Enhanced sludge dewatering and drying comparison of two linear polyelectrolytes co-conditioning with Polyaluminium chloride

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Abstract

This paper investigated the influences of Polyaluminium chloride (PAX) co-conditioning with linear polyelectrolytes on the sludge dewatering and drying performances.
Two linear polyelectrolytes were tested on sludge samples obtained from the Liege's WWTP. It was found that sludge conditioned with the couple PAX/ higher linear weight polyelectrolyte led to better dewatering results than with the lower molecular weight one.
Concerning drying, it appeared that the samples treated by the couple PAX/ polymers showed higher drying rates than samples conditioned by polymer without PAX adding, allowing a reduction of the drying time. This benefic effect was more pronounced with the high molecular weight polymer.

Keywords: Activated sludge treatment, sludge flocculation, dewatering, convective drying, X-ray microtomography, linear polymers, PAX

INTRODUCTION

According to the directive of the Council of European Communities concerning urban wastewater treatment [1, 2], municipalities will have to face with growing amounts of wastewater sludges. At the same time, the directive on waste landfill [3] has planned the progressive reduction of sludge disposal in dump sites until 2016 [4, 5]. At the present time, two major issues are used for sludge disposal: energy valorization through incineration and agriculture valorization through landspreading [5]. Excess sludge which is produced by the biological treatment of wastewater still contains more than 99% of water at the bottom of thickeners. Before being valorized, sludge has thus to be dewatered and, more and more often thermally dried. Sludge is a colloidal system in which small sludge particles form a stable suspension in water, making them very difficult to be separated from the water phase. To overcome this problem, the addition of chemical conditioners such as flocculants and/ or coagulants is often necessary to help the sludge particles to agglomerate into larger settleable flocs prior to solid-water separation usually by mechanical dewatering. Polyelectrolytes are often used to induce the formation of flocculated particle networks their most important characteristics are average molecular weight [6, 7] and charge density [8].
Depending on the dewatering technique, the so-called sludge cake reaches around 15 to 35 % DS. Thermal drying can then be used to remove totally or partially the remaining water, depending on sludge final use. This obviously reduces the mass and volume of waste and, consequently, the cost for storage, handling and transport. The removal of water to such a low level increases drastically the lower calorific value, transforming the sludge into an acceptable combustible.

Conditioning, dewatering and drying cannot be seen as independent steps. Indeed, some wastewater treatment plant managers have observed that, in some cases, the shear stresses underwent by the sludge in centrifuges will alter its drying behavior. Overdosage of conditioning polymer has also been referred to induce drying slowing down.
Furthermore, the strong decrease of the drying rate was obtained when the sludge was destructured due to pumping [9]. As thermal drying is highly energy consuming, this process still needs to be optimized [10] but

considering the whole chain effect including conditioning and dewatering. As just stated before, this effect is known to exist but there is a real lack of scientific papers concerning this issue [11, 12, 13].
In this context, the aim of this work was to study experimentally the influence of Polyaluminium chloride coagulant (PAC or PAX) co-conditioning with two types of linear polymers on dewatering performances and subsequent convective drying behaviour. The effect of wastewater sludge conditioning with PAX was proposed to become a technically feasible and very effective method to enhance sludge dewaterability and to avoid fouling problems during the course of drying [14].

MATERIALS AND METHODS

EXPERIMENTAL design

An experimental design was used to investigate the effect of input factors on a response variable. To have a better understanding of the dosing effect, five points of experiments were carried out in order to study the effect of Polyaluminium chloride coagulant combined to polyelectrolyte chemical on both the sludge dewatering process and drying behavior. The polyelectrolyte dose range, which covers the underdosing to overdosing area for the two polyelectrolytes ranges from the half to the double gram polyelectrolyte per kilogram of sludge dry matter, from the optimum dosage determined using CST (see below).
Concerning Polyaluminium chloride coagulant, a dose ranging above 8 g PAX/ kg_{DS} was shown to be ineffective, that permit to investigate the range below this value.
The tests were performed with sludge samples taken in the same week, stored in a vessel at room temperature of 25 °C and under continuous gentle stirring. The day 1 served to determine the optimum dosage prior to the measurement campaign by CST. The trials were performed in two weeks in random sequence, except for the central point (C) which was repeated 4 times respectively at the beginning and the end of each week which secured almost the same sludge characteristics. The range investigated for each parameter and experimental plan are presented in Table 1 and Figure 1.

