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ONDERZOEK NAAR HET VOORSPELLEN VAN DE ONTWATERBAARHEID VAN SLIB EN PE GEBRUIK

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TEN GELEIDE

STAPPEN NAAR DE VOORSPELLING VAN DE ONTWATERBAARHEID VAN ZUIVERINGSSLIB

De ontwatering en afzet van zuiveringsslib is een grote kostenpost voor waterschappen. Een goede ontwatering van het slib bespaart op kosten van transport en afzet maar gaat ten koste van de inzet van energie en chemicaliën. Waterschappen ervaren grote verschillen in ontwaterbaarheid tussen locaties maar ook gedurende het seizoen op een specifieke locatie. Dit onderzoek geeft inzicht in de belangrijkste factoren die de ontwaterbaarheid beïnvloeden maar laat ook zien dat de correlaties complex zijn. Regelmatige monitoring van relevante parameters kan helpen om een uitgebreidere dataset op te bouwen om deze complexe correlaties beter te ontwarren, bijvoorbeeld met artificiële intelligentie.

Waterschappen produceren jaarlijks 1,3 miljoen ton ontwaterd zuiveringsslib. Dit slib wordt voornamelijk verbrand en de kosten voor de afzet bedragen ruim €100 miljoen terwijl nog eens ruim € 20 miljoen aan kosten worden gemaakt voor het gebruik van chemicaliën voor de ontwatering. De ontwaterbaarheid van slib verschilt sterk van locatie tot locatie en van seizoen tot seizoen. Daardoor het is heel moeilijk vast te stellen of altijd de beste ontwateringsprestatie wordt bereikt. Er kan scherper aan de wind worden gevaren als waterschappen beter kunnen vaststellen wanneer de beste prestatie wordt bereikt en welke slibeigenschappen invloed hebben. Dat bespaart kosten.

In dit onderzoek zijn gedurende twee jaar slibmonsters genomen van het slib van twee representatieve rioolwaterzuiveringen. Deze monsters zijn uitgebreid gekarakteriseerd. Daarnaast zijn verschillende methoden getest om de ontwaterbaarheid vast te stellen en vervolgens zijn deze resultaten vergeleken met de ontwateringsresultaten op praktijkschaal.

De resultaten maken het mogelijk om een schifting aan te brengen in meetmethoden die relevant zijn gebleken en meetmethoden die minder duidelijke resultaten geven. Daarmee is een eerste basis gelegd. Wel blijken de correlaties tussen ontwaterbaarheid en slibparameters zwak. Daarom is het nodig om gedurende langere tijd een dataset op te bouwen voor de relevant gebleken parameters en ontwateringsresultaten. Omdat verschillende parameters tegelijk invloed kunnen hebben is het aan te bevelen om deze dataset te analyseren met methoden voor multivariate data, zoals bijvoorbeeld artificiële intelligentie. Door een verdere opvolging van de inzichten uit dit onderzoek kunnen waterschappen hopelijk steeds meer grip krijgen op een optimale slibontwatering en het beheersen van de kosten ervan.

Mark van der Werf
Directeur STOWA

SAMENVATTING

ONDERZOEK NAAR MEETMETHODEN OM SLIBONTWATERINGSRESULTAAT TE VOORSPELLEN

Tijdens biologische behandeling van huishoudelijk afvalwater in Nederland worden grote hoeveelheden slib geproduceerd. Dit slib wordt ontwaterd en vervolgens, vaak na vergisting en/of (biologisch) drogen, verbrand in slibverbrandingsinstallaties of mee gestookt bij andere verbrandingsprocessen. De kosten voor ontwatering, transport en afzet van slib bedragen 20-30% van de operationele kosten van Nederlandse rioolwaterzuiveringsinstallaties (rwzi's). Poly-elektrolyt (PE), gebruikt in slibontwatering, zorgt voor 10-15% van de milieu-impact van rwzi's. Het optimaliseren van slibontwatering, door het drogestofgehalte te verhogen en/of PE-verbruik te verlagen, kan dus aanzienlijke financiële voordelen brengen voor rwzi's en/of de milieu-impact verlagen.

Er is veel wetenschappelijk labonderzoek gedaan naar slibontwatering. In de literatuur worden diverse testen en meetmethoden beschreven die gelinkt worden aan de ontwaterbaarheid van slib en het gebruik van PE. Voorbeelden hiervan zijn de Capillary Suction Time, specifieke filtratie weerstand, Higgins centrifuge test en de Streaming Current Potential Test. Ook zijn er diverse methodes die specifieke eigenschappen van het slib kwantificeren, zoals metingen van de samenstelling, zetapotentiaal of reologie. In de wetenschappelijke literatuur wordt van een aantal van deze eigenschappen gesuggereerd dat deze eigenschappen een voorspeller zijn van de ontwateringseigenschappen van slib. Er is echter weinig onderzoek dat deze wetenschappelijke kennis ook daadwerkelijk toepast voor het verklaren van praktijkresultaten van ontwatering op rwzi's.

