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ORIGINAL ARTICLE

Utilization of fine powder in demolition concrete as recyclable coagulant in removing color from dye-bearing wastewater

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Abstract The fine powders in demolition concretes with Portland cement, high alumina cement or white silicate cement as binders were used as coagulants to treat wastewater containing disperse red S-GR (typical hydrophobic dye) or reactive brilliant blue X-BR (typical hydrophilic dye). The efficiency in removing a color from wastewater was examined at varying contact time, dosage and regeneration cycles. In addition, the coagulation sludge was characterized by thermal analyses and X-ray diffraction technique for recycling. High alumina cement can coagulate both dyes at a relative low dosage of 2–3 g/L, but regeneration performance was poor. The coagulation with white silicate cement reached a color removal of 98 % for disperse red S-GR at a dosage of 6 g/L and 90 % for reactive brilliant blue X-BR at a dosage of 18 g/L. Portland cement showed a rapid coagulation rate and >95 % color removal for both dyes even after seven cycles of coagulation and sludge calcination. The dosage of the coagulant required for active brilliant blue X-BR coagulation (10 g/L) was much higher than that for disperse red S-GR coagulation (2.5 g/L). It is concluded that Portland cement can be used as a recyclable coagulant in the treatment of dye-bearing wastewater.

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List of symbols

A	Al_2O_3
C	CaO
F	Fe_2O_3
H	H_2O
S	SiO_2
C_3S	Tricalcium silicate, $3CaO{\cdot}SiO_2$, Ca_3SiO_5
C_2S	Dicalcium silicate, $2CaO{\cdot}SiO_2$, Ca_2SiO_4
C_3	A tricalcium aluminate, $3CaO{\cdot}Al_2O_3$, $Ca_3Al_2O_6$
C_4AF	Ferrite, $CaO{\cdot}Al_2O_3Fe_2O_3$, Ca_2AlFeO_5
CA	$CaO{\cdot}Al_2O_3$, $CaAl_2O_4$
C–S–H	Calcium silicate hydrate gel
C–A–H	Calcium aluminate hydrate
CAH_{10}	$CaO{\cdot}Al_2O_3 {\cdot}10H_2O$
AH_3	$Al(OH)_3$

Introduction

The amount of demolition concrete is nearly 320 Mt/a in the United States, 510 Mt/a in Europe (Kumar and Morawska 2014; Mália et al. 2013; Kucukvar et al. 2014) and over 240 Mt/a in China (Li 2008; Shi and Xu 2006). The recycling of concrete is attempting to address the growing concrete waste stream in environmentally friendly manners by providing an alternative to landfills (Diaz and Otoma 2013). The Waste Framework Directive 2008/98/EC requires member states of European Union to take any necessary measures to recycle a minimum of 70 % construction and demolition (C&D) waste by 2020 (Kumar and Morawska 2014; Achillas et al. 2013).

Major concrete components must be processed separately to source recycled materials. If recycled concrete aggregate (RCA) is freed from hydrated cement paste residues, which would be detrimental to the performance of the new concrete (Li 2008; Serpell and Lopez 2013), substitution of natural stone materials by RCA can reach 100 % (Hu et al. 2010; Srour et al. 2013). Usually, 75 % of the original wastes in demolition concrete can be recovered as high-quality fine or coarse aggregates, leaving 25 % of fine powder comprised mostly of hydrated and non-reacted cement phases (Vivian et al. 2006; Zhang 2013; Al-Dwairi et al. 2014). Recycling of fine powder as recyclable coagulant for wastewater treatment holds high environmental value, reducing impacts from natural resources consumption, greenhouse gas emissions, and energy consumption. The fine fraction of recovered concrete-based demolition waste has proven to be an effective sorbent for aqueous heavy metal species by virtue of its ion exchangeable phases and high pH (Coleman et al. 2005; Chen et al. 2009). Recently, cement kiln dust was used to treat textile industrial effluents effectively (Mahmoued 2010). However, to the best knowledge of the authors, little work has been reported to apply fine powder in demolition concrete as a recyclable coagulant for the de-colorization of dyeing wastewater until now.

It is estimated that more than 100,000 commercially dyes with over 7×10^5 tons of dyestuffs produced annually (Anjaneyulu et al. 2005; Lu et al. 2010). Of these dyes, reactive dye and disperse dye are most extensively used in textile industry. As substantial amounts of dyes are usually present in textile industrial effluents, displaying a high organic load as indicated by high chemical oxygen demand (COD) and strong color, the pollution of dyes has been receiving much attention because of the growing concern over impacts of wastewater from dyeing houses on the aquatic environment. This has prompted the textile industry to investigate more appropriate and environmentally friendly on-site or in-plant treatment technologies to meet the stringent discharge consents (Sanz et al. 2013).

