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James Jihoon Kang
The University of Texas Rio Grande Valley, jihoon.kang@utrgv.edu

Jacob D. Wiseman

McKhenzy A. Welch

Richard A. McLaughlin

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CHEMICAL FLOCCULATION FOR REMOVING BENTONITE SPILLS IN WATER

Jihoon Kang[1]*, Jacob D. Wiseman[2], McKhenzy A. Welch[3], and Richard A. McLaughlin[4]

[1] School of Earth, Environmental and Marine Sciences, University of Texas Rio Grande Valley, Edinburg, Texas 78539, USA

[2]Wildlands Engineering, Inc., Asheville, North Carolina, 28806, USA

[3]Terracon Consultants, Inc., Charlotte, North Carolina, 28206, USA

[4]Department of Soil Science, North Carolina State University, Raleigh, North Carolina 27695, USA

ABSTRACT

A potential environmental impact associated with horizontal directional drilling is the inadvertent return of bentonite-based drilling fluid to the surface via naturally occurring fractures or fissures. This study investigated a range of flocculants consisting of water-soluble linear polyacrylamides (PAMs) differing in charge (anionic, neutral, and cationic), biopolymer (chitosan) and gypsum for treating the bentonite suspension that might release with runoff or into stream water. Laboratory jar test were conducted with a 0.4 % (*w*/*v*) bentonite suspension having an average initial turbidity of 1,217 nephelometric turbidity units (NTU). None of the PAMs by themselves were effective in flocculating bentonite suspension (> 690 NTU), but adding gypsum in a combination with anionic PAM enhanced the flocculation reaction (< 120 NTU). The biopolymer performed better than the PAMs alone by lowering turbidity to 437 NTU. A simple, passive dosing system was tested in which bentonite-laden turbid water was pumped into a length of pipe with a jute lining treated with the chemical and passed through a geotextile dewatering bag. Both the biopolymer and the combination of gypsum and anionic PAM were effective, reducing turbidity by 86-95 % relative to influent bentonite suspension (1,188 NTU). These results suggest that simple, inexpensive flocculation systems can be deployed to reduce turbidity caused by bentonite spills.

Keywords: Bentonite; Biopolymer; Gypsum; Inadvertent return; Polyacrylamide; Turbidity

INTRODUCTION

Horizontal directional drilling (HDD) is a steerable, trenchless method of installing underground pipelines and it is considered to be a less intrusive method than the traditional trenching for crossing a watercourse or wetland (Keykha et al. 2011). The HDD offers minimal surface disturbance because construction is limited to the established entry and exit sites for drilling equipment, which can be located outside of existing utilities and structures. However, an inadvertent return of drilling fluids to the surface ("frac-out" release) is a potential concern when the HDD is used under environmentally sensitive areas (Bennett and Wallin 2008; Osbak et al. 2011).

The HDD process involves a drilling mud primarily composed of bentonite clay and water (Caenn et al. 2011). When mixed with water, clay platelets in bentonite shift apart and attract water to the negative face, adsorbing 7 to 10 times their weight in water and swelling up to 18 times their dry volume (Pˇrikryl 2006). The specific surface area of fully dispersed bentonite slurry increases up to 600 to 800 m2 $^{g-1}$, which causes the drilling fluid to become viscous and thixotropic (Cater et al. 1986; Sparks 2003). Frac-out releases are typically caused by the pressurization of the borehole beyond the containment capability of the near-surface geologic materials and drilling fluids from the borehole can migrate through existing fissures or weak areas to the surface. In most cases, a frac-out tends to take the path of least resistance and surface in driveways, gardens and utility poles (Allouche 2001). If it occurs near or in a stream, a large volume of bentonite released can smother benthic invertebrates, aquatic plants, fish and their eggs similar to the negative impacts of sedimentation and turbidity (Newcombe and MacDonald 1991; Newcombe 2003; Cott et al. 2015). Proper contingency planning is critical for an effective response to the frac-out release (Allouche 2001; Murray et al. 2013)

