DOKUZ EYLÜL UNIVERSITY
GRADUTE SCHOOL OF NATURAL AND APPLIED SCIENCES

SWELLING AND SETTLING BEHAVIOR OF BENTONITES IN DIFFERENT PORE FLUIDS

by
Esra DİKİŞCİ

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İZMİR

SWELLING AND SETTLING BEHAVIOR OF BENTONITES IN DIFFERENT PORE FLUIDS

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We have read the thesis entitled **"SWELLING AND SETTLING BEHAVIOR OF BENTONITES IN DIFFERENT PORE FLUIDS"** completed by **ESRA DİKİŞÇİ** under supervision of **ASSOC. PROF. DR. A. HAKAN ÖREN** and we certify that in our opinion it is fully adequate, in scope and in quality, as a thesis for the degree of Master of Science.

Assoc. Prof. Dr. A.Hakan ÖREN

Supervisor

Prof. Dr. Yeliz Yükselen AKSOY

(Jury Member)

Prof. Dr. Mehmet Murat MONKUL

(Jury Member)

Prof.Dr. Kadriye ERTEKİN

Director

Graduate School of Natural and Applied Sciences

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SWELLING AND SETTLING BEHAVIOR OF BENTONITES IN DIFFERENT PORE FLUIDS

ABSTRACT

In this study, swell index tests were performed using 14 bentonites that have different properties and it was aimed to find out pore medium chemistry which can give similar swell index as in deionized water (DIW). For this purpose, swell index tests were conducted in NaCl and $CaCl_2$ concentrations ranging between 5 mM to 30 mM. At the end of these test, swell indices were found quite similar in NaCl solutions and 5 mM $CaCl_2$ with respect to those obtained with DIW. On the other hand, a slight reduction was observed in the swell indices with respect to DIW when 10 mM $CaCl_2$ solution was used.

As a result of sedimentation tests conducted after swell index tests, three different sedimentation behaviors were observed: Diffused Particle Settling (or Accumulation Settling, AS), Flocculation Settling (FS) and Mixed Settling (MS). It was also observed that the settling behavior of bentonites exhibiting AS type of settling in DIW changed to FS when the pore medium chemistry was changed. Since the FS behavior was observed for most bentonites in 10 mM $CaCl_2$ concentration, it was selected as the critical salt solution.

The swell index and sedimentation characteristics/final sediment volume (FSV) of bentonites were related with some physico-chemical properties of bentonites such as liquid limit, cation exchange capacity and specific surface area. Either observational or quantitative relationships, liquid limit, rather than the others, has fair correlation with the swell index and sedimentation characteristics/FSV. Increase in the swell index led to increase the liquid limit. The relationship with sedimentation was evaluated in two ways. Bentonites mostly exhibited AS type of settling in DIW and mostly FS type of settling in 10 mM $CaCl_2$ concentration. Thus, liquid limit decreased as the FSV increased in AS, whereas liquid limit increased as the FSV increased in FS.

The swell indices and sedimentation behaviors/FSVs were also related with the hydraulic conductivity. For this time, an opposite behavior of liquid limit was obtained. Hydraulic conductivity decreased as the swell index of bentonites increased. The relationship established with the sedimentation was done in two ways as well. Hydraulic conductivity increased when FSV increased in AS, whereas it decreased as the FSV increased in FS.

Keywords: Bentonite, geosynthetic clay liners, physico-chemical properties, sedimentation test, settling, swell index

FARKLI BOŞLUK SIVILARINDA BENTONİTİN ŞİŞME VE ÇÖKELME DAVRANIŞI

ÖZ

Bu çalışmada farklı özelliklere sahip 14 adet bentonit kullanılarak şişme indisi deneyleri yapılmış ve saf su ile benzer şişme indisi değeri veren ortam kimyasının bulunması amaçlanmıştır. Bu sebeple, 5 mM ile 30 mM arasında değişen NaCl ve $CaCl_2$ konsantrasyonlarında şişme indisi deneyleri yapılmıştır. Bu deneyler sonucunda, çalışmada kullanılan tüm NaCl çözeltileri ve 5 mM $CaCl_2$ ile yapılan deneylerden elde edilen şişme indisi değerleri saf su ile yapılanlar ile oldukça benzer bulunmuştur. Diğer yandan 10 mM $CaCl_2$ çözeltisi kullanılarak yapılan deneylerden elde edilen şişme indislerinde saf suya kıyasla az miktarda düşüş gözlemlenmiştir.

Şişme indisi deneylerini takiben yapılan çökelme deneyleri sonucunda ayrışık serbest çökelme (ASÇ), topaklanmış serbest çökelme (TSÇ) ve ayrışık+topaklanmış serbest çökelme (ATSÇ) olmak üzere 3 farklı çökelme davranışı gözlemlenmiştir. Saf suda ayrışık serbest çökelme davranışı sergileyen bentonitlerin, değişen ortam kimyası ile TSÇ'ye doğru evrildiği görülmüştür. Bentonitlerin birçoğu 10 mM $CaCl_2$ çözeltisinde TSÇ davranışı gösterdiği için bu konsantrasyon kritik konsantrasyon olarak belirlenmiştir.

Bentonitlerin şişme indisi ve çökelme davranışları/nihai çökelek hacmi bentonitlerin likit limit, katyon değişim kapasitesi ve özgül yüzey alanı gibi fiziko-kimyasal özellikleri ile ilişkilendirilmiştir. Hem gözlemsel hem de nicel olarak likit limitin, diğer parametrelere kıyasla, şişme indisi ve nihai çökelme davranışı/çökelek hacmiyle ilişkisi vasattır. Bentonitlerin şişme indisi artarken likit limitleri artmıştır. Çökelme ile ilişkilendirme iki şekilde ele alınmıştır. DS'de ekseriyetle ASÇ tipinde çökelme davranışı sergileyen bentonitler, 10 mM $CaCl_2$ çözeltisinde TSÇ şeklinde çökelmişlerdir. Dolayısıyla AS tipi çökelmede nihai çökelek hacmi arttıkça likit limit azalırken; TSÇ tipi çökelmede nihai çökelek hacmi arttıkça likit limit de artmıştır.

Şişme indisi ve çökelme davranışı/nihai çökelek hacmi ayrıca hidrolik iletkenlikle de ilişkilendirilmiştir. Bu kez likit limitle elde edilen ilişkinin tersi davranış elde edilmiştir. Bentonitlerin şişme indisi artarken hidrolik iletkenlikleri azalmıştır. Çökelme ile ilişkilendirme yine iki şekilde yapılmıştır. AS tipi çökelmede nihai çökelek hacmi arttıkça hidrolik iletkenlik artarken; nihai çökelme hacmi arttıkça hidrolik iletkenlik azalmıştır.

Anahtar kelimeler: Bentonit, geosentetik kil örtüler, fiziko-kimyasal özellikler, çökelme deneyi, çökme, şişme indisi

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CHAPTER ONE
INTRODUCTION

1.1 Background

Bentonites are materials that composed of different minerals. The main mineral in bentonites is montmorillonite. Bentonites can swell around ten times of their dry volumes when they are exposed to water (Jo, Katsumi, Benson & Edil, 2001; Kolstad, Benson & Edil, 2004). Thus, there are little pore spaces existed between the clay particles which prevent movement of the mobile water and provides low hydraulic conductivity for bentonite. Because of this capability, bentonite is placed between geotextiles to provide impermeability to the geosynthetic clay covers (GCLs) and therefore, a composite material having low hydraulic conductivity is obtained (Kong, Wu, Chai & Arulrajah, 2017).

GCLs are used in solid waste landfills, mine lagoons, tunnels and many other areas to isolate contaminants from the environment or to keep away intrusion of water to the structures (Petrov, Rowe & Quigle, 1997; Ruhl & Daniel, 1997; Shackelford, Benson, Katsumi, Edil & Lin, 2000). Since GCLs have low hydraulic conductivity, they are used as the hydraulic barrier for the landfill cover system as an alternative to compacted clay liners. Therefore, determination of the hydraulic conductivity has an important place for the GCLs, but long testing time is required for this purpose.

Swell index test is the most common test that gives the estimation of hydraulic conductivity in a rapid way. Swell index indicates the swelling ability of bentonites when DIW is used as the hydrating liquid (Scalia, Benson & Finnegan, 2019). Although the swell index test is a widely used parameter throughout the world, some difficulties can exist during its determination. For instance; some of the swollen bentonite particles may still remain suspended on the water surface without settling during 10 minutes waiting time or until addition of another 0.1 g sample. Then, these suspended particles together with 0.1 g added bentonites form aggregate and exhibit rapid settling which may cause air bubbles between the aggregates. The availability of air bubbles between the aggregates results in greater swell index value than its actual value. Moreover, uneven swelling of particles at the end of the test generally lead rough surface which allow inaccurate reading of swell index value as well. Thus, sedimentation test, an alternative test method, was proposed herein.

The proposed method can be used either in place or in combination of swell index. After performing the swell index, the sedimentation test can be run without using new sample. This test can be performed by shaking the swollen sample after recording the swell index value and allowed free settling of particles in the graduated cylinder. The sedimentation test is simple, cheap and rapid test method which can give additional information about the settling characteristics of bentonites. Settling behavior of bentonite can also give some information about the hydraulic conductivity of bentonites.

1.2 Scope of this study

The main objective of this study is to investigate the use of sedimentation test as an alternative test method for the interpretation of some engineering properties of bentonites. After recording the swell volume at the end of 24 hours in swell index test, graduated cylinder can be shaken slightly and then let the particles settle down freely. Hence, sedimentation behavior of the particles can be observed and eventually the final sediment volume can be recorded. The principal aim of this study is to obtain a critical salt solution which gives a similar swell index value as in deionized water (DIW), but particles should exhibit flocculation type of settling which is called as “flocculation sedimentation (FS)”.

There are different types of settling regimes like flocculation sedimentation (FS), accumulation sedimentation (AS) and mixed sedimentation (MS) which is the mixture of both settling types (Imai, 1980; Kaya, Ören & Yükselen 2006; Sridharan & Prakash, 2001). Although FS is a rapid type of settling, MS and AS may last long depending on the dispersed particles which settle slowly. Thus, the target settling type in this study was selected as FS because of its rapid settling nature. Since most bentonites served AS type of settling in DIW, pore medium chemistry was changed using salt solutions with monovalent and divalent cations to obtain FS. Weak concentrations (up to 30 mM) were used in this study, because it was desired to achieve similar swell indices in the salt solutions with respect to those in DIW.

To show the efficiency of sedimentation test method over swell index, a quantitative and observational relationships were established between the swell index/sedimentation characteristic/FSV and physico-chemical/hydraulic properties of bentonites.

1.3 Outline of the thesis

This thesis includes 6 chapters. Chapter One gives information about general overview and the objective of this thesis.

Chapter Two gives information about some material properties of the bentonites and experimental procedures according to ASTM standards.

Chapter Three compares the swell indices of bentonites obtained in different pore fluids such as DIW; 10, 20, 30 mM NaCl and 5 and 10 mM $CaCl_2$ solutions. This chapter also interprets the swell index results with respect to ionic strength (I) and Relative Abundance of Monovalent and Divalent Cations (RMD).

Chapter Four describes how sedimentation characteristics change depending on the pore fluid chemistry. Sedimentation was separated into two groups namely sedimentation behavior and final sediment volume (FSV). The results were evaluated based on these considerations.

Chapter Five describes the relationships between swell index and sedimentation characteristics/FSVs of bentonites and some properties of bentonites such as consistency limits, cation exchange capacity, specific surface area and hydraulic conductivity. The relationships were established as observational when sedimentation characteristics was considered and quantitative when FSV was taken into consideration.

Chapter Six includes summary and conclusions based on the results obtained from this study.

CHAPTER TWO
MATERIALS AND METHODS

2.1 Materials

Total of 14 bentonites were used in this study five of which had been taken from GCLs manufactured by the local producer and the rest had been gathered from the companies that are available in the local market in Turkey. These GCLs were produced placing the bentonite between woven and non-woven geotextiles by needle punching. Some bentonites were rich of sodium, some of polymer-treated and some were in their natural state and not subjected to any treatment.

Swell index tests and sedimentation tests were performed in 100 mL graduated cylinders and using deionized water (DIW), sodium chloride (NaCl) solution having 10, 20, 30 mM concentration and calcium chloride ($CaCl_2$) solution with 5, 10 mM. DIW was obtained from Mill-Q Gradient purifier water unit. Solutions were prepared by dissolving Merck brand NaCl and $CaCl_2$ salts in water. All solutions were prepared in 1 liter flasks and kept in room temperature throughout the test duration.

2.2 Methods

2.2.1 Index Properties

Grain size distribution of bentonites were determined by wet sieving method (ASTM:D422-63, 2007). Liquid limit tests were performed using Casagrande and Fall Cone test apparatus and plastic limit tests were done by manually rounding as described in ASTM:D4318-05 (2005). In order to provide homogeneous distribution of water throughout the bentonite particles, liquid and plastic limit tests were carried out after keeping the bentonites in plastic bags for 24 hours.

Specific gravity tests were determined by following the ASTM:D854-14 (2014). Due to high water adsorption and swelling tendency of bentonite, specific gravity tests were conducted on 25 g of samples. Deaeration process was applied using a vacuum pump for approximately 3 days until entrapped air between bentonite particles was completely taken.

The liquid limits of bentonites were 149-552% and plastic limits ranged from 34-60%. The specific gravity of these bentonites are between 2.64 and 2.70. Index properties of bentonites are summarized in Table 2.1. Note that Table 2.1 presents the original name of products such as YP-1, 1, 2, etc. In this study, however, the bentonites were categorized as GCL-1-5 and B-1-9.

Table 2.1 Index properties of bentonites

Bentonites	Name of Products	Specific Gravity	Liquid Limit (%)		Plastic Limit (%)
			Fall Cone	Casagrande	
GCL-1	YP-1	2.77	325	385	58
GCL-2	1	2.7	282	400	60
GCL-3	2	2.7	-	-	-
GCL-4	Ca-GCL	2.64	215	240	53
GCL-5	MLTY	2.67	258	280	36
B-1	Çanbensan-949	2.71	298	417	44
B-2	SC-2-362	-	120	128	53
B-3	Karakaya	2.69	388	529	38
B-4	SC-Ham Bentonit	-	117	121	-
B-5	SC-8033	-	96	100	57
B-6	Çanbensan-948	2.76	305	397	34
B-7	Eczacıbaşı-act	2.67	450	552	41
B-8	Eczacıbaşı-nonact	2.72	141	149	42
B-9	SC-14957	2.76	189	283	48

Table 2.2 Physico-chemical properties of bentonites

Bentonites	Swell Index (mL/2g)	Liquid Limit (%)	Plastic Limit (%)	SSA (m^2/g)	CEC (cmol/kg)
GCL-1	21	385	58	-	81
GCL-2	23	400	60	-	-
GCL-3	23	-	-	-	-
GCL-4	21	240	53	-	73
GCL-5	21	280	36	-	-
B-1	26	417	44	612	69
B-2	15	128	53	-	-
B-3	17	529	38	637	73
B-4	7	121	-	-	-
B-5	8	100	57	-	-
B-6	24	397	34	587	61
B-7	25	552	41	607	105
B-8	8	149	42	475	78
B-9	13	283	48	837	85

SSA: Specific surface area, CEC: Cation exchange capacity

2.2.2 Swell Index

Swell index tests were carried out using DIW and salt solutions having different molarities as described in ASTM:D5890-11 (2011). Bentonites collected from the GCLs were grinded using mortar to obtain powdered bentonites. Then, they were sieved from No. 200 sieve and oven-dried at 105 ° C. Since metal containers may pose corrosion, porcelain containers were used to eliminate the negative effect of corrosion on swell index. After drying, the graduated cylinder was initially filled with test liquid until 90 mL level. Then, 0.1 g of bentonite was weighed and poured in the cylinder over a period of approximately 30 seconds. After 10 minutes of waiting for bentonite swell, another 0.1 g of bentonite was poured into the graduated cylinder. This procedure was continued after all bentonite particles were poured. Subsequently, the liquid level in graduated cylinder was completed to 100 ml level. Top part of the graduated cylinder was closed with a parafilm to prevent evaporation. At the end of 2 hours of hydration, graduated cylinder was tilted with an angle of 45° to avoid inclined or irregular bentonite surface. Moreover, graduated cylinder was rotated back and forth slightly several times around its vertical direction. After 24 hours of hydration, the swell index value of bentonite was recorded. This test was replicated three times for each bentonite using the same test liquid.

2.2.3 Sedimentation Tests

The sedimentation tests were conducted immediately after swell index value had been recorded. The cylinder was vigorously shaken until it was ensured that there were no bentonite particles remained at the bottom of the cylinder. Once it was ensured, the shaking was continued a few more minutes. The suspension formed by shaking was allowed settling and sedimentation behavior was examined with time. Sedimentation volumes were recorded at the end of 1, 2, 5, 10, 15, 30, 60, 120, 240, 480 and 1440 minutes (sometimes with extended reading times). Sedimentation volume was recorded depending on time and plotted for each bentonite and salt solution. Since the swelling index experiments were repeated three times, the sedimentation tests were performed three times for each bentonite as well. The sedimentation behavior at varying concentrations of salt solutions and DI water was also observed during this study.

2.2.4 Chemical Analyses

The cation analyses of bentonites had been measured previously in the laboratory. These analysis had been conducted using 1 M ammonium acetate solutions as described in ASTM:D7503-10 (2010). Cation analysis had been conducted on the filtrate and PerkinElmer DV 2100 OPTIMA ICP-OES was used for this purpose. Then, bentonites were further washed with isopropanol and 1M potassium chloride to measure the cation exchange capacities (CECs). The filtrates were transferred to spectrophotometer and the nitrogen concentrations were determined (ASTM:D7503-10, 2010). The exchangeable mole fractions of bound cations and CECs are presented in Table 2.2.

The specific surface areas (SSAs) of bentonites were measured using methylene blue and filter paper. The details of about this test can be found in (Ören, Yükselen Aksoy, Önal & Demirkıran, 2018; Yukselen Aksoy & Kaya, 2010). The SSAs are also summarized in Table 2.2. Some values are not given in Table 2.2. Because chemical analyses were not carried out for all bentonites.

Table 2.3 Exchangeable cations, cation exchange capacities (CEC) and specific surface areas (SSA) of bentonites

Bentonites	Name of Products	Exchangeable Cations (%)				SSA (m^2/g)	CEC (cmol/kg)
		Na^+	K^+	Ca^{2+}	Mg^{2+}		
GCL-1	YP-1	72	2	19	8	-	81
GCL-2	1	-	-	-	-	-	-
GCL-3	2	-	-	-	-	-	-
GCL-4	Ca-GCL	70	4	23	3	-	73
GCL-5	MLTY	-	-			-	-
B-1	Çanbensan-949	55	3	30	12	612	69
B-2	SC-2-362	-	-	-	-	-	-
B-3	Karakaya	77	5	17	2	637	73
B-4	SC-Ham Bentonit	-	-	-	-	-	-
B-5	SC-8033	-	-	-	-	-	-
B-6	Çanbensan-948	59	2	27	11	587	61
B-7	Eczacıbaşı-act	71	6	15	8	607	105
B-8	Eczacıbaşı-nonact	7	7	41	44	475	78
B-9	SC-14957	77	1	14	8	837	85

2.2.5 Hydraulic Conductivity Test

There was no hydraulic conductivity test carried out in this study. However, an observational comparison was made between the hydraulic conductivities previously determined and sedimentation characteristics of bentonites in this study. Hydraulic conductivity tests had been run on artificially prepared (AP) and original GCLs which were reported elsewhere (Ören & Akar, 2017; Ören et al., 2018). Artificial GCLs had been prepared by placing bentonite between non-woven and woven geotextiles without needle-punching. The hydraulic conductivities of bentonites had been measured by permeating DIW and using flexible wall permeameters. Other testing details can be found in Ören and Akar, 2017 and Ören et al., 2018.

CHAPTER THREE
SWELL INDICES OF BENTONITES

3.1 Background

Bentonite is a clay that contains high amounts of montmorillonite and has a high water adsorbtion capacity. Swelling occurs when bentonite contacts with water and results in a decrease in the hydraulic conductivity. Swell index is the most important parameter affecting the quality of bentonite (CETCO, 1994). The determination of this parameter is significant because the swelling behavior of bentonite directly governs the hydraulic conductivity of GCL.

Several studies have shown that solution type and concentration have an influence on the swelling of bentonite (Di Emidio, Mazzieri, Verastegui-Flores, Van Impe & Bezuijen, 2015; Katsumi, Ishimori, Onikata & Fukagawa, 2008; Lee & Shackelford, 2005; Liu, Bouazza, Gates & Rowe, 2015; Ören et al., 2018; Rowe & Abdelatty, 2012). For example, Katsumi et al. (2008) used DIW and salt solutions containing monovalent, divalent and trivalent cations to study the effect of cation valence, pH and concentration on the swell index and hydraulic conductivity of GCLs. They used salt solutions with concentrations ranging between 5 mM and 1000 mM. They pointed out that the greater the concentration the lower is the swell index. Moreover, the highest swell index value was obtained in solutions with monovalent cations and the lowest swell index value was obtained in solutions with trivalent cations.

Recently, different from other studies, Scalia et al. (2019) reported an alternate method to determine the swell indices of bentonites from GCLs. In that study, they investigated the impact of several factors which may influence the swell index. These factors were particle size of bentonite, time between addition increments (1, 2, and 10 min), time of addition during each increment (1, 10, 30 s) and mass input during each increment (1.0, 0.5, 0.2, 0.1 g). Based on 252 alternative analyses, fast alternative swell test (FAST) and multiple alternative swell test (MAST) procedures were proposed. They noticed that the maximum percent error of these new procedures with respect to the standard method was 25%. However, FAST and MAST is faster than the standard method.

3.2 Swell Index in DIW

In this study, swell index tests were performed using DIW at first. Each tests were repeated three times. The pictures of samples after swell index tests can be seen between Figure A-1 and Figure A-14. As seen from Table 3.1, lowest and largest swell index values were obtained as 7 (B-4) and 26 mL/2g (B-1) with DIW. The swell indices of other bentonites were within the range of 8-25 mL/2g. Finding different range of swell indices could be attributed to differences in physical and chemical properties of bentonites. In other words, the reason for greater swell indices in DIW can be attributed mainly to: (i) smectite contents of bentonites and (ii) the exchangeable cations present on bentonites.

Table 3.1 Swell indices of bentonites in different solutions

Bentonites	DIW	10 mM NaCl	20 mM NaCl	30 mM NaCl	5 mM $CaCl_2$	10 mM $CaCl_2$
GCL-1	21	23	23	21.5	20.5	20
GCL-2	23	23	21	19.5	20	18.5
GCL-3	23	23	21	20	21	17
GCL-4	21	21	24	30	21	19
GCL-5	21	21	20	18.5	18	15
B-1	26	25	24	22	22	19.5
B-2	15	15	14	12.5	13	9
B-3	17	31	35	36	29	31
B-4	7	7	8	8	-	6.5
B-5	8	8	8	8	7	6
B-6	24	25	23	21	21	18.5
B-7	25	25	31	30	25	22.5
B-8	8	9	10	10.5	9	8
B-9	13	17.5	16	14.5	13	12.5

3.3 Swell Index in NaCl Solutions

The purpose of this study is to find out a critical salt solution and concentration which gives similar swell index value as in DIW. Sridharan et al. (1990) performed a study which investigates the sedimentation of soils under different concentrations levels of NaCl solution. They noticed that %0.025 (4.25 mM) NaCl gives similar sediment volumes close those in DIW. Based on Shridharan et al.'s (1990) study, the use of salt solution with monovalent cation was decided in this study. Jo et al. (2001) reported that the swell indices of the GCL bentonites did not differ from that of DIW when NaCl solutions were within the range of 5-25 mM. Based on all these considerations, the range of NaCl concentrations were limited between 10 and 30 mM herein.

The pictures of samples after swell index tests in 10, 20, 30 mM NaCl solutions are given from Figure A-1 to Figure A-14. Similar swell index values were obtained with 10 mM and 20 mM NaCl solutions when comparing to those obtained with DIW (Table 3.1). When the swell index tests were conducted with 30 mM NaCl solutions, some values were obtained similar to that of DIW and some of them were slightly less than that of DIW. The swell indices decreased to 19.5 mL/2g for GCL-2, 20 mL/2g for GCL-3, 18.5 mL/2g for GCL-5, 22 mL/2g for B-1, 12.5 mL/2g for B-2, 21 mL/2g for B-6 at this concentration.

On the other hand, the swell indices for GCL-4 were greater than those of DIW, when the solution concentrations were 20 mM and 30 mM NaCl. The exchange complex of GCL-4 bentonite moderately occupied by Ca^{2+} ions (23%) (Ören et al., 2018). Different from GCL-4, swell indices of B-3, B-8 and B-9 increased even in the 10 mM NaCl solution. The calcium concentration in the exchange complex was 17%, 41% and 14% for these bentonites, respectively. Lee et al. (2012) obtained similar findings which is explained by cation exchange between Ca^{+2} and Na^{+} in solution. The swell index of Ca^{2+} bentonite was obtained higher in dilute concentrations of NaCl solutions. In contrast, the calcium concentration in the exchange complex of B-1 and B-6 were 27 and 30%, respectively. However, the swell indices slightly decreased from 26 to 22 mL/2g and from 24 to 21 mL/2g as the NaCl concentration increased from 0 to 30 mM, respectively. Despite considerable amount of calcium in the exchange complex, the reason for this reduction in the swell indices of these bentonites are unknown.

The comparison of swell indices obtained in DIW and NaCl solutions were drawn using 1:1 lines in each graph and shown in Figure 3.4 a-c. Figure 3.4a shows that the swell indices in DIW corresponds well with the swell indices in 10 mM NaCl solution. As explained above, when the concentration increased to 20 and 30 mM, some bentonites placed over 1:1 line, indicating the greater swell indices in 20 and 30 mM than those of DIW.

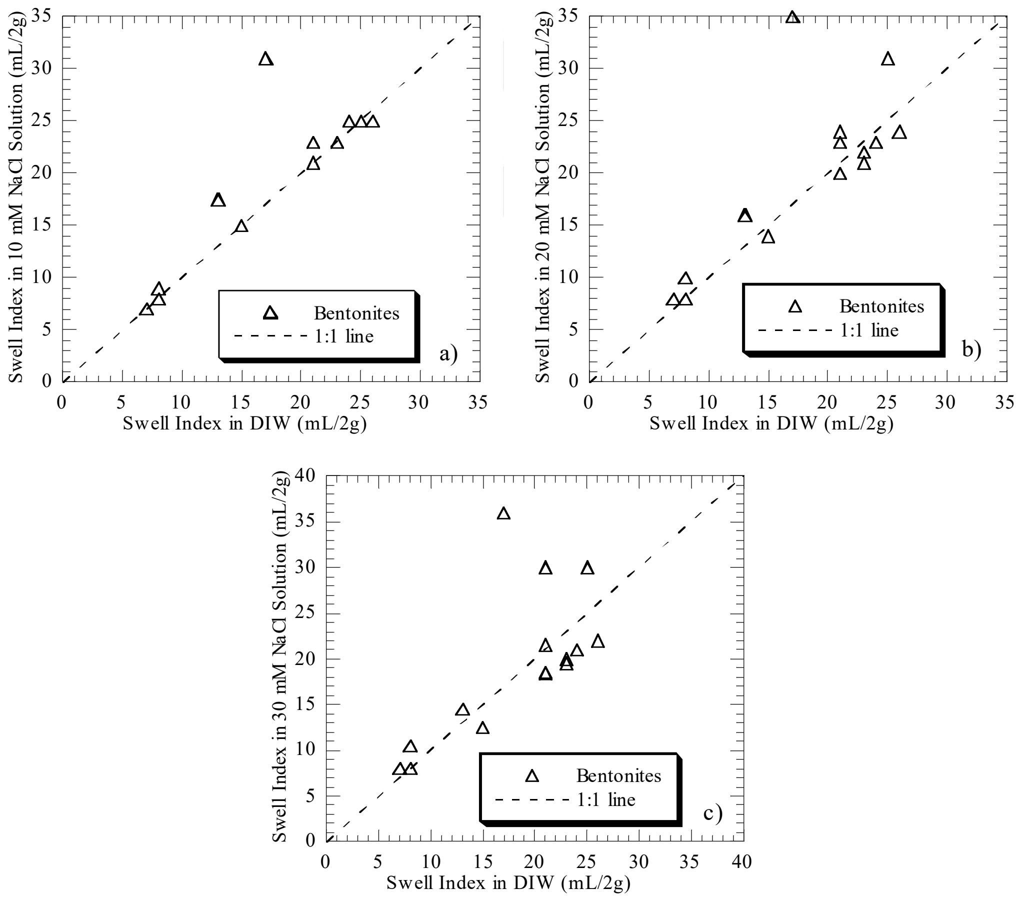

Figure 3.1 Comparison between the swell indices obtained from DIW and obtained from: a) 10 mM, b) 20 mM, c) 30 mM NaCl solutions

3.4 Swell Index in $CaCl_2$ Solutions

Similar to the previous section, it is aimed to find out a critical salt solution and concentration which gives similar swell index value as in DIW. Thus, the swell index tests were also replicated using salt solutions with divalent cations. For this purpose 5 and 10 mM $CaCl_2$ solution were used. The pictures of the samples in 5 and 10 mM $CaCl_2$ solutions are given in Appendix-A (Figure A-1-14). Swell indices were obtained in the range of 7-25 mL/2g at this concentration (Table 3.1). When these tests were performed in 5 mM $CaCl_2$ solution, the highest swell index value was obtained for B-7. On the other hand, the lowest swell index was obtained for B-5.