Most of the dyes have complicated aromatic structures that resist biological degradation in conventional wastewater treatment processes because they may exert significant toxicity toward the mixed microbial communities within biological treatment systems. Dyestuffs have potential for formation of toxic aromatic amines and their removal rates are very low during aerobic waste treatment. Anaerobic degradation products of some dyes are suspected of being carcinogenic and mutagenic (Shaw et al. 2002). Physical and chemical techniques such as coagulation (Kumar et al. 2008; Shi et al. 2007), adsorption (Lazaridis et al. 2003; Sakkayawong et al. 2005; Vimonses et al. 2010; Yu et al. 2001), filtration (Mahmoued 2010), membrane filtration (Bes-Piá et al. 2002; Zahrim et al. 2011), electrochemical

oxidation (Babu et al. 2007), photolysis, UV/H$_2$O$_2$, photocatalytic oxidation (Chu and Tsui 2002), electro-photocatalytic oxidation, ozonation and Fenton's reaction (Kang and Chang 1997) have been used for dyeing effluent treatment. Coagulation by the use of FeCl$_3$, Fe$_2$(SO$_4$)$_3$, FeSO$_4$, AlCl$_3$, Al$_2$(SO$_4$)$_3$, Ca(OH)$_2$, poly-aluminum chloride (PAC), poly-ferrite sulfate or polyelectrolytes (anionic, cationic, or nonionic polymers) is a feasible way of removing color from dyeing wastewater (Chu 2001; Hassan et al. 2009), but the sludge generated in coagulation needs to be treated through complicated handling and disposal procedures, usually by cement-based solidification/stabilization and landfill. The costs of the operation are high and landfill site is sanity. The proper disposal, recycling and reuse of coagulation sludge have become a significant environmental issue. On the other hand, the high cost of conventional coagulants has inspired a search for suitable low-cost coagulants. In this work, the fine powders in demolition concretes were used as coagulants to treat wastewater containing disperse red S-GR (typical hydrophobic dye) or reactive brilliant blue X-BR (typical hydrophilic dye). The coagulation sludge was characterized and the recycling of the coagulation sludge was discussed.

Experimental

Materials

Three waste concretes made from Portland cement, high alumina cement or white silicate cement were collected from demolition sites in Songjiang, Shanghai, China. The crushed and grounded concretes were sieved to obtain <30 μm of powders. For the interest of simplicity, they are denoted as Portland cement, alumina cement or white silicate cement herein below. The chemical compositions are shown in Table 1.

Two typical commercial dyes, namely disperse red S-GR and reactive brilliant blue X-BR, were selected to value the effectiveness of cements as recyclable coagulants. The formal is typical hydrophobic dye and the latter is typical hydrophilic dye. Their structures are depicted in Fig. 1. The dyes were dissolved in de-ionization water in a concentration of 143 mg/L (maximal solubility of disperse red S-GR) at a room temperature of approximately 20 °C for use.

Coagulation experiment

Bench-scale experiments were conducted using a standard six-beaker jar test apparatus (six paddle stirrer Model JJ-4, Jiangtan Co. Ltd.). Varying dosages of coagulant were added to the jars with 750 mL of dye solution in the

Table 1 Chemical compositions of fine powder in demolition concrete

Binder of concrete	SiO₂	Al₂O₃	Fe₂O₃	CaO	MgO	TiO₂	SO₃	Na₂O
Alumina cement (%)	6.74	51.99	2.23	34.75	0.81	0.37	0.18	0.31
Portland cement (%)	20.58	5.64	3.95	62.25	2.48	0.32	3.18	0.36
White silicate cement (%)	20.51	5.39	0.41	70.77	1.72	0.12	3.75	0.45
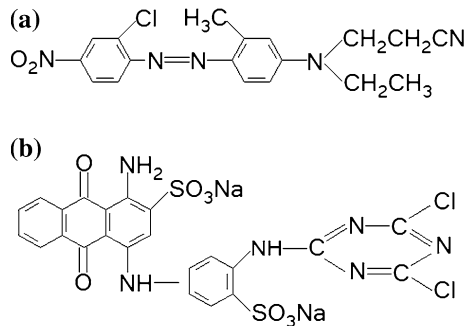
Fig. 1 Chemical structure of dyes used. **a** Disperse red S-GR. **b** Reactive brilliant blue X-BR

concentration of 143 mg/L at a room temperature of approximately 20 °C. Each sample was rapid stirred at impeller speed of 150 rpm for 30 min followed by slow stirring at impeller speed of 50 rpm for desired time, which corresponds to a suitable velocity gradient value "G" = 50 s⁻¹ for the flocculation process. Thereafter, the impeller was removed and the suspension was allowed to settle for 30 min. At the end of the settling period, the supernatant sample was withdrawn at the same depth (3 cm below the surface of the supernatant) and filtered through 0.45 μm membranes for analyses.

The dye concentration and color in the supernatant was determined spectrophotometrically using the calibration curves by monitoring the absorbance at λ_{max} 570 and 610 nm for disperse red dye and active brilliant blue X-BR, respectively. All spectrophotometric measurements of dyes were done with a Shimadzu 160A UV–vis spectrophotometer using 1.0 cm matched quartz cuvettes. In all experiments, the difference between the initial dye concentration (C_o) and the residual concentration (C_e) was used to calculate the dye removal. A digital pH/mV meter (Model PHS-3C) in conjunction with a combination glass electrode was used for all pH measurements.

Calcination of coagulation sludge

The sludge generated from the coagulation was filtered and then dried at 65 °C followed by the calcination at 850 °C for 2 h to obtain the recycling coagulant. The calcined product was ground to fine powders (<30 μm) in a pestle and mortar prior to the addition to dye aqueous solutions.