One potential method to treat bentonite suspension inadvertently released or collected in mud bits is to employ a flocculation approach. Practical treatment systems have been developed for controlling turbidity in construction site storm water, including a corrugated pipe lined with flocculant (Bhardwaj and McLaughlin 2008) and a drainage channel with a series of fiber check dams coated with flocculant prior to entering into a sediment basin or a filtering device such as geotextile dewatering bag (McLaughlin et al. 2009; Kang et al. 2014a, b; Kang and McLaughlin 2016a). Solid forms of polyacrylamide (granule, block) or biopolymers (granule or flakes filled in porous pouches) have been used for treating the turbid storm water in agricultural fields and construction sites (Sojka et al. 2007; Kang et al. 2014a,b,c; Kang and McLaughlin 2016a). The overall objective of this study was to find cost-effective flocculation methods for treating bentonite suspension. Specific objectives were to 1) evaluate various types of PAM products and a biopolymer in laboratory jar test and 2) evaluate dosing turbid water with solid flocculants in a simple treatment system consisting of a corrugated pipe with jute lining followed by a geotextile dewatering bag.

Material and Methods

Laboratory jar test

A powder form of bentonite (Wyo-Ben, Inc., Billings, MT, USA) was mixed with tap water overnight using a magnetic stirrer. The tap water had a pH of 7.6 with electrical conductivity at 310 µS cm^{-1} (Table 1). Initially bentonite suspensions were prepared at concentrations of 1 %, 2.5 %, and 5 % (*w/v*). Our preliminary tests found that ≥ 1 % bentonite suspensions were highly viscous and essentially a paste, resulting in limited liquid: solid separation of 10 % or less by volume. Thus, we limited our jar tests to0.4 % bentonite suspension. The bentonite suspension at 0.4 % resulted in an average turbidity of 1,217 nephelometric turbidity units (NTU) on average, such as might occur if concentrated bentonite suspension was diluted with runoff or stream water.

Table 1. pH and electrical conductivity (EC) of 0.4 % (*w/v*) bentonite suspensions prepared in tap water (jar test) and well water (pumped water test).

	Laboratory jar test		Pumped water test	
Property	Tap water	Bentonite suspension in tap water	Well water	Bentonite suspension in well water
pH	7.6	9.5	6.0	7.4
EC (µS cm^{-1})	310	490	155	382

Laboratory jar tests included three different PAMs and a biopolymer product. The three PAMs were high molecular weight PAMs but differing in charge: anionic PAM (APS 705, Applied Polymer Systems, Inc., Woodstock, GA, USA), neutral PAM (N300, NALCO Industries, Chicago, IL, USA) and cationic PAM with 50 % of charge density (C9909, NALCO Industries, Chicago, IL, USA). The APS 705 contains a proprietary mixture of medium- and high-molecular weight anionic PAM and it has been found to be effective in flocculating suspended sediments in North Carolina (NC), USA (McLaughlin and Bartholomew, 2007; Kang et al., 2016b). It is certified by North Carolina Department of Environment and Natural Resources (NCDENR) for stormwater treatment. The biopolymer was Dual Polymer System (HaloSource, Inc., Bothell, WA; referred to as "DPS" hereafter) that is also certified by the NCDENR for storm water treatment. The DPS consists of two agents, a charging agent (HaloKlear DBP-2100) and chitosan flocculant (HaloKlear GelFloc), and we used these two agents at equal ratio (1:1).The charging agent is formulated from proprietary natural biopolymers and is reported to form highly stable, strong bonds with chitosan (HaloKlear 2016). Once the optimal PAM type and its dose were determined, another set of jar tests was performed in a combination with gypsum ($CaSO_4 \cdot 2H_2O$). The gypsum was included in this study since the presence of Ca^{2+} in the water is known to shrink the electrical double layer surrounding soil particles and help bridge the anionic surfaces of soil particles and negatively charged PAM molecules, enhancing flocculation reaction (Sojka et al. 2007; Lee et al. 2010).

For turbidity measurement on bentonite suspensions, a sample was shaken for 10 s and the turbidity reading was taken after 30s of settling using an Analite NEP 260 turbidity probe (McVan Instruments, Melbourne, Australia). The 30 s waiting time was used to reduce variations produced by the turbulence in the sample bottle after shaking and this has been employed as a standardized procedure in previous studies (McCaleb and McLaughlin 2008; Kang et al. 2013; Kang et al. 2014a,b,c). Measured turbidity readings were corrected using a standard curve generated with formazin solutions of known turbidity.

