate strength and residual strength of reclamation sands. Considering that the 24h ultimate strength of the sand mould is the most important in actual use, the 24h ultimate strength of the sand sample is the main evaluation index, and the effect of falling sand after casting, the residual strength should not be too large. Meet the performance requirements of new sand.

The L_9 (3^4) four-factor three-level orthogonal table was generated by Minitab software. The factor levels of the orthogonal experimental were showed in Table 2.

Table 2.
The orthogonal factor level of CO_2 hardened sodium silicate used sand reclamation technology

level	Factor			
	CaO amount (A)	Sodium silicate amount (B)	Water amount (C)	Reclamation time (D)
1	0.5%	4.0%	4.0%	2.0h
2	1.0%	5.0%	5.0%	12.0h
3	1.5%	6.0%	6.0%	24.0h

3. Results and discussion

3.1. Reclaimed reagent of CaO, $Ca(OH)_2$ and $Ba(OH)_2$ effect

3.1.1. Reclaimed sands strength

The reclamation effects of CaO, $Ca(OH)_2$ and $Ba(OH)_2$ were shown in Fig. 1. and the strength of used CO_2 sodium silicate sands was compared with the reclamation sands of three different reagents. Compared to the used sands, the strength of CaO, $Ca(OH)_2$, and $Ba(OH)_2$ reclamation grains of sand was improved. the results showed that the three alkaline earth metals can improve the properties of CO_2 sodium silicate sand to a certain extent. The initial strength of CaO and $Ca(OH)_2$ reclaimed sands was close to that of new sand, The 24h strength of CaO reclaimed sands was most relative to that of new sands, which was higher than that of used sands. The residual strength of CaO and $Ba(OH)_2$ reclaimed sands was the lowest.

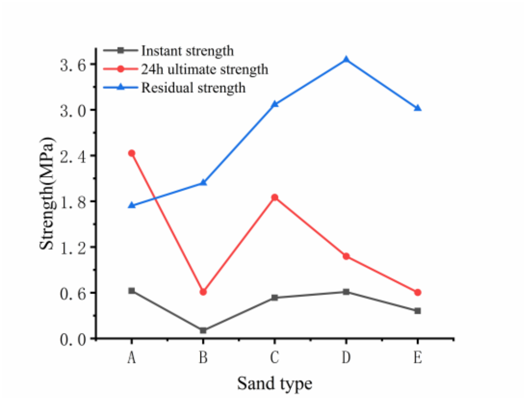
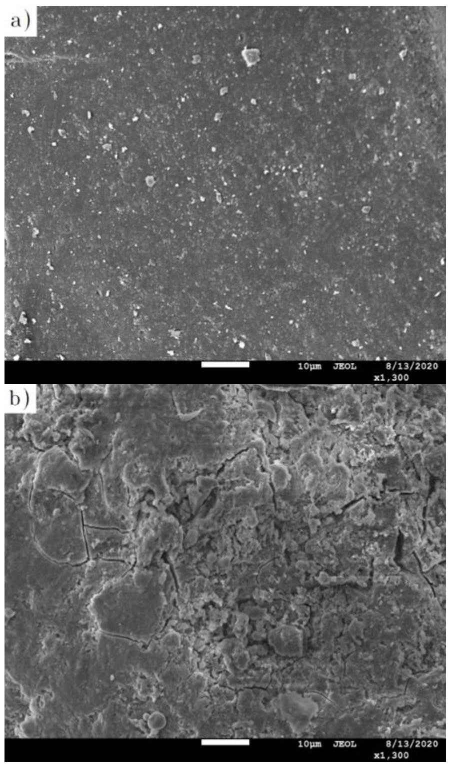


Fig. 1. Strength comparison of reclaimed sands with three reagents:A) New sands, B) Used sands, C)CaO reclaimed sands, D) $Ca(OH)_2$ reclaimed sands and E) $Ba(OH)_2$ reclaimed sands

3.1.2. Micro-structure analysis (SEM analysis)

The used sands and five kinds of sands were analysed by scanning electron microscopy (SEM), as shown in Fig. 2. The surface of the used sands were covered with adhesive film and a large number of columnar carbonate crystals. Compared with used sands, the surface of $Ca(OH)_2$ reclaimed sands was smooth, and other crystals remained on the surface, the surface of $Ba(OH)_2$ reclaimed sands was uneven, and a large number of different crystal substances remained. The surface of CaO reclaimed sands was clean, without rod-like crystals, with the cleanest surface and good reclamation effect.