When the swell index tests were conducted with 5 mM $CaCl_2$ solution, most values were obtained similar to that of DIW and few of them were less than that of DIW. The swell indices remarkably decreased to 18 mL/2g for GCL-5, 22 mL/2g for B-1 and 21 mL/2g for B-6 at this concentration (Table 3.1). On the other hand, swell index values for almost all of the bentonites were obtained significantly less than those of DIW when 10 mM $CaCl_2$ solution was used (Table 3.1). Also some values were obtained similar to that of DIW at this concentration.

These reductions in swell index values may be related to chemical composition of bentonites. However, most of the bentonites that show a decrement trend with respect to DIW have no chemical analyses (Table 2.2). Thus, only B-1 and B-6 can be evaluated in view of exchangeable cations. Indeed, Table 2.2 presents that the Na^{+}-Ca^{2+} concentrations in B-1 and B-6 were determined as 55-30% and 59-27%, respectively. This shows that the Ca^{2+} concentration in the bentonites were significantly high which might have been further increased in 5 and 10 mM $CaCl_2$ solutions, resulting in a reduction in the swell indices of bentonites. This reduction is possibly due to the suppression of thickness of diffuse double layer surrounding the particles.

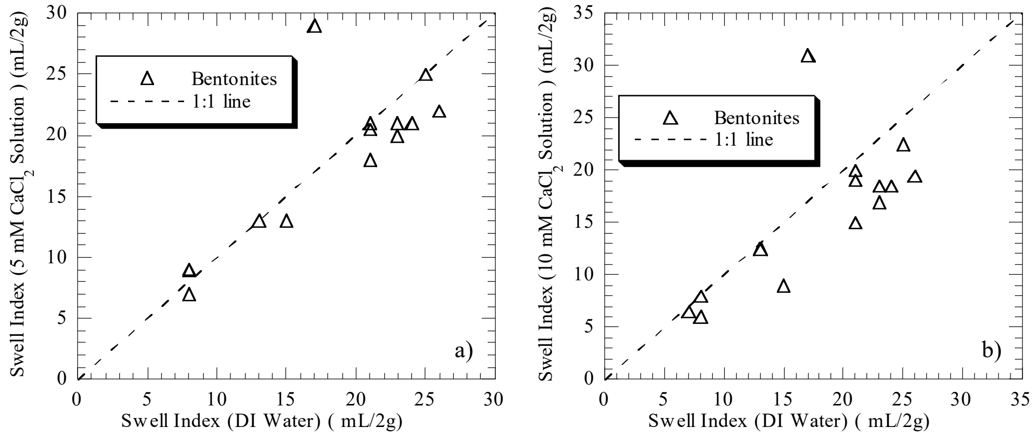

Figure 3.2 Comparison between the swell indices obtained from DIW and obtained from: a) 5 mM and b) 10 mM $CaCl_2$ solutions

The comparison of swell indices obtained in DIW and $CaCl_2$ solutions are shown in Figure 3.6 a-b. Figure 3.6a depicts that the swell indices in DIW corresponds fairly well with the swell indices in 5 mM $CaCl_2$ solution. As explained above, swell index values were obtained significantly decreased in 10 mM $CaCl_2$ concentration and therefore, most bentonites placed below the 1:1 line, indicating lower swell indices were obtained in 10 mM $CaCl_2$ than in DIW.

3.5 Interpreting the Swell Index with Ionic Strength and RMD

Swell index tests were performed using DIW, monovalent and divalent salt solutions with concentrations ranging from 5 mM to 30 mM as mentioned in the previous sections. A graphical comparison of the swell indices with monovalent and divalent salt solutions as a functions of ionic strength (I) and are shown in Figure 3.7a for GCLs and Figure 3.7b for bentonites. Ionic strengths of the solutions were calculated as in the following equation:

$$I = \frac{1}{2}\sum_{i=1}^{n} c_i\, z_i^2 \qquad (3.1)$$

where, c is concentration and z is valence of the ions present in the solution. Since only the influence of the cations on the swell index of bentonites have been investigated, ionic strengths were calculated by taking into consideration only the cation part of the solution. It means chloride (Cl^-) was not taken into account while calculating the ionic strength. Based on this equation, 10, 20 and 30 mM NaCl and 5 and 10 mM $CaCl_2$ correspond to 5, 10, 15, 10, 20 mM ionic strength, respectively. The swell indices of GCLs decreased as the I increased (Figure 3.7a). However, such kind of tendency was not seen when bentonites are considered (Figure 3.7b).

The Relative Abundance of Monovalent and Divalent Cations (RMD) of the solutions were also calculated as in the following:

$$RMD = \frac{M_M}{\sqrt{M_D}} \tag{3.2}$$

where, M_M is the total molarity of the monovalent cations and M_D is the total molarity of the polyvalent cations. Based on this equation, RMD in NaCl solutions are calculated as ∞, whereas it is 0 in $CaCl_2$ solutions. Generally, the higher the RMD, the higher is the swell index which, in turn, means that the monovalent cations are dominant the in the solution.

Figure 3.7a-b show that swell indices obtained in $CaCl_2$ solution prepared at ionic strength of 10 mM are less than those obtained in NaCl solution at the same ionic strength. The difference between the swell indices in NaCl and $CaCl_2$ solution was obtained approximately 2 mL for the GCLs and bentonites which is due to huge difference between the RMD values (∞ and 0). This shows that the presence of divalent cations in $CaCl_2$ suppressed the thickness of diffuse double layer surrounding the bentonite particles, resulting in a decrease in swell indices although both solutions had the same ionic strength (10 mM).

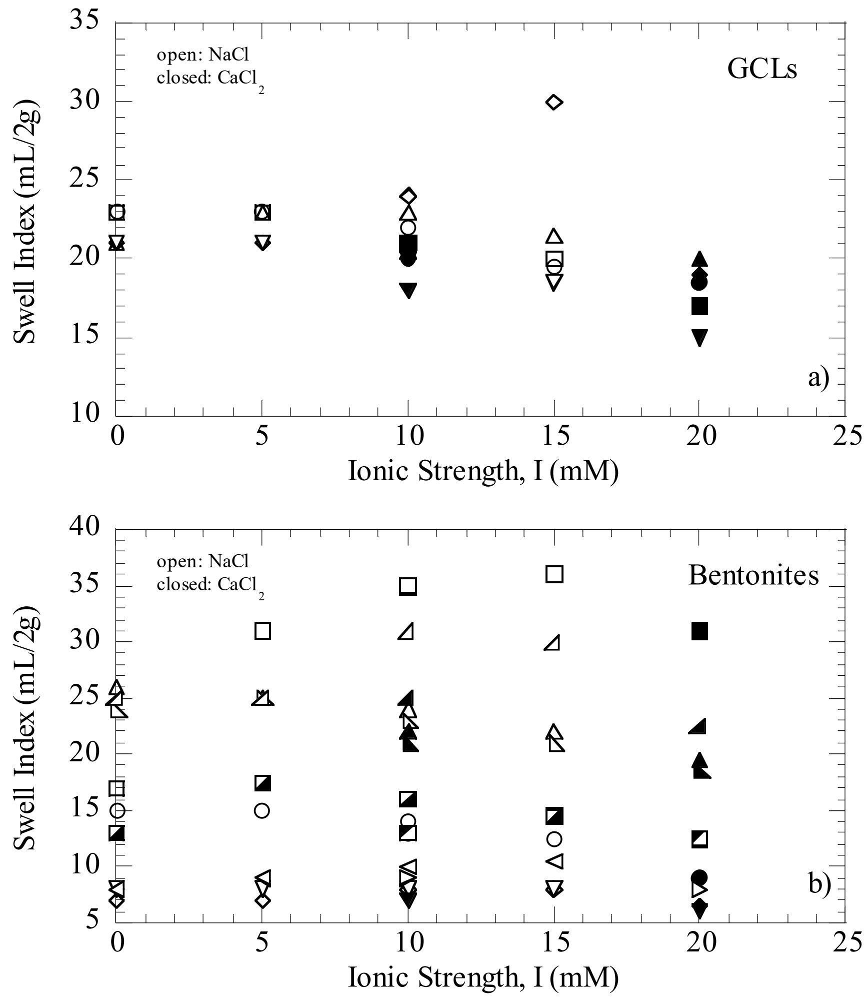

Figure 3.3 Evaluation of the swell indices as a function of ionic strength for: a) GCLs and b) bentonites

3.6 Conclusions

In this chapter swell index tests were conducted using DIW; 10, 20, 30 mM NaCl and 5 and 10 mM CaCl2 solutions. The aim was to find out a critical salt solution which gives similar swell index value as with DIW. The results were also evaluated in terms of ionic strength (I) and RMD. The results are summarized below:

- Swell indices of GCLs and bentonites were obtained similar to that of DIW when 10, 20, 30 mM NaCl and 5 mM $CaCl_2$ solutions were used.

- In the case of 10 mM $CaCl_2$ solution, however, the swell indices started to become lower than that had in DIW.

CHAPTER FOUR
SEDIMENTATION BEHAVIOR OF BENTONITES AFTER SWELL INDEX TEST

4.1 Background

The sedimentation behavior may change depending on the solid/liquid ratio, clay mineralogy and pore medium chemistry (solution type, concentration and pH). Sedimentation formation mechanism consist of four categories in the work done by Imai (1980) depending on the water content (or solid/liquid ratio (S/L)): (i) Dispersed free settling (observed at low S/L), (ii) flocculated free settling (observed at medium S/L), (iii) zone settling (consist of flocculation, settling and consolidation stages and observed at high S/L), and (iv) consolidation settling (observed at very high S/L).

Sridharan & Prakash (2001) examined the sedimentation behavior depending on clay minerology under the name of dispersed free settling and flocculated free settling by reducing the behaviors into two which have distinct characteristics from those proposed by (Imai, 1980).

Kaya et al. (2006) have suggested another type of settling as "mixed settling" in which the heavier particles settle immediately, and remaining particles have form flocs (or aggregates) to serve flocculation settling. This type of settling can be seen in the study of Sridharan & Prakash (2001) as well.

Sedimentation characteristics of any type of soil basically depend on two main factors such as solid to liquid ratio and pore medium chemistry. Imai (1980) showed how sedimentation characteristics change with the solid to liquid ratio and pore fluid chemistry of the medium. Increase in the concentration of the pore medium or solid to liquid ratio led to facilitate the flocculation type of settling. In addition to this, some factors like particles size, clay contents, and smectite content have an influence on the sedimentation characteristics as well. Sridharan & Prakash (1999, 2001) have suggested that the sedimentation under different physico-chemical environments are strongly influenced by the clay mineralogy of soils. Kaya et al. investigated (2006) the settling characteristics of kaolin clay under different concentration levels and pH conditions and reported that there is fair relationship within these factors.

Although several studies with sedimentation tests have been conducted so far, most of these studies had been applied on natural soils or clays (Sridharan et al., 1986, 1988; Sridharan & Prakash 1999; Ören, 2014). In this study, however, the sedimentation behavior of 2 g of bentonite was evaluated. Swell index test is conducted with 2 g of bentonite and immediately after recording the swell volume, sedimentation test can be carried out by vigorously shaken the graduated cylinder. Then, the settling regimes of the particle can be observed and finally the sediment volumes can be recorded.

4.2 Sedimentation Characteristics in DIW

After 24 hours of swelling, the swell indices of bentonites were recorded. Then, the graduated cylinders were shaken to examine the sedimentation behavior of bentonites. Sedimentation experiments were conducted initially using DIW. The sedimentation characteristics of bentonites were categorized into three different regimes. In the first type of settling, the particles settled down quickly and accumulated at the bottom of the graduated cylinder. Since accumulation was in upward direction, the sediment volume of the particles increased with time. Accumulated layer was followed by dispersed particle layer in the graduated cylinder and long time is necessary for sedimentation of these particles. This type of settling is called as accumulated sedimentation (AS) and is shown in Figure 4.1a. The surface separating the dispersed bentonite particles from the accumulated part can seen clearly from this figure. In the flocculated type of settling, the particles immediately formed aggregates and flocculated structure start to settle down together. Thus, a decrease in the sediment volume is observed from top to bottom over time. A sharp interface is also observed between the sediment surface and supernatant solution which was DIW (Figure 4.1b). Different from AS behavior, water above the dispersed surface is clear. In other words, there is no dispersed bentonite particle above the sediment. In the third type of settling, the bentonite particles cannot find enough time to flocculate and therefore some particles settle down rapidly at the beginning of the experiment and called as mixed settling (Figure 4.1c). In this form of sedimentation characteristic, sharp interface is observed between sediment at the base and dispersed part and this interface disappear after a while. Since the suspended particle density relative to the AS behavior is greater in this behavior, the remaining particles find sufficient time to flocculate and begin to settle from top to bottom, as in the flocculated type of settling.

The sedimentation tests were carried out three times. The time-dependent representation of the sedimentation curves is given in Figure 4.2a-c. Although the accumulated thickness of the sediments increased with time in AS (Figure 4.2a), a decrease in sediment thickness was obtained in FS (Figure 4.2b). Generally, it is expected to have larger sediment volumes for FS than for AS. Mixed sedimentation includes both flocculation and accumulation sedimentation behaviors. Indeed, the sedimentation thickness initially increased from bottom of the cylinder because of accumulation type of settling (Figure 4.2c). However, sometime after beginning of the test, the sharp interface between accumulated sediments and dispersed particles disappeared. Thus, it was unable to read the sediment thickness. Since the particles started to settle downward, this transition part observed during sedimentation test is shown in dashed line in Figure 4.2c.

Based on sedimentation tests with DIW, the average of final sediment volumes (FSVs) recorded at the end of the tests are given in Table 4.1. Table 4.1 also summarizes the sedimentation characteristics of bentonites which are shown in parentheses. As presented in Table 4.1, nine samples exhibited AS type, while four samples showed MS type of settling. FS behavior was observed only for B-1.

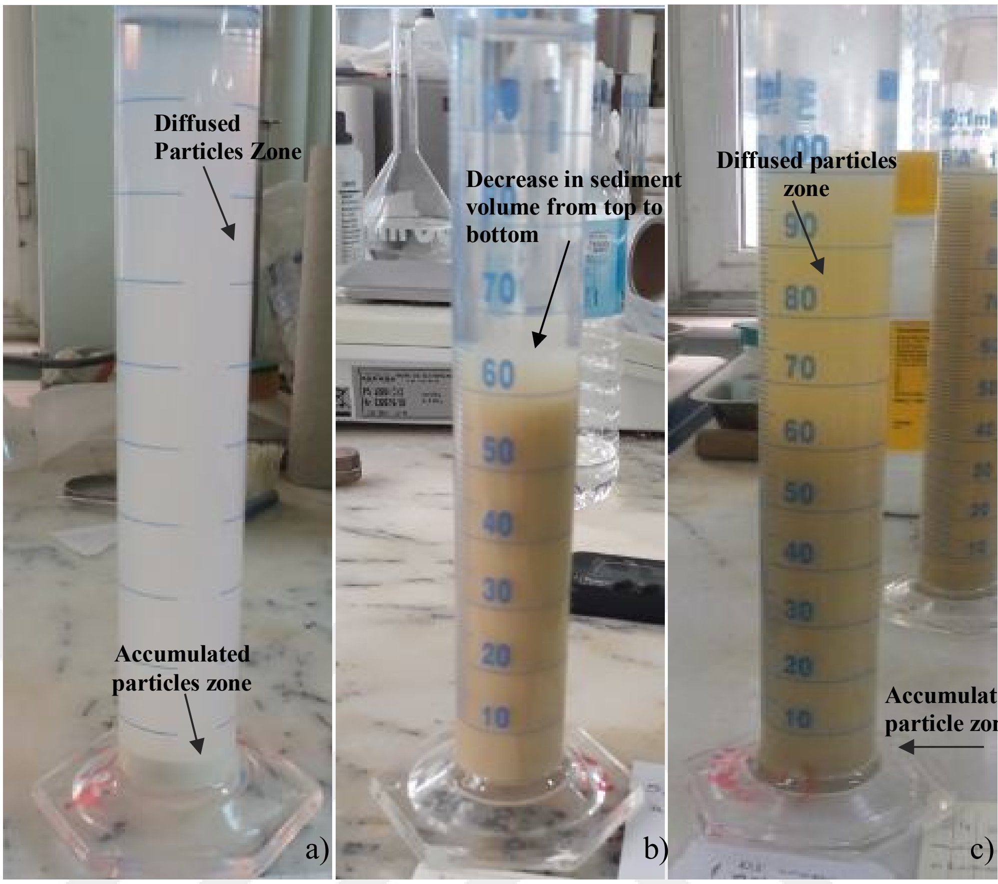

Figure 4.1 Sedimentation behavior of bentonites: a) Accumulation sedimentation (AS), b) flocculated sedimentation (FS) and c) mixed sedimentation (MS) (Personal archive, 2019)

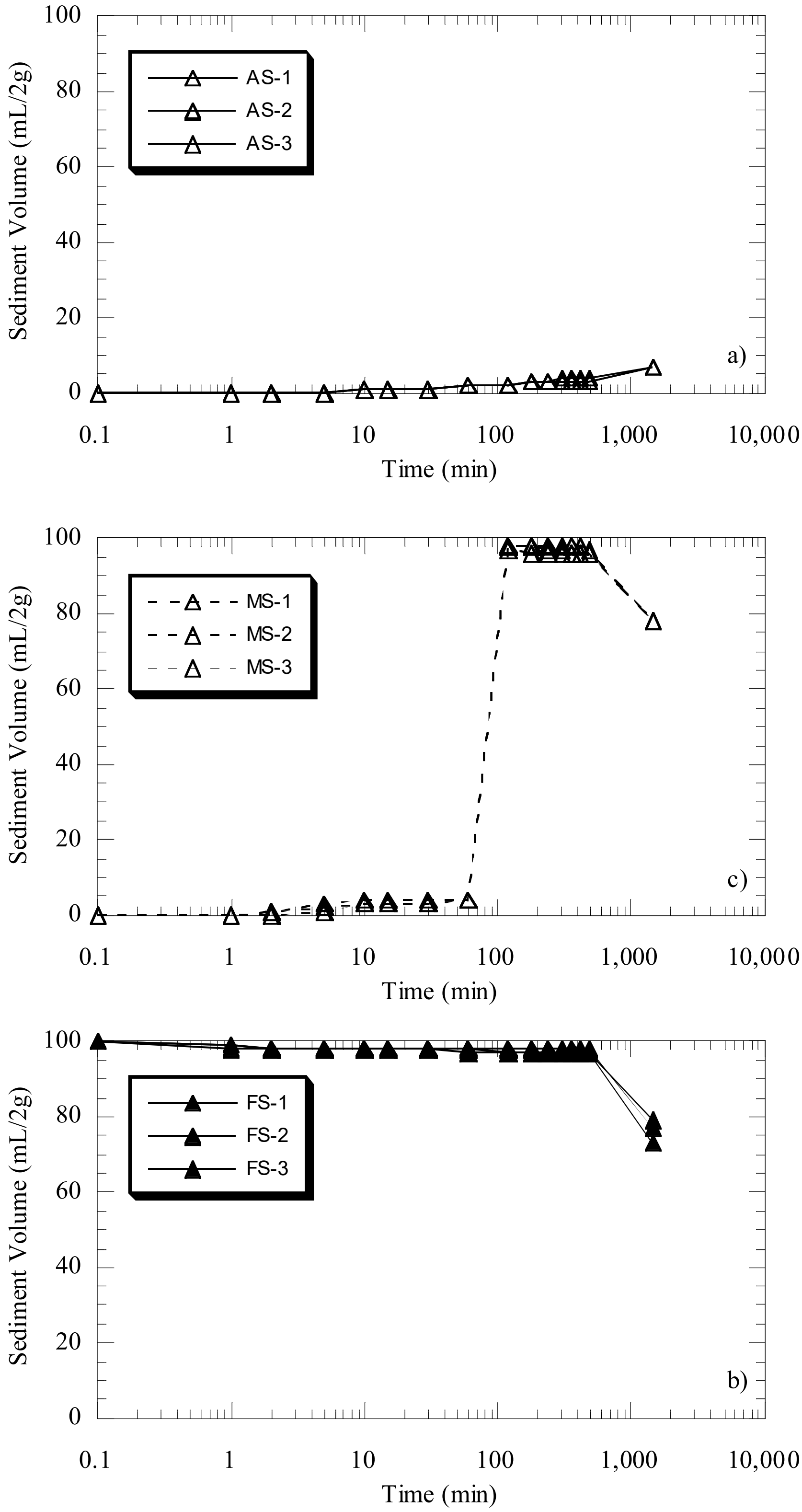

Figure 4.2 Graphical representation of sediment volume as a function of time: a) Accumulated sedimentation (AS), b) flocculated sedimentation (FS) and b) mixed sedimentation (MS)

Table 4.1 Final sediment volumes (FSVs) and sedimentation characteristics of bentonites in different solutions

Bentonites	DIW	10 mM NaCl	20 mM NaCl	30 mM NaCl	5 mM $CaCl_2$	10 mM $CaCl_2$
GCL-1	7 (AS)	52 (FS)	40 (FS)	71 (FS)	44 (FS)	34 (FS)
GCL-2	59 (MS)	40 (FS)	27 (FS)	22 (FS)	27 (FS)	18.5 (FS)
GCL-3	78 (MS)	37 (FS)	31 (FS)	-	31 (FS)	22.5 (FS)
GCL-4	4 (AS)	nd. (MS)	nd. (MS)	38 (FS)	15 (AS)	35 (MS)
GCL-5	5 (AS)	45 (FS)	30 (FS)	26 (FS)	30 (FS)	22 (FS)
B-1	76 (FS)	74 (FS)	36 (FS)	30 (FS)	30 (FS)	23 (FS)
B-2	7 (AS)	23 (AS)	19 (MS)	17 (MS)	9.5 (AS)	10 (MS)
B-3	nd. (MS)	nd. (MS)	nd. (MS)	-	nd. (AS)	77 (FS)
B-4	8 (AS)	9 (AS)	9 (MS)	10 (MS)	-	8 (MS)
B-5	8 (AS)	9 (AS)	10 (MS)	10 (MS)	8 (MS)	-
B-6	95 (MS)	82 (FS)	50 (FS)	40 (FS)	48 (FS)	20 (FS)
B-7	4 (AS)	5 (AS)	57 (FS)	nd. (AS)	10 (AS)	37 (FS)
B-8	6 (AS)	10 (AS)	11 (MS)	11 (MS)	10 (MS)	10.5 (FS)
B-9	8 (AS)	44 (MS)	20 (FS)	-	42 (MS)	16.5 (FS)

nd: not determined, final sediment volume could not have been read.

4.3 Sedimentation Characteristics in NaCl Solutions

The purpose of this study is to find out pore medium chemistry for bentonite which has common swell index value similar to DIW and shows FS type of settling. For this reason, sedimentation tests were performed with NaCl solution with concentrations ranging from 10 mM to 30 mM after swell index test by vigorously shaking the graduated cylinder.

Many sedimentation graphs can be drawn for each bentonite and solution. The sedimentation behaviors are summarized with final sediment volumes in Table 4.1. Since the sedimentation tests were repeated three times for each bentonite, the final sediment volumes were calculated taking the average of three experiments. The sedimentation behaviors of all samples are plotted as a function of time and given under Appendix-B.

Among others, 5 samples (B-2, B-4, B-5, B-7, B-8) exhibiting AS behavior in DIW continued to show the same behavior in 10 mM NaCl (Table 4.1). Similarly, the sedimentation behaviors of B-1 and B-3 did not change in 10 mM NaCl. In contrast, the sedimentation behavior changed from AS to MS for GCL-4, whereas that turned directly from AS to FS for GCL-1 and GCL-5 in 10 mM NaCl. In addition to these, GCL-2, GCL-3 and B-6 changed its sedimentation behavior from MS to FS (Table 4.1). It was observed that the sedimentation behavior of bentonites changed from AS to MS or FS, when the solution concentration was further increased (Table 4.1). It was expected because increasing the NaCl concentration from 10 to 30 mM also increases the ionic strength of the solution.

The swell index versus sedimentation behavior of bentonites are also shown in Figure 4.3a-d. When experiments were conducted using DIW, it was observed that the swell indices of bentonites exhibiting AS type of sedimentation behavior were in a wide range (8.0-25.0 mL/2g). On the other hand, the swell indices of bentonites showing MS and FS were in a limited range which are more than the average of swell indices obtained with AS (Figure 4.3a).

Similar swell indices were obtained for most bentonites and sedimentation behaviors changed from AS to MS or FS when the NaCl concentrations were increased from 10 mM to 30 mM (Figure 4.3b-d). Although 9 bentonites showed AS type of settling in DIW, no bentonite was observed showing AS in 20 mM NaCl solution. Interestingly, there was one bentonite serving AS in 30 mM NaCl solution.

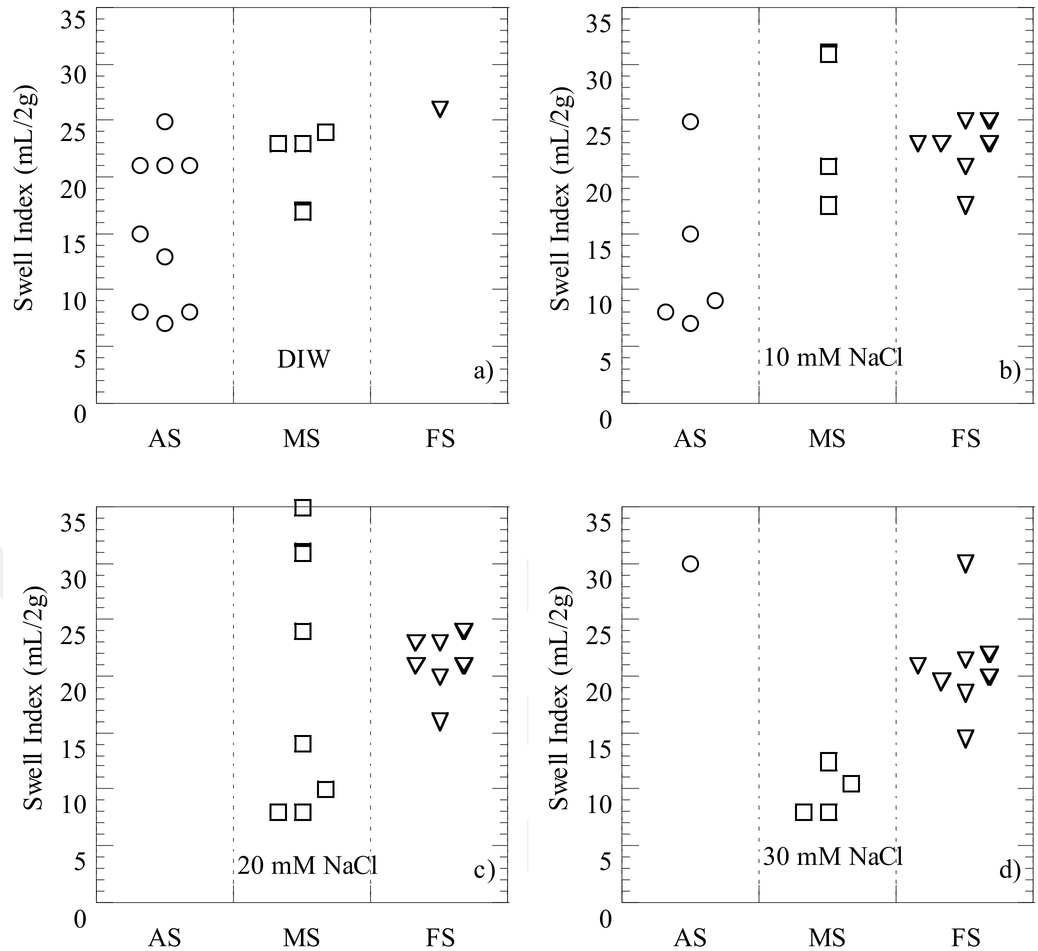

Figure 4.3 Graphical representation of swell indices of bentonites with respect to sedimentation behaviors: a) DIW, b) 10 mM NaCl, c) 20 mM NaCl and d) 30 mM NaCl

4.4 Sedimentation Characteristics in $CaCl_2$ Solutions

Bentonites were subjected to sedimentation tests after swell index tests with $CaCl_2$ solutions. For this reason, sedimentation tests were conducted with 5 mM and 10 mM concentrations. Since 28 sedimentation graphs can be drawn as a function of time [14×2=28 graphs for 14 GCLs and bentonites and for two concentrations), only the sedimentation behavior were discussed herein and shown in Table 4.1.