Table 1. Experiments days design

MONDAY	CST [s]
TUESDAY	C_1
WEDNESDAY	A
THURSDAY	B
FRIDAY	C_2
MONDAY	CST [s]
TUESDAY	C_3
WEDNESDAY	D
THURSDAY	E
FRIDAY	C_4

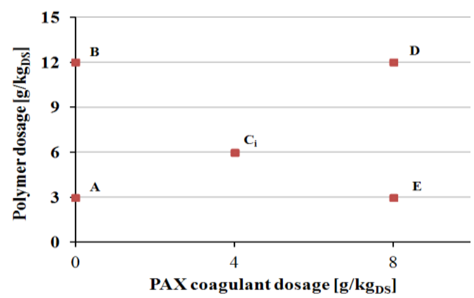


Figure 1: Design of experiments representation.

Sludge samples characteristics and conditioners

The study was performed on activated sludge samples collected after thickening from the wastewater treatment plant of the Grosses-Battes, located closed to University of Liège (Belgium). The dry solids (DS) and volatiles solids (VS) content were respectively determined by drying the wet material at 105 °C during 24 h, and then calcinated the dried residue at 550 °C during 2 hours, and weighing. Table 2 presents dry solids (DS) and volatiles solids (VS) contents characteristics, determined according Standard Methods [15]. Three replicated tests were carried out to evaluate the reliability of the experiments.
Two cationic polymers were obtained as 40 wt % active substance from the Société Nationale Française (SNF) supplier in emulsion form. The one referenced as 640 LH was a linear polymer with a low molecular weight, whereas the other (640 CT) was a linear polyelectrolyte with a high molecular weight. Both polymers were obtained with a high charge density.

These charged organic polymers were gained a large market share over the last decades in sludge treatment, since they can be dosed in much lower quantities than inorganic flocculants such as lime and iron chloride [16]. Furthermore, these organic polyelectrolytes were easily biodegradable and can be obtained at economic costs.
Concerning the Polyaluminium chloride coagulant used in this paper, it was commercially available PAX-14 from Kemira Rotterdam (basicity 26 ± 6%; density of 1.3 kg/L; Al concentration of 7.2 ± 0.3 wt %). PAX solution was characterized by the presence of the highly charged tridecameric polymer or polycation $[AlO_4Al_{12}(OH)_{24}(H_2O)_{12}]^{7+}$, in short referred to as the Al_{13} polymer [17, 18]. The Al_{13} has the so called Keggin crystal structure composed of a tetrahedral $Al(O)_4$ center surrounded by 12 octahedrally coordinated Al atoms with bridging hydroxides and water molecules.

Table 2: Sludge characteristics

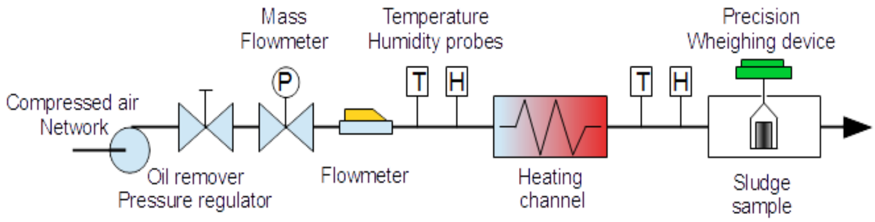
Trials points	Dry solids [%]	Volatiles solids [%]
A	0.87±0.01	40.1±1.1
B	0.78±0.02	38.3±0.3
D	0.89±0.04	39.66±0.2
E	0.77±0.01	39.19±0.2
C_1	0.91±0.01	38.36±0.3
C_2	0.84±0.01	41.37±0.3
C_3	0.80±0.01	37.81±0.1
C_4	0.75±0.01	40.26±0.4

