Recycling water glass from wet reclamation sewage of waste sodium silicate-bonded sand

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Abstract: Wet reclamation of waste sodium silicate-bonded sand produces much alkaline sewage and causes pollution. Recycling water glass from wet reclamation sewage of the waste sodium silicate-bonded sand can solve pollution issues and generate economic benefits. In this work, the wet reclamation sewage was filtered, and the filtrate was causticized with a quicklime powder to produce a lye. The effects of causticization temperature, causticization time, and the amount of quicklime powder on the causticization rate were studied. The lye was used to dissolve the silica in the filtration residue to prepare a sodium silicate solution. The effects of the mass of filtration residue, dissolution temperature, and dissolution time on sodium silicate modulus were studied. Finally, the recycled water glass was obtained by concentrating the sodium silicate solution, and the bonding strength of the recycled water glass was tested. The results showed that the causticization rate could be improved by increasing the amount of quicklime powder, causticization temperature, and causticization time, and the highest causticization rate was above 92%. Amorphous silica in the filtration residue dissolved in the lye. Increasing the amount of the filtration residue, dissolution temperature, and dissolution time could improve the sodium silicate modulus. The bonding strength of the recycled water glass was close to that of commercial water glass. The recycled water glass could be used as a substitute for the commercial water glass.

Key words: waste sodium silicate-bonded sand; wet reclamation; sewage treatment; causticization; recycling water glass

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Sodium silicate-bonded sand casting is popular due to its advantages including good casting quality, low cost, and non-toxicity^[1-2]. Currently, millions of tons of waste sodium silicate-bonded sand are produced in China every year, and cause serious environmental problems if not properly treated^[3]. The waste sodium silicate-bonded sand can partially replace ordinary sand for concrete or it can be used as building filler^[4-6], but as the amount of the waste sodium silicate-bonded sand is great, most of it still needs to be reused. The waste sodium silicate-bonded sand should be reclaimed before reuse. Reclaiming the waste sodium silicate-bonded sand refers to removing the impurities such as residual binder on the surfaces of the waste sand

grains to regain a state close to that of new sand. Currently, the main methods for reclaiming the waste sodium silicate-bonded sand are wet reclamation and dry reclamation^[1-3, 7-9]. The reclaimed sand obtained by wet reclamation is better than that obtained by dry reclamation in quality, and can directly replace the new sand^[10]. Wet reclamation refers to transferring the impurities from the surfaces of the waste sand grains into water, and then separating the sand grains and the water to remove the impurities. However, wet reclamation produces much alkaline sewage, which has to be well treated. If the problem of the wet reclamation sewage is solved, wet reclamation will be an environmentally friendly way to reclaim the waste sodium silicate-bonded sand.

Water glass (sodium silicate) is used as a binder in the sodium silicate-bonded sand casting, and is usually hardened with carbon dioxide or esters. Therefore, the impurities on the surfaces of the waste sand grains mainly include sodium carbonate, sodium hydrogencarbonate, sodium acetate (hardened with esters), and sodium silicate, and cause the wet

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reclamation sewage to be alkaline. At present, the treatment process of wet reclamation sewage includes neutralizing, and then precipitating or filtering^[11-12]. The treated sewage contains many soluble salts and will cause environmental problems if directly discharged^[13]. During the wet reclamation process, most of the binder components enter into the water. Recycling water glass from the wet reclamation sewage can solve environmental problems and generate economic benefits.

In this work, aiming at recycling water glass from the wet reclamation sewage of waste sodium silicate-bonded sand, the wet reclamation sewage was first filtered, and the filtrate was causticized^[14-15] with a quicklime powder and then converted into a lye. The effects of causticization temperature, causticization time, and the amount of quicklime powder on the causticization rate were investigated. The lye was used to dissolve the filtration residue, and a low concentration sodium silicate solution and a dissolution residue were obtained after the filtration. The effects of the filtration residue to lye ratio, dissolution temperature, and dissolution time on the sodium silicate modulus were studied. The XRD analysis and component analysis of the filtration residue and the dissolution residue were performed. Finally, the recycled water glass was obtained by concentrating the low concentration sodium silicate solution. The bonding strengths of the recycled water glass and commercial water glass were measured.