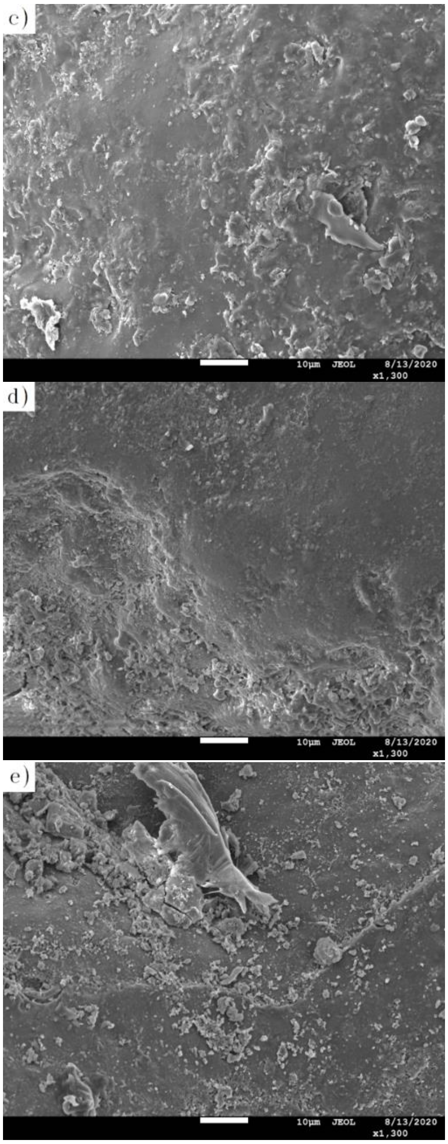


Fig. 2. Surface morphology of used sands and five kinds of reclaimed sands: a) New sands, b) Used sands, c) CaO reclaimed sands, d) $Ca(OH)_2$ reclaimed sands and e) $Ba(OH)_2$ reclaimed sand

3.1.3. Choose the optimal reagent

Results from experiment analysis indicated the best effect of using CaO for reclamation. On this basis, the CaO reagent was used as the treatment reagent. The effects of CaO, the amount of water added, and the reclamation time on the reclamation properties of CO_2 sodium silicate used sands were further studied.

3.2. Single-factor experiment of CaO

3.2.1. Effect of CaO amount on compressive strength of samples

The effect of CaO amount on the compressive strength of the sample was shown in Fig. 3. When the amount of CaO was between 0.5% and 1.5%, the instant strength, 24h ultimate strength, and residual strength of the reclaimed sands rose progressively with the increased of the added amount, then showed a trend of decrease. More than 1.5% will deteriorate the moisture resistance of the sample, resulting in a rapid decline in strength. The instant strength, 24h ultimate strength and residual strengths peaked at 0.686MPa, 4.335MPa and 4.317MPa, respectively. When the CaO content was 1.5%. A certain amount of CaO was needed in the reclamation process. Adding CaO up to 1.5% will make the sodium silicate remaining in the used sands restore its bonding properties. Therefore, three additions of 0.5, 1.0 and 1.5% were selected as the three levels of the orthogonal experiment.