In dit STOWA onderzoek is gepoogd om dit gat tussen wetenschap en praktijk op het gebied van slibontwatering te verkleinen. Welke meetwaarden kunnen we relateren aan een verbetering van slibontwatering in de praktijk? En is deze meting gemakkelijk te doen, bijvoorbeeld door een procesoperator in het lab van de rwzi? Het uiteindelijke doel is om te begrijpen aan welke knoppen gedraaid kan worden om slibontwatering in de praktijk te verbeteren en PE verbruik te verminderen.

Methoden

In dit project is gedurende twee jaar de ontwatering van slib op twee geselecteerde rwzi's in Nederland gevolgd. Dit waren rwzi Harnaschpolder, in het gebied van Hoogheemraadschap Delfland en rwzi Bath, in het gebied van Waterschap Brabantse Delta. Zesmaal zijn er monsters genomen van diverse meetpunten rond de ontwatering. Er zijn door TU Delft en Royal HaskoningDHV diverse analyses op uitgevoerd. De resultaten van deze labtesten zijn vergeleken met de praktijkresultaten. Allereerst is onderzocht welke meetmethoden voor de ontwatering van slib op labschaal correleren met de resultaten op praktijkschaal (droogte van de koek en PE verbruik). Vervolgens is onderzocht of veranderingen in slibeigenschappen correleerden met het ontwateringsresultaat, zowel op labschaal als praktijkschaal. Op basis van deze analyse worden aanbevelingen gedaan voor geschikte meetmethoden om het ontwateringsresultaat of PE gebruik te monitoren.

Daarnaast is de samenstelling van het gebruikte PE op de zuiveringen meerdere malen gemeten, om uit te sluiten dat een variatie in samenstelling invloed had op de ontwateringsresultaten.

RESULTATEN

Vergelijking drogestofgehalte slib na ontwatering en PE-verbruik in lab versus praktijk

Er zijn drie verschillende ontwateringstesten gedaan op labschaal waarvan verwacht werd dat die zouden correleren met de droogte van de koek na ontwatering. Dit waren de Higgins centrifuge test, de Specific Resistance to Filtration (SRF) en de Capillary Suction Time (CST). Het droge stofgehalte van het slib na de Higgins test correleerde het meest met de praktijkresultaten van de ontwatering op rwzi Harnaschpolder en rwzi Bath. Ook al zijn de resultaten in het lab altijd wat beter, de trend was hetzelfde. Een eenduidige relatie tussen de resultaten van de Higginstest en een voorspelling van het te behalen resultaat in de praktijk is echter nog niet mogelijk gebleken, onder andere door het beperkte aantal meetpunten. SRF en CST waren redelijk goed aan de resultaten van Harnaschpolder te relateren, maar niet aan die van Bath. De Higgins test lijkt dus in dit onderzoek het meest geschikt om ontwatering in het lab na te bootsen.

Om het PE verbruik te kunnen te volgen zijn in het lab verschillende testen uitgevoerd: PE dosering tijdens de Higgins test en de Streaming Current Potential (SP) test. De labresultaten suggereerden grote verschillen in het optimale PE verbruik voor de verschillende monsters, terwijl de PE verbruiken in de praktijkinstallaties slechts weinig varieerden gedurende de proef. Dit zou kunnen komen doordat er niet met zekerheid te zeggen is of de PE dosering aan de praktijkinstallaties goed geoptimaliseerd was tijdens monstername. Daardoor kan niet met zekerheid gezegd worden dat de labtesten een goede voorspeller zijn voor het PE-verbruik op praktijkschaal. Het onderzoek laat echter wel zien dat de PE consumptie zoals die met de Higgins test wordt bepaald correleerde met andere slibeigenschappen waarvan aangenomen wordt dat zij invloed hebben op het PE gebruik. Deze correlatie was minder sterk voor de SP test en daarom concludeert dit onderzoek dat de Higgins PE test de meeste potentie heeft als voorspeller voor de ontwaterbaarheid van slib en het PE gebruik.

Eigenschappen van het slib die invloed hebben op het ontwateringsresultaat en PE verbruik

Er zijn verschillende metingen aan het slib gedaan om te kijken of er verbanden gevonden konden worden met het ontwateringsresultaat en het PE verbruik enerzijds en de samenstelling van het slib anderzijds. Er zijn een aantal correlaties gevonden. Bij Bath slib leek het colloïdale COD gehalte (zie paragraaf 1.3.2) in de vloeistoffase van het slib goed te relateren aan het droge stofgehalte. De hoeveelheid Extracellular polymeric substances (EPS, polymeren gebonden aan de cellen van het slib) ook, maar het verband was minder sterk. Ook kalium in de vloeistoffase van het slib en totaal magnesium in het slib zelf waren goed te relateren aan het droge stofgehalte. Bij Harnaschpolder was alleen een verband te vinden tussen de viscositeit van het slib en het ontwateringsresultaat. Het natriumgehalte in het rejectiewater was de enige parameter die gerelateerd kon worden aan het droge stofgehalte van het slib van beide rwzi's, maar er kon niet worden afgeleid dat een bepaald natrium gehalte een bepaalde ontwateringsgraad geeft.

