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Mineralogy of Laterite Samples from Viet Nam and Their Use as Flocculating Agents for the Removal of Clay Suspensions

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The existence of clay suspended particles is one of main factors increasing turbidity in water. Flocculation is a common technique used to eliminate suspended materials from water. Currently, a variety of flocculating agents have been found as commercial products and applied to purify water. Most of agents works well and bring positive effects on environment. However, the use of some agents has shown problems relating to economic benefits and environment. The treatment cost and environmental safety evoke the increasing interests in not only developing countries but also in developed countries. To solve these problems, many researches have been carried out in the past decades focusing on improving materials for treatment. As a following tendency, the present study aims to understand mineralogy of the laterite samples located in different areas in Viet Nam and investigate their use as flocculating agents for clay suspended particle removal. Three laterite samples were collected to use for experiments in this study. The laterite sample collected in Thach That region (1–TT) performed the best flocculation efficiency followed by the laterite sample collected in Hoa Lac region (2–HL). The sample collected in Quynh Tam commune (3–HL) worked less efficiently than the other two samples. Geothite and hematite are considered to be the main components that are responsible for flocculation action. The study also suggested that the presence of clay sized quarts has an adverse effect on flocculation action.

Key words: clay particle, flocculation, geothite, hematite, laterite, water processing

INTRODUCTION

The suspended clay particles in water are main pollutants that cause turbidity in water environment. Removal of suspended particles is an essential operation in water treatment. Flocculation technique is a common way that is used to remove suspended materials from natural waters. In flocculation process, flocculating agents are added to overcome electrostatic repulsion and promote coalescence of the suspended particles to form large flocks that settle down rapidly (Tripathy and De Ranjan, 2006).

The synthetic agents, such as poly–diallyldimethylammoniumchloride (PDADMAC or Cat–Floc), used either alone or in combination with alum, have been reported to be effective in flocculation of negatively charged suspended particles in water (O'Melia, 1972; Faust and Aly, 1983). Long chain synthetic polymers are also popular flocculating agents to remove suspended materials via bridging and electrostatic patch mechanism (Tripathy and De Ranjan, 2006). Zhang *et al.* (2010) reported that a microbial polymer was effective for flocculating synthetic kaolinite suspensions. Chitosan, a product made from crab shell, is very effective to remove organic suspended materials (Tripathy and De Ranjan, 2006) with less negative effects on the environment.

The flocculating agents have brought undoubted benefits in water treatment. However, negative effects on the environment have been found by the use of some agents. Aluminum salts and synthetic polymers cause possible physiological effects on organisms (Nakanishi and Wada, 2007) as well as aluminum accumulation in the environment and its connection with Alzheimer disease (Stauber *et al.*, 1999). Some natural substances such as chitosan and carboxymethyl cellulose are nontoxic to environment, available from renewable resources and very effective for pollutant removal (Divakaran and Pillai, 2001) but they are not stable against microbial attack during storage (Tripathy and De Rajan, 2006; Zhang *et al.*, 2010) and may increase the biological oxygen demand of processed water if high dose is used. Therefore, flocculating agents with high treatment efficiency and less negative effects on the environment is needed for advanced water processing in open aquatic systems.

Natural flocculants derived from soil were reported by Nga *et al.* (2012b), which were effective in removing pollutants from water at a low treatment cost. The flocculant made from volcanic soil was found to be as effective as PAC for flocculating suspended clay particles in some natural reservoirs but their flocculation efficiency largely depended on clay mineralogy (Kuchibune *et al.*, 2006; Nga *et al.*, 2012a). Laterite, the *in–situ* weathering and decomposition products of rocks, is abundant in the tropical and sub–tropical regions and rich in sesquioxides (Fe_2O_3 or Al_2O_3). Thus they are possibly applied for water treatment (Blight, 1997; Nga *et al.*, 2012b). The study of Udoeyo *et al.* (2010) indicated strong absorption capacity for heavy metals of laterite materi-

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Table 1. Source of soil and clay samples

Sample name	Source
T5	B22 horizon of Haplustox in Nakhon Ratchasima, Thailand
F	Ap horizon of Fluvaquents in Fukuoka, Japan
Kaolinite	Iriki mine, Kagoshima, Japan
Montmorillonite	Kunipia–F supplied by Kunimine Co., Tokyo, Japan

als. Lateritic soils have been used as effective sorbents for arsenic in groundwater (Maji *et al*., 2006; Rahman *et al*., 2008; Maiti *et al*., 2010). In addition, laterite adsorbs phosphorous in natural lakes and landfills (Zhang *et al*., 2011). Nga *et al*. (2012b) investigated removal capacity of kaolinite from water by laterite material that was collected in Viet Nam.