Characterization of coagulation sludge

X-ray diffraction

The samples were oven dried at 65 °C and manually ground to fine powders (<30 μm) in a pestle and mortar prior to X-ray diffraction (XRD) analyses. The XRD analyses were conducted using a Rigaku D/Max 2000 diffractometer operated at 40 kV and 30 mA (CuK-alpha radiation) in the range 5–70° 2θ at scanning rate of 1° 2θ per minute. Once the diffraction patterns were obtained, both manual matching of the peak positions and a computer-aided search for the compounds were performed with the current version of the international powder data file (ICDD-JCPDS).

Thermal analyses

The coagulation sludge was oven dried at 65 °C until constant weight was achieved and ground with an agate mortar and pestle to less than 30 μm. Sample of 20 mg was packed in the crucible of 5.8 mm diameter and 4 mm high and made from rhodium–platinum. TG/DTG curves were obtained on DT-40 Thermal Analyzer in the temperature range of 30–900 °C with a heating rate of 10 °C/min under flowing nitrogen (40 cm³/min). The CaCO₃ contents were determined at 700–850 °C and calculated using the following equation:

$$CC(\%) = WL_{CC}(\%) \times MW_{CC}/MW_C \tag{1}$$

where CC(%) is the content of CaCO₃, $WL_{CC}(\%)$ is the weight loss occurred during the decomposition of calcium carbonate, MW_C and MW_{CC} are the molar weight of carbon dioxide and calcium carbonate.

Results and discussion

Coagulation performance of fine powder in demolition concrete

A series of coagulation experiments were performed varying the dosage of fine powder in demolition concrete and the process performance was evaluated on the basis of the removal efficiency of dyes and volume of sludge. The coagulation was achieved in a jar test through perikinetic phase (fast agitation 150 rpm for 30 min), orthokinetic phase (slow agitation 50 rpm for 4 h), and a phase of

settling (30 min). Figure 2 shows the effect of fine powder dosages on the efficiency of dye removal. Interestingly, dye removal varied significantly with the nature of cements, structure of dyes and dosage of fine powders. The dosage of silicate cements required for hydrophobic dye coagulation was much lower than that for hydrophilic dye coagulation. The optimal dosage of alumina cement for both dyes was around 3 g/L, lower than those of silicate cements. At the optimal dosage, the supernatant solution was almost colorless and displayed a dye concentration of 3 ∼ 5 mg/L, indicating an efficiency of dye removal exceeding 98 %. Note that the coagulation efficiency of white silicate cement for reactive brilliant blue X-BR was only 90 %. This indicated that the coagulation of hydrophobic dye with cement phases was more effective than that of hydrophilic dye.

The re-stabilization of disperse red S-GR dye was obvious at the over dose of alumina cement or Portland cement (Fig. 2). Theoretically, hydrophobic dyes (e.g., disperse red S-GR) have an extremely low solubility in water, and they can be treated as ''colloidal particles''. Upon coagulation at suitable doses, agglomeration occurs and dye particles are destabilized as a consequence of van der Waals' force attraction and can be removed by sedimentation. However, at an overdose of coagulant, an
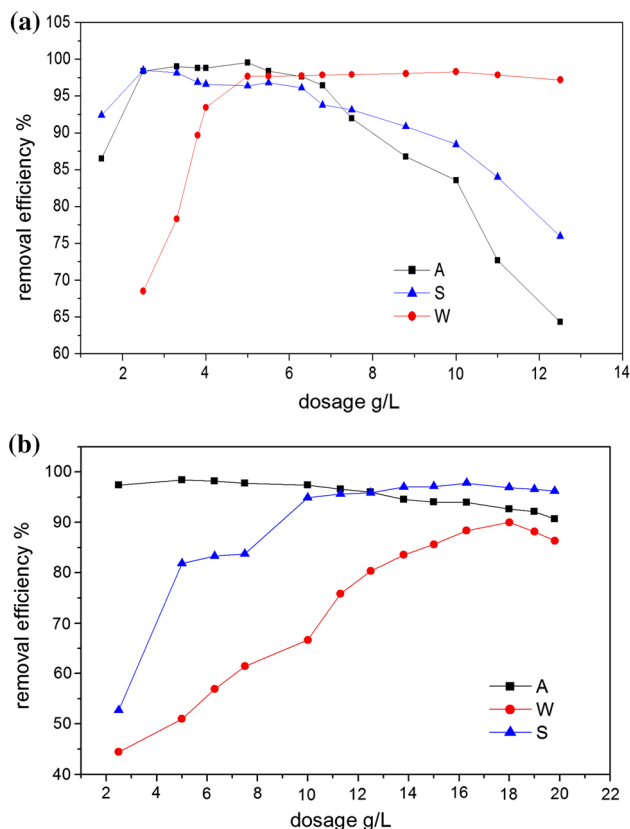
electrostatic repulsive force is established between the dye particles due to the accumulation of silicate, calcium, iron and aluminum ions on the particle surface.

Figure 3 shows the setting performance of the coagulation sludge, which possessed superior thickening and dewatering characteristics and was suitable for filter pressing. It can be efficiently dewatered without the addition of polymers, which is necessary for the conditioning of the sludge produced from ferric (ferrous) salts or alum coagulation. The volume of the coagulation sludge with Portland cement was much less compared with the volume of the coagulation sludge with other two cements.