Pumped water test with bentonite suspension

The pumped water test was conducted at the Sediment and Erosion Control Research and Education Facility (SECREF) in Raleigh, NC, USA. A 3.6-m long, corrugated drainage pipe (25 cm in diameter) cut in half was installed on a supporting frame and lined with jute netting to retain the flocculants (Fig. 1). The jute netting had a mesh-spacing of approximately 2.5 cm × 2.5 cm with 60 % open area and its weight was 0.35 kg m^{-2} (Green Resource Inc., Clayton, NC, USA). The netting has been used as a medium for granular PAM dosing in construction site drainage channels as the PAM sticks to the fibers but dissolves slowly into flowing water(McLaughlin et al. 2009; Kang et al. 2014a,b). A 60-cm long plastic funnel was installed to direct the water leaving the pipe into a geotextilede watering bag (60 cm × 60 cm) constructed for this study using the same material used for commercial dewatering bags (Dirtbag™, ACF Environmental Inc., Richmond, VA, USA). The bag was fastened with a built-in strap at the bottom of plastic funnel. The technical specifications of the dewatering bag are presented in Table 2 (ACF Environmental Inc. 2016).

Fig. 1 Experimental setup to treat bentonite suspension. The bentonite suspension (0.4 % *w*/*v*) was in the black tub at the top of the hill and was pumped into the half-cut pipe lined with jute netting, which was then funneled into a geotextile dewatering bag.