When sedimentation tests were conducted using 5 mM $CaCl_2$ concentration, 3 samples (B-2, B-7, GCL-4) showed AS behavior in DIW continued to show the same behavior at this concentration (Table 4.1 and Figure 4.4a). Similar to this finding, sedimentation behavior of B-1 was obtained the same with the DIW (i.e. FS). On the other hand, sedimentation behavior changed from AS to MS for B-5, B-8, B-9 and from AS to FS for GCL-1 and GCL-5. Also, sedimentation behavior changed from MS to FS for GCL-2, GCL-3 and B-6 (Table 4.1).

Most bentonites showed FS behavior in 10 mM $CaCl_2$ solution, while others showed MS at this concentration (Figure 4.4b). The sedimentation behavior of bentonites changed from AS to FS or MS when the solution concentration was increased from 5 to 10 mM. The FS behavior observed on most bentonites at this concentration can be attributed to the reduction of thickness of diffuse double layer surrounding the particles, resulting rapid settling of particles in the form of flocs. Based on these experiments, 10 mM $CaCl_2$ solution can be chosen as the critical concentration at which the bentonites showed FS type of settling.

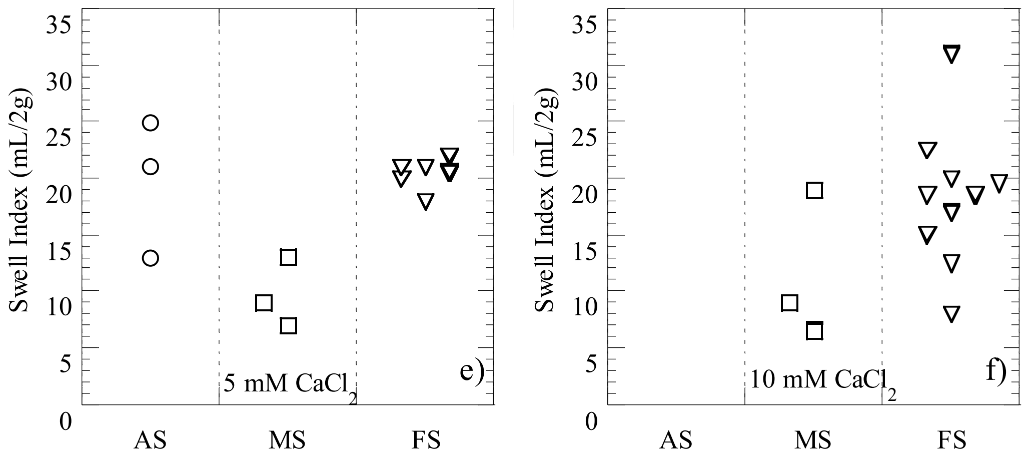

Figure 4.4 Graphical representation of swell indices of bentonites with respect to sedimentation behaviors: a) 5 mM $CaCl_2$ and b) 10 mM $CaCl_2$

4.5 Interpreting the Final Sediment Volumes (FSVs) with Ionic Strength and RMD

The comparison of the FSVs as a functions of ionic strength is shown in Figure 4.5a for GCLs and Figure 4.5b for bentonites. The sediment volume decreased as the ionic strength increased for both GCLs and bentonites. Since RMD of 5 mM $CaCl_2$ solution is 0 (zero) and that of 20 mM NaCl solution is infinity (∞), the final sediment volumes obtained in 5 mM $CaCl_2$ is less than those obtained in 20 mM NaCl. The greater the RMD, the greater is the swell or sediment volume.

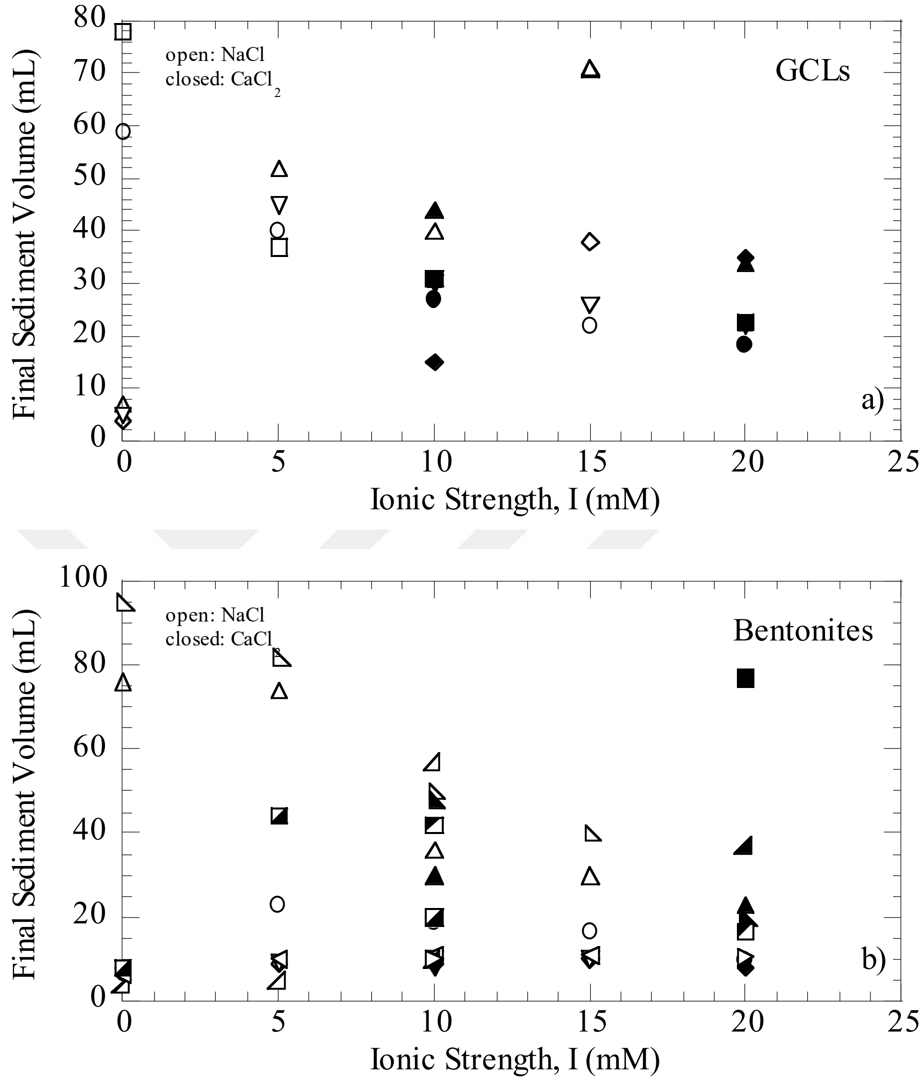

Figure 4.5 Evaluation of final sediment volumes as a function of ionic strength for: a) GCLs and b) bentonites

4.6 Conclusions

After swell index tests, the sedimentation tests were initiated by vigorously shaken the graduated cylinders. During the tests, the sedimentation characteristics were observed as well as the final sediment volumes were recorded at the end of the test. The significant points obtained so far is summarized below:

- Among the samples, 9 bentonites exhibited AS type of settling while the others showed MS and FS in DIW. It was observed that the sedimentation behavior of bentonites changed from AS to MS or FS with increasing concentration from 10 mM to 30 mM with NaCl.

- When increasing the $CaCl_2$ concentration from 5 mM to 10 mM, the sedimentation behavior of bentonites changed from AS to FS or MS.

- Since swell indices were more or less the same as in DIW and FS type of settling was observed in 10 mM $CaCl_2$ solution, this concentration was determined as the critical concentration.

- FSV decreased with an increase in the ionic strength for both GCLs and bentonites.

CHAPTER FIVE
RELATIONSHIPS BETWEEN SWELL INDEX/FINAL SEDIMENT VOLUMES AND SOME PROPERTIES OF BENTONITES

5.1 Background

Determining the hydraulic conductivity of bentonites is a time consuming process. The test that is to be conducted on bentonite is complex and the equipment used during testing is expensive (especially, when flexible wall permeameter is used). Although hydraulic conductivity test must be performed in every case, some other tests are also carried out to support the results or to estimate the hydraulic conductivity of bentonite in a simple and quick way. One of the most significant test that has been considered so far is swell index test. In that test, the swelling ability of bentonite can be determined within one day using different liquids. Then, the tendency of swelling in these liquids can be compared with the hydraulic conductivity of the bentonite. However, swell index test has some handicaps such as: (i) rapid settling of flocculated particles from surface of the liquid to down of the graduated cylinder, (ii) presence of entrapped air within the swollen particles, and (iii) irregular surface of settled bentonite that make it hard to record the correct value of swell index. To eliminate the effect of these factors, this study suggests a new test method for the interpretation of hydraulic conductivity of GCLs. The proposed methodology is sedimentation test which can be applied after swell index test by vigorously shaking the graduated cylinder. This test method does not need to prepare any new sample and since the same sample is used in swell index and sedimentation test, the correlation efficiency of swell index and sedimentation behavior (or sediment volume) with different properties of bentonite can be compared more realistically within each other.

In this chapter, observational relationships were also established between swell index category/sedimentation characteristics and some engineering properties of bentonites. The physicochemical/hydraulic properties were determined using DIW. Moreover, the quantitative relationships between swell indices/final sediment volumes and physico-chemical/hydraulic properties of bentonites were established and the results were discussed.

5.2 Observational Comparison between Swell Index and Some Properties of Bentonites

The summary of test results obtained for the bentonites and GCLs are presented in Table 5.1. Since the sedimentation behavior is like AS, MS, and FS type, the swell index of bentonites were resembled to sedimentation characteristics by categorizing the swell indices into three groups. In the first category, the range of swell index was taken as 7-13 mL/2g (average of 9 mL/2g). In the second and third category, the ranges of swell indices were 15-21 and 23-26 mL/2g, respectively (average of 19 and 24 mL/2g, respectively). This categorization allows one to establish observational relationships for the swell index. Hence, the performance comparison with respect to sedimentation behavior can be done.

Table 5.1 Swell index and sedimentation behavior

Bentonites	Swell Index	Category Level	Sedimentation Behavior	Liquid Limit (%)	Plastic Limit (%)	Hydraulic Conductivity (%)
B-4	7	1	AS	121	-	-
B-5	8	1	AS	100	57	-
B-8	8	1	AS	149	42	3.0×10^{-11}
B-9	13	1	AS	283	48	1.3×10^{-11}
GCL-1	21	2	AS	385	58	5.8×10^{-11}
GCL-4	21	2	AS	240	53	3.2×10^{-11}
GCL-5	21	2	AS	280	36	3.8×10^{-11}
B-2	15	2	AS	128	53	-
B-3	17	2	MS	529	38	6.1×10^{-12}
GCL-2	23	3	MS	400	60	1.8×10^{-11}
GCL-3	23	3	MS	-	-	-
B-1	26	3	FS	417	44	8.1×10^{-12}
B-6	24	3	MS	397	34	5.9×10^{-12}
B-7	25	3	AS	552	41	5.2×10^{-12}

As given in Table 5.1, the same swell index might have either the same or different sedimentation behavior. Although GCL-1, GCL-4, and GCL-5 had swell index of 21 mL/2g, the sedimentation characteristics were in accumulation type. Only the sedimentation characteristic of B-3 was obtained MS in this category. In contrast, B-1, B-6, and B-7 had an average of 25 mL/2g swell index. However, the settling type of these bentonites were totally different within each other.

The category based evaluation of swell index is initially examined for the consistency limits of bentonites. As can be seen in Figure 5.1a, the liquid limit of bentonite increased when category level increased from 1 to 3. Note that swell index increases with the category level. In contrast, there is no such kind of relationship between swell index category and plastic limit (Figure 5.1b).

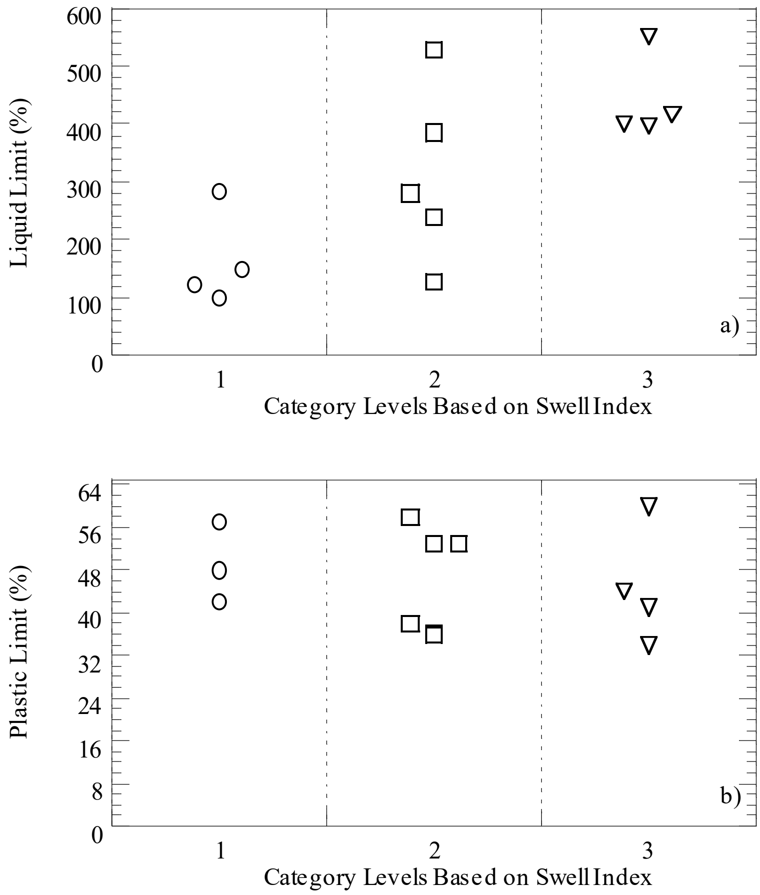

Figure 5.1 Observational relationship between swell index category a) liquid limit and b) plastic limit

From Table 5.1, it can be seen that the bentonites with the same swell index may have different hydraulic conductivities. The difference between the hydraulic conductivity of bentonites is 9.5 times at most. This amount of difference in the hydraulic conductivity is quite significant. There is only two hydraulic conductivity data in Category 1 and the difference is only 2.3 times. In this category, all bentonites exhibited AS type of settling. In Category 2, however, the difference between the hydraulic conductivities of three bentonites, which had AS type of settling, is a factor of 1.8. When settling regime changed to MS (B-3), the hydraulic conductivity reduced to 6.1×10^{-12} m/s or the difference between the hydraulic conductivity of bentonite with MS and with AS (average was taken) became a factor 7.0. In Category 3, the difference between the hydraulic conductivities is also significant (3.5 times). The settling regime in this category changes from AS to FS, indicating sedimentation behavior may be more effective than swell index while interpreting the hydraulic conductivity.

The hydraulic conductivity of GCLs are plotted as a functions of swell index category in Figure 5.2. Figure 5.2 shows that tendency between the swell index category and hydraulic conductivity is in "reverse V shape". The hydraulic conductivity increased when category level increased from 1 to 2. Then, it decreased an average of 3.6 times (from 3.4×10^{-11} m/s to 9.3×10^{-12} m/s) at Category 3.

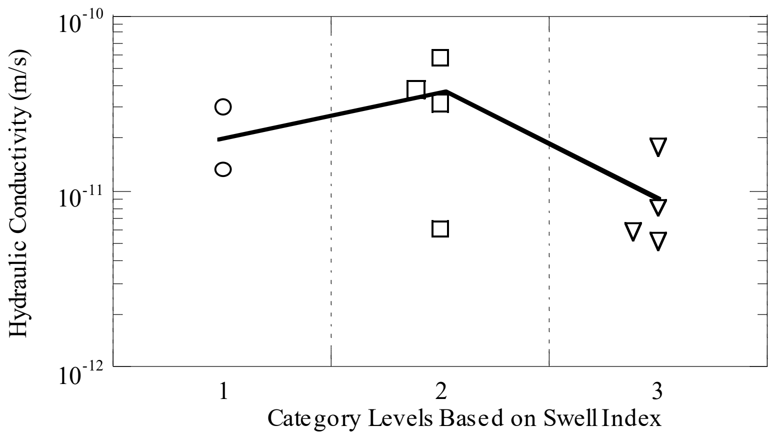

Figure 5.2 Observational relationship between swell index category and hydraulic conductivity

5.3 Observational Relationships between Sedimentation Characteristics and Some Properties of Bentonites

Since sediment characteristics of bentonites were different within each other, the relationship was established by considering sediment behavior rather than sediment volumes. Because different settling regimes have different sediment volumes. Therefore, giving the tendency with a quantitative correlation is avoided herein. The interpretation of sedimentation characteristics depends on visual observations. Thus, this kind of relationship is defined as "observational" as previously done when hydraulic conductivity is evaluated in terms of swell index category.

The consistency limits are one of an important index property of the soils that have wide applicability in geotechnical engineering. Therefore, sedimentation characteristics were initially evaluated by taking into account the index properties (i.e. liquid and plastic limits) of bentonites. For example, the sedimentation behavior was just AS type when the liquid limit was less than 300% (Figure 5.3a) or the plastic limit was more than 45% (Figure 5.3b). In contrast, AS, MS or FS type of settling was observed when the liquid limit was more than 300% (Figure 5.3a) or plastic limit was less than 45% (Figure 5.3b).

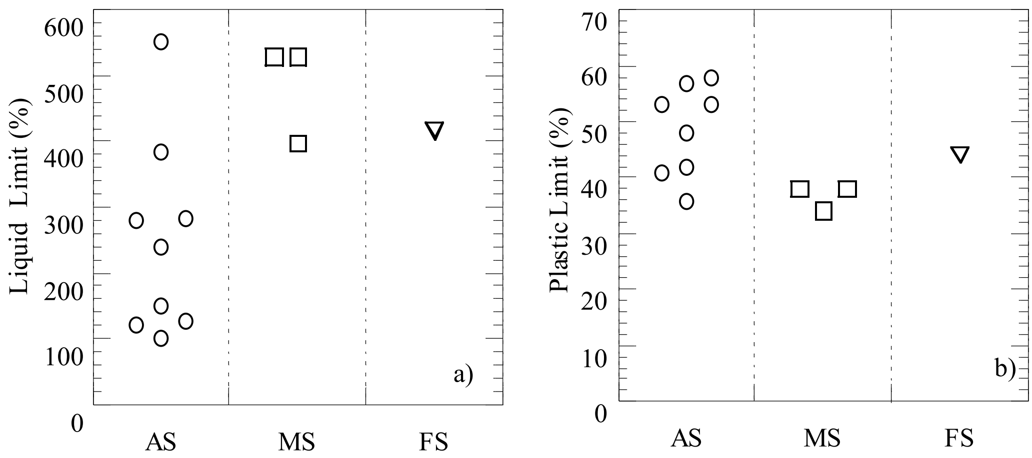

Figure 5.3 Categorizing the sedimentation behavior based on: a) liquid and b) plastic limits of bentonites

Only 9 bentonites could be evaluated when hydraulic conductivity is of interest (Figure 5.4). As can be seen from Figure 5.4, the hydraulic conductivity of bentonites showing AS behavior is higher than the bentonites exhibiting FS behavior. In other words, there is a decreasing trend in the hydraulic conductivity from AS to FS.

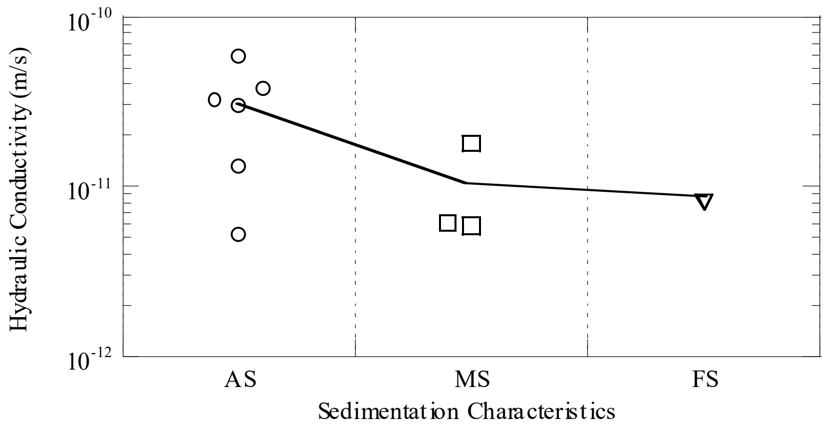

Figure 5.4 Relationship between the hydraulic conductivity and sedimentation behavior of bentonites

5.4 Quantitative Relationships between Swell Index and Some Properties of Bentonites

The swell indices of bentonites are correlated with some properties of bentonites such as consistency limits, specific surface area (SSA), cation exchange capacity (CEC) and hydraulic conductivity.

The liquid limit values obtained from Casagrande method is higher than those obtained from Fall Cone method (Table 2.1). Since all bentonites used in this study mainly consist of montmorillonite, liquid limit is controlled by viscous shear resistance with the effect of viscous nature of diffuse double layer (Sridharan, 2000). In the fall cone test method, viscous shear cannot resist the movement of the cone, resulting lower liquid limit. Thus, Casagrande test method is preferred and correlated with swell indices of bentonites which is shown in Figure 5.5a. It is seen that swell indices linearly increased as the liquid limit of bentonite increased. The coefficient of determination (i.e. R^2) is ~0.6.

The swell indices are also correlated with plastic limits of bentonites. Although there is a decreasing trend of swell index with plastic limit (Figure 5.5b), the data is so scattered and therefore, poor relationship was obtained between swell index and plastic limits of bentonites (R^2 is close to 0).

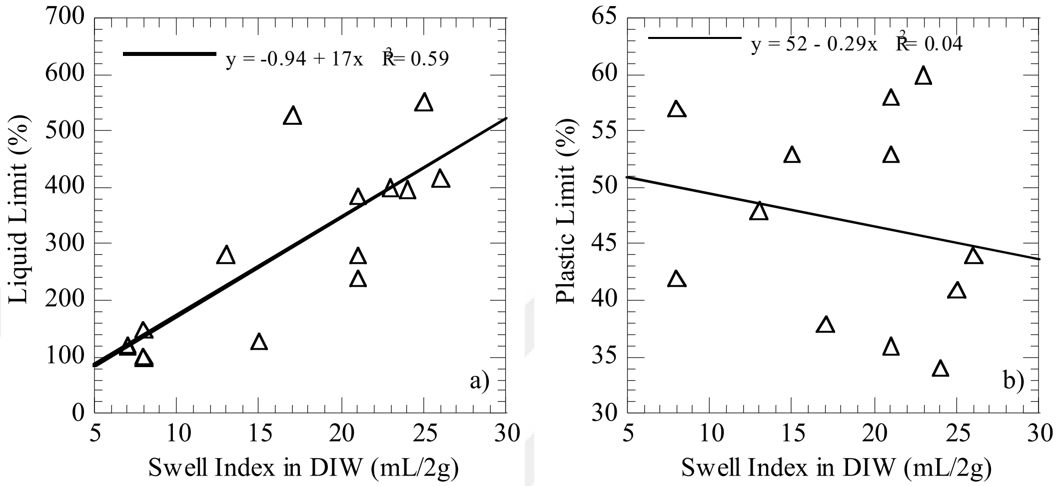

Figure 5.5 Relationship between swell index and: a) liquid limits and b) plastic limits of bentonites

The physico-chemical parameters of bentonites are also evaluated with the swell indices (Figure 5.6a-b). Like liquid limit, an increasing trend was obtained between swell index and SSA. There is one data out of this trend which has been highlighted with a question mark (Figure 5.6a). This data was not included into the dataset while drawing the tendency. Contrary to SSA, there is no relationship exists between CEC and swell index (Figure 5.6b). Since limited data is available for CEC, it may not be convenient to give a coefficient of determination value.

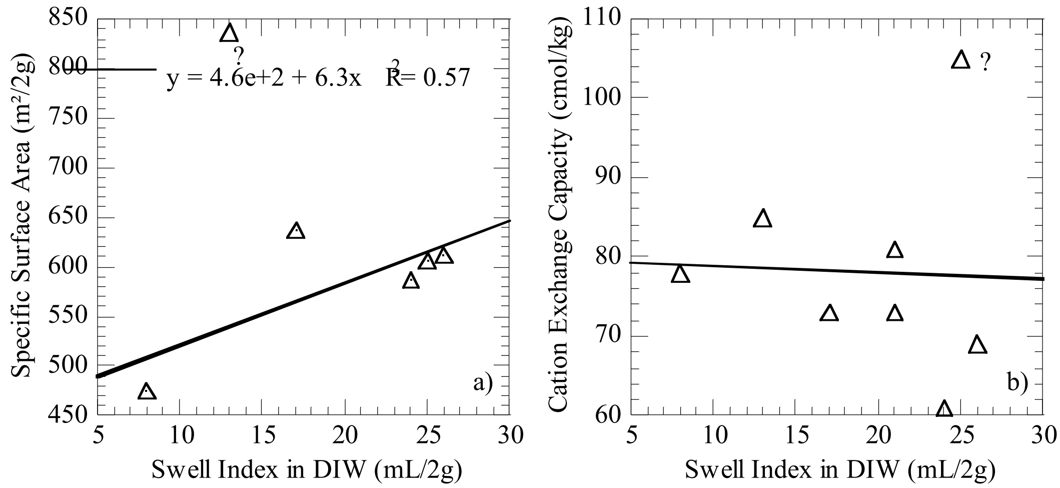

Figure 5.6 Relationship between swell indices: a) specific surface area and b) cation exchange capacity of bentonite

The relationship between the swell index and hydraulic conductivity of bentonites is shown in Figure 5.7. There are two distinct tendencies in Figure 5.7 which has V shape. One passes through B-8 and the other passes through GCL-4. However, in both tendencies, the hydraulic conductivities had a decreasing trend with increasing swell index. Since two tendencies were available, no R^2 value was included.

Another important finding is that, although the hydraulic conductivities of bentonite GCL-4 and B-8 are almost the same, the swell indices are quite different (8 and 21 mL/2g). Therefore, it is clear that swell index alone is not sufficient to interpret the hydraulic conductivity.

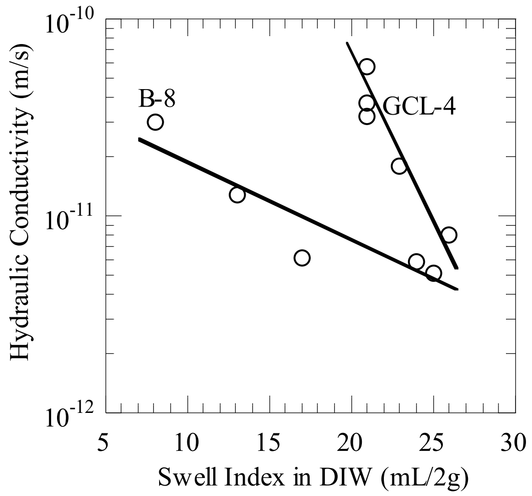

Figure 5.7 Relationship between swell index and hydraulic conductivity of bentonites

5.5 Quantitative Relationships between Final Sediment Volumes (FSV) and Some Properties of Bentonites

Since there were three types of settling regime (AS, MS, and FS), the final sediment volumes (FSVs) of bentonites were different from each other. AS had less, whereas FS had greater FSVs in DIW. Thus, when FSVs were intended to correlate with the some properties of bentonites, only AS type of settling was taken into consideration. As presented in Table 2.1, 9 bentonites, which served AS type of settling, are correlated with the consistency limits (Figure 5.8a-b). The relationship between FSV and liquid limit was poor (R^2=0.24) when compared to the relationship between swell index and liquid limit (Figure 5.5a). However, although better correlation was obtained between FSV and plastic limit when compared to swell index and plastic limit, it is still poor (R^2=0.27).

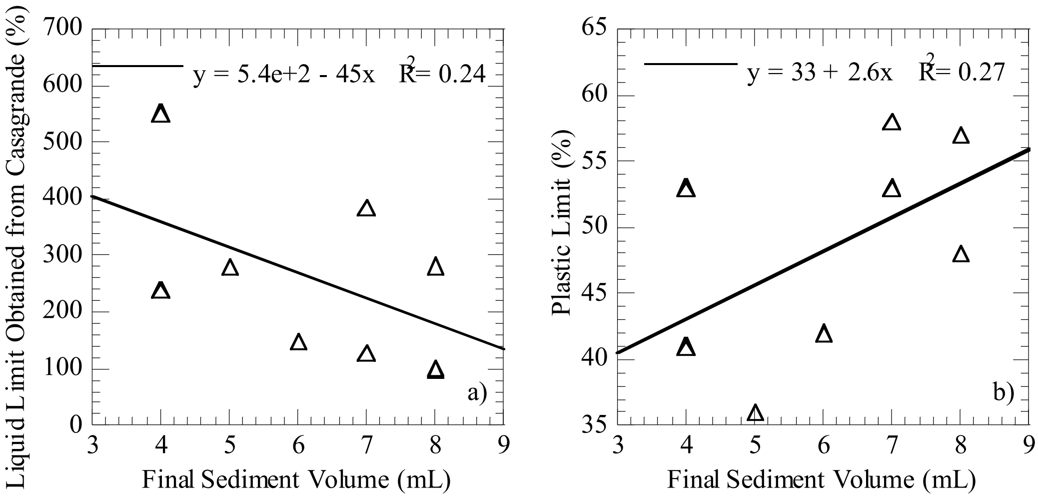

Figure 5.8 Relationship between final sediment volumes: a) liquid limit obtained from casagrande and b) plastic limit

The relationships between SSA/CEC and FSV cannot be drawn because of scarce data available. Unlike swell index, neither SSA versus FSV nor CEC versus FSV graphs are plotted.