The pH variations during the dye coagulation are presented in Figs. 4 and 5 at a dosage of 3 g/L. The pH increased with contact time and the removal efficiency of dyes increased correspondingly. As is well known, the pH variation reflected the effect of dye molecules on cement phase hydration. It can be seen that both dyes, especially disperse red S-GR, retarded the hydration of cement
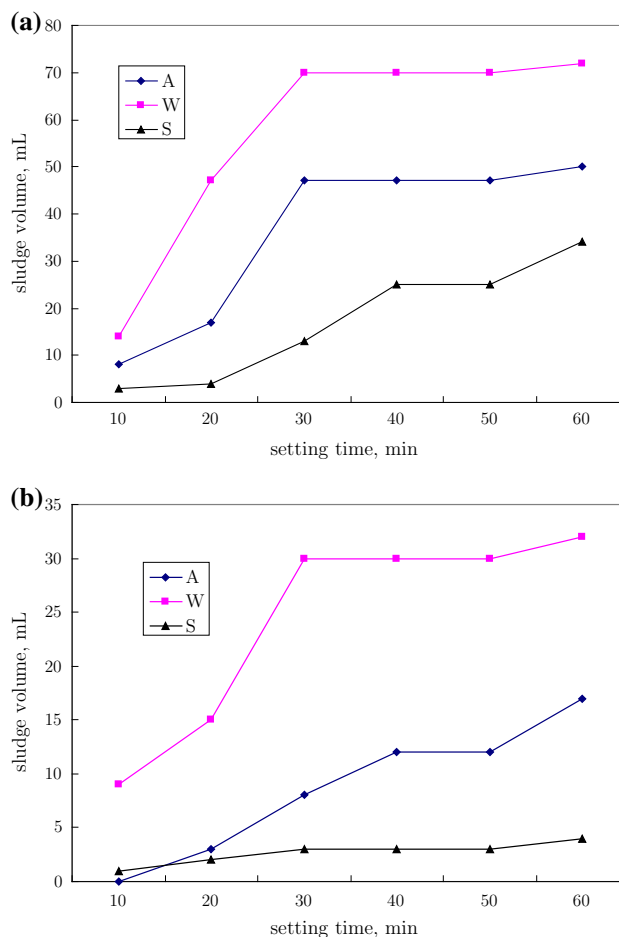

Fig. 2 Effect of coagulant dosage on the efficiency of dye removal. a Disperse red S-GR. b Reactive brilliant blue X-BR. (A alumina cement, W white silicate cement, S Portland cement)

Fig. 3 Setting performance of the coagulation sludge. a Disperse red S-GR. (A alumina cement 2.25 g/L, W white silicate cement 6.58 g/L, S Portland cement 2.53 g/L). b Rreactive brilliant blue X-BR. (A alumina cement 2.55 g/L, W white silicate cement 17.51 g/L, S Portland cement 10.53 g/L)
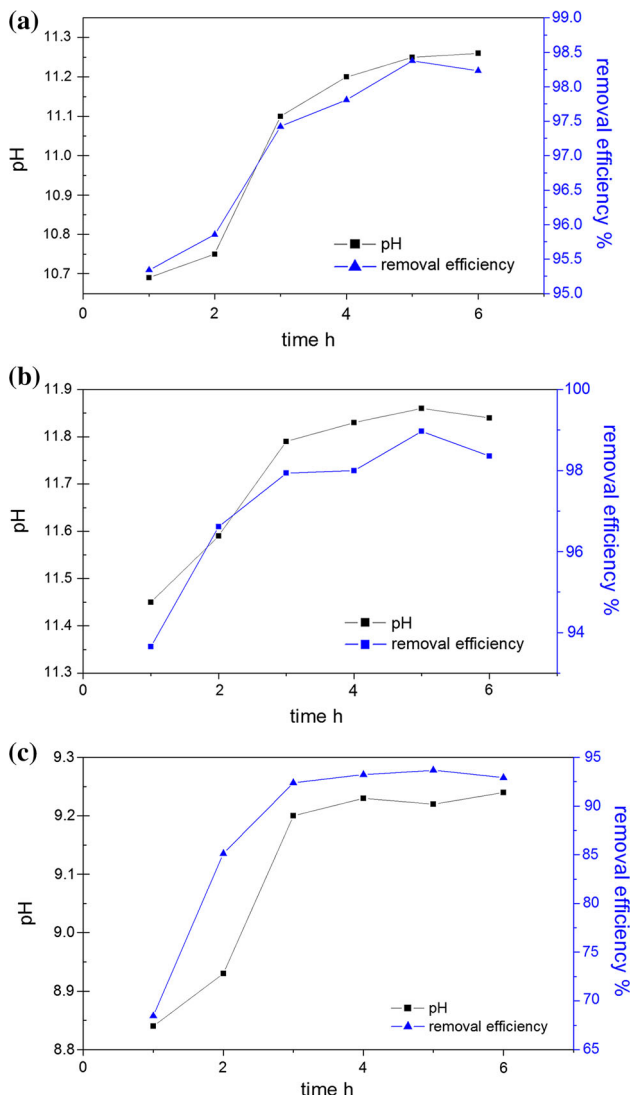
Fig. 4 pH variations during disperse red S-GR coagulation. **a** Alumina cement. **b** Portland cement. **c** White silicate cement

phases, as the suspension pH values of cement hydration were close to 12.4 and 11.5 immediately for silicate cements and alumina cement when they contacted tap water. According to the pH variation rate, the retarding effect of hydrophilic active brilliant blue X-BR was less significant compared with that of hydrophobic disperse red S-GR. This is because the adsorption of hydrophobic dyes on the surface of cement particles slowed down or prevented the interior of the particles from hydrating.