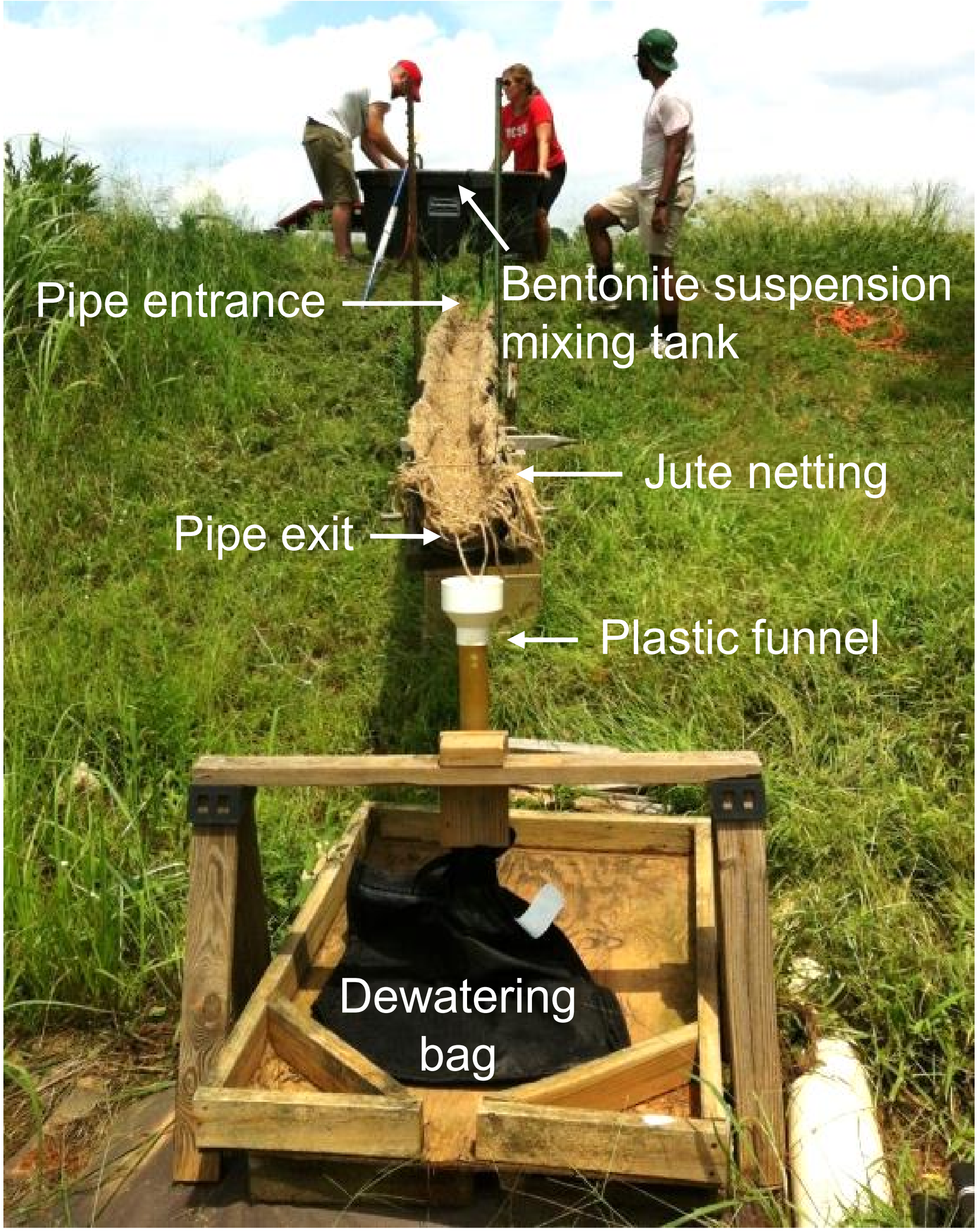


Table 2. Specifications of geotextile dewatering bag used in this study (adapted from ACF Environmental Inc., 2016).

Property	Test method[a]	Result
Weight (oz yd^{-1})	ASTM D-3776	8
Grab tensile strength (lbs)	ASTM D-4632	205
Mullen burst (psi)	ASTM D-3786	350
Static puncture strength (lbs)	ASTM D-4833	110
UV resistance (%)	ASTM D-4355	70
Flow rate (gal min^{-1} ft^{-2})	ASTM D-4491	110
Apparent opening size retained (%)	ASTM D-4751	80

[a] Standard method from the American Society of the International Association for Testing and Materials (ASTM).

A total of three treatments were tested: 1) control with no flocculant, 2) DPS consisting of a charging agent sock (DBP-2100) followed by a chitosan sock (GelFloc), and 3) dry gypsum (1.3 kg) + anionic PAM (100 g) applied on jute netting. Treatment 2) and 3) were the most effective in removing turbidity in the jar testing. The charging agent sock in the DPS was deployed in the upstream of chitosan sock, enabling to form stable strong bonds with the chitosan socks at the downstream (HaloKlear 2016). Each sock was 1.8 m in length and segmented in eight pouches. For treatment 3, a commercial gypsum (SoftCal Pellets Soil Conditioner, Austinville Limestone Co., Austinville, VA, USA) and granular anionic PAM were applied to jute netting pre-wetted to enhance PAM retention (“stickiness”). The mass ratio between gypsum and anionic PAM was determined from the optimal doses in the jar tests. The gypsum was a synthetic form derived from flue gas treatment of coal-burning power plants and had a dark color in appearance.

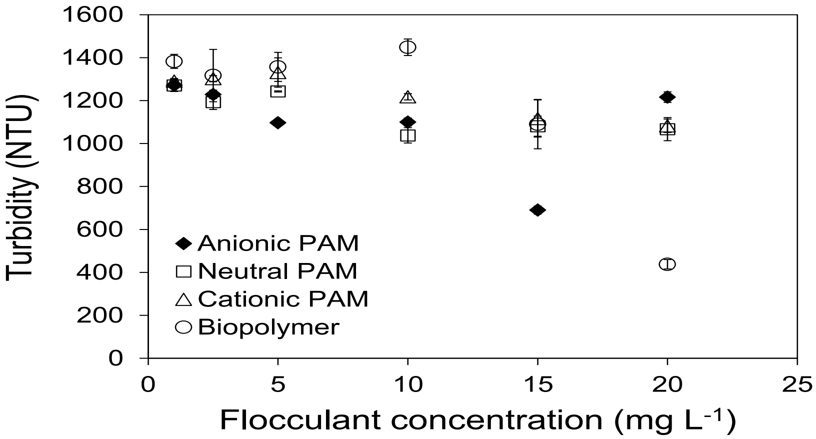
For each test, 322 L of bentonite suspension at 0.4 % of solid content were prepared with local well water (Table 1; Fig. 1). The suspension was pumped into the pipe entrance using a sump pump approximately at 15 L min^{-1}. Each test ran for 7.6 min and water samples were collected every 30 s until 5-min mark and one last sample at 7.6 min at the pipe entrance, pipe exit, and dewatering bag exit. All samples were analyzed for turbidity following the identical procedures used in the jar tests. We recorded clogging time of the dewatering bag for each test. Clogging was considered to occur once the flow backed up 58 cm up in the tube, equivalent to metric measurement for 0.8 pound-per-inch (psi).