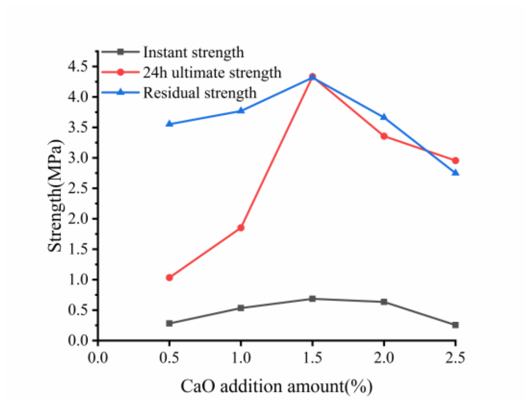


Fig. 3. Effect of CaO amount on strength of CO_2 sodium silicate sands

3.2.2. Effect of water amount on compressive strength of samples

The effect of the water amount on the compressive strength of the sample was shown in Fig. 4. When the addition amount of water was between 2.0% and 6.0%, the 24h ultimate strength and residual strength of the reclaimed sands rose progressively with the increase of the additional content when the amount of water was more than 6.0%, it showed a trend of reduced. The initial strength reaches the peak of 0.722MPa at 4.0%, and the 24h ultimate strength and residual strength reach the peaks of 4.626MPa and 4.423MPa, respectively, at 6.0%. It was demonstrated that the reclamation process requires a certain amount of water to participate in the reaction, when the amount of water added was appropriate, CaO was reacted with Na_2CO_3 and SiO_3^{2-} and activated the residual sodium silicate in the used sands. The amount of water was too low; the reaction would not be sufficient, excessive addition of water causes bicarbonate to appear on the surface of the reclaimed sands, which deteriorates the performance of the reclaimed sands. Considering the 24h ultimate strength and residual strength, three water addition measures of 4.0, 5.0, and 6.0% were selected as the three levels of the orthogonal experiment.

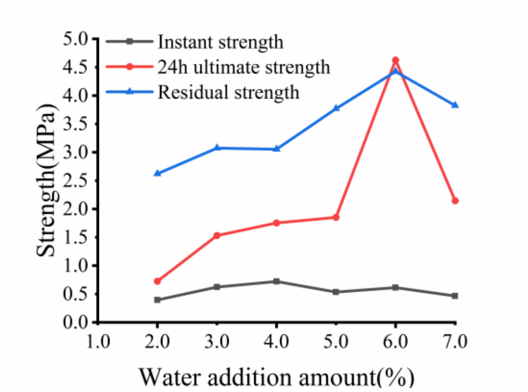


Fig. 4. Effect of water amount on strength of CO_2 sodium silicate sands

3.2.3. Effect of reclamation time on compressive strength of samples

The effect of reclamation time on the compressive strength of samples was shown in Fig. 5. When the reclamation time was between 0h-48.0h, the instant, 24h ultimate, and residual strengths of reclaimed sands rose progressively with the increase of reclamation time. When the reclamation time changed from 2.0h-24.0h, the initial strength increased from 0.358MPa to 0.534MPa, and the 24h ultimate strength increased from 1.015MPa to 1.852MPa, the residual strength increased significantly from 3.274MPa to 3.769MPa.During 24.0h-48.0h, the properties of reclaimed sands did not change significantly. It was founded that the reclamation process takes a certain time; if the time were too short, the reaction would not be sufficient, and if the reclamation time were extended, the reaction would not continue. Considering the reclamation period and the performance improvement rate, three times of 2.0, 12.0, and 24.0 h were selected as the three levels of the orthogonal experiment.