Sludge conditioning and dewatering process

Before the sludge was used for dewatering tests, it was conditioned in the laboratory, aimed at mimicking similar operating conditions. The chemical conditioning implies the addition of undiluted amount of PAX in combination with cationic polymer [19].
Classical jar test device was used to mix the PAX/ polymer with the sludge. More specially we gently mixing 600 mL of sludge in a beaker of 800 mL, PAX was added while stirring was applied to the mixture sludge (typically 0; 300; 600 μL depending on the experiment) during 1 minute at 120 rpm, then a defined quantity of the diluted polymer solution (prepared the day before its use) was added rapidly and further shearing was applied at the same rotation speed and time (120 rpm during 1 min) to promote PAX/ polymer dispersion. After this period, the rotation speed was reduced and the sludge was gently shaken at 40 rpm during 3 min to promote flocs growth. Once the supernatant removed, the sludge can be used in dewatering stage.
After conditioning, the dewatering process was realized by using a normalized filtration-expression cell (AFNOR 1979).The pressure on the piston was applied and controlled by pressurized air. It was fixed at 5 bars. The mass of collected filtrate was recorded every 10 seconds on the personal computer linked to a precision balance device that measure collected filtrate. The filtration was stopped after a time fixed at 1 h for all experiments then, the specific resistance to filtration was evaluated by using the Carman-Kozeny equation[20].
Before drying, sludge cakes obtained after mechanical dewatering were extruded through a circular die of 14 mm of diameter and cut at a height of 14 mm, yielding cylindrical samples with mass of approximately 2.5 g, as used in several industrial belt dryers.
The time that the filtrate requires to travel a fixed distance in the filter paper is referred to as Capillary Suction Time (CST). The whole purpose of CST is to determine dewatering characteristics of a given sludge rapidly and easily. A large CST is usually indicator of poor sludge dewaterability.
A conditioned sludge sample was placed in the sample container. As water migrates through the Whatman filter and reaches the first probe, it activates the timer. When water reaches the second probe, the timer deactivates. The time interval between timer activation and deactivation is the CST [s]. CST measurements were done three times each with a Triton Electronics 304 M CSTmeter. CST was plotted

versus chemical dosage. The dosage that gives the fastest time is the optimum chemical dosage. For the two cationic polyelectrolytes, the CST test results were closed to 6 g/ kg_{DS} for both polymers.

Convective Drying Rig
Convective drying experiments (



Fi2) were carried out in a so-called 'micro-drier' specially designed for handling small extruded samples with a mass between 0.5 and 5 g. The micro-drier is a classical convective rig controlled in relative humidity, temperature and air velocity, which has already been described in detail in a previous paper [21]. Drying curves representing the drying rate (kg s^{-1}) versus the water content on a dry basis W (kg kg^{-1}) were calculated from these mass versus time data. Dividing the drying rate by the external exchange area yields the so-called Krisher's curves commonly used to study drying, i.e., the drying flux (kg m^{-2} s^{-1}) versus water content (kg kg^{-1}). Results reported in this study refer to the following operating conditions: temperature of 130°C, superficial velocity of 1 m.$s^{-1}$and the absolute humidity of the air fixed at 0.005 kg_{water}/ kg_{DS}.

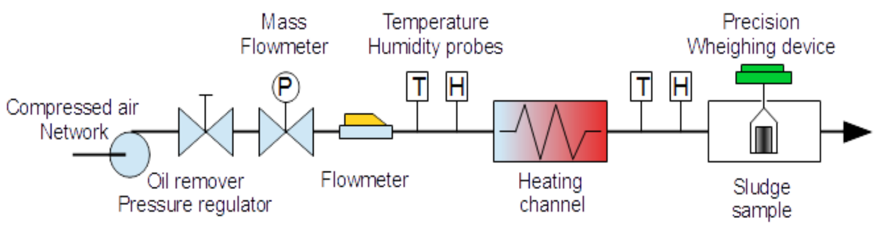


Figure 2: Scheme of the convective micro-dryer.

X-ray Microtomography
To determine Krischer's curves, it is necessary to know the exchange surface developed by the sludge sample, assumed to be the external sample surface. Following a method developed inside the laboratory [20-22], it was evaluated by using X-ray microtomography, acting as a medical scanner. This method allows the determination of shrinkage curves from series of 2D cross sections images of the samples. This results in a reduction of the effective surface exchange in course of time. The X-ray microtomographic device used in this study was a "Skyscan-1074 X-ray scanner". The X-ray source operates at 40 kV and 1 mA. The detector was a 2D, 768x576 pixels, and 8-bit X-ray camera giving image with a pixel size of 41µm. The following sequence was repeated several times during a drying experiment: drying interruption-tomographic analysis- drying resumption. These interruptions have been proved to have no impact on the drying kinetics [21].