1 Experimental procedure

1.1 Process of recycling water glass

Figure 1 shows the process of recycling water glass from the wet reclamation sewage. After the quicklime powder is added into the filtrate, the following reactions occur:

where equation (1) is the digestion reaction, and equation (2) is the causticization reaction. After the lye is mixed with the filtration residue, the following reaction occurs:

1.2 Materials

The sewage used in the experiment was the wet reclamation sewage of waste sodium silicate-bonded sand hardened with carbon dioxide. The sewage had been filtered using filter membranes with a mean pore diameter of 5 μm, and the filtration residue was dried at 110 °C and then ground. Table 1 shows the main solutes in the filtrate. The sodium silicate in the filtrate is in a low concentration and can be ignored. Table 2 shows the main components of the filtration residue, and Fig. 2 shows the XRD analysis results of the filtration residue. The calcium carbonate and alumina in the filtration residue were derived from the waste sodium silicate-bonded sand. The effective CaO content of the quicklime powder was

Fig. 1: Flow chart for recycling water glass from wet reclamation sewage

Table 1: Main solutes in filtrate

Solute	Na ₂ CO ₃	NaHCO ₃	Na ₂ SiO ₃
Concentration (g·L ⁻¹)	92.9	31.7	<1

Table 2: Main components in filtration residue (wt.%)

Si	Ca	Na	Zr	Fe	Al	K	Mg
72.2303	8.3843	6.4956	3.8354	3.5366	2.8052	0.8927	0.4561

Fig. 2: XRD pattern of filtration residue

95%. The other reagents used were of analytical reagent grade. Da-lin sand at 50–100 mesh, commercial sodium silicate (modulus 2.19, density 1.54 g·mL⁻¹), and commercial ester were used in the bonding strength test.

1.3 Methods

1.3.1 Causticization

The filtrate (100.0 mL) was put into a beaker, and the beaker was put in a water bath and heated to 30–90 °C. The quicklime

powder (7.4–9.6 g) was added into the filtrate and the mixture was stirred for 1–15 min. The mixture was at last filtered. The lye obtained was titrated to measure the normality of sodium hydroxide and sodium carbonate with phenolphthalein and methyl orange as indicators. The efficiency of the causticizing process was evaluated by the causticization rate, which was calculated as follows:

$$\text{Causticization rate} = \frac{N_{\text{NaOH}}}{N_{\text{NaOH}} + N_{\text{Na}_2\text{CO}_3}} \quad (6)$$

where N_{NaOH} is the normality of sodium hydroxide in the lye, and $N_{\text{Na}_2\text{CO}_3}$ is the normality of sodium carbonate in the lye.

1.3.2 Dissolution

The lye (30.0 mL) and the filtration residue (6–15 g) were added into a beaker and mixed. The beaker was then bathed at 50–90 °C for 10–60 min. The mixture was filtered at last and a low concentration sodium silicate solution and a dissolution residue were obtained. The modulus of sodium silicate solution was measured. The dissolution residue was washed, dried, and ground. The dissolution residue was subjected to XRD analysis and component analysis using an X-ray diffractometer (XRD, Empyrean, PANalytical B.V., Holland) and a wavelength dispersive X-ray fluorescence spectrometer (WD-XRF, XRF-1800, Shimadzu, Japan), respectively.

1.3.3 Bonding strength test

The recycled water glass was obtained by concentrating the low concentration sodium silicate solution. The bonding strengths of the recycled water glass were measured and the bonding strengths of the commercial water glass with the same modulus and density were also measured for comparison. The Da-lin sand and the commercial ester were mixed in a sand mixer for 60 s, and then the water glass was added into the mixer and mixed for 30 s. The commercial ester used was 0.3% of the Da-lin sand by mass, and the water glass used was 3% of the Da-lin sand by mass. The mixture was made into cylindrical samples (30 mm in diameter and 30 mm in height) and the compressive strengths of the samples were measured.