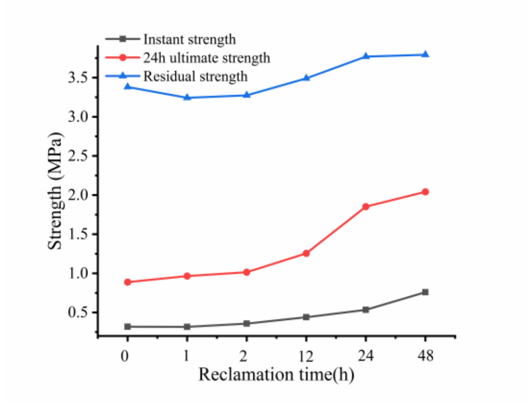


Fig. 5. Effect of reclamation time on strength of CO_2 sodium silicate sands

3.3. Orthogonal experiment Range analysis

Table 3.
$L_9(3^4)$ orthogonal designed table and its results

	A(%)	B(%)	C(%)	D(h)	Instant strength(MPa)	24h ultimate strength(MPa)	Residual strength (MPa)
1	0.5	4.0	4.0	2.0	0.027	0.192	0.812
2	0.5	5.0	5.0	12.0	0.257	1.465	2.775
3	0.5	6.0	6.0	24.0	0.304	1.809	5.016
4	1.0	4.0	5.0	24.0	0.748	1.468	3.133
5	1.0	5.0	6.0	2.0	0.329	1.894	3.203
6	1.0	6.0	4.0	12.0	0.678	2.190	3.538
7	1.5	4.0	6.0	12.0	0.674	3.354	4.020
8	1.5	5.0	4.0	24.0	0.551	1.540	3.359
9	1.5	6.0	5.0	2.0	0.342	3.261	4.017

Table 4.
Range analysis results

	level	A	B	C	D	
Instant strength (MPa)	1	0.588	1.449	1.256	0.698	3.91
	2	1.755	1.137	1.347	1.609	
	3	1.567	1.324	1.307	1.603	
	R	0.389	0.104	0.030	0.303	
24h ultimate strength (MPa)	1	3.466	5.014	3.922	5.347	17.173
	2	5.552	4.899	6.194	7.009	
	3	8.155	7.260	7.057	4.817	
	R	1.563	0.787	1.045	0.730	
Residual strength (MPa)	1	8.603	7.965	7.709	8.032	29.873
	2	9.874	9.337	9.925	10.333	
	3	11.396	12.571	12.239	11.508	
	R	0.931	1.535	1.51	1.159	

The orthogonal experimental results were shown in Table 3. In this work, through the range analysis of Table 3 data, Table 4 was obtained. Table 4 was the range analysis of three evaluation indexes.

It can be concluded from Table 3 and Table 4 that the influence relationship of various factors on the initial strength of reclaimed sand was as follows: the amount of CaO added > the reclamation time > the amount of sodium silicate added > the amount of water added. The optimal combination was as follows: the addition amount of CaO was 1.0%, the addition amount of sodium silicate was 4.0%, the addition amount of water was 5.0%, and the reclamation time was 12.0h. The influence relationship of various factors on the 24h ultimate strength of reclaimed sand was as follows: the amount of CaO added> the amount of water added > the amount of sodium silicate added > the reclamation time. The optimal combination was as follows: the addition amount of CaO was 1.5%, the addition amount of sodium silicate was 6.0%, the addition amount of water was 6.0%, and the reclamation time was 12.0h. The influence relationship of various factors on the residual strength of reclaimed sand was as follows: the amount of sodium silicate added > the amount of water added > the reclamation time > the amount of CaO added. The optimal combination is: the amount of CaO was 0.5%, the amount of sodium silicate was 4.0%, the amount of water was 4.0%, and the reclamation time was 2.0h. Considering the 24h ultimate strength, the residual strength should not be too large. The optimal combination of CaO reclamation CO_2 hardened sodium silicate sand was as follows: the addition of CaO was 1.5%, sodium silicate was 4.0%, the addition of water was 6.0%, and the reclamation time was 12.0h.