The laterite is an abundantly distributed in many regions in Viet Nam due to strong laterization. The characteristics of laterite would be different depending on regional climate and geology. The laterite materials are of high potential use in environmental treatments, but the laterite materials in Viet Nam have not been received due attention. Mineralogy of materials has been considered as an important factor affecting the removal of suspended particles (Nga *et al*., 2012a). Therefore, the present study aims to investigate mineralogy of laterite samples from Viet Nam and their use as a flocculating agent to remove suspended clay particles.

Fig. 1. Location of sampling site.

MATERIALS AND METHODS

Laterite samples

Three laterite samples, namely 1–TT, 2–HL and 3–QT, were collected from lateric soil zones in Viet Nam indicated in Fig 1. The 1–TT sample was collected from a layer at a depth of 1.5 m in Thach That town where completely weathered laterite distributes. The original sample is a hard block with dark yellow color. The 2–HL sample was found in excavated soil at a construction site. It is a hard block containing red solid gravels. The 3–QT sample was taken from a laterite profile at a depth of 0.5 m from the surface. The raw sample was a hard block with black grey color.

The original samples were washed with tap water and air–dried at a room temperature. They were gently crushed to pass through a 2–mm screen. Aliquot of the treated samples was mixed well with distilled water, and adjusted to pH 5–6 with 0.1 mol L^{-1} HCl and sonicated. The prepared three flocculant samples had dry matter content of 131, 127 and 91 g/Lfor1–TT, 2–HL and 3–QT, respectively.

Clay suspensions

Clay suspensions used for flocculation tests were prepared from two soil samples and two specimen clays (Table 1) following the procedure described by Nga *et al*. (2012a). A five–gram portion of the soil or clay sample was mixed with 1 L of deionized water and sonicated for 15 min after pH adjustment to about 8 with 0.1 mol/L NaOH. The dispersed suspension was put into sedimentation cylinder and the fraction having equivalent spherical diameter of $< 2\,\mu$m was separated by siphoning. This step was repeated using several portions of the samples to obtain sufficient amount of clay suspension for flocculation tests. The separated stock clay suspensions were kept in a polypropylene bottle with fitted stopper and diluted with deionized water just before use.

Mineralogy and surface characteristic analysis of clay suspensions and laterite samples

Mineralogy of the laterite samples were analyzed for four components (clay, silt, fine sand and coarse sand) by X–ray diffraction (RINT–2000V, Rigaku) following the procedure of Nga *et al*. (2012a).

Mineralogical analysis of the clay suspensions by X–ray diffraction was carried out following the procedure described by Wada and Umegaki (2001) with a Cu K$_\alpha$ radiation at 40 kV and 20 mA.

Exchangeable cation composition was determined by 1 mol/L NH$_4$OAc extraction. For clay suspensions, the principle of Nga *et al*. (2012) was applied. A clay suspension containing 200 mg of clay was placed on a 0.1 μm membrane filter and washed with 50 mL of the NH$_4$OAc solution under suction. For laterite samples, the method of Muramoto *et al.* (1992) was applied. A Two gram portion of a laterite sample was extracted three times with 30 mL the NH_4OAc solution. The extract was analyzed for Ca^{2+}, Mg^{2+}, Na^+ and K^+ by atomic absorption spectroscopy (Z2300, Hitachi, Japan).

Particle size distribution of the laterite samples was determined by siphoning method. The air–dried samples after organic matter digestion were separated by sonication for 15 minutes. Clay, silt and sand fractions were collected after respective settling time by siphoning.

Surface charge of laterite samples was determined by serial potentiometric titration method. A Five gram portion of the sample was equilibrated with NaCl solutions having different pHs for 4 hours keeping ionic strength at 0.005, 0.02 or 0.1 mol/L. Proton and hydroxide ion adsorption by laterite was calculated from pH difference between before and after reaction, then pH_{PZSE} were determined from the intersection point of the curves where the proton hydroxide adsorption were plotted against pH at different ionic strengths. The surface charge was set to zero at pH_{PZSE} and for other pH surface charges were determined by shifting the proton and hydroxide adsorption isotherms.