Results and Discussion

Laboratory jar test

The flocculants were not particularly effective in reducing bentonite turbidity compared to our experience with turbidity generated from soils (Fig. 2). Anionic PAM and biopolymer were the top two, but needed higher dose (15-20 mg /L) than those for typical soil suspensions (≤ 1 mg/L) (McLaughlin and Bartholomew2007) and the turbidity was much higher (> 400 NTU)than typical discharge standards of 50 NTU or less. Several factors may play role in making bentonite difficult to flocculate compared to normal sediment suspensions. Commercial bentonite mainly consists of smectite (usually > 60%) and other impurities such as quartz, feldspar, and mica (Permien and Lagaly 1995). Smectite isa group in the broad family of 2:1 (silica: aluminum) clay minerals and has a high cation exchange capacity (CEC, 80-150 cmol kg^{-1}) due to substantial isomorphic substitutions in the octahedral layers (permanent charge) and the presence of fully expanded inter layers that promote exchange of ions (Sparks 2003). The swelling nature of bentonite is due to the presence of unbalanced charges and CEC produced by sodium (Na)-based bentonite. Expanding layer silicates such as smectite has extensive internal and external surface, having more than 20-times greater specific surface area (600-800 m^2 g^{-1}) than that of non-expanding layer silicate such as kaolinite (7-30 m^2 g^{-1}) (Sparks 2003). Deng et al. (2006) compared the relative adsorption of PAM with three different clay minerals (smectite, illite, and kaolinite) and found that the adsorption of PAM on clay minerals was correlated to the surface areas of the clay. Their study indicated that flocculation was likely to be less favorable for the clays with larger surface areas (e.g., smectite). McLaughlin and Bartholomew (2007) also found that kaolinitic clays were more readily flocculated by anionic PAM than smectitic clays.

Fig. 2 Turbidity of bentonite suspension as a function of flocculant concentration. Error bars indicate the standard error of three replicates.



Soil stabilization with gypsum has been studied to improve the engineering properties of expansive soils (Nalbantoglu and Gucbilmez 2001). Basic mechanism is based on the gypsum-clay reaction in that excess Ca from gypsum replace monovalent cations in the clay and change the electrical charge density around the clay particles (Sojka et al. 2007). Replacement of monovalent Na^+ by Ca^{2+} may lead to a reduction in diffused double layer thickness leading to decrease in liquid limit, plasticity, and swelling pressure of the expansive soils (Ameta et al. 2007). In our study, gypsum alone did not reduce turbidity in the bentonite suspensions (data not shown). However, the highest doses of gypsum enhanced turbidity reduction by anionic PAM when the PAM was dosed at 15 mg/L (Fig. 3). Figure 4 shows the nature of bentonite suspensions with and without gypsum + anionic PAM treatment after 1-h settling.

Fig. 3 Turbidity of bentonite suspensions as a function of gypsum concentration in the presence of anionic PAM solution. Error bars indicate the standard error of three replicates.