Coagulation performance of calcined sludge

The coagulation sludge was calcined at 850 °C for 2 h and reused in the coagulation of dye solutions. At calcined sludge dosage of alumina cement 2.25 g/L, white silicate cement 6.58 g/L or Portland cement 2.53 g/L, effects of
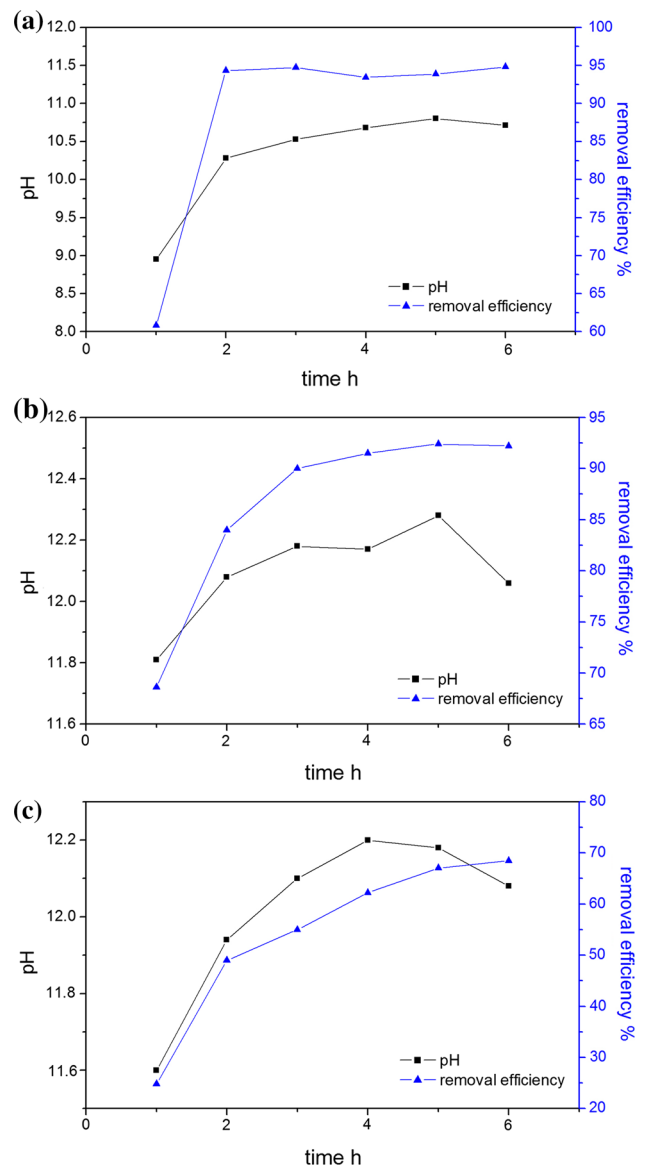

Fig. 5 pH variations during reactive brilliant blue X-BR coagulation. **a** Alumina cement. **b** Portland cement. **c** White silicate cement

regeneration cycles of coagulation–calcination on the removal of disperse red S-GR dye are shown in Fig. 6a. At calcined sludge dosage of alumina cement 2.55 g/L, white silicate cement 17.51 g/L or Portland cement 10.53 g/L, effects of regeneration cycles of coagulation–calcination on the removal of reactive brilliant blue X-BR are shown in Fig. 6b. It can be seen that recycling sludge showed excellent performance in the coagulation except coagulation sludge with alumina cement.

Sludge characterization and coagulation mechanism

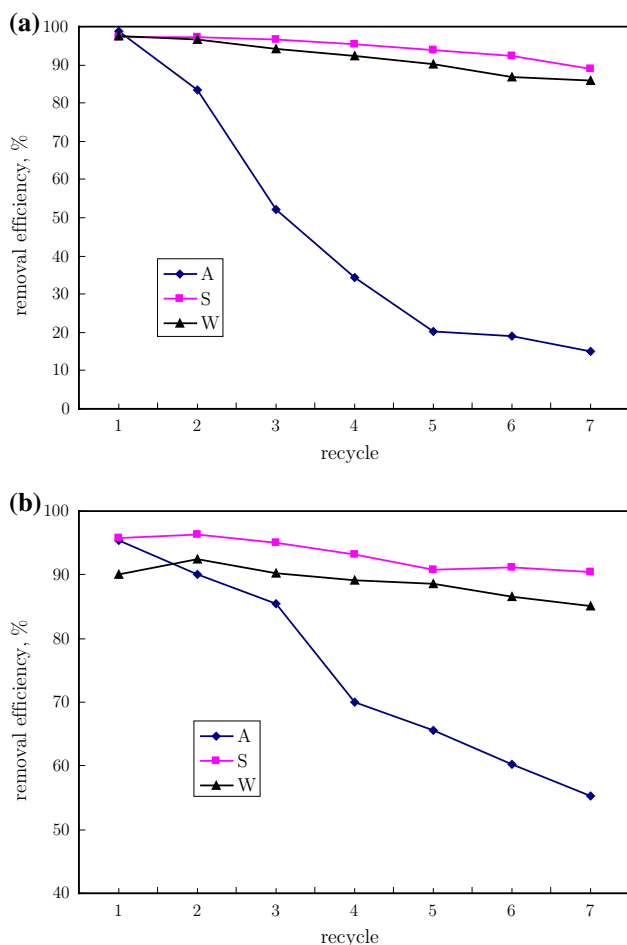Diffractograms of cement, coagulation sludge and regenerated coagulant (calcined sludge in the fifth cycle of