Thermal analyses

Differential thermal and gravimetric analysis (DTA–TG) was carried out simultaneously using the system of Rigaku Thermo plus EVO. The samples were heated from 20 to 1000°C for 100 minutes. The amount of sample used in each test was 10 mg.

Flocculation experiments

The flocculation experiments were performed by a jar tester (MJS–3H, Miyamoto Co., Japan) in a room at 20–24°C following the procedure of Nga *et al.* (2012a). A four hundred mL portion of a 500 mg/L clay suspension was placed in a 500 ml tall beaker and the impeller rod was put in the suspension. Immediately after the addition of predetermined dose of laterite flocculants, the suspension was stirred at a speed of 300 rpm for 3 min and at 50 rpm for 5 min. The flocks were allowed to settle for 30 min. A 10 ml portion of the supernatant was collected from 2 cm below the water surface and absorbance was measured at a wave length of 660 nm by a spectrophotometer (U1900, Hitachi, Japan). All of the tests were carried out in triplicate.

RESULTS AND DISCUSSION

Mineralogy and surface charge characteristics of laterite samples

X–ray diffraction patterns of the Mg–saturated and glycerol–solvated clay fractions and fine sand fractions of three laterite samples are shown in Fig. 2. The clay fractions of the 1–TT and 2–HL samples showed fairly strong peaks at 0.7, 0.418, 0.35 and 0.269 nm, indicating that these samples contain kaolinite and goethite and hematite. In addition, the 1–TT sample showed weak peak at 1.4 nm, indicating that it contain small amount of non–expandable 2:1 layer silicates. The weak peak at

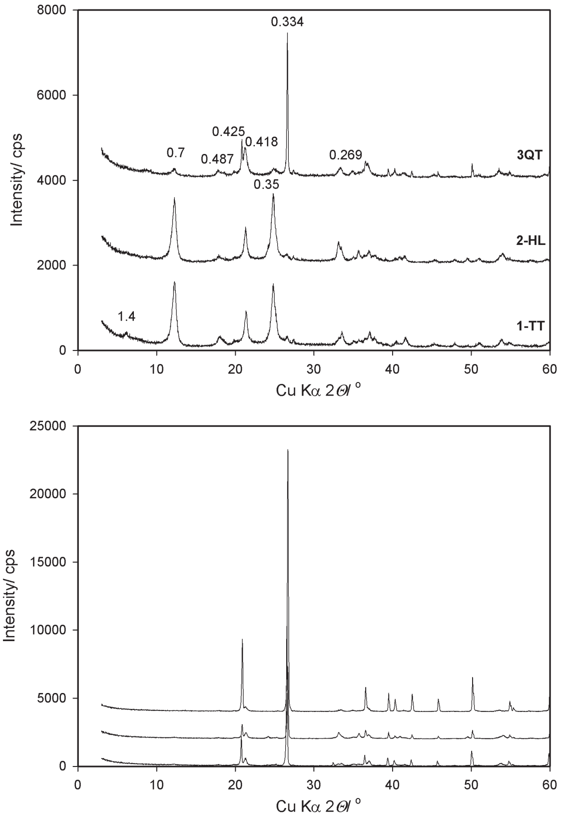

Fig. 2. X–ray diffraction pattern of laterite samples: Clay fraction (top) and Fine sand fraction bottom.