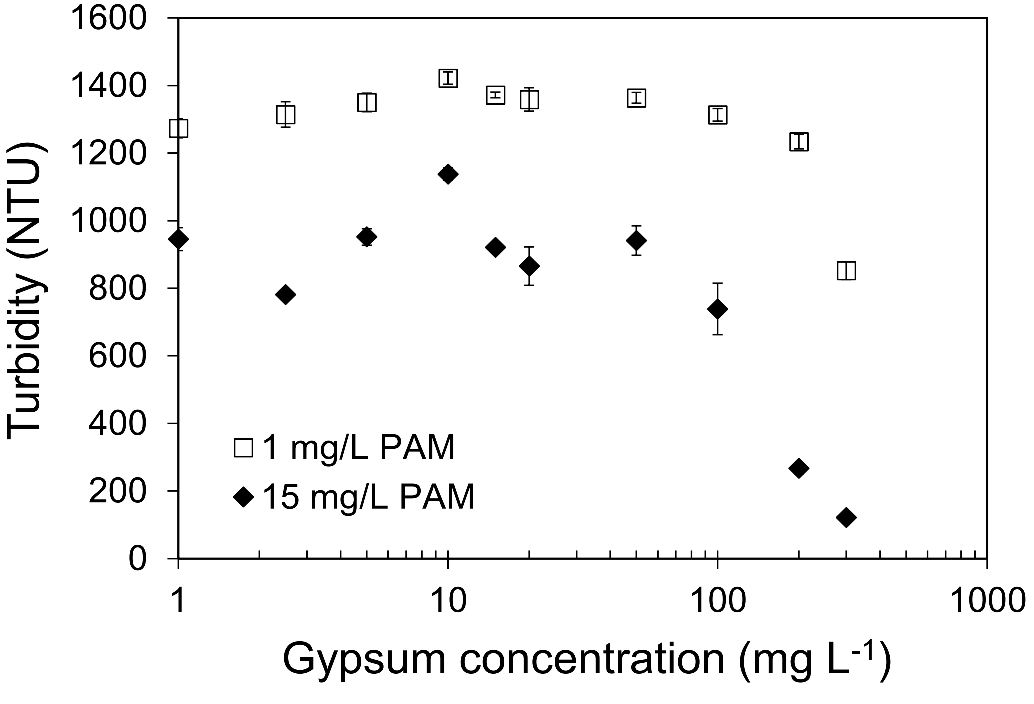
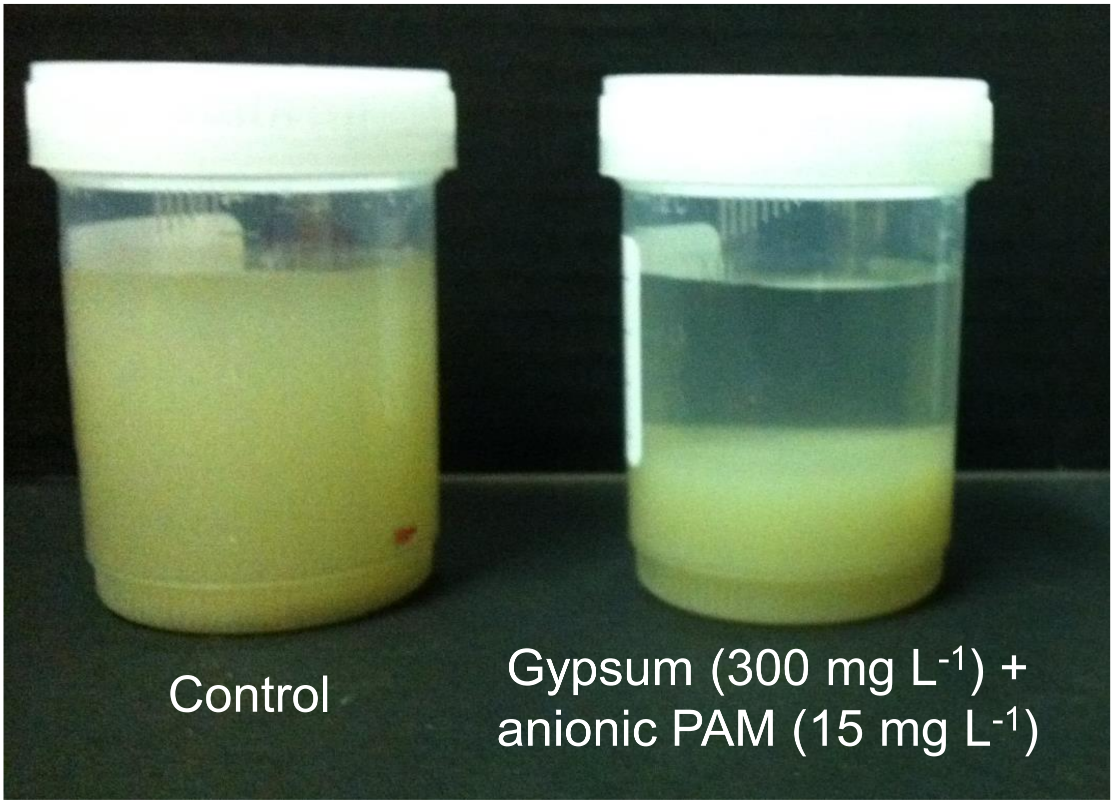


Fig. 4 Bentonite suspension (0.4 % *w*/*v*) separated by gypsum + anionic PAM treatment after 1-h settling.