There are 6 data available to give a relationship between FSV and hydraulic conductivity of bentonites which served AS type of settling and ranged between 4 and 8 mL. Instead of giving a trend line, the tendency between these two parameters is shown by lines which were drawn through upper and lower boundaries (Figure 5.9). Figure 5.9 shows that increase in the FSV increased the hydraulic conductivity. However, similar to swell index data (Figure 5.7), although sedimentation behavior of bentonites are the same, the difference between hydraulic conductivities were around one order of magnitude.

Figure 5.9 should be kept into mind. Because the hydraulic conductivity of bentonites exhibiting AS increases with an increase in the FSV. Thus, it can be said that the higher the FSV in AS, the higher is the hydraulic conductivity. Higher FSV in AS means that most of the particles are accumulated at the bottom of the cylinder while others are dispersed over the accumulated sediment. If test duration was increased until all particles settled, then the FSV would change negligibly. This shows that the colloidal particles that dispersed over the accumulated sediments are in less amount (or low density) which are not able to control or govern the hydraulic conductivity. In contrast, when the FSV in AS is lower, this means that more particles are dispersed over the sediment, resulting in a lower hydraulic conductivity (Figure 5.9). The liquid limit versus FSV relationship given in Figure 5.8a also proves the above argument. The liquid limit decreases with an increase in the FSV (for AS), indicating that dispersed particles not only controls the hydraulic conductivity, but also controls the liquid limit.

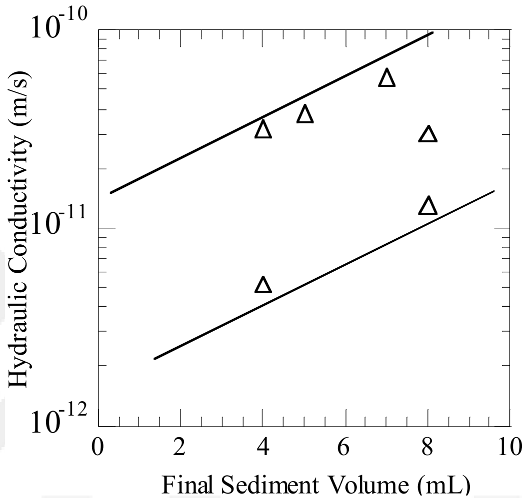

Figure 5.9 Relationship between final sediment volumes and hydraulic conductivity

5.6 Quantitative Relationships between Final Sediment Volumes (FSV) and Some Properties of Bentonites by Considering the Critical Concentration (10 mM $CaCl_2$ Solution)

Based on the swell index results obtained so far, the critical salt solution was determined as 10 mM $CaCl_2$. Now, it may be reasonable to make a quantitative relationship between the hydraulic conductivity and sedimentation behavior. Because, except GCL-4, B-2, B-4 and B-5 (Table 4.1), FS behavior was observed for most of the bentonites at this concentration. Thus, relationships were established only with the bentonites that exhibit FS type of settling.

The relationship between FSV and liquid limit is given in Figure 5.10a. Contrary to Figure 5.8a, FSV increased with an increase in the liquid limit. This is an important finding, because the relationship between FSV and liquid limit changes depending on the settling regime. In the case of AS, the greater the FSV, the lower is the liquid limit (Figure 5.8a). In the case of flocculation settling, however, the greater the FSV, the greater is the liquid limit (Figure 5.10a).

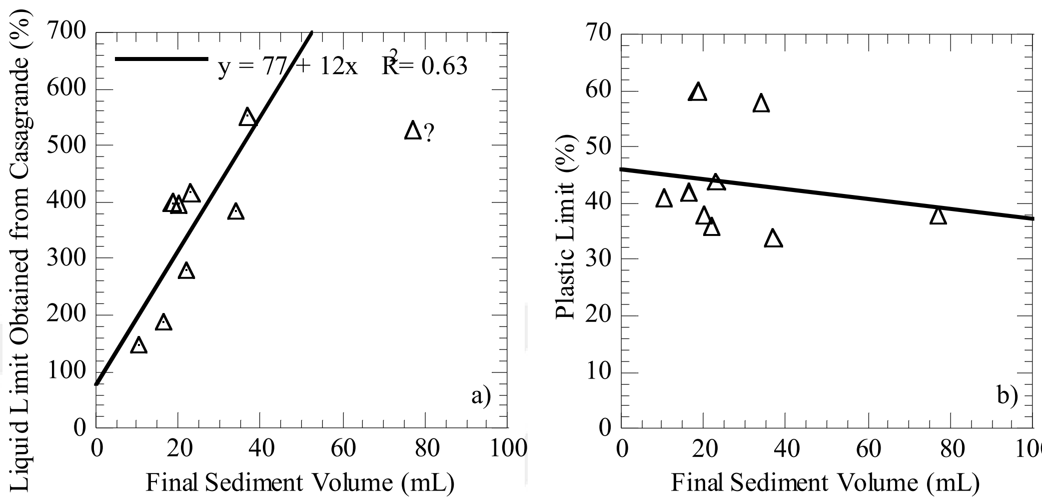

Figure 5.10 Relationship between final sediment volumes: a) liquid limit obtained from casagrande and b) plastic limit

In DIW, the density of the dispersed particles in AS is less when FSV is high or liquid limit is low (Figure 5.8a). This situation is also shown with a schematic representation in Figure 5.11. When testing liquid is changed from DIW to 10 mM $CaCl_2$ solution, dispersed particles tend to settle quickly together with the other particles or settle immediately after the accumulated sediment formed at the bottom of the cylinder. This is because that the thickness of adsorbed water layers surrounding the dispersed particles are compressed with the divalent calcium ions present in 10 mM $CaCl_2$. Thus, the contribution of dispersed particles to FSV is expected to be negligible when 10 mM $CaCl_2$ solution is used (Figure 5.11). In contrast, the density of dispersed particles in AS is high when FSV is low or liquid limit is high (Figure 5.8a). When 10 mM $CaCl_2$ solution is used, the contribution of dispersed particles is significant now. Although double layer surrounding the dispersed particles is compressed by the calcium ions, the flocculated structure of the particles leads greater sediment volume occurrence at the bottom of the cylinder (Figure 5.11). Therefore, greater FSVs is obtained in FS.

The efficiency of proposed methodology can be compared in two ways by considering the R^2. In the first case, the R^2 obtained from liquid limit-FSV relationships shown in Figure 5.8a and Figure 5.10a can be compared. If this will be done, then it can be seen that the R^2 in Figure 5.10a is greater than the R^2 in Figure 5.8a (0.63 vs. 0.24). This shows that liquid limits can be correlated well with using 10 mM $CaCl_2$ than using DIW. In the second case, the efficiency of sedimentation test over swell index test can be evaluated when liquid limit is the independent variable. The R^2 in Figure 5.5a is 0.59 which is slightly less than 0.63 in Figure 5.10a, indicating the success of sedimentation test with respect to swell index test while correlating with liquid limit.

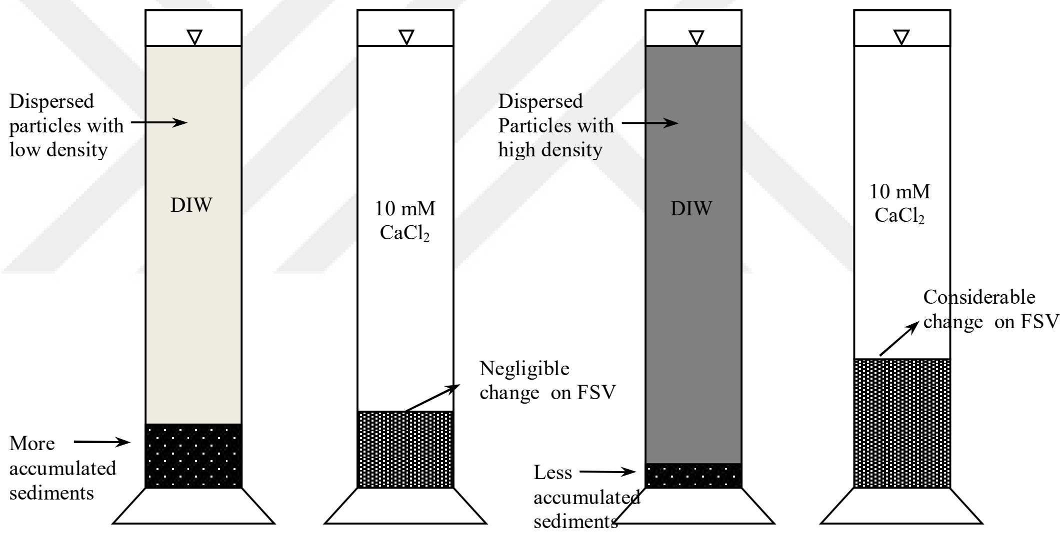

Figure 5.11 Schematic representation of change in settling regime by 10 mM $CaCl_2$ solution

Similar to the previous findings (Figure 5.5b and Figure 5.8b), poor relationship exists between plastic limit and FSV (Figure 5.10b). Thus, no correlation equation or R^2value was written in Figure 5.10b.

Similar to liquid limit, an increasing relationship was obtained between FSV and SSA (Figure 5.12a). The data which was shown by question mark is out of this trend and therefore it was not taken into the consideration when adding tendency. When this correlation efficiency is compared with swell index (Figure 5.6a), it can be seen that R^2 obtained from swell index is greater than R^2 obtained from FSV (0.57 vs. 0.48). To draw a solid conclusion, however, the data set should be increased and therefore, the proposed equation should be used with care. It was also obtained that the cation exchange capacity increased as the FSV increased (Figure 5.12b). One of a data was highlighted with question mark since it was placed far from the trend line, thus it was not included in the relationship. Similar to plastic limit, CEC gives poor relationship with swell index and FSV as well and the R^2 value was not embedded in Figure 5.12b.

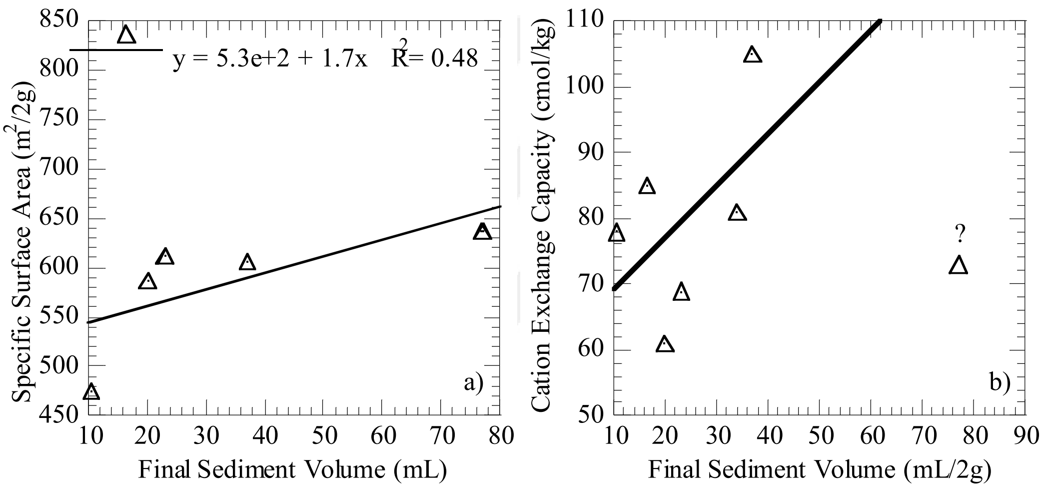

Figure 5.12 Relationship between final sediment volumes: a) specific surface area and b) final sediment volume

The correlation efficiency was finally evaluated between FSV and hydraulic conductivity. Figure 5.13 shows that hydraulic conductivity decreased as the FSV increased. Although poor correlation was obtained (R^2= 0.021), the data were less scattered in comparison to the data shown in Figure 5.9 (AS type of settling) and Figure 5.7 (swell index). This tendency can be easily compared with Figure 5.9 where the hydraulic conductivity increased as the FSV increased in the case of AS type of settling. The relationships obtained with AS can be summarized as liquid limit decreases (Figure 5.8a) and hydraulic conductivity (Figure 5.9) increases as the FSV increases. In contrast, the liquid limit increases (Figure 5.10a) and hydraulic conductivity decreases (Figure 5.13) as the FSV increased. Like liquid limit, this is one of another significant finding that the relationship established using hydraulic conductivity is affected from the sedimentation type. Despite larger FSV, the hydraulic conductivity is lower in FS type of settling than in the AS type of settling.

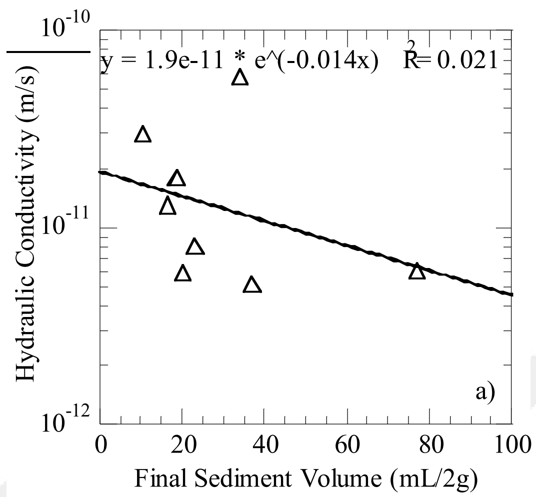

Figure 5.13 Relationship between final sediment volumes and hydraulic conductivity

5.7 Conclusions

Based on the findings, observational and quantitative relationships were established between swell indices/final sediment volumes (FSVs) and physico-chemical/hydraulic properties. The results are discussed below:

- When the swell index values were divided into three category levels to make observational comparison with the sedimentation characteristics, it was seen that the relationship between sedimentation characteristics and hydraulic conductivity is better than that between swell index category and hydraulic conductivity.

- When physico-chemical parameters of bentonites were evaluated with the swell indices, an increasing relationship was obtained between liquid limit/specific surface area and swell index.

- No significant relationship was existed between CEC and swell index.

- Two distinct tendencies, which is similar to V shape, was obtained between swell index and hydraulic conductivity of bentonites. The data were scattered and hence, R^2 value was not given.

- In the case of AS, liquid limit decreased with an increase in the FSV. This relationship can be accepted as fair. However, poor relationship was obtained between FSV and plastic limit.

- An increasing trend was obtained between FSV and hydraulic conductivity of bentonites exhibiting AS type of settling.

- Opposite relationships were obtained when the correlation efficiency was assessed using the data gathered from 10 mM $CaCl_2$ solution which mostly exhibited FS type of settling. Liquid limit increased, while hydraulic conductivity decreased as the FSV increased. This shows that correlation efficiency depends on the sedimentation regime.

CHAPTER SIX
SUMMARY AND CONCLUSIONS

This study investigates the use of sedimentation test method to interpret the hydraulic conductivity test results which can be applied easily after performing swell index test. Initially, swell index tests were performed in DIW using 14 bentonites five of which were taken from the GCLs. Then, sedimentation tests were conducted immediately after recording the swell index value. In these experiments, NaCl solution with concentrations ranging from 10 mM to 30 mM and $CaCl_2$ solution having 5 mM and 10 mM concentration were used. Based on these experiments, bentonites served different settling regimes, namely accumulation, flocculation and mixed (AS, FS, and MS).

The aim was to find out the critical salt concentration which gives similar swell index value as with DIW. Some relationships were established between the swell index/sedimentation characteristics/FSV and some engineering properties of bentonites such as consistency limits, cation exchange capacity, specific surface area and hydraulic conductivity. These relationships were considered just in case of results obtained in DIW. The sedimentation behavior is mainly AS type in DIW and thus liquid limit decreased and hydraulic conductivity increased as the FSV increased.

Among other salt solutions, 10 mM $CaCl_2$ was selected as the critical salt solution. Since swell index in $CaCl_2$ and DIW were similar within each other, the relationships were established once more using the sedimentation characteristics and FSV. The sedimentation characteristics turned to FS and FSV significantly changed in $CaCl_2$. The results showed that liquid limit increased and hydraulic conductivity decreased as the FSV increased which is contrary to the previous finding. Therefore, it is important to note that such correlations depend on the settling regimes.

Different from other studies, observational relationships were established between swell indices/sedimentation characteristics and hydraulic properties. To make adequate comparison with the sedimentation characteristics, the swell index values were divided into three category levels. It was seen that the observational relationship between sedimentation characteristics and hydraulic conductivity is better than that between swell index and hydraulic conductivity.

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APPENDICES

APPENDIX-A: Pictures of Bentonites After Swell Index Test

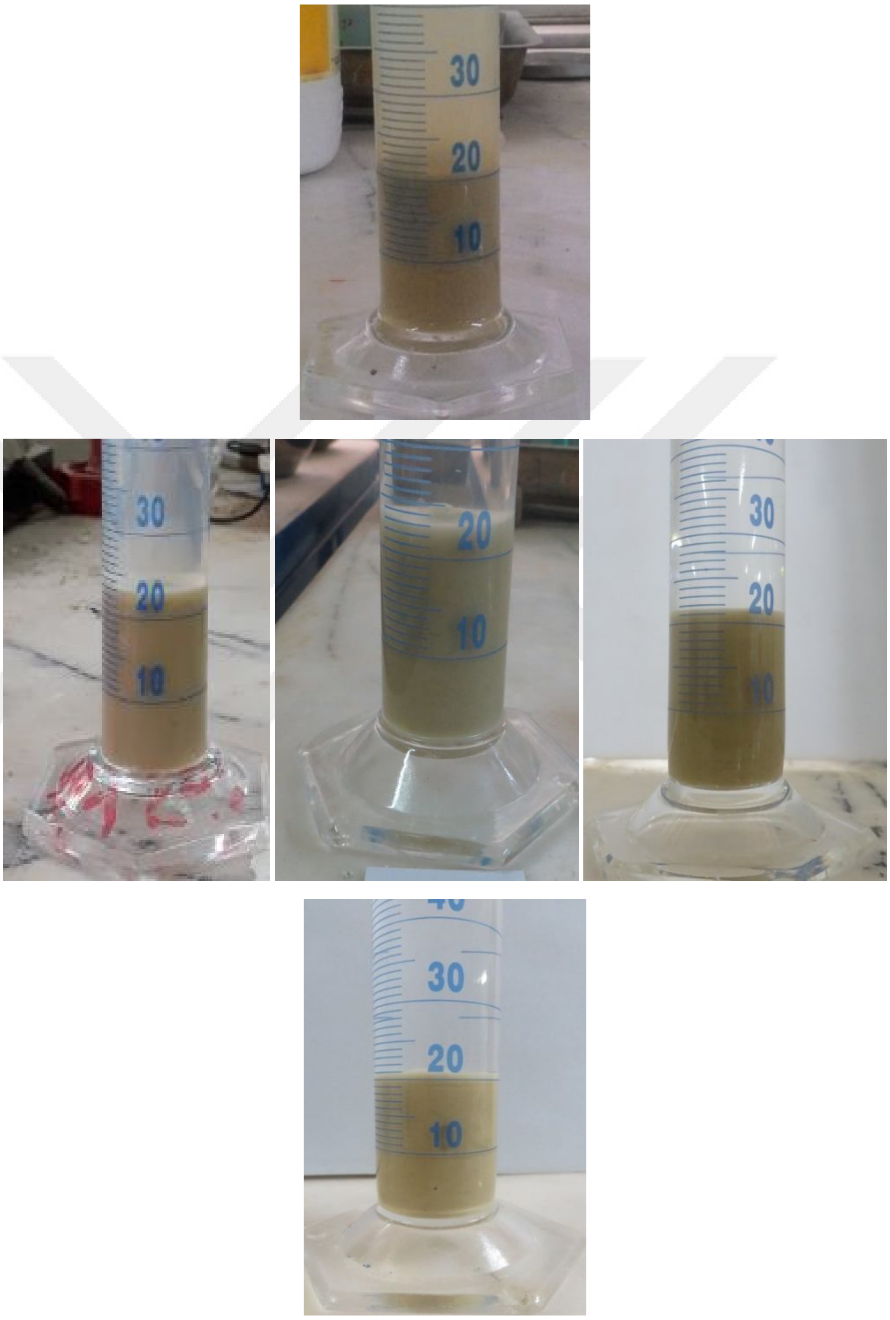


Figure A-1. Swell index of GCL-1 in: a) Deionized water (DIW), b) 10 mM NaCl, c) 20 mM NaCl d) 30 mM NaCl, e) 5 mM $CaCl_2$, f) 10 mM $CaCl_2$ (Personal archive, 2019)

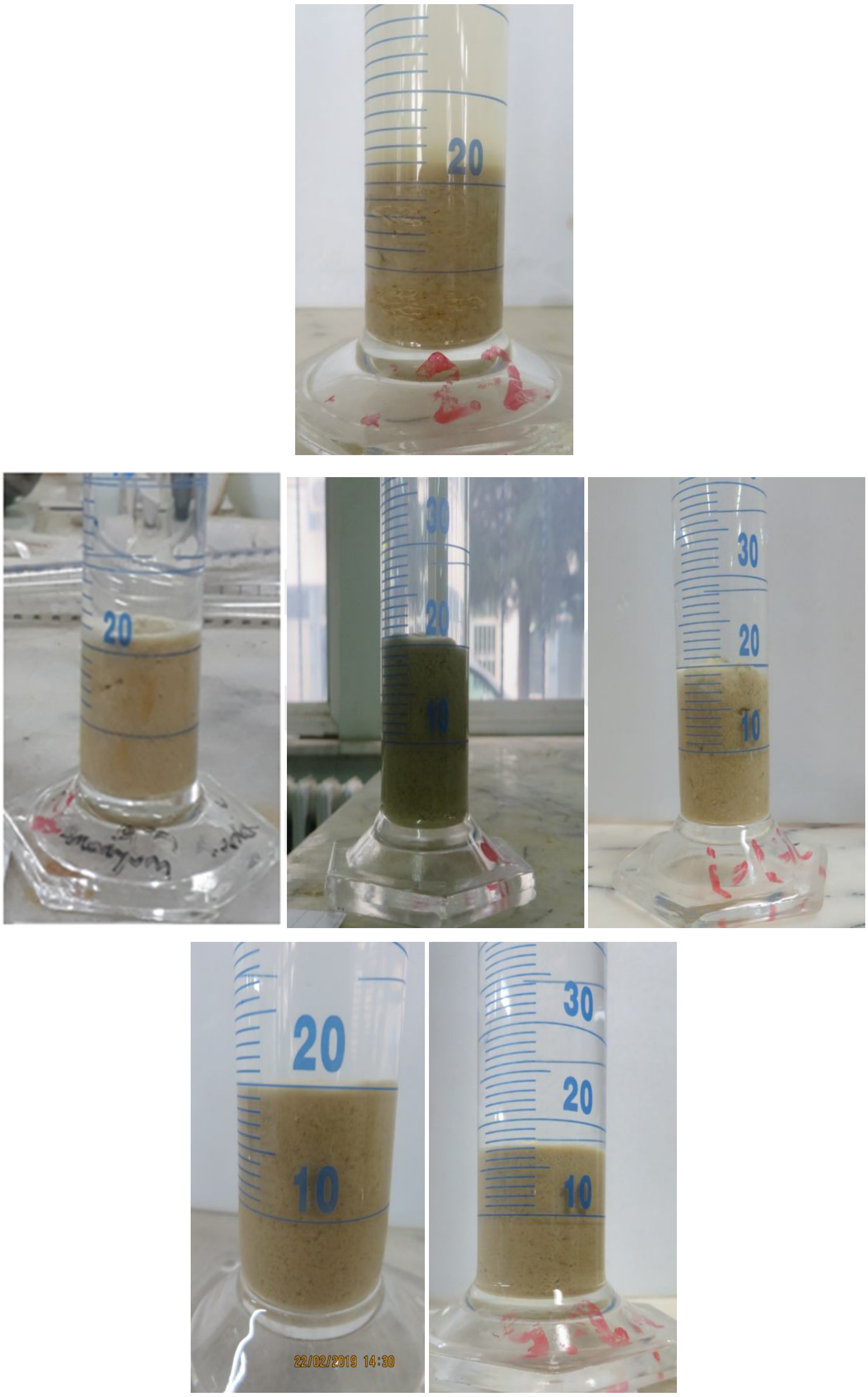


Figure A-2. Swell index of GCL-2 in: a) Deionized water (DIW), b) 10 mM NaCl, c) 20 mM NaCl d) 30 mM NaCl, e) 5 mM $CaCl_2$, f) 10 mM $CaCl_2$ (Personal archive, 2019)

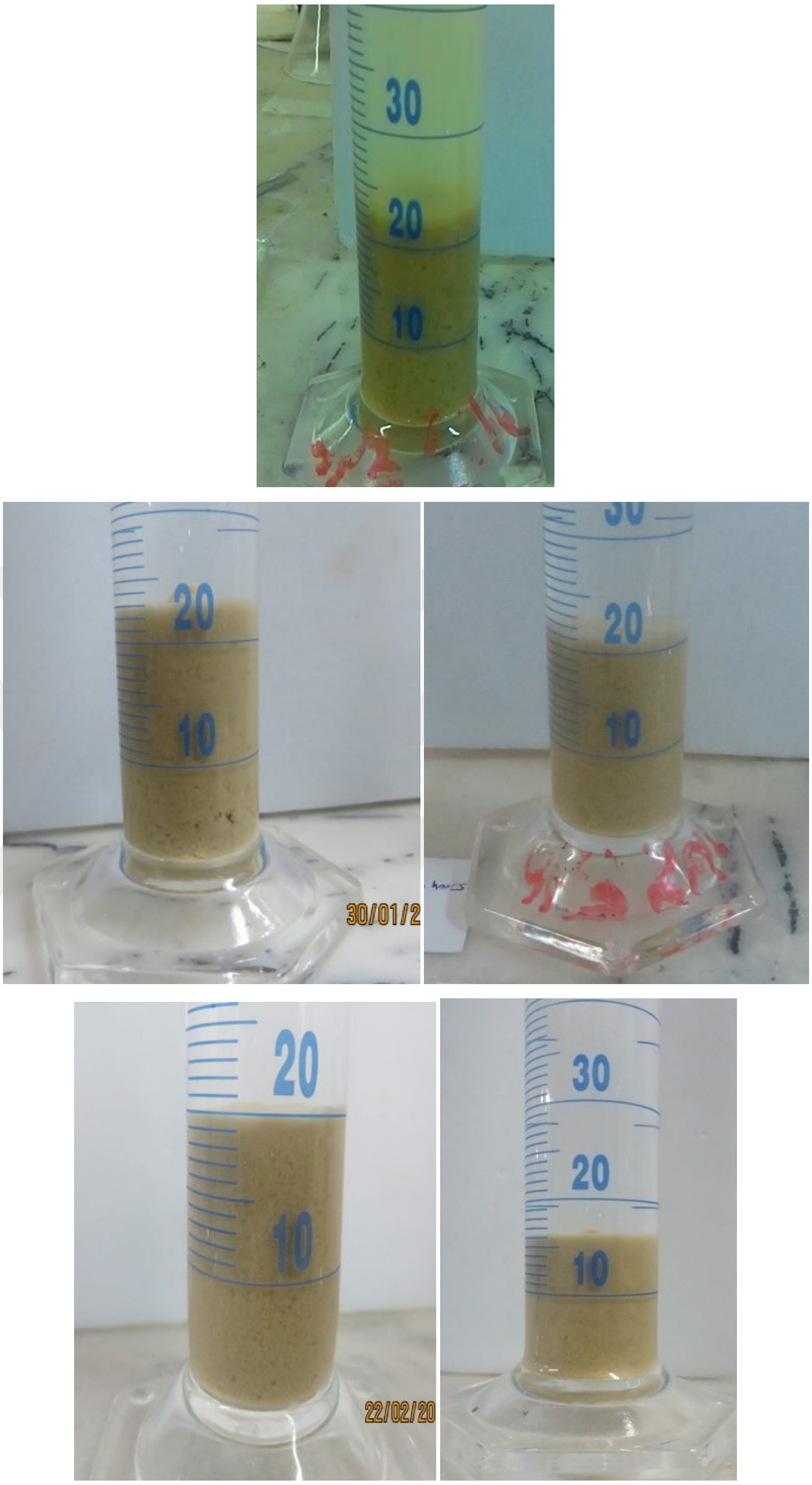


Figure A-3. Swell index of GCL-3 in: a) Deionized water (DIW), b) 10 mM NaCl, c) 20 mM NaCl d) 30 mM NaCl, e) 10 mM $CaCl_2$ (Personal archive, 2019)