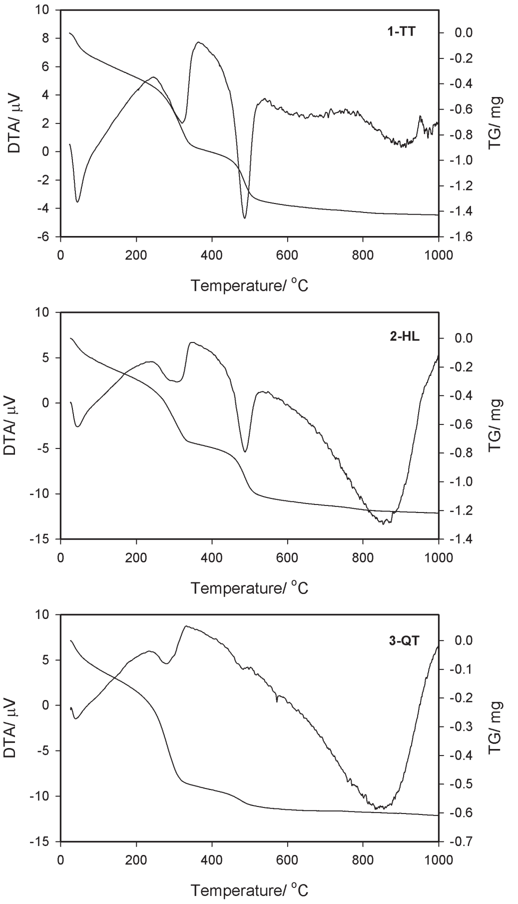

Fig. 3. Results of differential thermal analysis and thermogravimetric analysis.

0.487 nm indicates that all the samples contain gibbsite. The 3–QT sample showed a significantly different mineralogy in that it is dominated by quartz and contained much less amount of kaolinite but it contained goethite and hematite. The dominance of 0.425 and 0.334 nm peaks in the X–ray diffraction patters for fine sand samples indicate that the major mineral in the fine sand fraction is quartz. The small but distinct peak at 0.418 and 2.69 nm shows that the fine sand fractions also contain goethite and hematite. The silt and coarse sand fractions exhibited similar X–ray patterns.

The results of thermal analyses are shown in Fig. 3. All the samples showed clear endothermic peaks centered at around 50, 320°C and corresponding weight losses. The 1–TT and 2–HL samples exhibited an additional large endothermic peak at 480°C but the 3–QT sample showed only a faint peak at that temperature. The end thermic peak at 50°C obviously came from desorption of adsorbed water. And the peaks at 320 and 480°C are assigned to dehydration reaction of hydroxide minerals and kaolinite, respectively. Since goethite dehydrates at lower temperature than gibbsite, the endothermic peak centered at around 320°C suggests that the contribution of gibbsite was larger. These results harmonize with X–ray diffraction patterns shown in Fig. 1, particularly in that the 3–QT sample showed very weak diffraction peak at 0.7 nm. The 2–HL and 3–QT samples showed large endothermic peaks at around 840°C but they did not accompany weight loss. The origin of these endothermic peaks was not identified.

The dehydration reaction of gibbsite and kaolinite are expressed as

$$2Al(OH)_3 = Al_2O_3 + 3H_2O \tag{1}$$

and

$$Si_4Al_4O_{10}(OH)_8 = SiO_2 . Al_2O_3 + 4H_2O \tag{2}$$

The weight loss from 200 to 400°C was about 0.5 mg for all three samples. If this came from the dehydration of gibbsite, the stoichiometry in the reaction (1) gives the gibbsite weight of 1.4 mg in 10 mg sample. Similarly, the weight of kaolinite in is estimated to be 1.4 mg for the 1–TT and 2–HL samples and 0.71 mg for the 3–QT sample.

Basic chemical properties are summarized in Table 2. The pH (H_2O) was in a range from 5.3 to 5.6 and the pH (KCl) was similar, indicating that the samples contained little exchangeable Al. The 1–TT sample showed the highest clay content of 67% followed by the 2–HL and 3–QT samples. The clay content of the 3–QT sample was only 30% and it had fairly high sand content. Although the clay content was high, the effective cation exchange capacity, i. e. the sum of exchangeable cations, was very low. This is a reflection of the virtual lack in 2:1 type layer

Table 2. Some physical and chemical properties of laterite samples

No	pH_{H2O}	pH_{kcl}	TOC (%)	Clay (%)	Silt (%)	Fine sand (%)	Coarse sand (%)	ECEC ($cmol_c$/ kg)
1–TT	5.30	5.03	0.18	67.42	6.29	7.71	18.58	1.36
2–HL	5.56	4.95	0.13	57.03	6.24	9.89	26.83	1.00
3–QT	5.35	5.18	0.13	30.93	8.04	24.65	36.38	0.77