RESULTS AND DISCUSSION

Impact of sludge conditioning on the dewatering process
The dewatering of a sludge working under constant pressure is well described by t/V versus V plot [23].The filtration phase is characterized by the linear part (Figure 3a). It corresponds to the formation of a cake due to the accumulation of the solid particles on the surface of a filter medium. The second part represents the expression phase. It describes the removal of water by cake squeezing. The ability of the forming cake to

let the water go through is commonly characterized, during the filtration phase, by the SRF. This parameter is calculated by the slope of the linear part according to the following equation [24]:

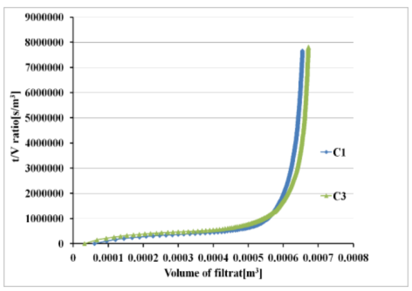
$$\frac{t}{V} = \mu . SRF . \frac{C}{2PA^2} V + \frac{\mu Rf}{PA}$$

Figure 3a already shows the dewatering repeatability test at the central point of PAX/ 640 LH conditioners. Only two replicates of sampling central point were presented. The dewatering behavior seems to be repeatable. However, negligible differences can be noticed at the dewatering expression phase.

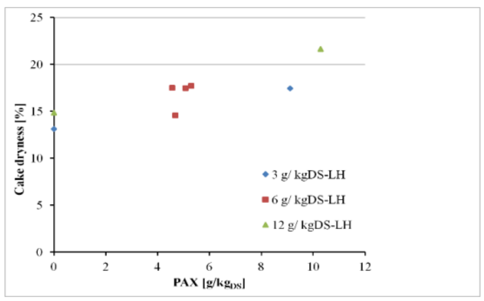
Sludge cake dryness obtained after a constant pressure filtration is another important parameter than can useful to describe the efficiency of the dewatering performances [25].

Figures 3b and 3c show the dry solids content of dewatering cake obtained for the two types of conditioning sludge of the whole points of experimental design.

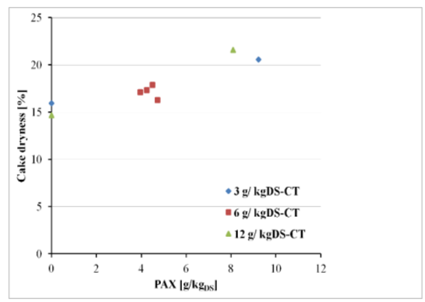
The cake dryness of the dewatering cake increases with increasing simultaneously Polyaluminium chloride and flocculant dosage for both series of experiments. It means that PAX was contributed to improve solids capture. Additional explanation might be found in a higher porosity of sludge, i.e. more cavities in the interface between the sludge were developed after PAX conditioning.



(a): Dewatering curves PAX/LH Repeatability test



(b) PAX/ 640 LH



(c) PAX/ 640 CT

Figure 3: Impact of PAX/ polymer on cake dryness

Concerning the dewatering stage, several observations can be made from the data reported in Table 3. First they show that the RSF values obtained for the PAX/ 640 CT conditioners are lower than those obtained for the PAX/ 640 LH.That could possibly lead to a better filtration performance, but it is not sufficient to conclude.

In fact, the importance of SRF data in the particular case of sludge dewatering is relative because a large amount of water is not removed by filtration, but by expression [23]. Generally, at industrial scale the efficiency of the dewatering is quantified in terms of final dry solid content (which is crucial data for the following treatments).

To complete the physical interpretation of RSF which it is sometimes mentioned to use with caution, the information about the volume of filtrate removed during the filtration phase and the total volume of dewatering process can give some interesting information. This information can be obtained by regarding the ratio of the volume of filtrate removed at the end of the filtration phase (V_{carman}) over the volume of filtrate removed at the end of the whole dewatering.

Regarding the values V_{carman}/V_{total} ratio reported in Table 3, sludge conditioning with couple PAX/ 640 CT exhibits a much higher ratio V_{carman}/V_{total} than sludge conditioning with PAX/ 640 LH conditioners. This remark means that, a huge amount of water was released in the wastewater sludge by this protocol.

We can notice that the D point of experiment seems to correspond an overdosing regarding its high RSF value. In fact, relatively small size flocs were obtained for this experimental point.