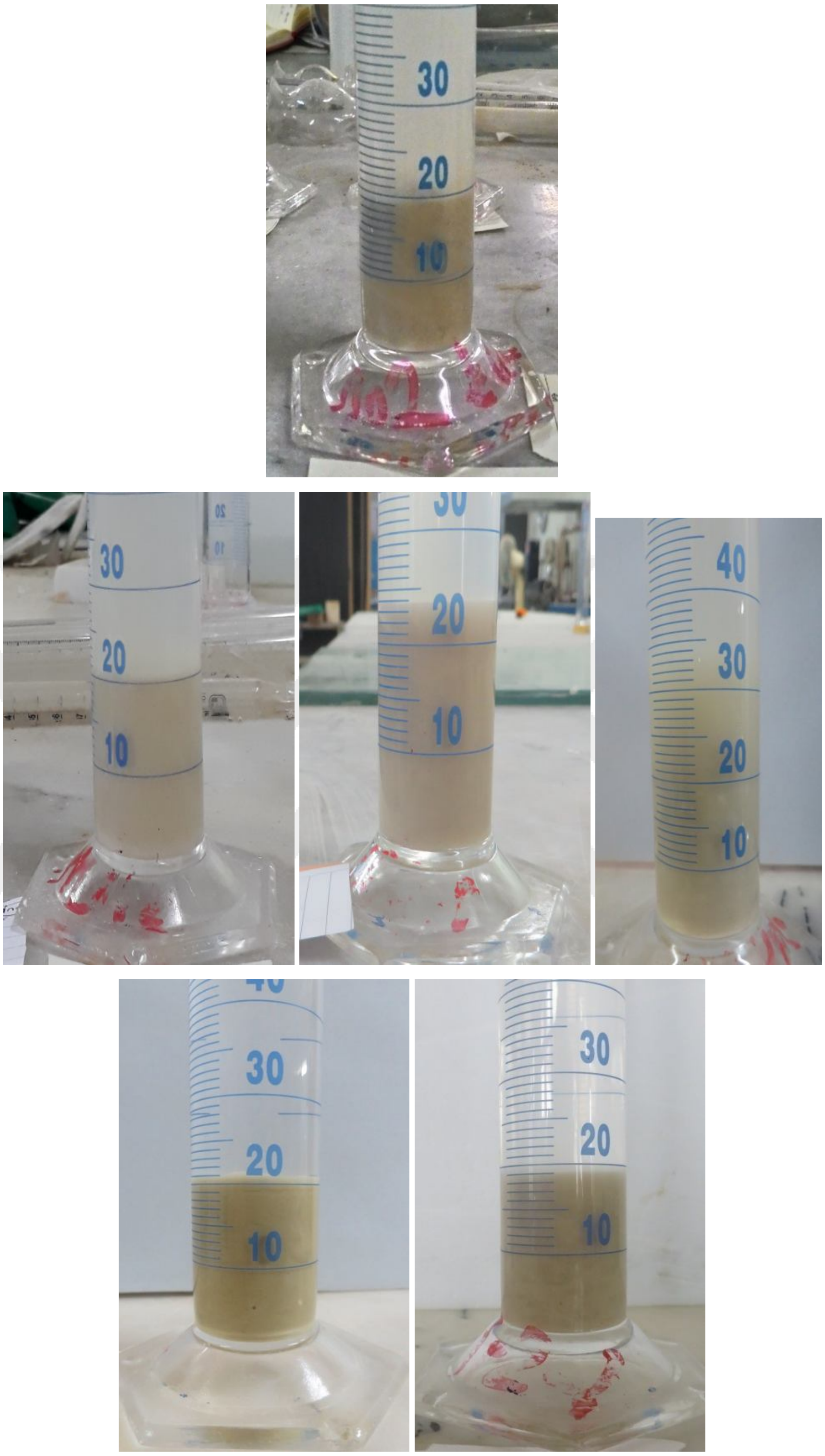


Figure A-4. Swell index of GCL-4 in: a) Deionized water (DIW), b) 10 mM NaCl, c) 20 mM NaCl d) 30 mM NaCl, e) 5 mM $CaCl_2$, f) 10 mM $CaCl_2$ (Personal archive, 2019)

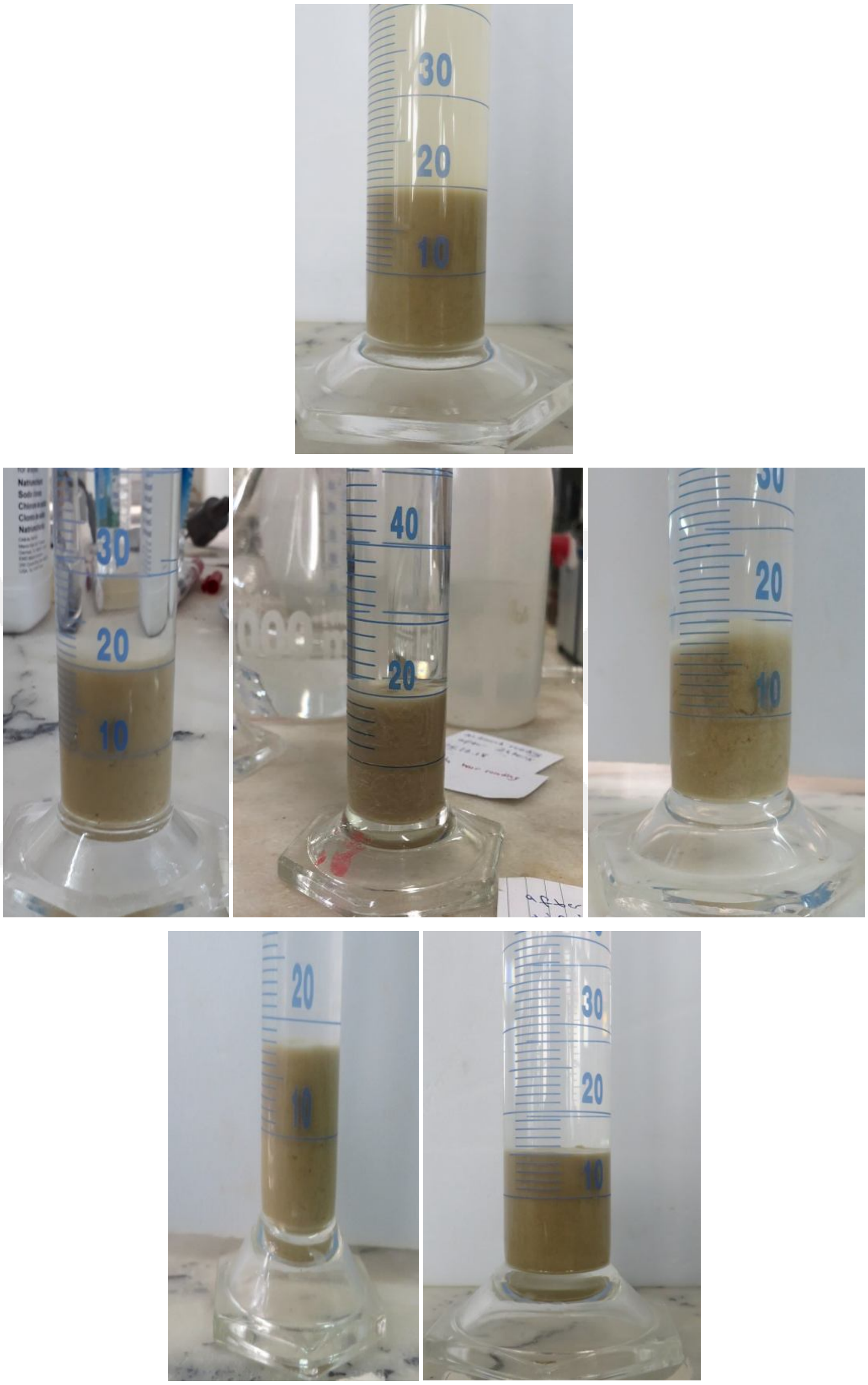


Figure A-5. Swell index of GCL-5 in: a) Deionized water (DIW), b) 10 mM NaCl, c) 20 mM NaCl d) 30 mM NaCl, e) 5 mM $CaCl_2$, f) 10 mM $CaCl_2$ (Personal archive, 2019)

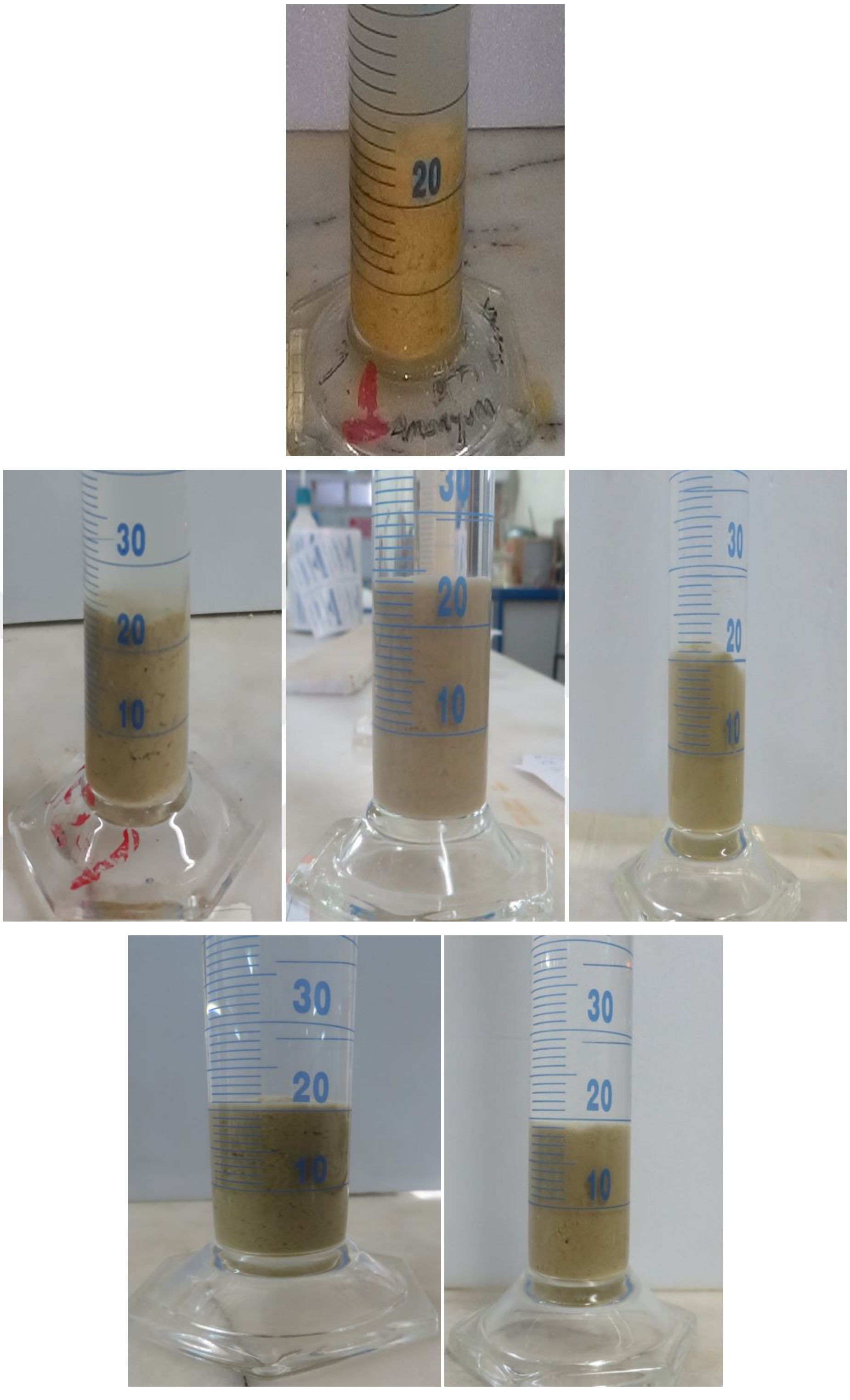


Figure A-6. Swell index of B-1 in: a) Deionized water (DIW), b) 10 mM NaCl, c) 20 mM NaCl d) 30 mM NaCl, e) 5 mM $CaCl_2$, f) 10 mM $CaCl_2$ (Personal archive, 2019)

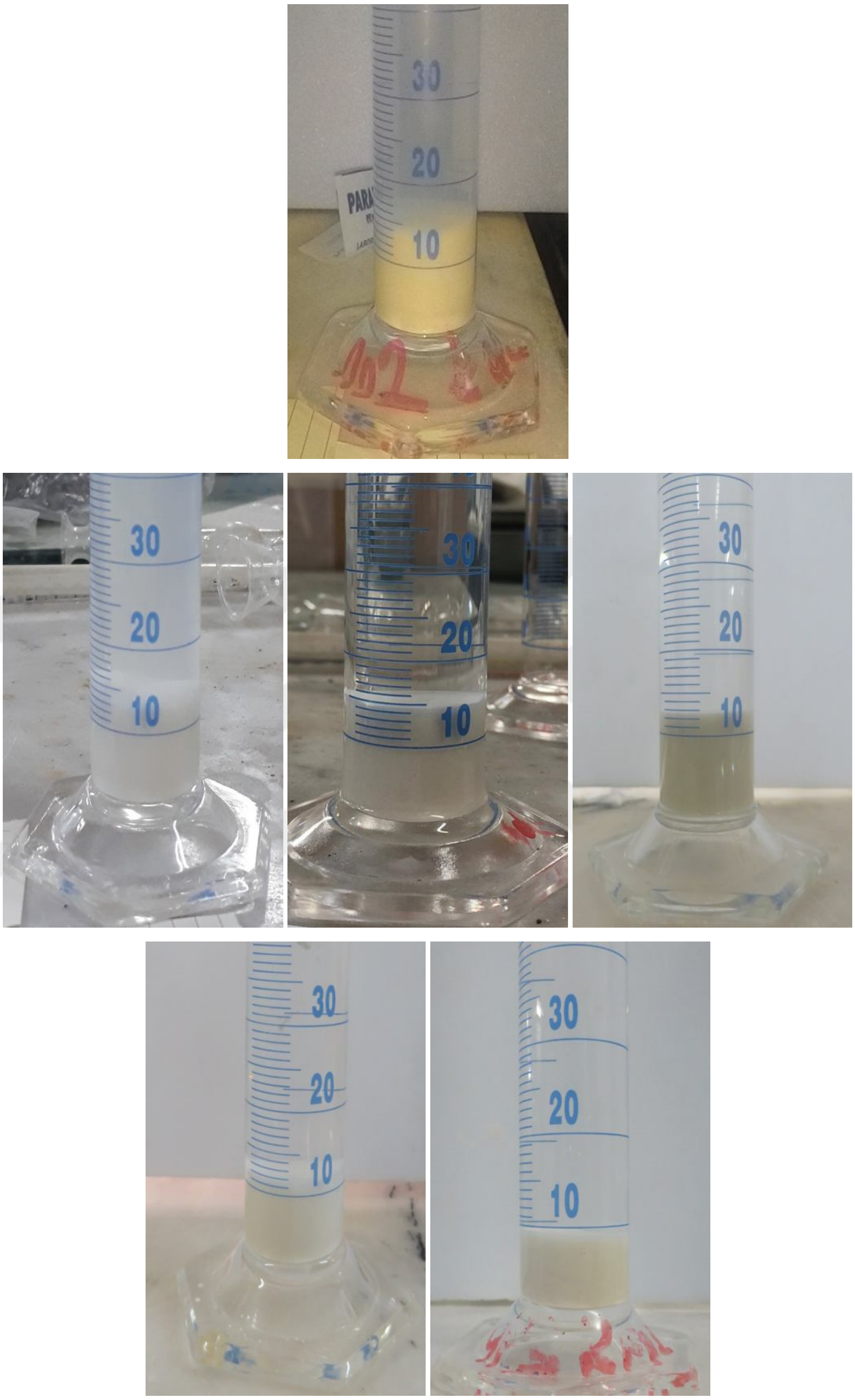


Figure A-7. Swell index of B-2 in: a) Deionized water (DIW), b) 10 mM NaCl, c) 20 mM NaCl d) 30 mM NaCl, e) 5 mM $CaCl_2$, f) 10 mM $CaCl_2$ (Personal archive, 2019)

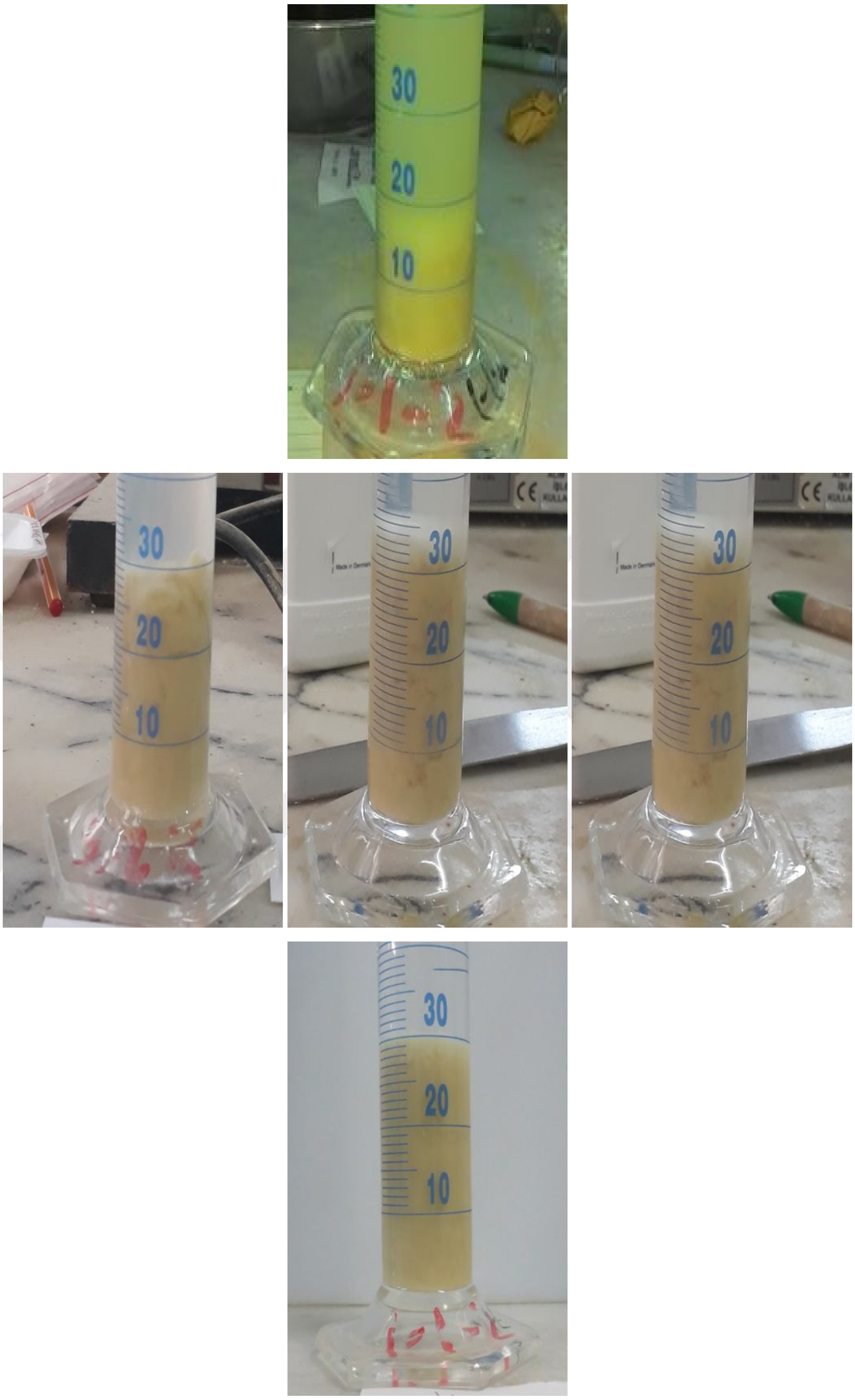


Figure A-8. Swell index of B-3 in: a) Deionized water (DIW), b) 10 mM NaCl, c) 20 mM NaCl d) 30 mM NaCl, e) 10 mM $CaCl_2$ (Personal archive, 2019)

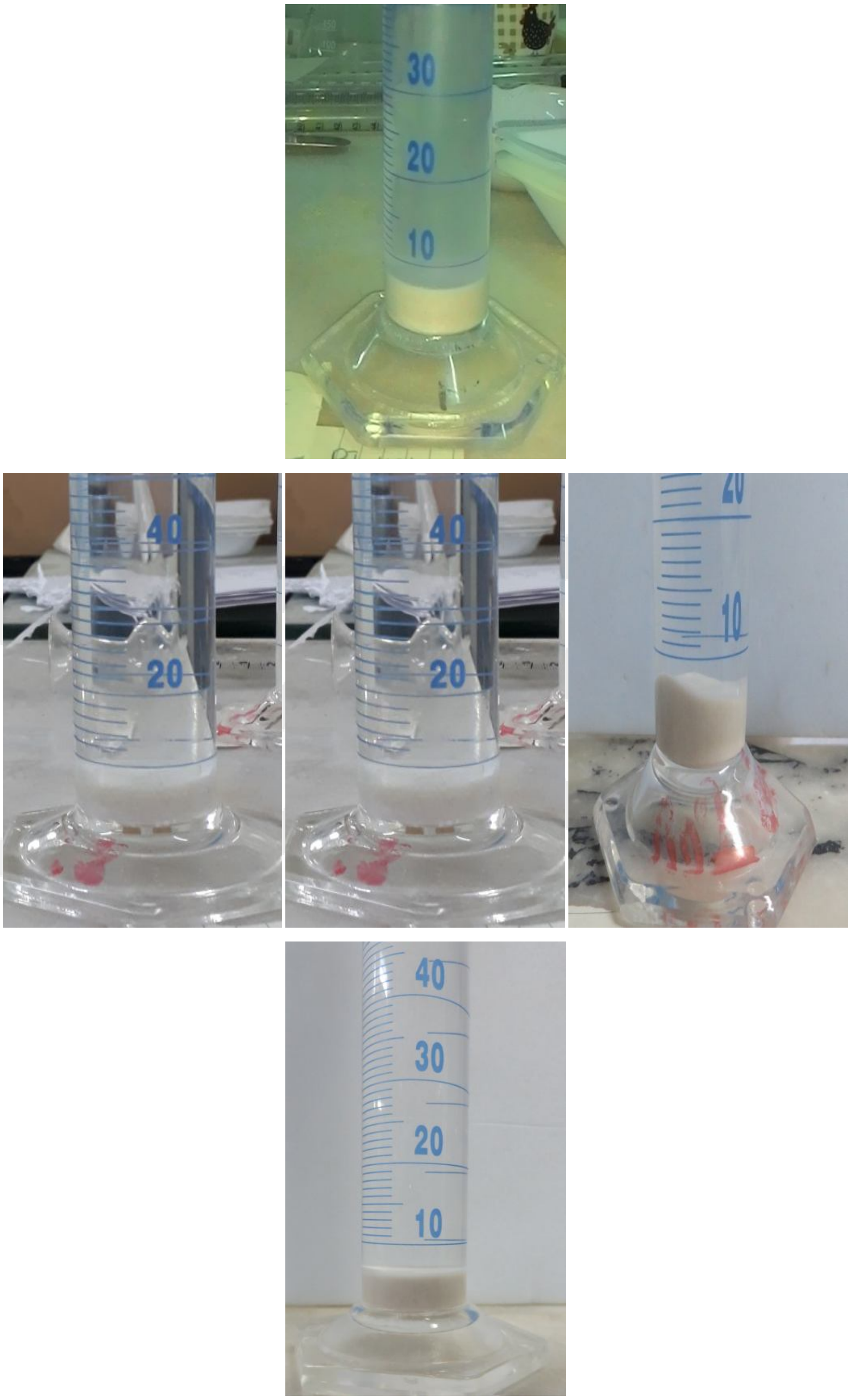


Figure A-9. Swell index of B-4 in: a) Deionized water (DIW), b) 10 mM NaCl c) 20 mM NaCl, d) 30 mM NaCl, e) 10 mM $CaCl_2$ (Personal archive, 2019)

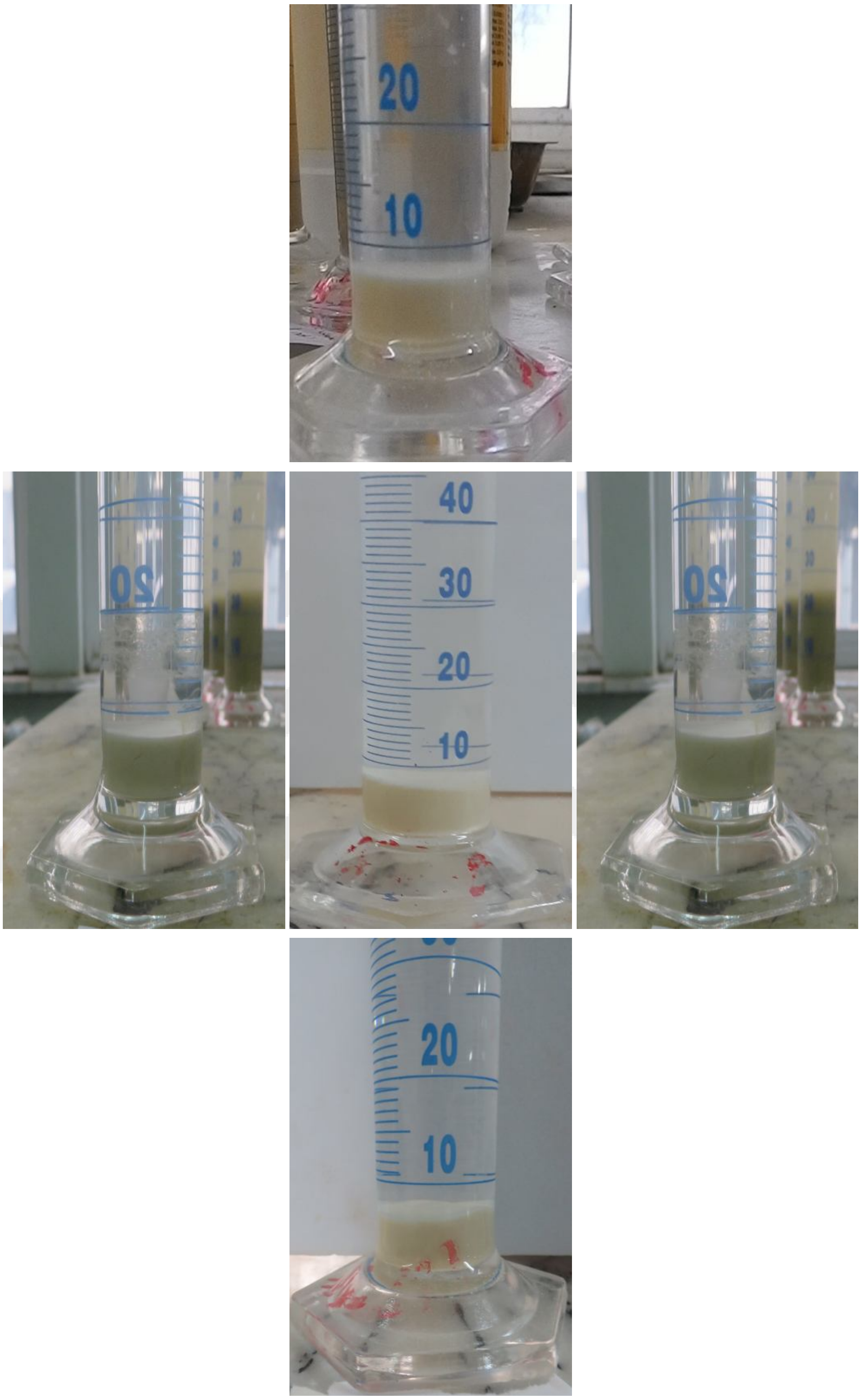


Figure A-10. Swell index of B-5 in: a) Deionized water (DIW), b) 10 mM NaCl c) 20 mM NaCl d) 30 mM NaCl, e) 5 mM $CaCl_2$, f) 10 mM $CaCl_2$ (Personal archive, 2019)