Fig. 6 Effect of regeneration cycles on dye removal. a Disperse red S-GR. b Reactive brilliant blue X-BR. (A alumina cement, W white silicate cement, S Portland cement)

coagulation–calcination) are shown in Fig. 7. There was no significant difference between cement and regenerated coagulants. However, the difference in cement and the coagulation sludge was noticeable.

In the coagulation sludge with alumina cement, the absence of X-ray reflection peaks in the coagulation sludge at 16°, 22°, 24°, 29.5°, 35°, 36° and 46° (2θ) may imply that the amount of CA phase reduced due to the hydration in coagulation. C–A–H dehydrated in the calcination, resulting in the formation of CA and recurrence of these peaks.

As shown in Fig. 7b, the main diffractogram differences between Portland cement, coagulation sludge and the regenerated coagulant were in double peaks around 32° (2θ), which were attributed to X-ray reflections of C_3S and C_2S. Note that the calcination of the sludge resulted in the reformation of C_3S and C_2S due to dehydration of C–S–H, as reflection peaks of C_3S and C_2S around 32° were absent in the coagulation sludge but present in the regenerated coagulant. The residual cement phases such as C_4AF can
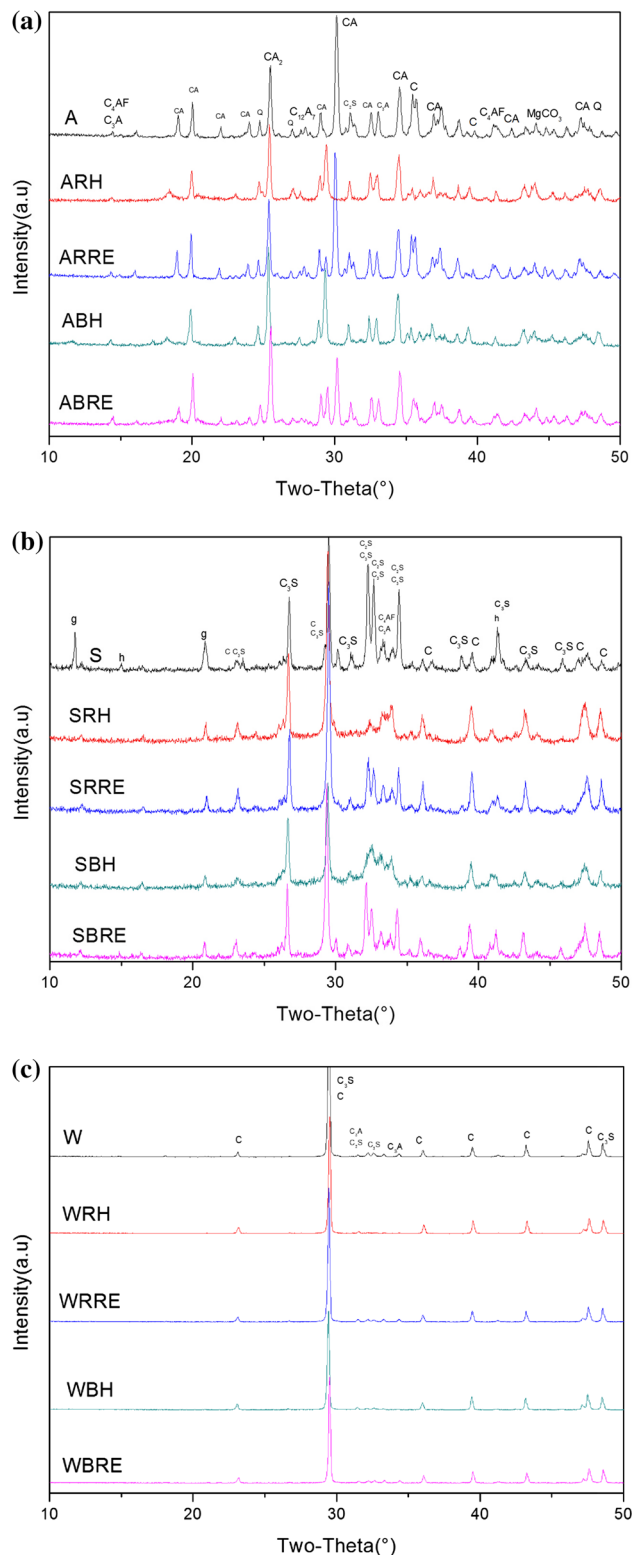

Fig. 7 Diffractograms of cement, coagulation sludge and the regenerated coagulant. a Alumina cement. b Portland cement. c White silicate cement. (A stands for alumina cement, S for Portland cement, W for white cement; R stands for disperse red S-GR, B for active brilliant blue X-BR; H stands for hydration, RE for calcined sludge (regenerated coagulant); C stands for calcite, g for gypsum, h for hemi-hydrate, Q for quartz)

be identified in all samples, as the hydration of these phases was very slow (see Fig. 7b). It should be pointed out that peaks of gypsum (11.6º, 20.7º, 23.4º, 31.8º, 32.9º, 42.2º, 2θ) and hemi-hydrate (14.7º, 25.7º, 29.7º, 42.2º, 2θ) were absent in the coagulation sludge and regenerated coagulant, indicating that these phases dissolved during the coagulation process.