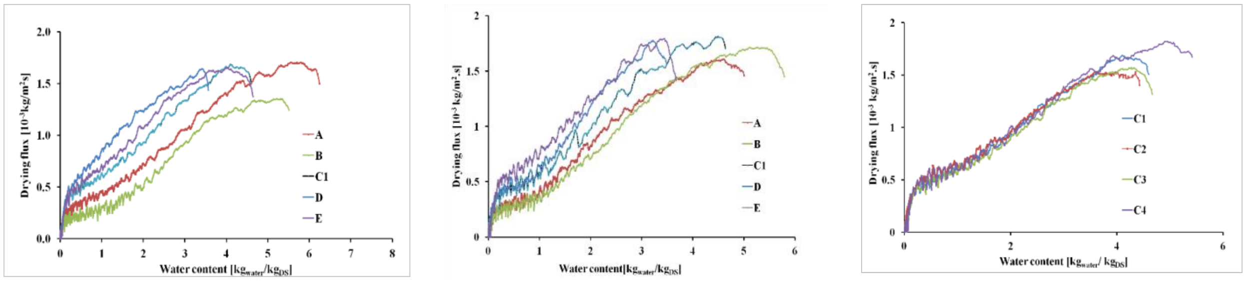
Considering V_{carman}/V_{total}, RSF values and sludge siccity reported in Table 3, we can assert that higher performances of dewatering were obtained by the combination of PAX/ 640 CT conditioners, i.e. with the one having the highest molecular weight.
This behaviour must be correlated to the mechanisms of flocculation. For large molecular weight polymer the flocculation is governed by bridging, whereas patch phenomena prevail for lower molecular weight polymer [26]. In the first case, because of their large size, the polymers may mostly adsorb on the outer easily accessible charges of the initial aggregates. Consequently, large flocs containing a large amount of intrafloc water tend to be generated. For lower molecular weight polymer, initial aggregates' charge sites are largely neutralized and the water retention inside the flocs in thus lowered.

Table 3: Filtration characteristics after dewatering

	Trials points	Cake dryness [%]	SRF [10^{13} m/kg]	V_{Carman} [mL]	V_{total} [mL]	V_{Carman}/V_{total} [-]
PAX 640 LH conditioning	A	13.21±0.12	2.57	246.1	711.4	0.35
	B	14.86±0.03	1.02	650.9	700.4	0.93
	D	21.40±0.40	16.1	58	779.8	0.07
	E	17.30±0.20	7.46	89.7	749.8	0.12
	C_1	17.30±0.20	4.71	157.5	658.9	0.24
	C_2	17.72±0.01	6.67	123	760.6	0.16
	C_3	17.30±0.20	4.6	150.6	654.6	0.23
	C_4	14.70±0.30	3.11	201.5	761.3	0.26
PAX 640 CT conditioning	A	15.96±0.01	0.59	575	724.4	0.79
	B	14.43±0.33	1.49	560.5	691.8	0.81
	D	21.40±0.30	11.3	130.4	773.5	0.17
	E	20.60±0.10	6.63	192.3	772.4	0.25
	C_1	17.00±0.10	1.19	496.8	654.5	0.76
	C_2	17.54±0.29	3.4	263.1	729.8	0.36
	C_3	17.80±0.20	1.53	474.1	671.4	0.71
	C_4	16.40±0.20	4.65	271.2	749.5	0.36

Impact of sludge conditioning on the convective drying behavior

For the two series of experiments, classical drying curves were obtained which consist at representing the drying flux versus water content, depicted in Figures 4a and 4b for respectively PAX/ 640 LH and PAX/ 640 CT set up. These figures clearly show the impact of the dual PAX/ polymers on the drying kinetics. The drying flux was calculated by dividing the drying rate by the external exchange area obtained by using X-ray microtomography. This illustration of drying flux is called the Krisher's curve commonly used to understand drying phenomena [11].
For sludge such a curve can be divided into three conventional phases [21]: The first phase; is a short period called adaptation period or preheating period during which the product adapts its behavior to the new applied conditions. The second period is called the constant drying flux rate, during this period the supplied heat serves to the evaporation of the product water. The operation was done at constant surface product temperature. The last phase is the falling drying flux period, ending with stabilization in moisture content at an equilibrium value corresponding to the end of drying.
It can be clearly remarked that both conditioning treatment accelerate the drying process: for a same given water content, the drying kinetic was higher in presence of PAX adding. It means that water is more freely and then more easily removed in the presence of PAX added (see Figures 4a and 4b).
Moreover, results also show that this benefic effect of PAX is more marked in the case of PAX/ 640 CT conditions, implying that the molecular weight and structure of the flocculant agent can play a role by improvement of the drying behavior.