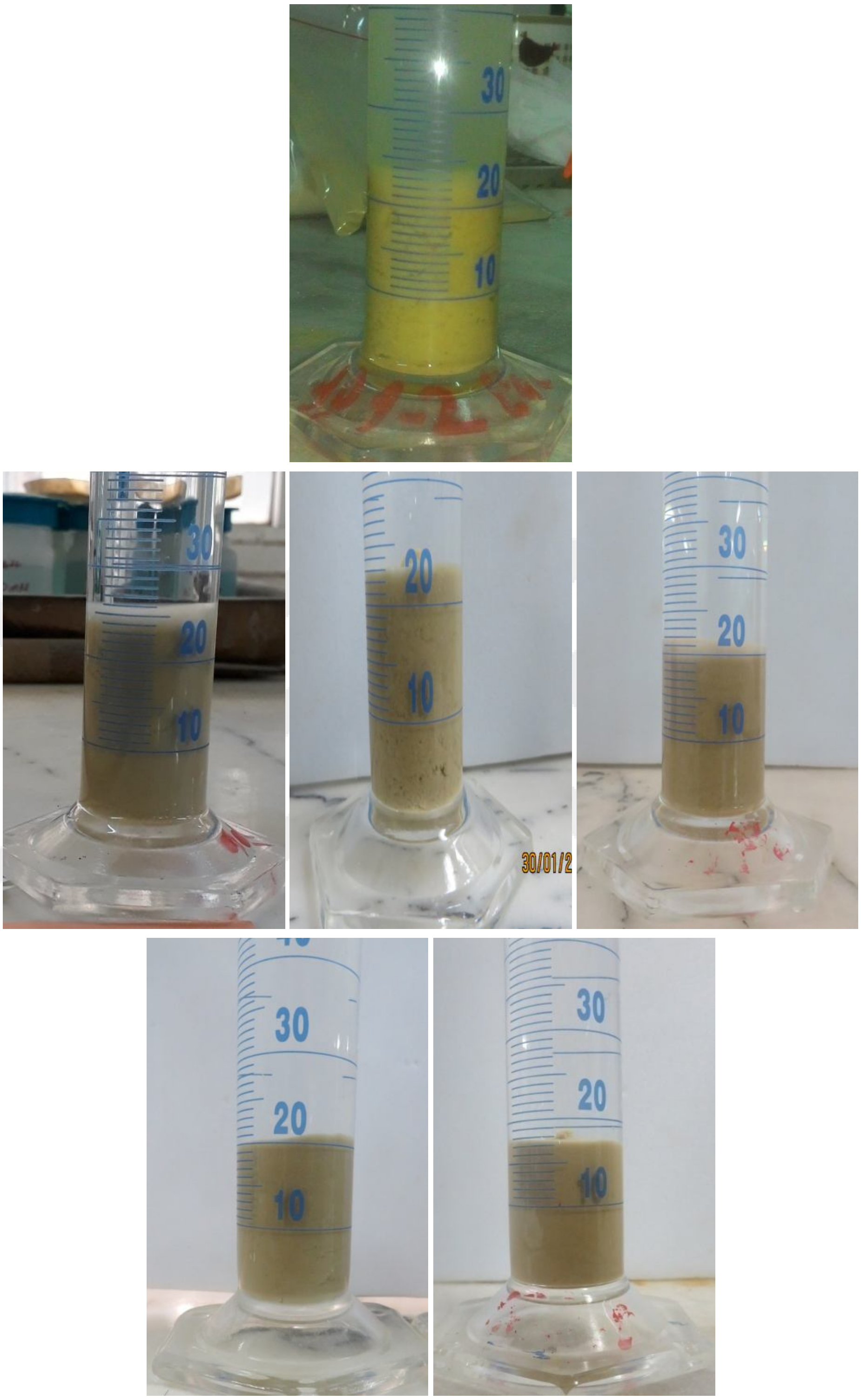


Figure A-11. Swell index of B-6 in: a) Deionized water (DIW), b) 10 mM NaCl c) 20 mM NaCl d) 30 mM NaCl, e) 5 mM $CaCl_2$, f) 10 mM $CaCl_2$ (Personal archive, 2019)

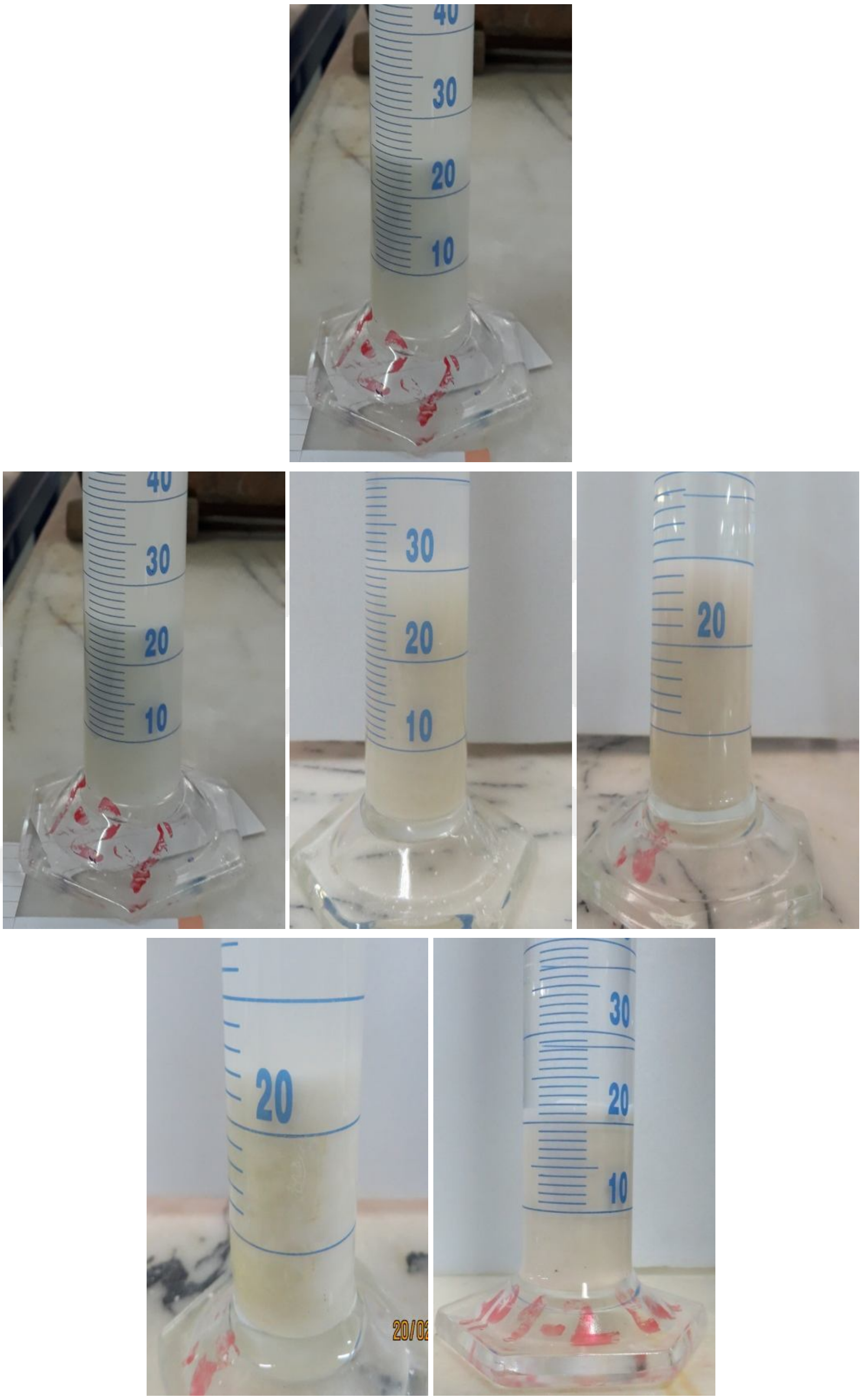


Figure A-12. Swell index of B-7 in: a) Deionized water (DIW), b) 10 mM NaCl, c) 20 mM NaCl d) 30 mM NaCl, e) 5 mM $CaCl_2$, f) 10 mM $CaCl_2$ (Personal archive, 2019)

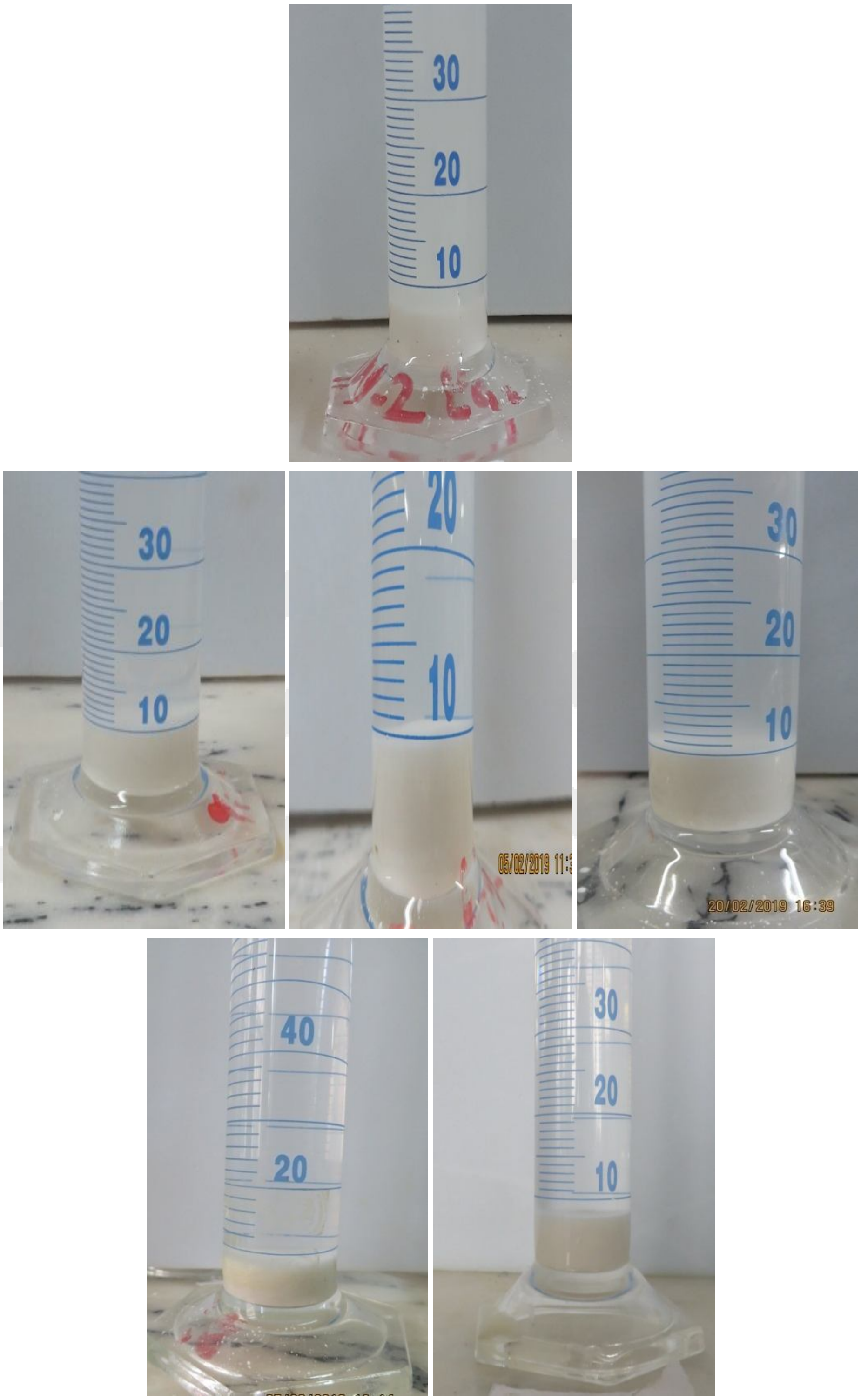


Figure A-13. Swell index of B-8 in: a) Deionized water (DIW), b) 10 mM NaCl, c) 20 mM NaCl d) 30 mM NaCl, e) 5 mM $CaCl_2$, f) 10 mM $CaCl_2$ (Personal archive, 2019)

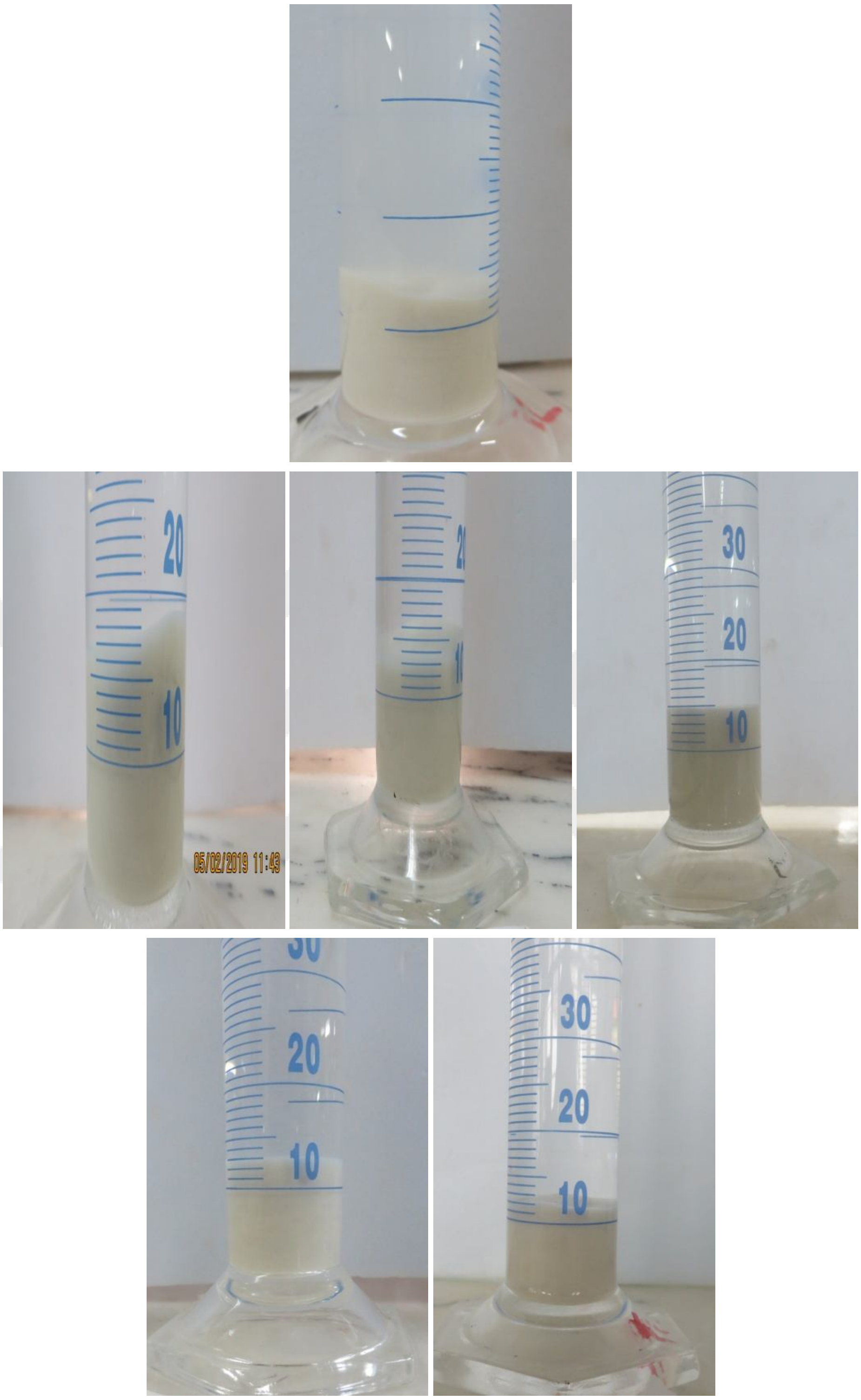


Figure A-14. Swell index of B-9 in: a) Deionized water (DIW), b) 10 mM NaCl c) 20 mM NaCl d) 30 mM NaCl, e) 5 mM $CaCl_2$, f) 10 mM $CaCl_2$ (Personal archive, 2019)

APPENDIX-B: Sediment Volume Versus Time Graph for Each Bentonite

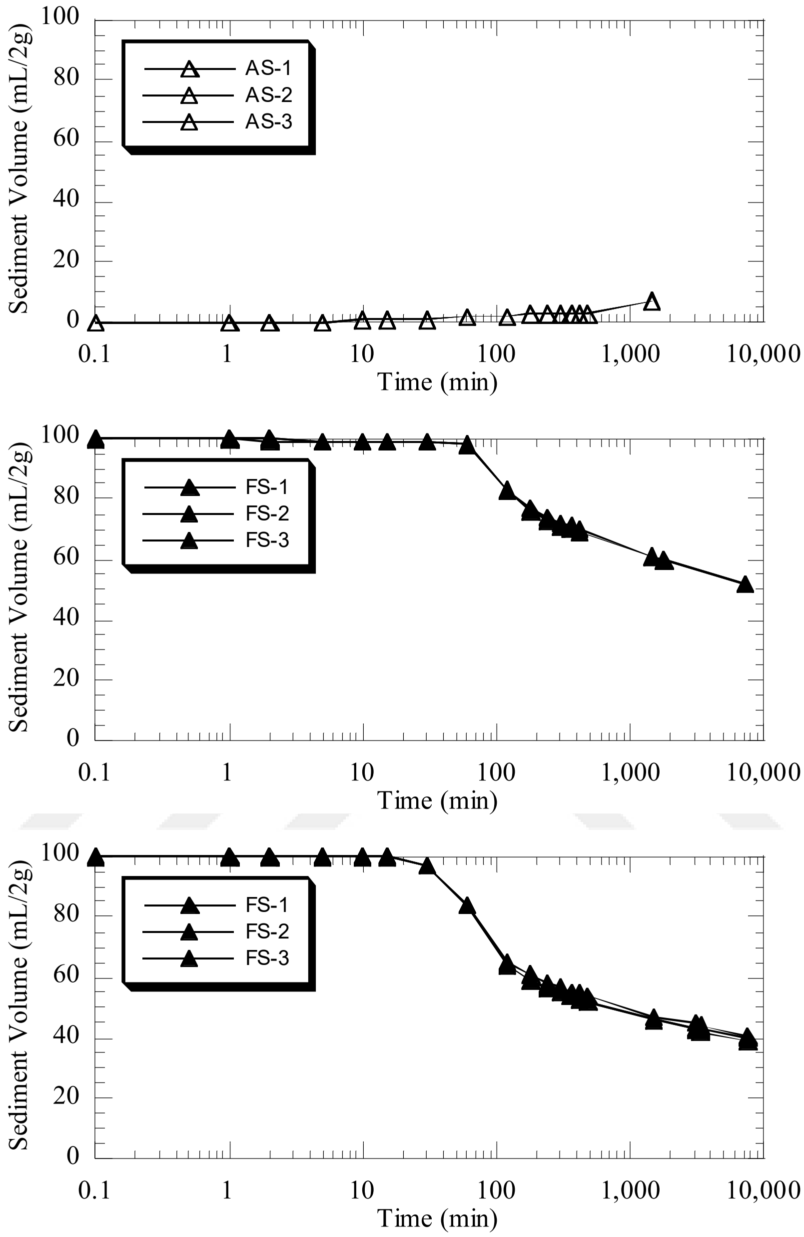


Figure B-1. Sediment volume versus time graph for GCL-1 in: a) Deionized water (DIW), b) 10 mM NaCl, c) 20 mM NaCl, d) 30 mM NaCl, e) 5 mM $CaCl_2$, f) 10 mM $CaCl_2$

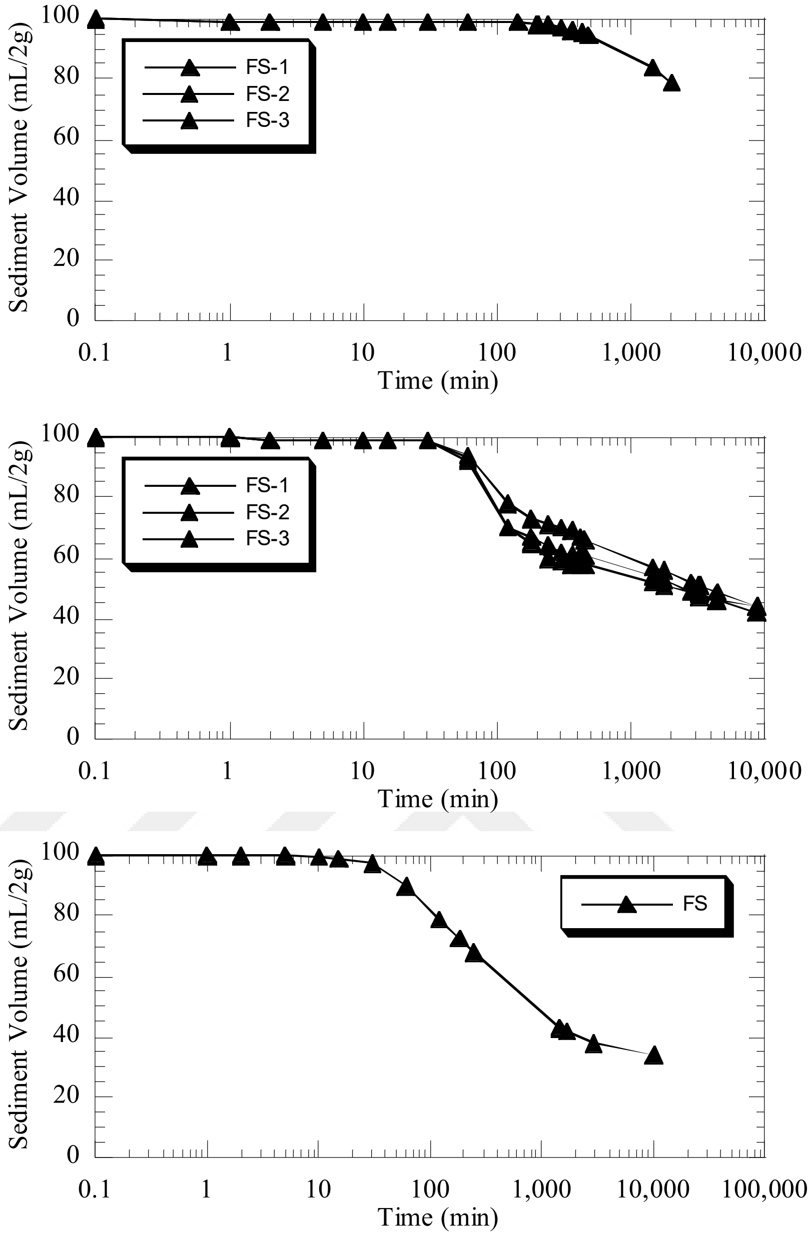


Figure B-1. continues

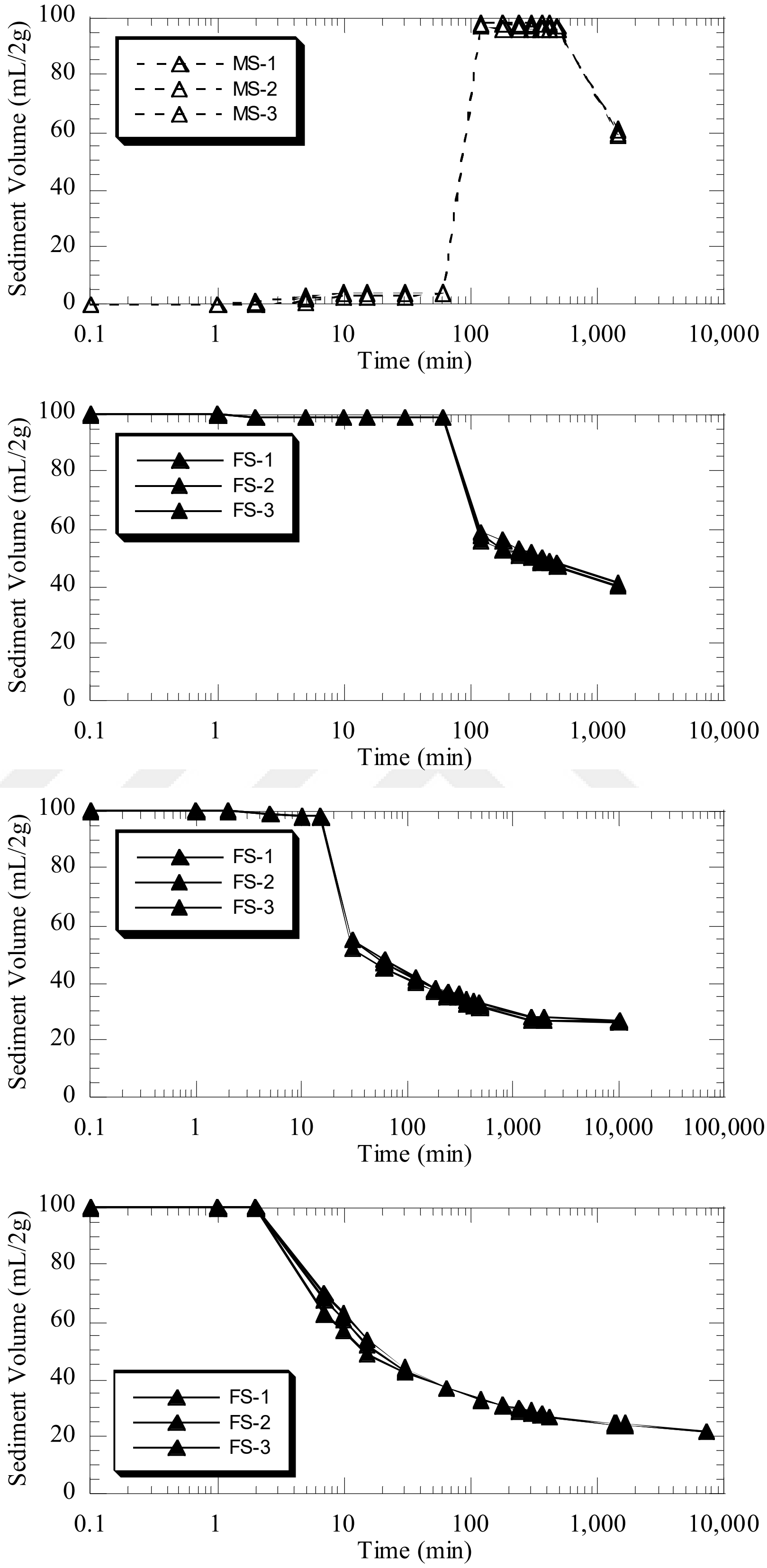
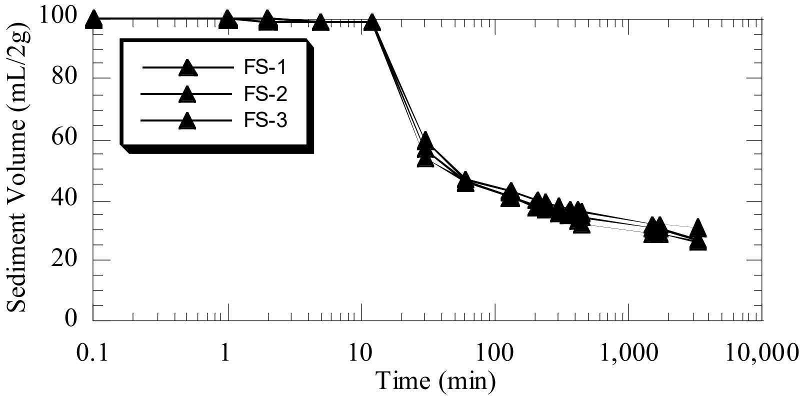


Figure B-2. Sediment volume versus time graph for GCL-2 in: a) Deionized water (DIW), b) 10 mM NaCl, c) 20 mM NaCl, d) 30 mM NaCl, e) 5 mM $CaCl_2$, f) 10 mM $CaCl_2$