It is noted from Fig. 7c that the diffractograms of white silicate cement, coagulation sludge and the regenerated coagulant varied at X-ray reflections of C_3S around 32º

(2θ). Obviously, the calcination of the sludge resulted in the reformation of C_3S due to dehydration of C–S–H.

(A stands for alumina cement, S for Portland cement, W for white cement; R stands for disperse red S-GR, B for active brilliant blue X-BR; H stands for hydration, RE for calcined sludge (regenerated coagulant); C stands for calcite, g for gypsum, h for hemi-hydrate, Q for quartz).

The TG curves and simultaneous DTG curves of coagulation sludge are presented in Fig. 8. In Figs. 8a and b, the mass loss at 50–310 °C was attributed to the evaporation of
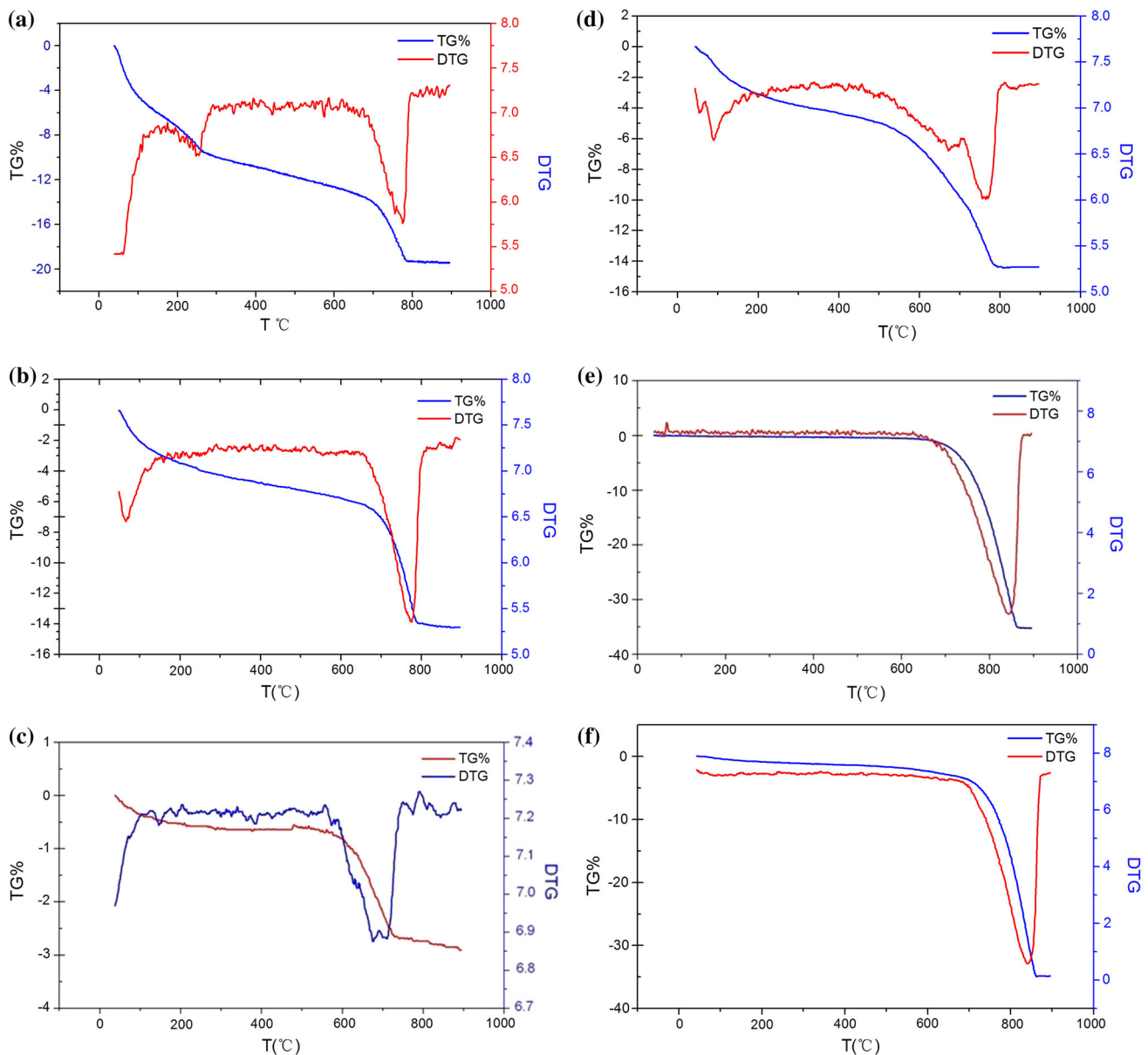