4. Validation experiments and recycling use

Three batches of validation experiments were carried out according to the optimal combination scheme of CaO hardened sodium silicate reclamation further to investigate the reliability and stability of the reclamation scheme. 500.0g of used sand was reclaimed according to the addition amount of CaO 1.5%, the addition amount of sodium silicate of 4.0%, and the water addition amount of 6.0%, and the reclamation time was 12.0h to obtain regenerated sand. Then sand samples are made as above. The compressive strength of the sand samples was measured. The compressive strength of three batches of validation experiments is shown in Fig. 6. The compressive strength of the sand samples verified by three batches of experiments(Three validation experiments are represented by 1#, 2#, and 3#.) was consistent with that of the orthogonal experiment. As shown in Fig. 7, compared to the used sands, the surface of reclaimed sands was smooth and neat, and there were no large-area sands dropping phenomena. The contents of carbonate 7.9kg/m^3 and silicate 1.4kg/m^3 in CO_2 hardened sodium silicate sand. The carbonates and silicate contents were 1.2kg/m^3 and 0.3kg/m^3 in CaO reclaimed sands, respectively. The carbonate and silicate content of the CaO reclaimed sands decreased by 82.2% and 75.0%, respectively, compared with the used sands. The results show that the optimized reclamation process is stable and feasible, and can effectively reduce the content of carbonate and silicate, and The reclaiming effect of sodium silicate sands was ideal.

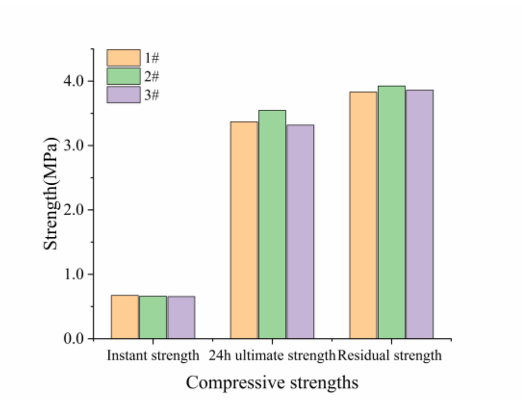


Fig. 6. Validation experiments strength results

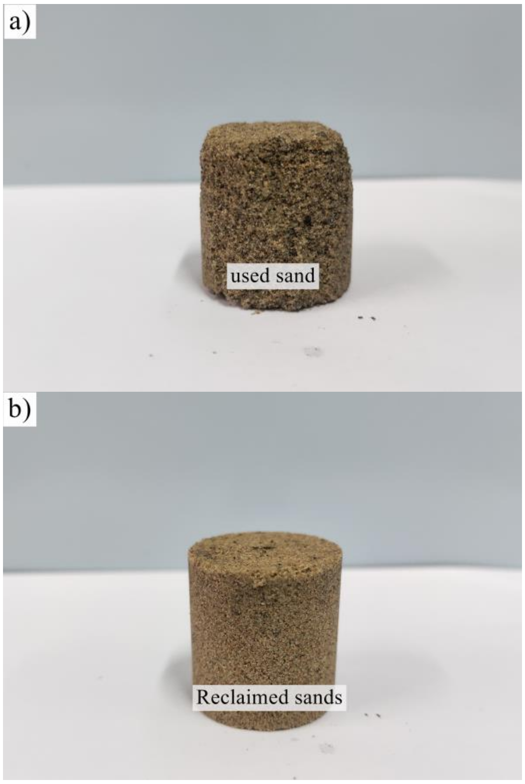
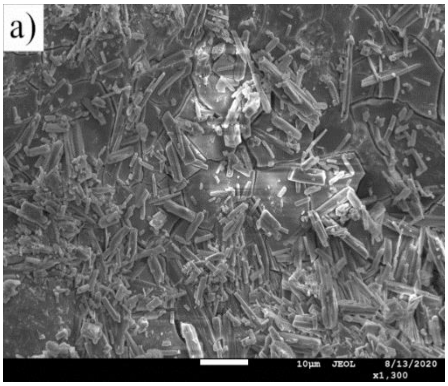


Fig. 7. Sands samples with a height and diameter of 30 mm (30 × Φ30 mm): a) Used sands, b) Reclaimed sands

The surface morphology of CaO reclaimed sands and used sands was observed by scanning electron microscope, as shown in Fig.8. As can be seen from Fig. 8. a), when the used sodium silicate sands surface was not reclaimed, there were a large number of strip carbonate crystals left on the surface, and the crystals covered a wide area in disorder. The whole surface was uneven, and some cracked. It can be seen from Fig. 8. b) that there were only a tiny amount of strip-shaped carbonate crystals on the surface of the reclaimed sands, and the surface was smooth, without large area cracks and powder accumulation. Thus, the CaO reclaimed sands had better properties.