2 Results and discussion

2.1 Causticization

2.1.1 Effect of causticization temperature on causticization rate

The amount of quicklime powder was indicated as the ratio of the amount of effective calcium oxide in the quicklime powder to theoretical demand [$n(\text{CaO}) : n(\text{Na}_2\text{CO}_3 + \text{NaHCO}_3)$], which was 1.2 : 1 in this experiment, and the causticization time was 15 min. As shown in Fig. 3, the causticization rate increases with increasing the causticization temperature. When the causticization temperature is below 60 °C, the causticization rate is below 50%, and the causticization rate is about 90% when the causticization temperature is over 70 °C. When the causticization temperature is low, the digestion reaction proceeds slowly, and the calcium carbonate precipitates generated by the causticization reaction cover the

quicklime powder and stop the digestion reaction. Therefore, the digestion reaction cannot proceed thoroughly, and the causticization rate is low in the lack of calcium hydroxide. When the causticizing temperature gets high, the digestion reaction is fast, making it difficult for the calcium carbonate precipitates to cover the quicklime powder in time. So the digestion reaction proceeds relatively thoroughly and increases the causticization rate. Since the causticization reaction is reversible, the causticization rate cannot reach 100%.

Fig. 3: Effect of causticization temperature on causticization rate

2.1.2 Effect of causticization time on causticization rate

As shown in Fig. 4, the causticization rate for 90 °C rises to 92.2% in 3 min, and then reaches 93.0% in 15 min, indicating the causticization reaction is basically completed in 3 min at 90 °C. The causticization rate for 80 °C reaches 82.8% in 3 min, and then rises to 92.8% in 15 min, which means 15 min is needed for the causticization reaction to complete at 80 °C. The heat released by the digestion reaction at 90 °C boils the filtrate, making it difficult for the calcium carbonate precipitates to cover the quicklime powder, so the digestion reaction proceeds rapidly. The filtrate is not boiled by the digestion reaction at 80 °C, so the calcium carbonate precipitates slow the digestion reaction down. Therefore, the causticization reaction takes a longer time to

Fig. 4: Effect of causticization time on causticization rate

complete. The causticization temperature should be 90 °C and the causticization time should be 3 min, so that a high causticization rate can be achieved within a short time.

2.1.3 Effect of amount of quicklime powder on causticization rate

As shown in Fig. 5, when the amount of quicklime powder is 1:1 (theoretical demand), the causticization rate is just 81.1%. The digestion reaction cannot be thorough because of the calcium carbonate precipitates when it happens in the filtrate, so the causticization reaction is not thorough in lack of calcium hydroxide. As the amount of quicklime powder increases, more calcium hydroxide is produced by the digestion reaction and the causticization rate increases. As the causticization rate does not change much when the amount of quicklime powder rise from 1.2 : 1 to 1.3 : 1, the amount of quicklime powder should be 1.2 : 1, so that the quicklime powder can be saved.

Fig. 5: Effect of amount of quicklime powder on causticization rate

2.2 Dissolution

2.2.1 Effect of mass of filtration residue on sodium silicate modulus

The dissolution temperature was 90 °C and the dissolution time was 1 h in this experiment. As shown in Fig. 6, as the mass of filtration residue increases, the sodium silicate modulus increases from 1.12 for 6 g to 2.25 for 15 g, and the sodium silicate modulus is roughly proportional to the mass of filtration residue, indicating that the proportion of silica dissolved in the filtration residue is substantially unchanged. When the mass of filtration residue is 15 g, the sodium silicate modulus is 2.25, which is within the range of sodium silicate modulus commonly used in casting. Therefore, in the subsequent experiments, the mass of the filtration residue was 15 g.

2.2.2 Effect of dissolution temperature on sodium silicate modulus

The dissolution time was 1 h in this experiment. As shown in Fig. 7, the sodium silicate modulus increases from 0.97

Fig. 6: Effect of mass of filtration residue on sodium silicate modulus

to 2.25 with increasing the dissolution temperature from 50 °C to 90 °C. The dissolution of silica in the lye is slow at a low temperature, so not much silica is dissolved within 1 h, resulting in a low sodium silicate modulus. When the temperature gets higher, the dissolution of silica performs faster, so more silica is dissolved in 1 h and the sodium silicate modulus increases.