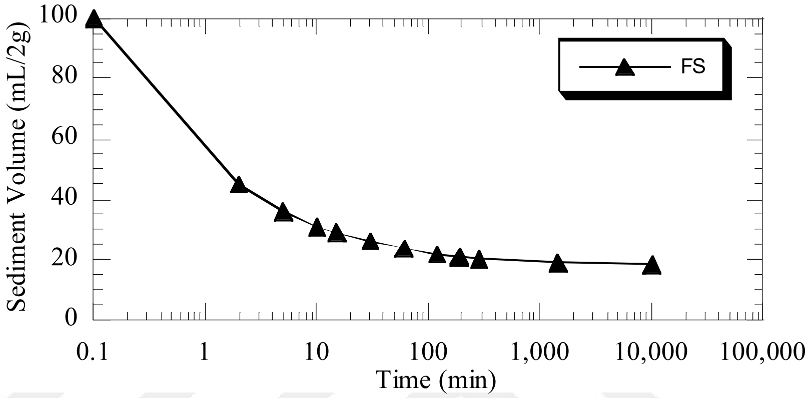


Figure B-2. continues

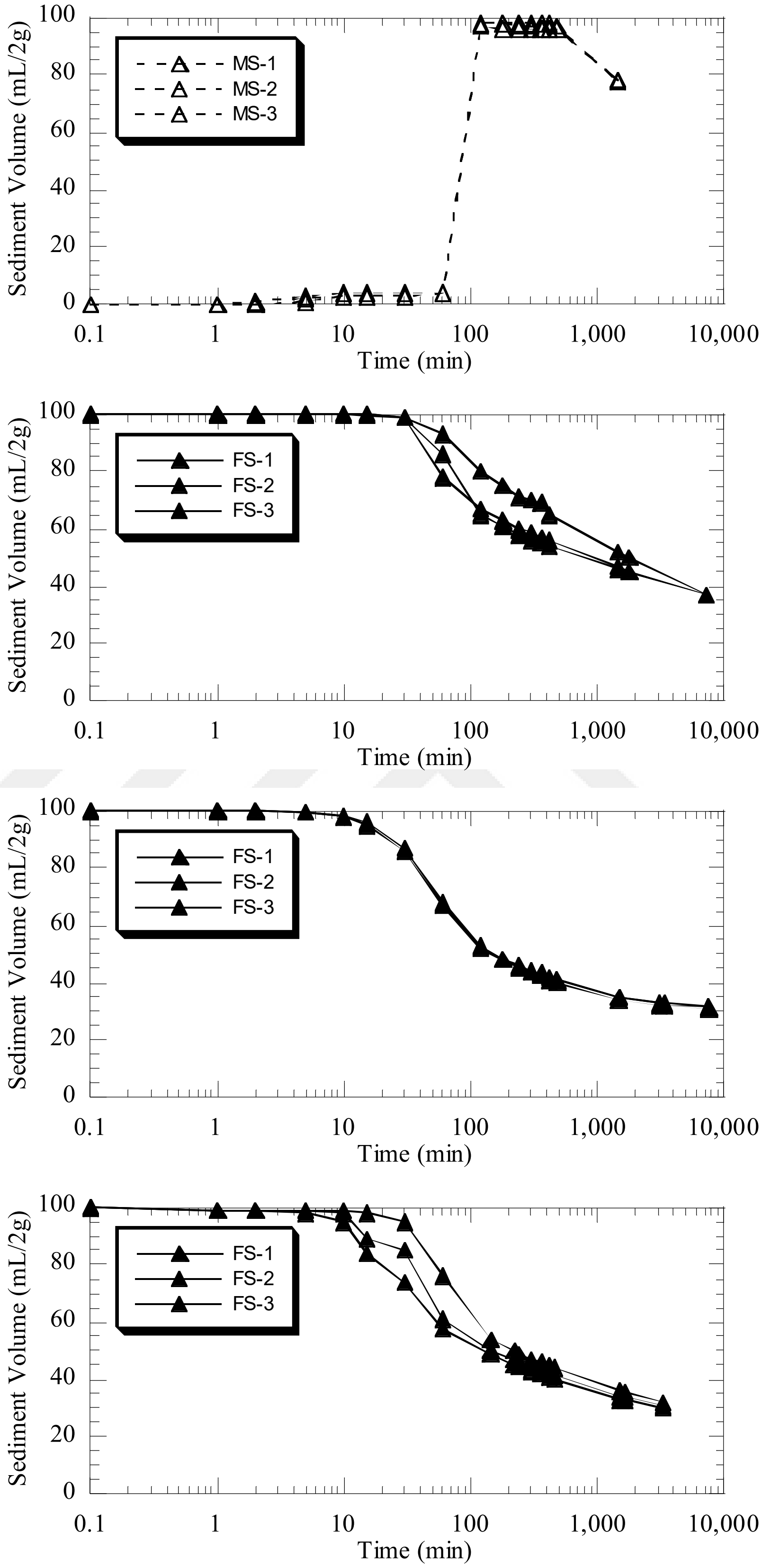


Figure B-3. Sediment volume versus time graph for GCL-3 in: a) Deionized water (DIW), b) 10 mM NaCl, c) 20 mM NaCl, d) 5 mM $CaCl_2$, e) 10 mM $CaCl_2$

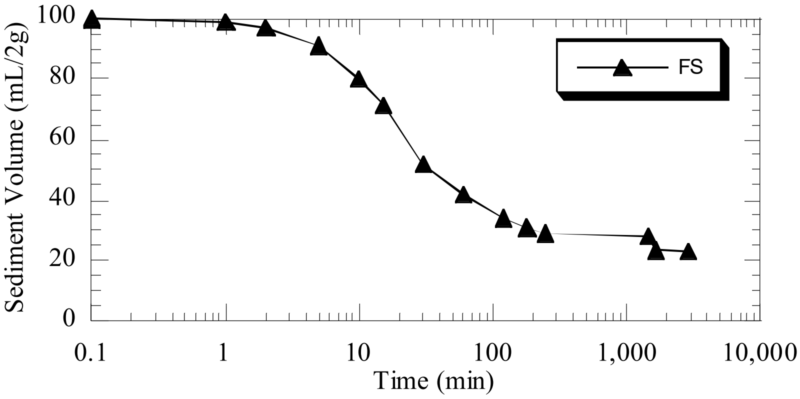


Figure B-3. continues

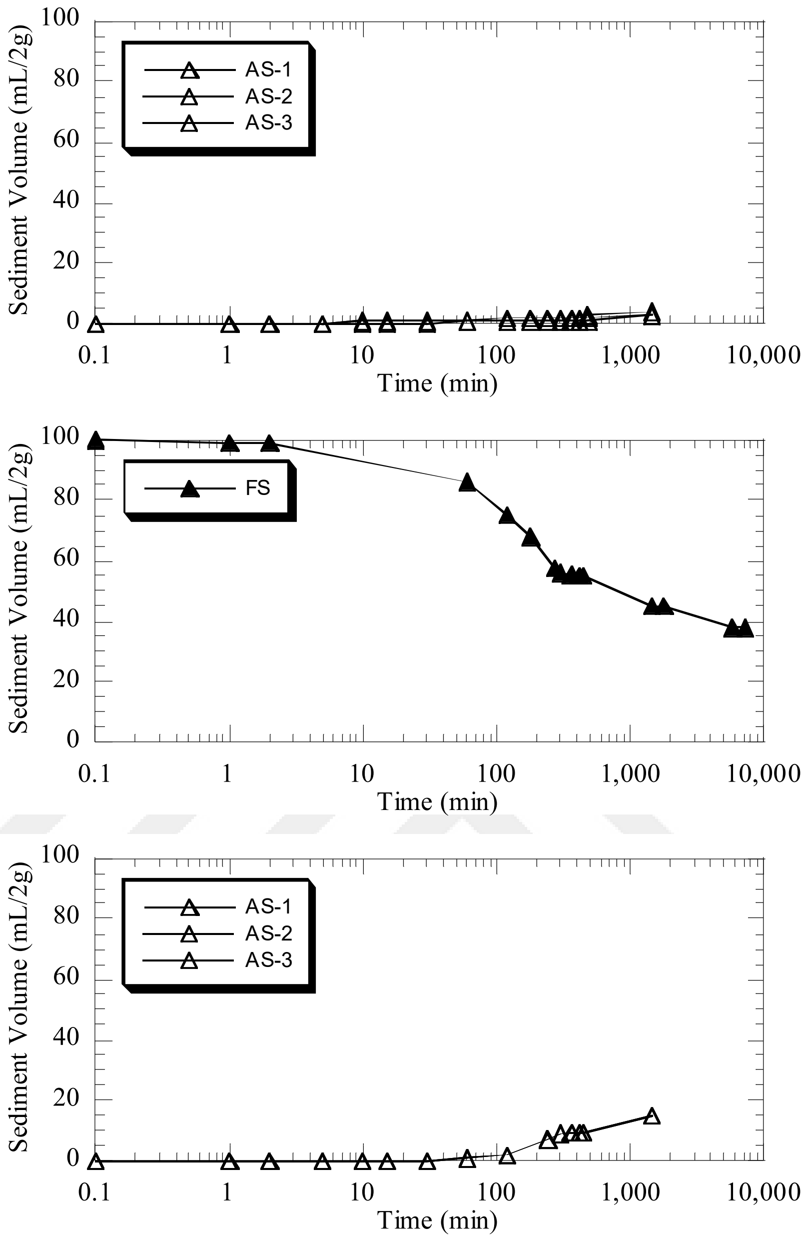


Figure B-4. Sediment volume versus time graph for GCL-4 in: a) Deionized water (DIW), b) 30 mM NaCl, c) 5 mM $CaCl_2$

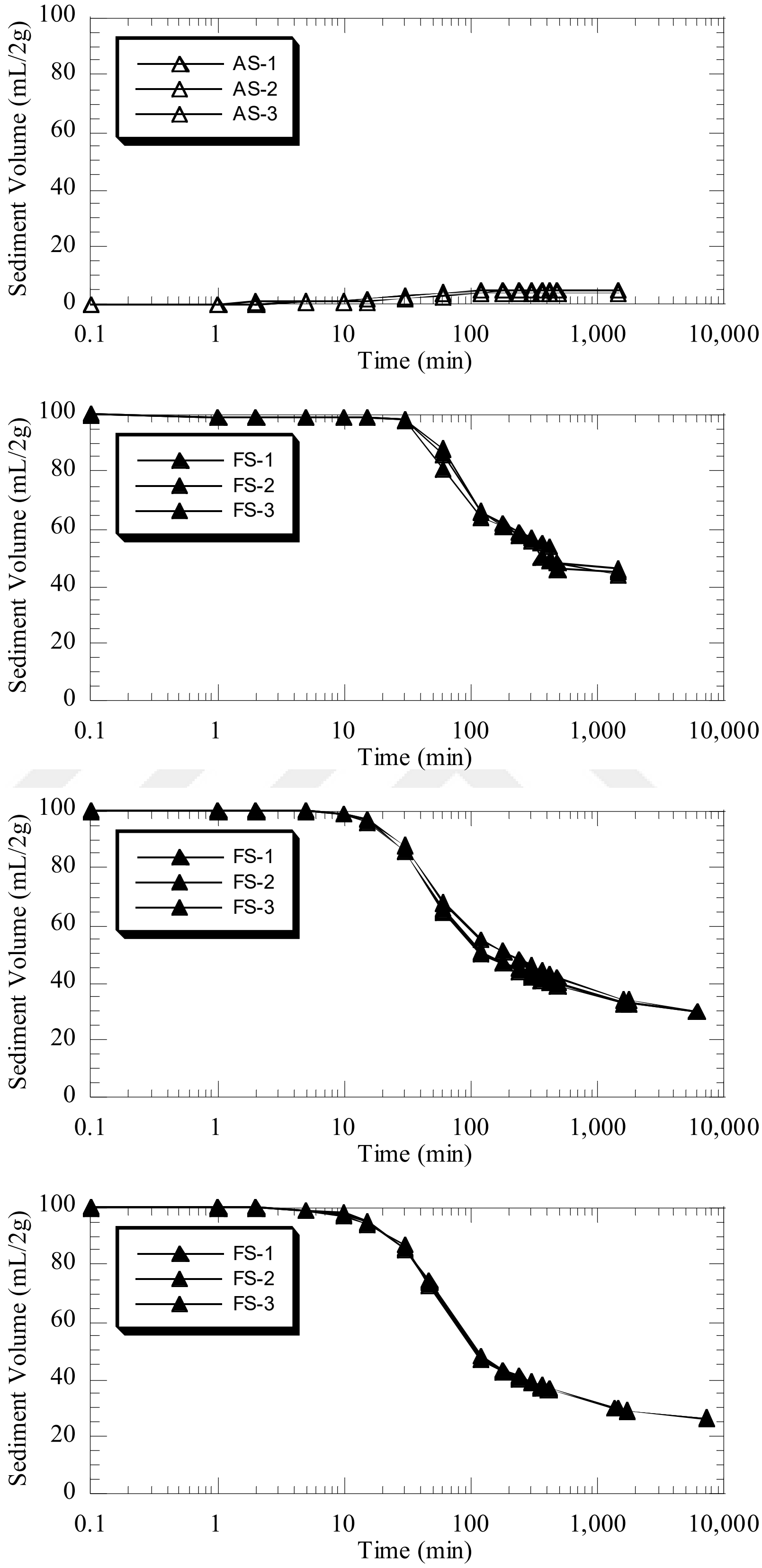


Figure B-5. Sediment volume versus time graph for GCL-5 in: a) Deionized water (DIW), b) 10 mM NaCl, c) 20 mM NaCl, d) 30 mM NaCl, e) 5 mM $CaCl_2$, f) 10 mM CaCl

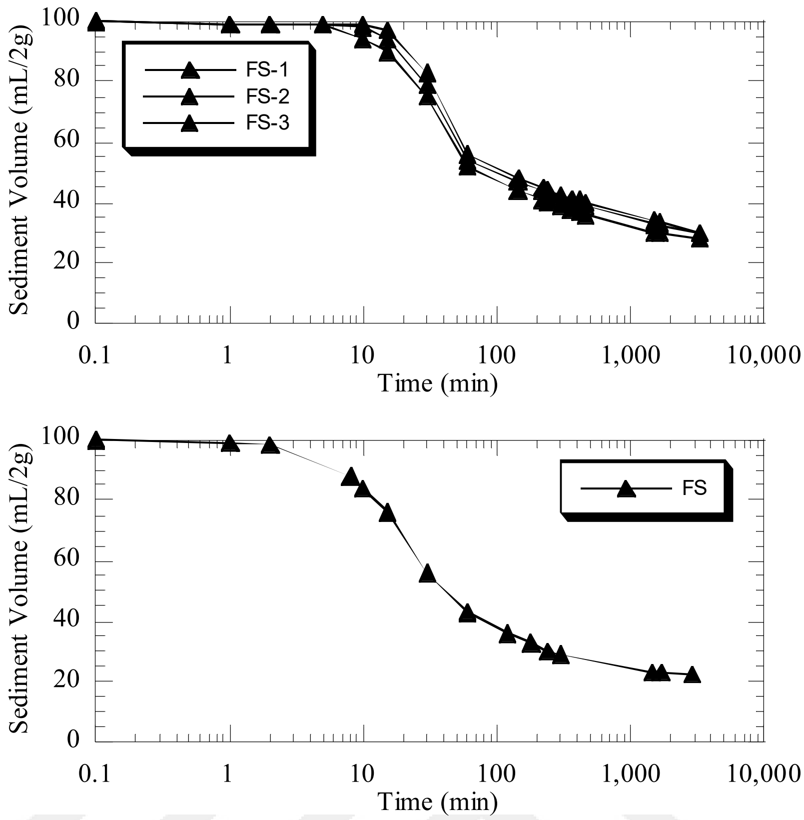


Figure B-5. continues

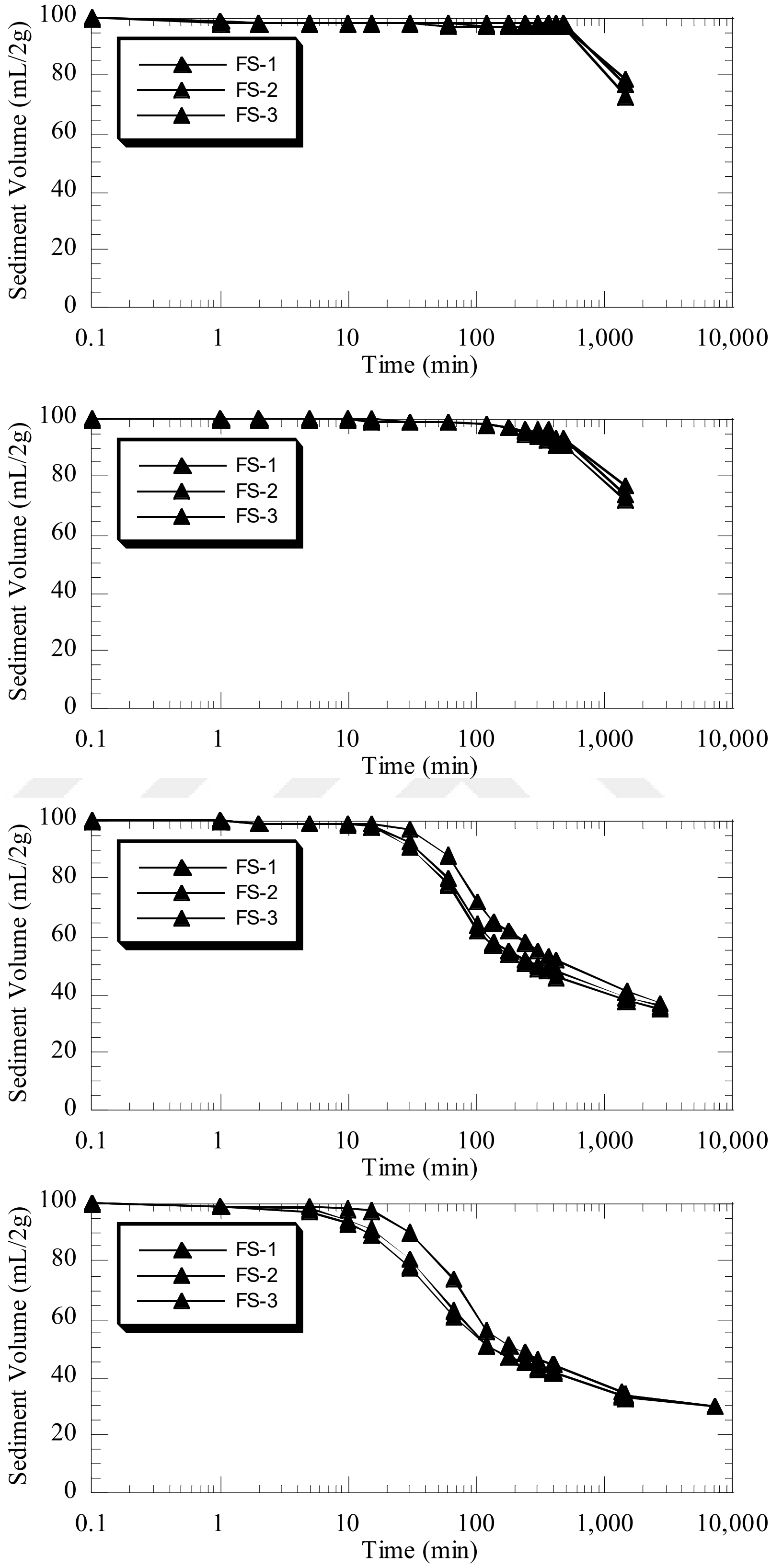
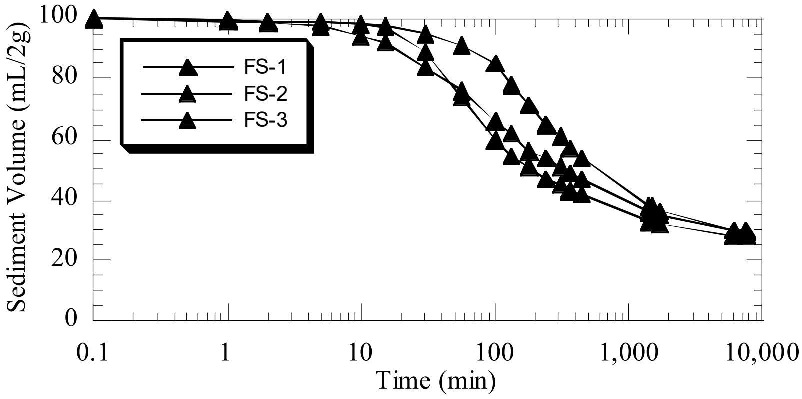


Figure B-6. Sediment volume versus time graph for B-1 in: a) Deionized water (DIW), b) 10 mM NaCl, c) 20 mM NaCl, d) 30 mM NaCl, e) 5 mM $CaCl_2$, f) 10 mM $CaCl_2$



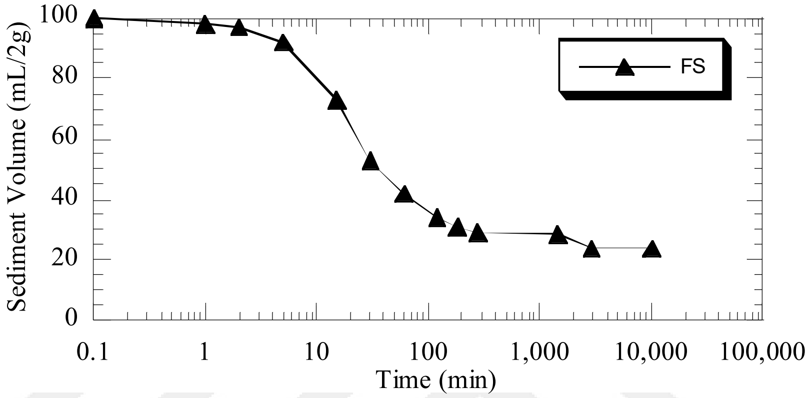


Figure B-6. continues

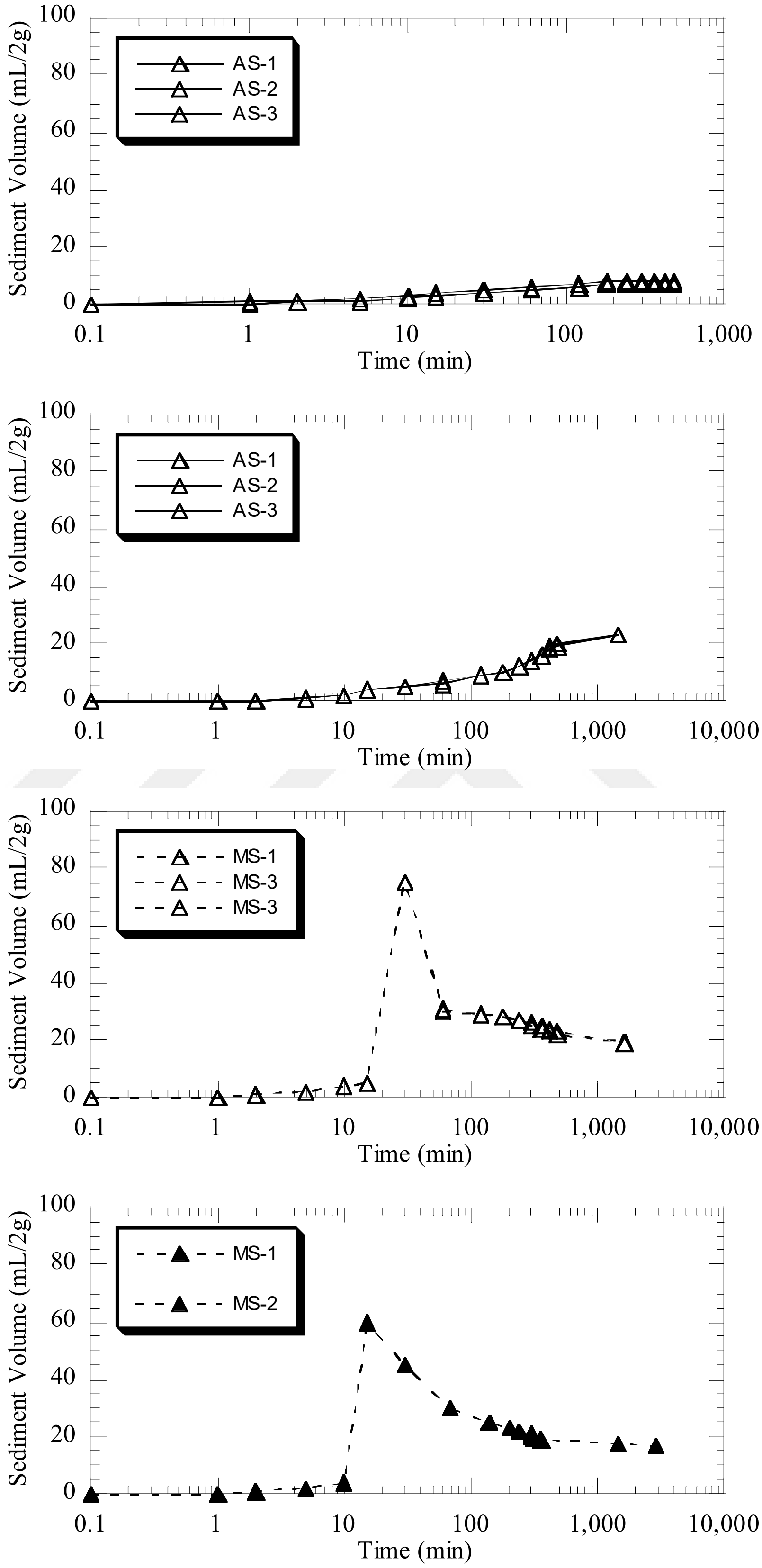


Figure B-7. Sediment volume versus time graph for B-2 in: a) Deionized water (DIW), b) 10 mM NaCl, c) 20 mM NaCl, d) 30 mM NaCl, e) 5 mM $CaCl_2$, f) 10 mM C

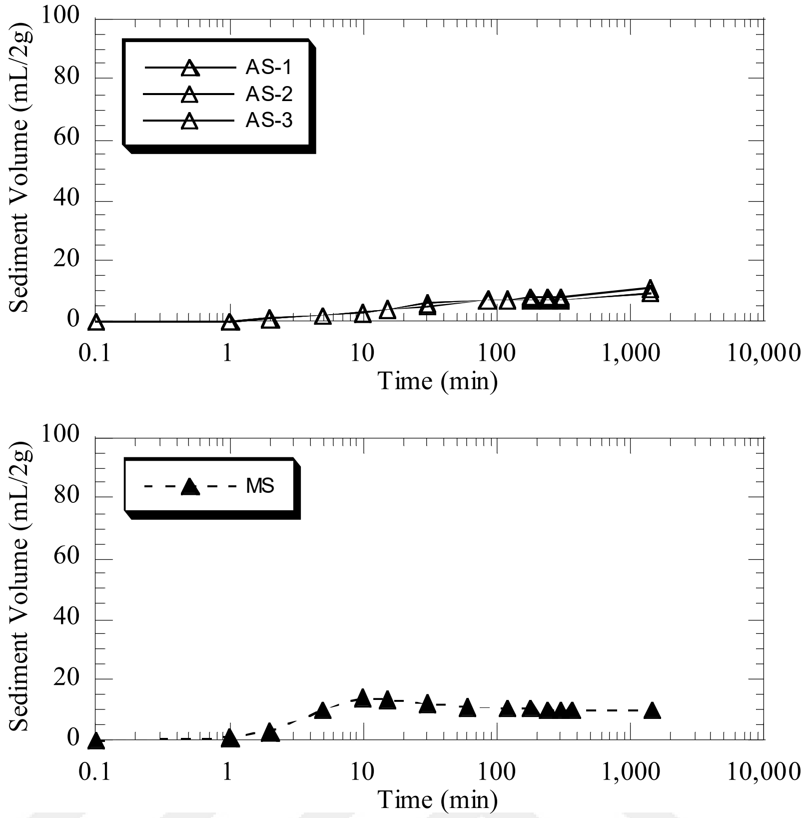


Figure B-7. continues

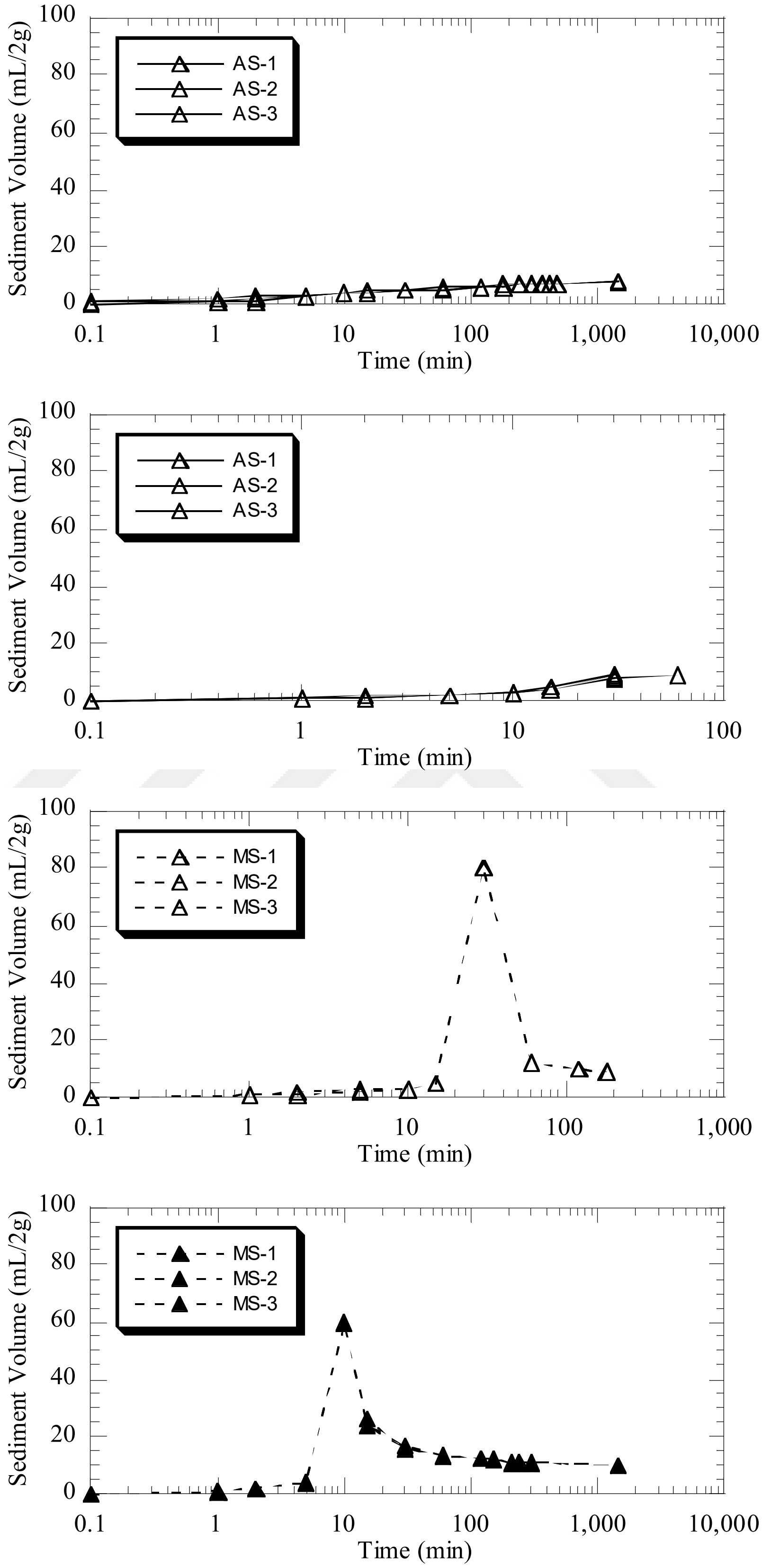
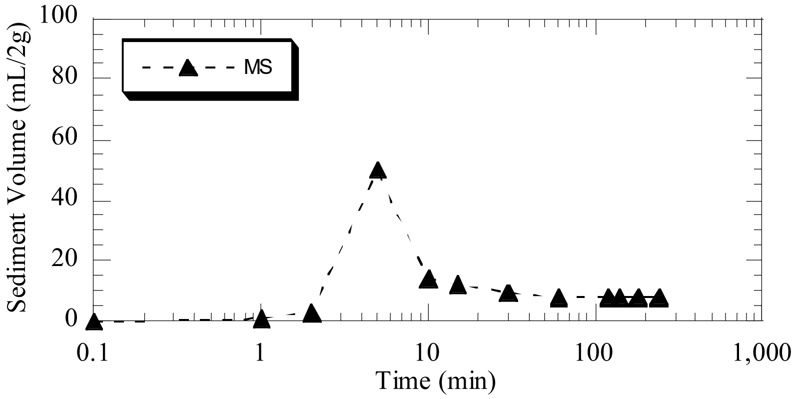