(a): PAX/ 640 LH (b): PAX/ 640 CT (c): PAX/ 640 LH central point's repeatability test

Figure 4: Krisher's curves of experimental points

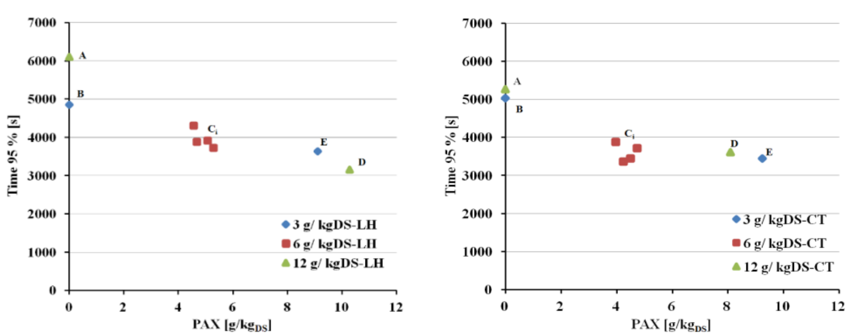
The reliability of the experiment was evaluated on four replicated tests carried out on the central point for the two series of experimental design. A good repeatability was obtained. Thus, the comparison which has been made with the others experimental design points can be objective.

For the sake of clarity only one series of drying curves repeatability test, conducted on sludge conditioned by the couple PAX/ 640 LH were presented in Figure 4c. The results obtained from the PAX/ 640 CT conditioner lead to the same remarks.

Impact of sludge conditioning on the convective drying time

In order to see how the combination of couple PAX/ polymers dosage can be influence the drying behavior, drying time was plotted versus Polyaluminium chloride and polymers dosages. (depicted in Figures 5a and 5b).

For both series of experiments, it can be observed that the drying time decreases when PAX/ flocculant dosage increase. Particularly, when the polymer concentration is at its low and high level, example for respectively A (0; 3 g/kg_{DS}) and B (0; 12 g/kg_{DS}) points, the drying time is longer as about 5 000 seconds. This time begins to reduce when PAX was added.



(a): PAX/ 640 LH conditioning (b): PAX/640 CT conditioning

Figure 5: Impact of PAX/polymer on drying time

Table 4: Impact of conditioning on drying time

Trials	Time 95 % [s]		Ratio
	PAX/LH	PAX/CT	t_{CT}/t_{LH}
A	4850	5030	1.04
B	6100	5265	0.86
D	3155	3615	1.15
E	3640	3450	0.95
C_1	3920	3885	0.99
C_2	3720	3360	0.90
C_3	4310	3490	0.81
C_4	3880	3715	0.96

Table 4 shows the values of drying times for the two experiments set up required to achieve 95 % of water samples removed. They indicate that the drying time is shorter by conditioning sludge samples with PAX/ 640 CT conditioners. We could notice that, for B test, drying time was most higher for the second way conditioning, may be due for this point was suspected to be an overdosing of PAX concentration.

About the ratio of drying time for each experiment PAX/ chemical testing, it was respectively found to be closed to $(min/\ max)_{LH}$= 0.52 and $(min/\ max)_{CT}$= 0.64.This observation indicated that, by choosing the right pair conditions can provide a better performance on the drying time reduction. The corresponding benefic effect on the drying time reduction was estimated at 12 %.

In conclusion, PAX can improve significantly the drying process when it is combined by linear polymers. This positive effect is most pronounced when the cationic polyelectrolyte conditioner is with high molecular weight.

CONCLUSIONS

Based on a considered experimental design, this work was an attempt to put in evidence the impact of sludge co-conditioning by Polyaluminium chloride and linear molecular weight polymers on both dewatering and drying behaviours. Higher molecular weight polyelectrolyte was shown to yield flocculation and dewatering process than the lower molecular weight one. PAX appears to be profitable to contribute at this process. About drying, the positive effect of PAX/ polymer addition was shown on the drying kinetics. Consequently a decrease of the drying time was observed. Furthermore, better results were obtained with linear polymer with high molecular weight.
Future works will be done by investigating the use of cross linked polymer and their relationship with textural rheological properties, including drying and shrinkage sludge modeling to simulate the experimental results.

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