Table 3. Approximate mineralogical composition of laterite samples

Sample	Fraction	Major minerals
1–TT	Clay	Gibbiste, Kaolinite, Goethite, Hematite, Vermiculite
	Silt	Quartz, Kaolinite, Goethite, Hematite
	Fine sand	Quazt; Geothite, Hematite
	Coarse sand	Quartz, Goethite, Hematite
2–HL	Clay	Gibbsite, Kaolinite, Goethite, Hematite
	Silt	Quartz, Goethite, Hematite
	Fine sand	Quartz, Goethite, Hematite
	Coarse sand	Quartz, Goethite, Hematite
3–QT	Clay	Gibbiste, Goethite, Hematite, Quartz, Kaolinite
	Silt	Quazt, Goethite, Hematite
	Fine sand	Quazt, Goethite, Hematite
	Coarse sand	Quatz, Goethite, Hematiet

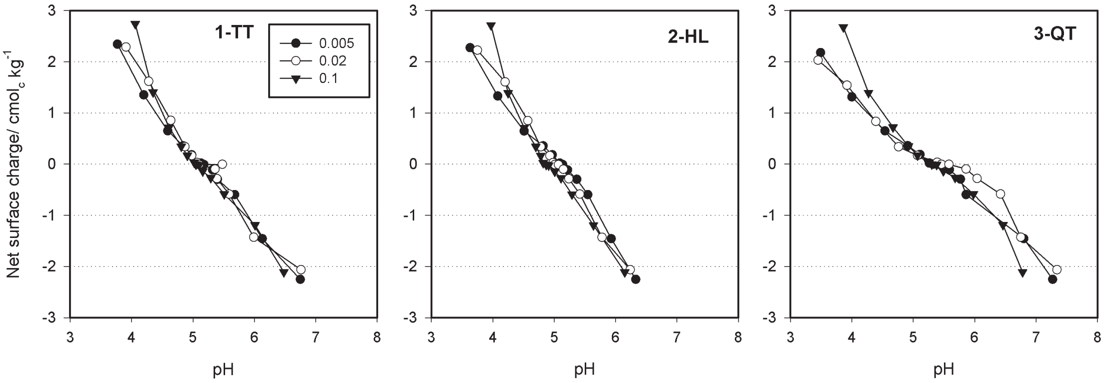

Fig. 4. Surface charge characteristics of laterite samples.

silicate minerals as indicated by X–ray diffraction (Fig. 2).

Fig. 4 shows the surface charge characteristics of laterite samples used in the present study, where the net surface charge was plotted against solution pH. The net charge decreased as pH increased and the sign of the net charge reversed at pHs 5.1, 5.0 and 5.4 for the 1–TT, 2–HL and 3–QT samples, respectively. These pH values, i. e. the point of zero net charge (PZNC) were not much different from pH values measured in water (Table 2). Since PZNC for goethite and gibbsite lie around pH 8, the observed lower PZNC values may be due to the presence of kaolinite and quartz.

Based on the mineralogical and chemical data, the approximate mineralogical composition of the laterite samples is summarized in Table 3.

Mineralogy and colloidal properties of clay suspensions

The X–ray diffraction patterns of the soil clay samples used for the flocculation tests are shown in Fig. 5. The strong peak at 0.7 and 0.35 nm in the T5 sample indicate that kaolinite predominates. The broad at 1.7–1.9 nm and 1.0 nm in the F sample show that it contains smectite and micaseous minerals. The approximate contents of layer silicate minerals and quartz that were estimated by the procedure described by Mosslehuddin (1998), Nguyen (2008) and Wada and Greenland (1970) were already reported by Nga *et al.* (2012a).

Properties of the clay suspensions used for the flocculation tests were summarized in Table 4. The suspension pH ranged from 6.81 to 6.86. The electric conductivity was low and not much different among samples. The T5 sample had the smallest average particle diameter of 86 nm, while the kaolinite sample had the largest average diameter of 384 nm. The ECEC of the suspended clays was in a large range from 3.7 to 99 cmol$_c$/kg. The fraction of Na was the largest among the exchangeable cations because NaOH was added for pH adjustment (Nga *et al.*, 2012a).