Figure B-8. Sediment volume versus time graph for B-4 in: a) Deionized water (DIW), b) 10 mM NaCl, c) 20 mM NaCl, d) 30 mM NaCl, e) 10 mM $CaCl_2$



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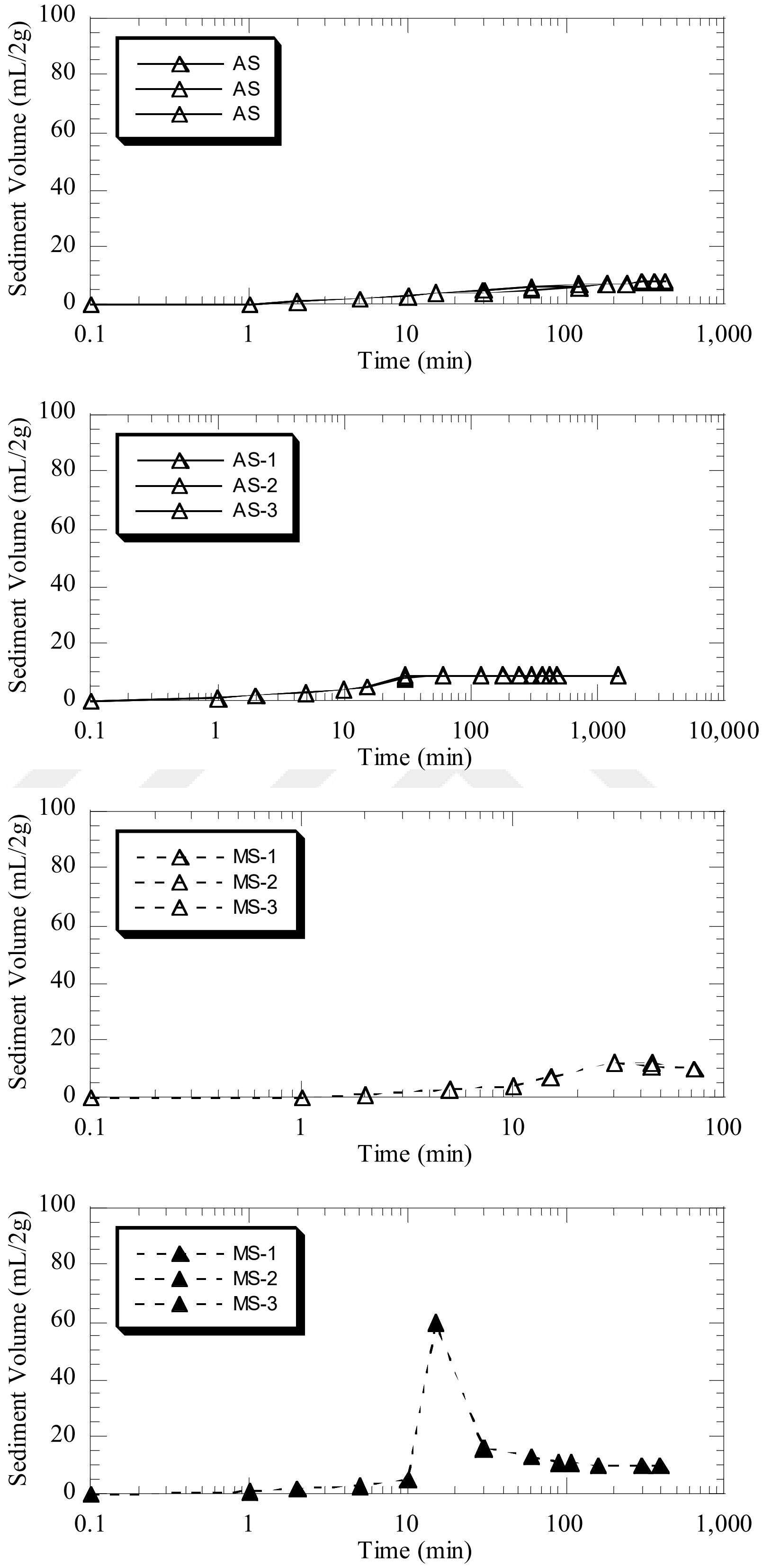


Figure B-9. Sediment volume versus time graph for B-5 in: a) Deionized water (DIW), b) 10 mM NaCl, c) 20 mM NaCl, d) 30 mM NaCl, e) 5 mM $CaCl_2$

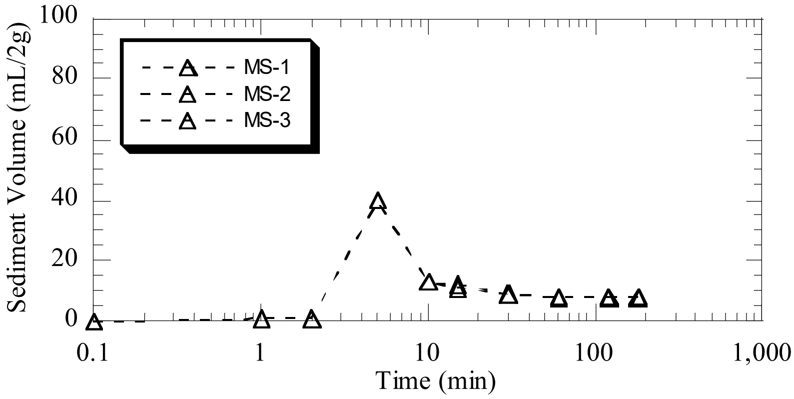


Figure B-9. continues

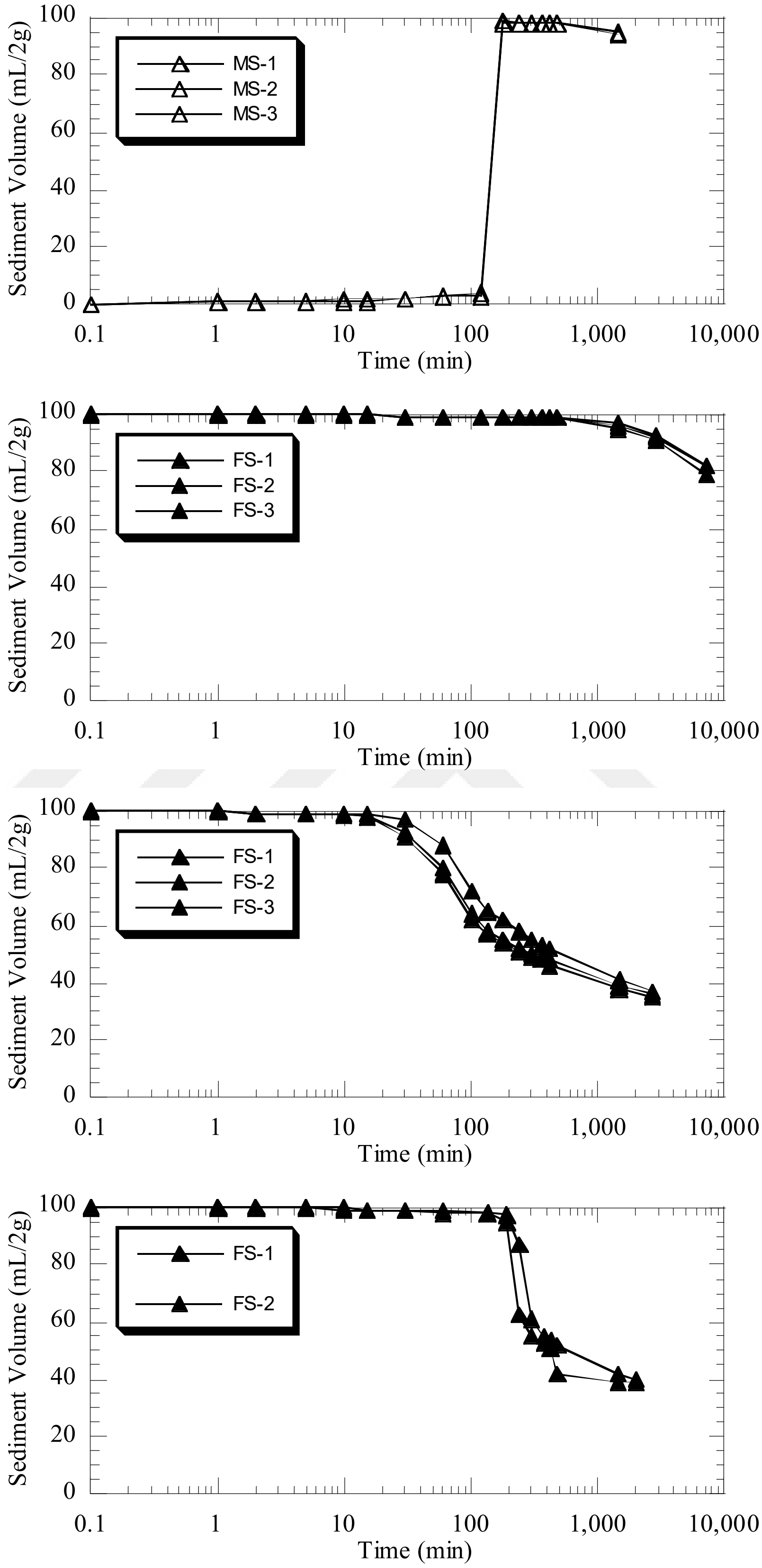
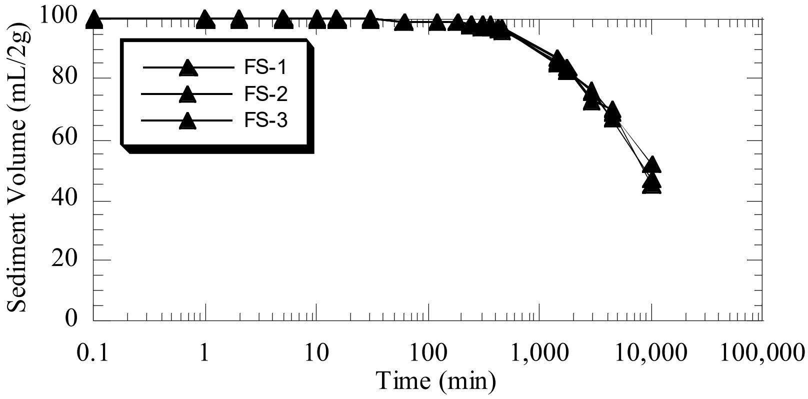
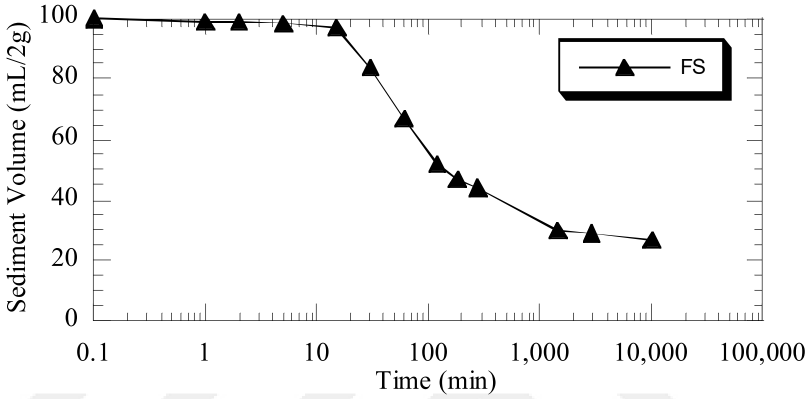


Figure B-10. Sediment volume versus time graph for B-6 in: a) Deionized water (DIW), b) 10 mM NaCl, c) 20 mM NaCl, d) 30 mM NaCl, e) 5 mM $CaCl_2$, f) 10 mM $CaCl_2$





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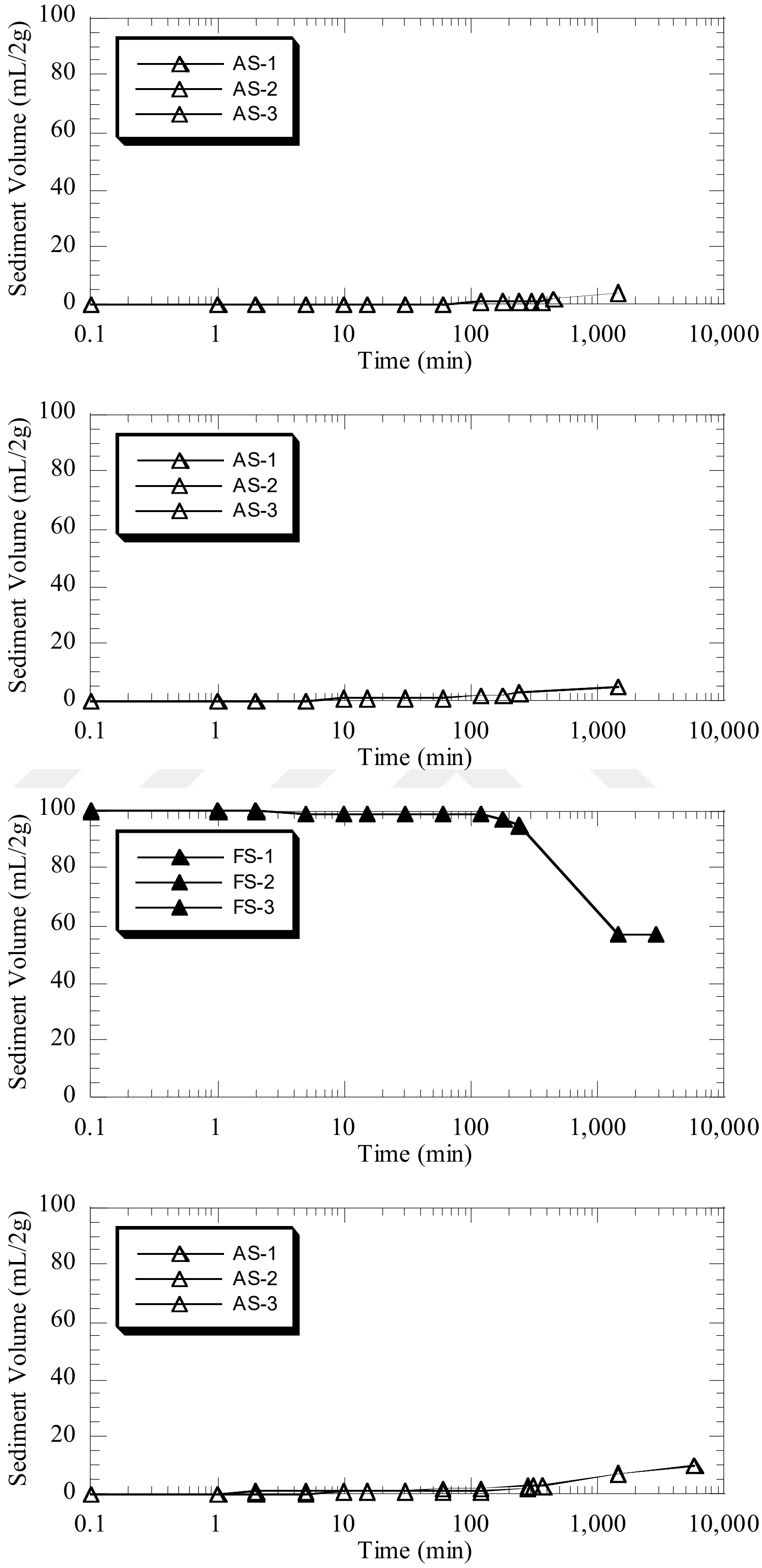


Figure B-11. Sediment volume versus time graph for B-7 in: a) Deionized water (DIW), b) 10 mM NaCl, c) 20 mM NaCl, d) 5 mM $CaCl_2$, e) 10 mM $CaCl_2$

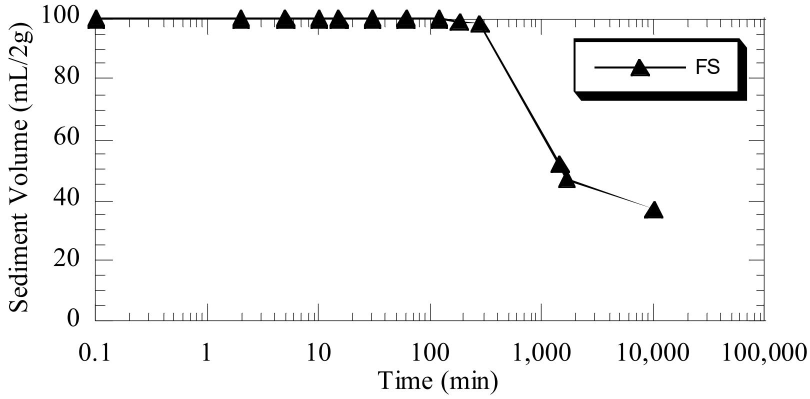


Figure B-11. continues

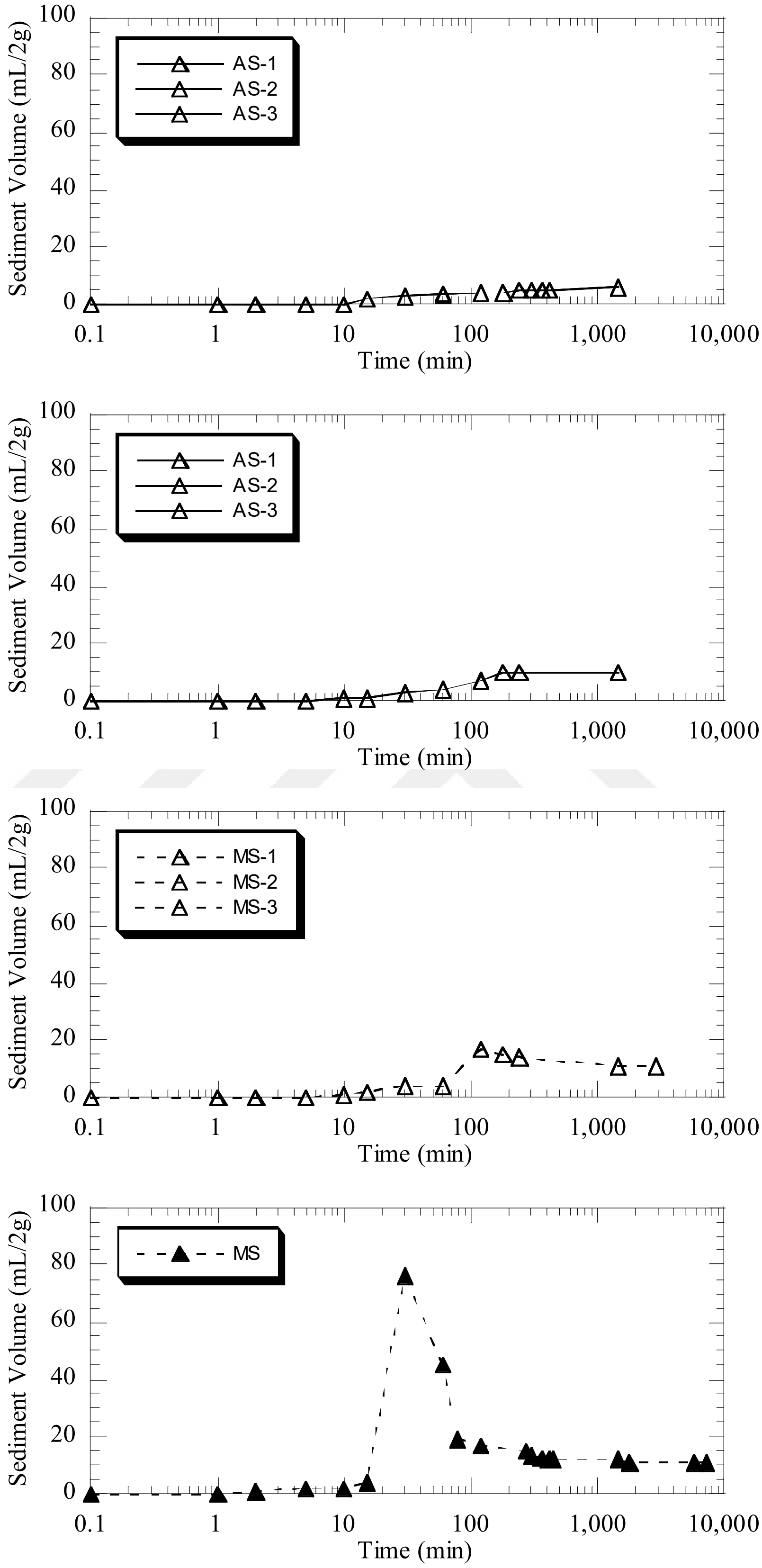
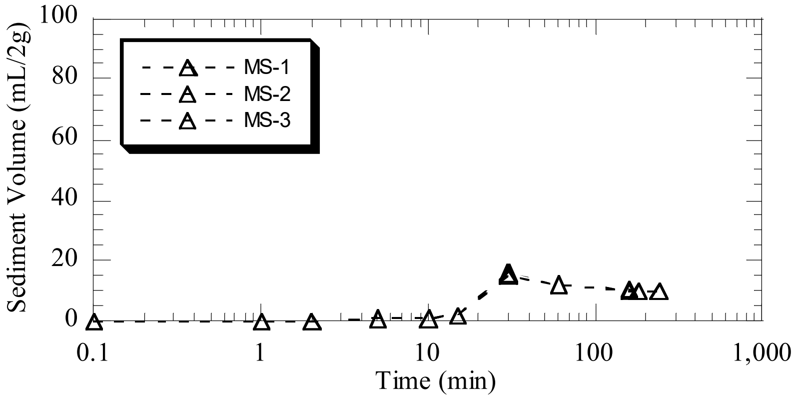
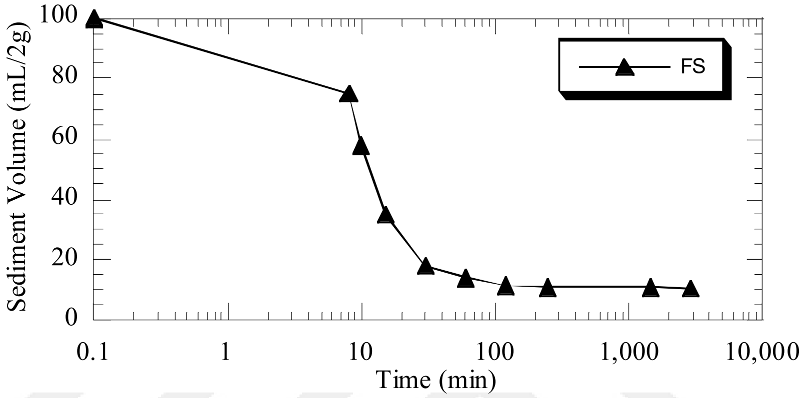


Figure B-12. Sediment volume versus time graph for B-8 in: a) Deionized water (DIW), b) 10 mM NaCl, c) 20 mM NaCl, d) 5 mM $CaCl_2$, e) 10 mM $CaCl_2$





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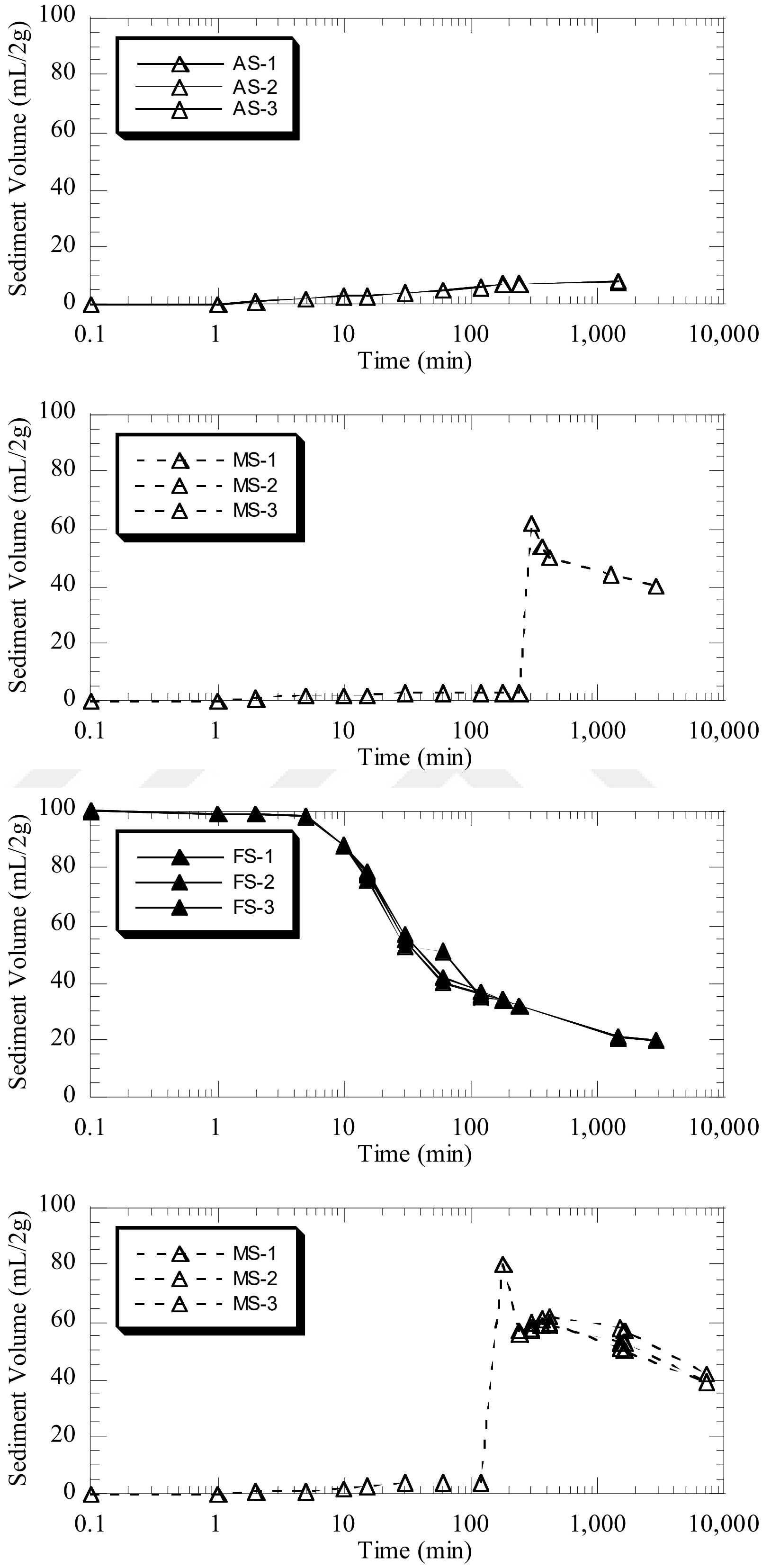
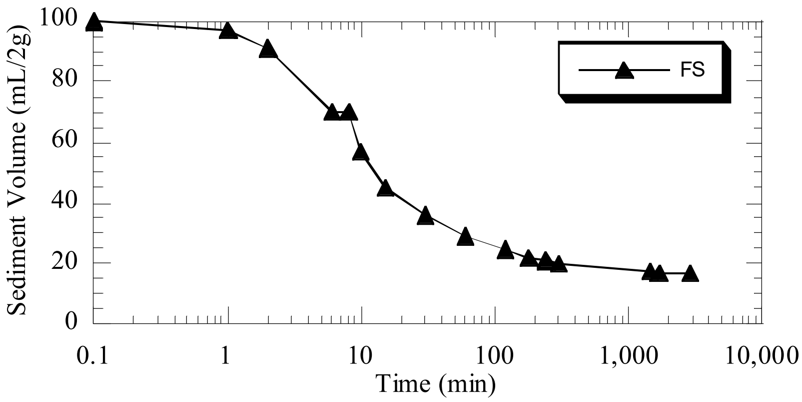


Figure B-13. Sediment volume versus time graph for B-9 in: a) Deionized water (DIW), b) 10 mM NaCl, c) 20 mM NaCl, d) 5 mM $CaCl_2$, e) 10 mM $CaCl_2$



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