Fig. 8 TG/DTG curves of the coagulation sludge. **a** Disperse red S-GR coagulation sludge with alumina cement. **b** Reactive brilliant blue X-BR coagulation sludge with alumina cement. **c** Disperse red S-GR coagulation sludge with Portland cement. **d** Reactive brilliant blue X-BR coagulation sludge with Portland cement. **e** Disperse red S-GR coagulation sludge with white silicate cement. **f** Reactive brilliant blue X-BR coagulation sludge with white silicate cement

bound water and the decomposition of C–A–H (e.g., CAH_{10}). The mass loss at 300–610 °C was due to dehydration of AH_3 and the mass loss at 700–810 °C was due to decomposition of calcite. The total mass loss in the active brilliant blue X-BR coagulation sludge was 15 %, whereas the total mass loss in disperse red S-GR coagulation sludge with alumina cement was 20 %. The total mass loss reflected the hydration degree of cement, as all samples were dry prior to the thermal analyses. It can be considered that the difference in the hydration degree of alumina cement in the coagulation of both dyes was insignificant.

In Figs. 8c and d, the mass loss at 50–110 °C was attributed to the evaporation of physical and chemical bound water, and the mass loss peak at 120–320 °C due to dehydration of C–S–H and decomposition of ettringite ($6CaO·Al_2O_3·3SO_3·32H_2O$), which is water insoluble produced by the reactions of gypsum or anhydrite with tricalcium aluminates and water during the early hydration of Portland cement (Carmona-Quiroga and Blanco-Varela 2013). The mass loss at 600–810 °C was evident, which could be attributed to the decomposition of calcium carbonate. For the disperse red S-GR coagulation sludge with Portland cement, the total mass loss was only 3 %, indicating that the hydration degree of Portland cement was very low. In Fig. 8d, the total mass loss of reactive brilliant blue X-BR coagulation sludge with Portland cement was 15 %. The retarding effect of disperse red S-GR on Portland cement hydration was much stronger than that of reactive brilliant blue X-BR, in agreement with pH variation mentioned above.

The TG/DTG curves of the coagulation sludge for both dyes with white silicate cement were similar (see Figs. 8e and f). The step of mass loss in TG curves corresponding to decomposition of calcite was noticed. According to the amount of mass loss, the calcite content was close to 90 %.

As hydration of cement phases results in the formation of hydrolyzed ions of calcium, silicate, iron and aluminum, cement acts as a composite coagulant and is more effective than aluminum or iron salt for dye coagulation (Coleman et al. 2005; Mahmoued 2010). During the dye coagulation, the removal of dyes could be accomplished by a complex process involving one or more mechanisms such as precipitation, enmeshment, surface charge neutralization and adsorption on the surfaces of residual cement particles, aluminum hydroxide, ferrite hydroxide and other cement hydration products. Although the broad principles of action of conventional coagulants are reasonably well understood, there are still some uncertainties regarding the nature of the active species of hydrolyzed cement.

The principal hydration products of silicate cement are C–S–H and $Ca(OH)_2$, whereas the principal hydration products of alumina cement are C–A–H and $Al(OH)_3$. The dehydration of C–S–H and C–A–H resulted in the reformation of C_3S, C_2S and CA (see Fig. 7). The regeneration performance of coagulation sludge with silicate cement including white silicate and Portland cement was better than that of coagulation sludge with alumina cement. This may be attributed to the difference of reactivity of reformed cement phases to water. Reformed C_3S reacts rapidly with water to produce a gelatinous mass of calcium silicate hydrate gel, which possesses excellent capability of enmeshment and adsorption for dyes in wastewater. Furthermore, the high pH due to the hydration of C_3S resulted in precipitation of dyes. Reformed CA reacts with water to form C–A–H which is inhibited due to a dense layer formation of $Al(OH)_3$, as calcium is dissolved more rapidly. It is reported that the hydration of CA stopped although 27 % CA is remaining due to a dense layer of hydration products surrounding non-reacted remaining CA (Klaus et al. 2013, 2015). We assume that CA dissolution decreases strongly after the dense hydrate layer reaches certain thickness. In consequence, the coagulation efficiency reduced with the increase of coagulation–calcination cycles.

The crushing of demolition concrete to produce recycled aggregate gives rise to a large volume of hydrated cement phase-rich fine powder. It is expected that this fine powder may replace conventional coagulant in the treatment of dyeing wastewater treatment or dyestuff-making wastewater in industrial application. This can be economically viable and an interesting alternative for recycling of fines derived from RCA production. The various advantages associated with the coagulation process include local availability of materials, simplicity, flexibility and reliability of the system; and recycling characteristics of the coagulation sludge. Additionally, the coagulation sludge was of high pH and chemically stabilized, and can be buried directly after many cycles of coagulation–calcination.

Conclusions

One hydrophobic and one hydrophilic dye were used as model pollutants to examine the performance of coagulation with fines of demolition concrete. The removal of the dyes increased with the increase of coagulation contact time. The optimal dosage of coagulant was dependent on the nature of dye and cement. The sludge resulting from the coagulation with silicate cements can be recycled at least seven cycles of coagulation–calcination. The recycling of concrete fine powders into economically viable coagulants may help in minimizing the impact of demolition concrete and dyeing wastewater to the environment.

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