Fig. 7: Effect of dissolution temperature on sodium silicate modulus

2.2.3 Effect of dissolution time on sodium silicate modulus

As shown in Fig. 8, the sodium silicate modulus rapidly rises to 2.19 in 20 min and only reaches 2.25 in 60 min at 90 °C; while the sodium silicate modulus rises to 2.00 in 20 min and reaches 2.19 in 60 min at 80 °C. It is indicated that the dissolution of silica occurs mainly in the first 20 min at high temperatures. The dissolution temperature should be 90 °C and the dissolution time should be 20 min. The sodium silicate modulus in this condition is 2.19, which is still in the range of sodium silicate modulus commonly used in casting.

2.2.4 Analysis of dissolution process

Table 3 shows the content of the main elements in the

Fig. 8: Effect of dissolution time on sodium silicate modulus

Table 3: Contents of main elements in dissolution residue (wt.%)

Element	Si	Ca	Zr	Fe	Al	Na	K	Mg
wt. %	47.2138	19.0259	9.0505	7.7499	7.0701	4.0753	1.7652	1.3665

Fig. 9: XRD patterns of (a) amorphous silica, (b) filtration residue, and (c) dissolution residue

the dissolution residue is due to the fact that soluble sodium salts were washed away when the dissolution residue was washed. Therefore, the substance dissolved in the lye is mainly amorphous silica.

2.3 Bonding strength

The recycled water glass was prepared by concentrating the low concentration sodium silicate solution (modulus 2.19) obtained in the dissolution experiment to a density of $1.54 \text{ g} \cdot \text{mL}^{-1}$. The moduli and densities of the recycled water glass and the commercial water glass are the same. Figure 10 shows the recycled water glass and the commercial water glass. The recycled water glass is brown because the waste sodium silicate-bonded sand contains soluble colored

dissolution residue. Figure 9 shows XRD patterns of amorphous silica, the filtration residue, and the dissolution residue. The comparison between Fig. 9(a) and (b) shows that the filtration residue contains amorphous silica in addition to crystalline substances. The comparison between Figs. 9(b) and (c) shows that the dissolution residue is substantially free of amorphous silica and contains the same crystalline substances as the filtration residue, indicating that the amorphous silica in the filtration residue is dissolved during the dissolution process while the crystalline substances are not. The comparison between Table 2 and Table 3 shows that the content of silicon in the dissolution residue is significantly less than that in the filtration residue, while the content of other elements except for silicon and sodium rises to more than twice the amount in the filtration residue. The decrease in the content of sodium in

impurities, which are dissolved in the wet reclamation sewage. The colored impurities cannot be removed by filtration and causticization, resulting in coloration of the recycled water glass.

As shown in Fig. 11, the bonding strength of the recycled water glass is slightly lower than that of the commercial water glass. The lye obtained by causticizing the filtrate and the filtration residue both contain sodium carbonate, so the recycled water glass contains sodium silicate, which may affect the bonding strength of the recycled water glass. The results in Fig. 11 show that the sodium carbonate has little effect on the bonding strength of the recycled water glass. Therefore, the recycled water glass can be used as a substitute for the commercial water glass.

Fig. 10: (a) Recycled water glass, (b) Commercial water glass

Fig. 11: Bonding strength of recycled water glass and commercial water glass

3 Conclusions

(1) The filtrate of the wet reclamation sewage was successfully causticized with a quicklime powder, and the highest causticization rate was over 92%. The causticization rate increased with increasing the amount of quicklime powder, causticization temperature, and causticization time.

(2) The lye obtained by causticizing the filtrate of the wet reclamation sewage could dissolve amorphous silica in the filtration residue to prepare a sodium silicate solution. Increasing the mass of filtration residue, dissolution temperature, and dissolution time improved the sodium silicate modulus.

(3) The recycled water glass was obtained by concentrating the sodium silicate solution, and the bonding strength of the recycled water glass was close to that of the commercial water glass with the same modulus and density. The recycled water glass can be used as a substitute for the commercial water glass.

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