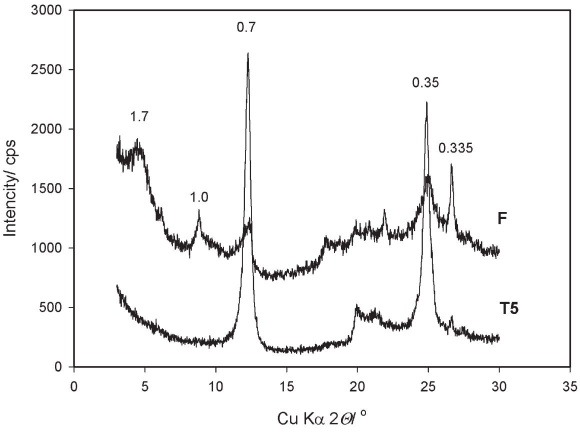

Fig. 5. X–ray diffraction patterns of soil clay samples used for flocculation tests.

Flocculation tests

The fluffy flocks formed when the flocculants were added and clay suspensions were vigorously stirred at a speed of 300 rpm. During the continued stirring at 50 rpm, the flocks grew in size and they loosely deposited on the bottom of the beaker after 30 min of the standing time. A 10 mL portion of the supernatant was sampled from a depth of 2 cm from the surface and its absorbance at 660 nm was measured. The results of the flocculation experiments are graphically presented in Fig. 6, where the averaged relative absorbance at three replications was plotted against the flocculant dose. In general, the absorbance decreased as the flocculant dose increased and then increased again past optimum dose. The re–increase in absorbance is probably due to the presence of the excess flocculent and/or re–dispersed clay particles (Nga *et al.*, 2012a). This observation was also in good agreement with the results by Nga *et al.* (2012b) on the kaolinite removal from water by volcanic and laterite materials. The montmorillonite suspension showed different response when the 3–QT slurry was added in that the absorbance exceeded that of the original montmorillonite suspension by about 20% (Fig. 6).

The lowest relative absorbance of about 0.01 indicates that 99% of the suspended clay particles were removed from the suspension. Considering the pHs of the clay suspensions and the PZNC the mutual flocculation of the added laterite and clay particles were due to the charge neutralization. The suspension pHs during flocculation experiment were close to those of the original clay suspensions, i. e. around 6.8, because the volume of the added flocculants remained mostly $< 10\%$ of the volume of the clay suspension. Since the PZNCs are around 5, the net surface charge is negative at round pH 6.8. This is seemingly contradictory to the charge neutralization. But it is not unreasonable to expect the mechanism because gibbsite and goethite would have had positive surface charge even at pH 7. Follett (1965) proved that electrostatic attraction force was dominated in retention of amorphous and colloidal ferric hydroxides

Table 4. Colloidal properties of clay samples

Samples	pH	EC/ dS m^{-1}	Exchangeable cations/cmol$_c$kg^{-1}				Average particle size/nm	Average zeta potential/mV
			Na$^+$	K$^+$	Ca^{+2}	Mg^{2+}		
T5	6.82	0.02	3.35	0.40	1.68	0.72	86	–45.1
F	6.81	0.03	4.95	2.11	17.22	5.53	187	–36.6
Montmorillonite	6.86	0.04	77.41	1.96	14.83	4.82	224	–55.7
Kaolinite	6.86	0.04	2.75	0.21	0.40	0.31	384	16.7