Pumped water test with bentonite suspension

Mean turbidity in the entrance samples (influent turbidity) was 1,188 ± 15 (mean ± standard error) when averaged across three tests. With no chemical treatment in the system, some turbidity reduction was evident initially as the influent bentonite suspension flowed along in the jute netting and dewatering bag (Fig. 5). However, turbidity at all sample points converged and became similar level to influent after 5 min. Both flocculant treatments were effective in reducing turbidity at dewatering bag exit while DPS showed a sequential turbidity reduction by sampling locations (i.e., entrance > pipe exit > dewatering bag) (Fig. 6). This result indicated that dewatering system like a geotextile bag is critical when biopolymer product is used as a passive dosing method. The gypsum + anionic PAM treatment maintained very low turbidity at pipe exit (< 40 NTU), suggesting that it was very effective in forming flocs with bentonite at pipe exit. The gypsum material entered into sediment bag, however, appeared to increase turbidity by continuously releasing its color, thus increasing the exit turbidity (Fig. 6; Fig. 7). The laboratory jar tests involved a reagent-grade gypsum which had no color.

Fig. 5 Turbidity of bentonite suspensions at three different sampling locations with control treatment (no flocculant added).

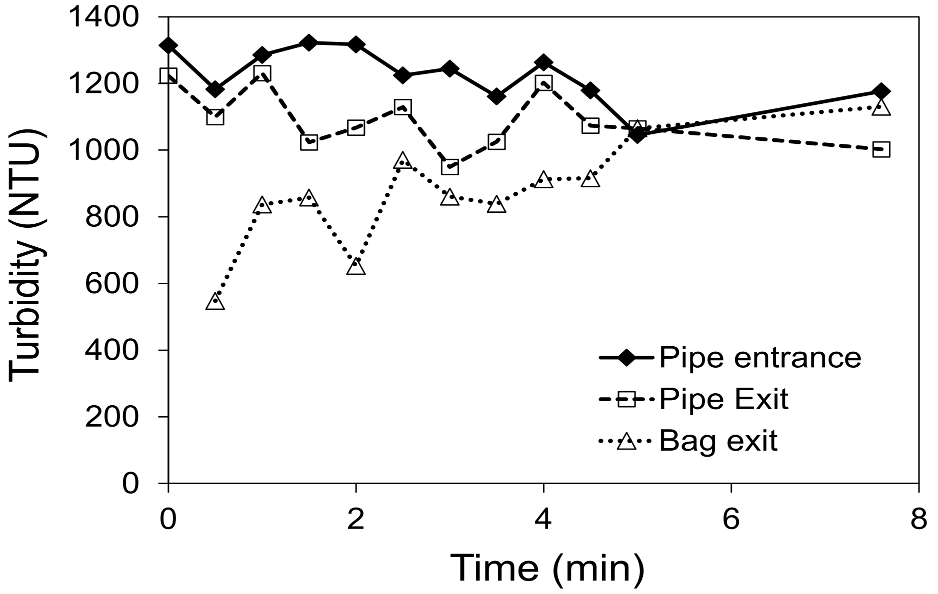
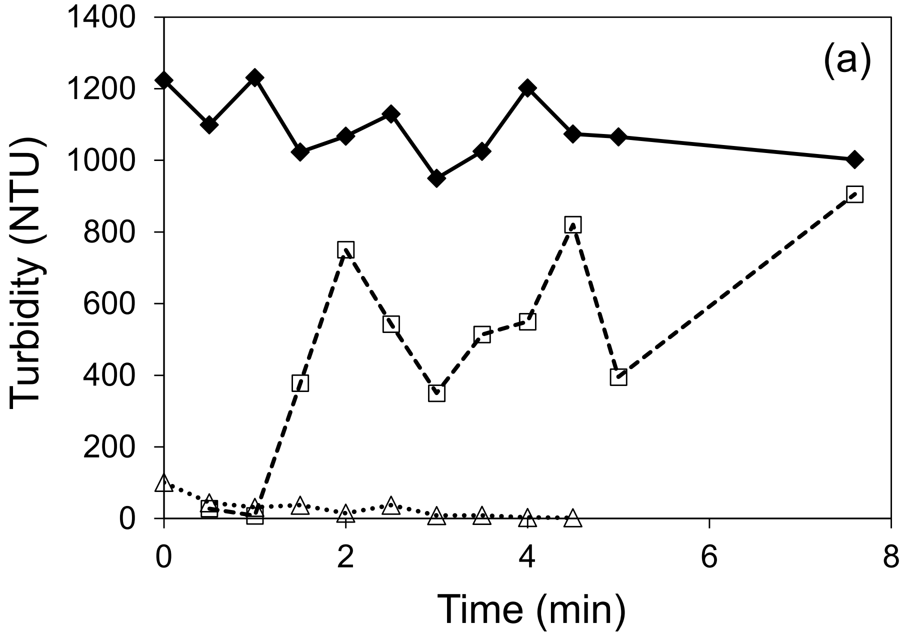