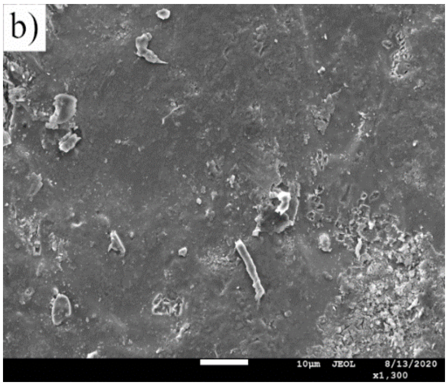


Fig. 8. Surface morphology of sands: a) Used sands, b) Reclaimed sands

To study the recycling of reclaimed sands, CaO powder and water were used to reclaim the used sands, and sodium silicate was added to make samples. After drying, crushing, and screening, the sand samples were reclaimed by repeating the reclamation process three times. The actual properties of the reclaimed were an experiment and compared with the new sands, the used sands, and the first reclaimed. The experiment results were shown in Fig. 9.

As shown in Fig. 9, Reclaimed sands with two recycled: initial strength was close to that of new sand, 24h ultimate strength was higher than the new sands, much higher than the used sands; residual strength was more elevated than used sands. At the same time, the first and second reclaiming sands can meet the performance requirements; the results of the two times are consistent, and the performance of the third recycled sands was close to the used sands, significantly lower than the new sands. The carbonates and silicates in used sand can be effectively removed by adding CaO powder and water during the first two cycles. At the same time, the crushing and screening before reclaimed can remove the formation of harmful material precipitates. With the increase of reclaimed, the accumulation of carbonate content and total alkali content in the sand. The properties of the third recycled sands had resulted in a decline, close to the used sands, and the reclaiming effect was not ideal. The performance of the reclaimed sand in which the used sand has been recycled twice in succession can meet the requirements.

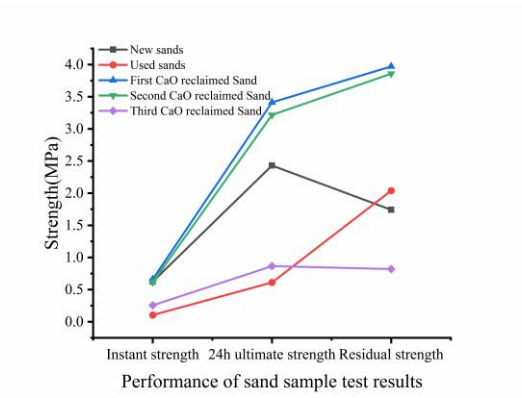


Fig. 9. Cyclic strength comparison of reclaimed sands

5. Conclusions

1) A method of reclaiming CO_2 hardened sodium silicate sand with CaO powder was developed, which restored the performance of the reclaimed sands. The addition of CaO had a significant effect on the initial and 24h ultimate strengths; the amount of sodium silicate added became more pronounced on the residual strength. The strength of CaO reclaimed sands was closest to that of new sands, which was far greater than higher than that of used sands.
2) According to the orthogonal experiment, the instant strength, 24h ultimate strength, and residual strength of the reclaimed sands were increased by 536.5%, 458.1%, and 89.8%, respectively. At the same time, the removal rate for the silicate can reach 75.0%, and the removal rate of carbonate reached 82.2%. When the amount of CaO was 1.5%, sodium silicate was 4.0%, water was 6.0%, and reclamation time was 12.0h. the surface of reclamation sands was smooth, without large area cracks and powder accumulation.

Acknowledgement

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