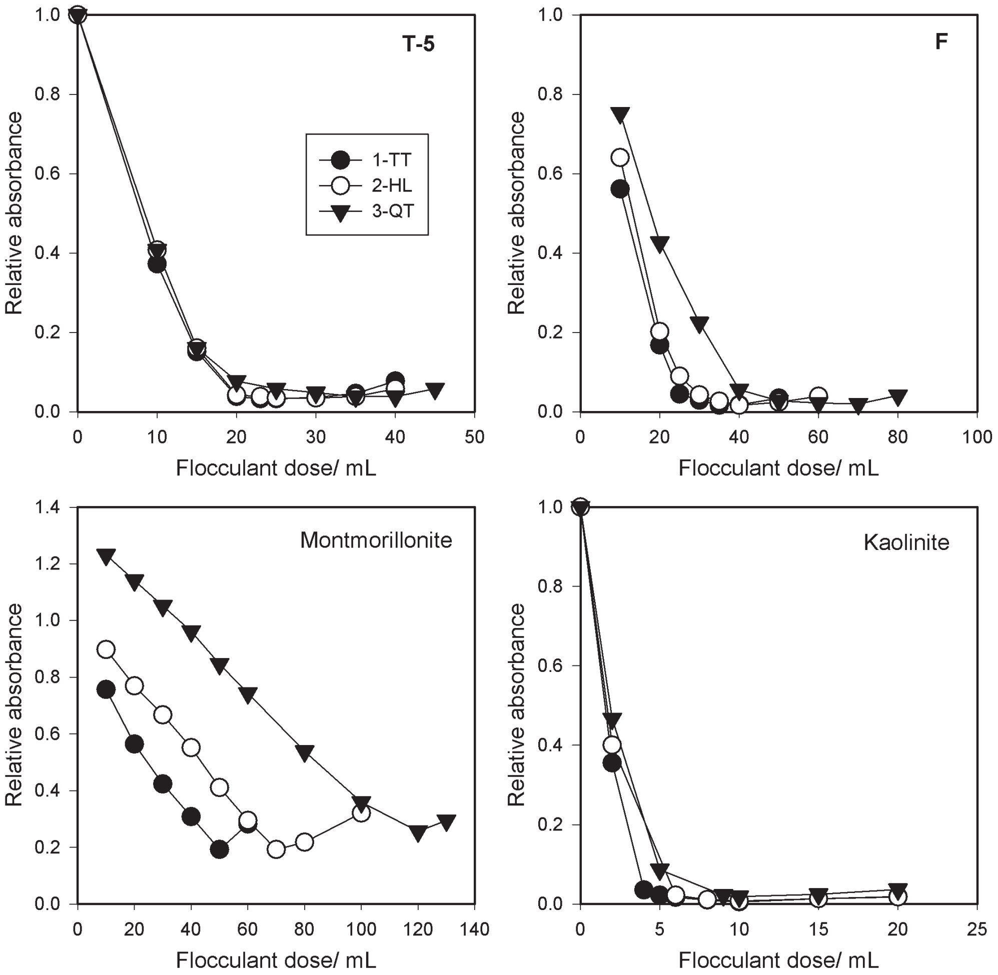

Fig.6. Results of flocculation tests showing the relative absorbance of the suspension after flocculant addition and flocculant dose.

by kaolinite. He also concluded that no bonding rearrangement occurred upon sorption of ferric colloids onto kaolinite. Weiss and Russow (1963) also indicated a similar association behavior of positively charged AgI particles by clay particles.

The results of the flocculation tests indicated that the amount of flocculant required to achieve the lowest absorbance was different for clay suspensions. The respective optimum dose of the flocculant made from 1–TT, 2–HL and 3–QT were 50, 70 and 120 ml for montmorillonite, 35, 40 and 60 ml for the F sample, 20, 25 and 35 ml for T5 sample and 5, 6, and 9 ml for kaolinite. Nga *et al.* (2012a) reported that the mineralogical characteristics of clay suspensions affected the flocculation efficiency. According their experimental results, suspensions containing smectite required larger flocculant dose (Nga *et al.*, 2012a). The present results are in accordance with theirs in that the T5 sample and kaolinite having low ECEC needed small amount of flocculants whereas montmorillonite and the F clay sample needed much more flocculant dose. Another factor for the very high flocculation efficiency for kaolinitic suspensions is that kaolinite has PZNC of about 3.0 as interpreted by Nga *et al.* (2012b) and Horikawa (1976).

The flocculation performance of the three laterite flocculants was obviously different. The 1–TT sample gave the highest flocculation efficiency followed by the 2–HL sample, and 3–QT and this trend was common to the results from all four clay suspensions. The trend was marked for montmorillonite (Fig. 6). The gibbsite and goethite content of the 3–QT laterite seems comparable with those of other laterite samples (Figs. 2 and 3). The surface charge characteristics for the three laterite samples were also similar (Fig. 4). One possible reason for the observed lower flocculation efficiency is the presence of a significant amount of clay sized quartz as indicated by the X–ray diffraction pattern in Fig. 2. The fine quartz particles may have left in the clay suspensions without interacting with negatively charged clay particles. This may explain the relative absorbance > 1 observed for montmorillonite (Fig. 6).

CONCLUSIONS

The flocculants made from laterites in Viet Nam are able to remove suspended clay particles via charge neutralization mechanism. The flocculation efficiency significantly depends on mineralogy and surface charge characteristics of both clay suspensions and laterite flocculants. Iron oxide minerals, e. g., geothite and hematite in the laterite are considered to be the factors contributing to flocculation.

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