Fig. 6 Turbidity of bentonite suspension affected by flocculant treatments: (a) pipe exit samples and (b) sediment bag exit samples.



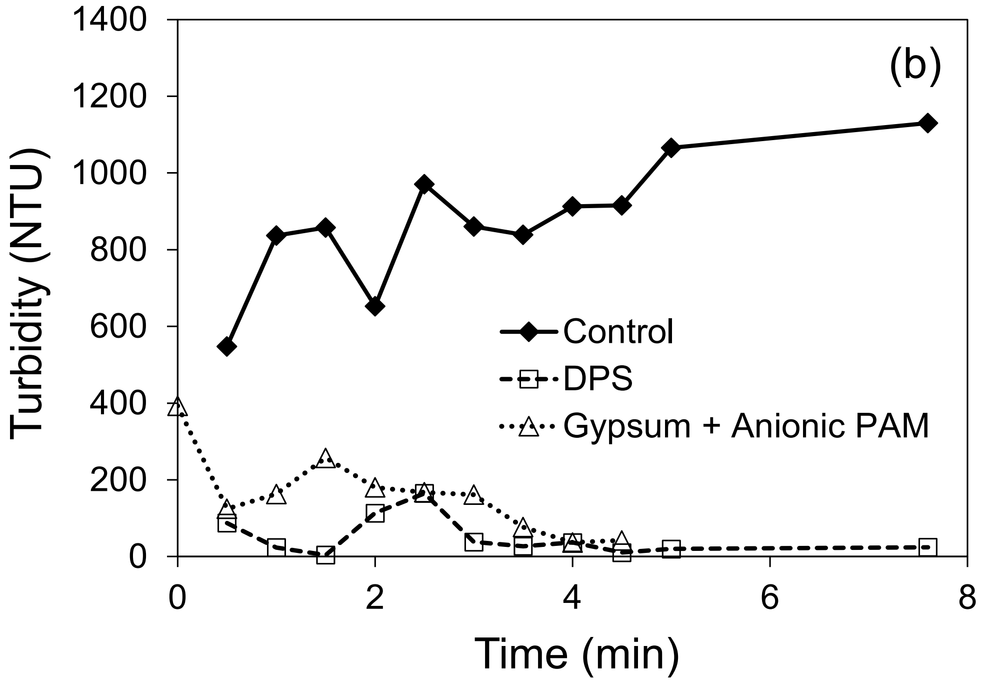


Figure 7 shows the appearance of the inside of sediment bags after tests. For control treatment, there was minimal bentonite captured, indicating poor filtration capacity of the dewatering bag against fine bentonite suspensions. There was no serious clogging observed in the dewatering bag from control treatment while DPS and gypsum + anionic PAM began to show clogging at 7 and 3.5 min, respectively. When comparing DPS vs. gypsum + anionic PAM, bentonite flocs from DPS looked relatively compact and dense, indicating they may have been able to pass water through the cake on the inside of the bag compared to the gypsum-PAM flocs.

Fig. 7 Inside of dewatering bags after pumped water tests.

CONCLUSIONS

Bentonite concentrations ≥ 1 % (*w*/*v*) had very little settling regardless of chemical treatment. More diluted bentonite (0.4%) was poorly flocculated by PAMs alone, but adding gypsum in a combination with anionic PAM reduced turbidity by 90% in the jar test. The DPS, a commercial biopolymer product alone, performed relatively well in flocculating the bentonite suspension. Our pumped water test confirmed that either biopolymer or gypsum + anionic PAM could be an effective passive dosing method when combined with physical dewatering system in treating diluted bentonite suspension which might arise from runoff or fugitive bentonite discharges into streams.

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