Er is een sterk verband gevonden tussen het calcium en het aluminium gehalte van het slib van beide rwzi's en het PE-verbruik, evenals de concentraties colloïdaal COD en humuszuren. Ook de relatie tussen de ratio monovalente en divalente ionen en PE-verbruik leek sterk voor beide rwzi's. Bij Harnaschpolder leek een verband te bestaan tussen het ijzergehalte van het slib en de magnesiumconcentratie in de vloeistoffase en het PE-verbruik. Bij rwzi Bath leken de deeltjesgrootte en de slibviscositeit van invloed te zijn. Uit voorgaande blijkt dat er wel relaties zijn tussen gemeten parameters en het drogestofgehalte of het PE-verbruik, maar dat deze relaties niet eenduidig zijn. Zo kan uit de relaties nog niet eenduidig worden afgeleid dat een bepaalde verhoging of verlaging van een gemeten parameter altijd overeenkomt met een bepaalde wijziging in drogestofgehalte of PE-verbruik. Dat dit nog niet eenduidig kan wordt veroorzaakt door o.a. het beperkte aantal monsterpunten, de niet altijd geoptimaliseerde instellingen van de apparatuur en de verschillen tussen de beide slibsoorten.

Variatie in compositie PE

Er is geen significant verschil in samenstelling van het PE gemeten gedurende de twee testjaren, dit wil zeggen dat de afwijkingen bij de gemeten resultaten binnen de foutenmarge van de meetmethode vielen.

DISCUSSIE EN CONCLUSIES

Bij de start van het onderzoek was de verwachting dat op 4 vragen een antwoord kon worden gegeven.

1. Geven de onderstaande labschaal testen, die genoemd worden in de literatuur, een correlatie met fullscale slib ontwateringsresultaten?
    - Capillary suction time (CST)
    - Specific resistance to filtration (SRF)
    - PE-verbruik in de streaming current potential test
    - Modified Higgins centrifuge test

➔ Conclusie: Alleen de Higgins testmethode gaf over het hele onderzoek voor beide slibsoorten een redelijk goed beeld van wat haalbaar is in praktijk. Doordat de omstandigheden in het lab altijd geoptimaliseerd werden, was de vergelijking met de praktijk minder goed, omdat het daar niet mogelijk was eerst een optimalisatie onderzoek te doen voorafgaand aan de monstername. Voor de eerste 3 testen werd geen eenduidig verband gevonden voor beide slibsoorten.

2. Welk van de volgende slibkarakteristieken kunnen worden gelinkt aan fullscale ontwateringsresultaten
    - Organisch stofgehalte (Volatile Solids = VS),
    - Chemisch zuurstof verbruik (COD),
    - Extracellulaire polymere stoffen (EPS))
    - Nutriënten (stikstof en fosfor)
    - Kationen (monovalent en divalente)
    - Deeltjesgrootte verdeling (PSD)
    - Viscositeit
    - Oppervlakte lading (Zeta potential)

➔ Conclusie: Enkele van deze slibkarakteristieken konden gelinkt worden aan de fullscale ontwateringsresultaten van één of beide slibsoorten, zie onderstaande tabel. De gevonden relaties tussen de slibkarakteristieken en het drogestofgehalte en/of het PE-verbruik waren echter niet altijd eenduidig. Op basis van het beperkte aantal metingen en slechts 2 slibsoorten kan nog niet geconcludeerd worden dat deze slibkarakteristieken gebruikt kunnen worden om het ontwateringsresultaat mee te voorspellen. Mogelijk dat met een combinatie van slibkarakteristieken het wel mogelijk is meer over de relaties te zeggen. Om dit wel te kunnen vaststellen, is meer onderzoek nodig.

		SRF	CST	Higgins	SP	COD in water	colloidaal COD	EPS	K in water	Mg in slib	Na in water	Calcium in slib	Aluminium in slib	hunus	Fe in slib	Mg in water	viscositeit	deeltjesgrootte	ratio mono/ divalente ionen
Harnaschpolder	PE	±	±	+	-	-	+	-	-	-	-	+	+	+	+	+	-	-	+
	DS-koek	±	±	+	-	-	-	-	-	-	+	-	-	-	-	-	-	-	-
Bath	PE	-	-	+	-	-	+	-	-	-	-	+	+	+	-	-	+	+	+
	DS-koek	-	-	+	-	+	-	±	+	+	+	-	-	-	-	-	-	-	-

3. Welke parameters kunnen worden gebruikt om de ontwaterbaarheid te voorspellen en zijn deze eenvoudig te meten in het lab?

➔ Conclusie: Ja er is een methode die gebruikt kan worden om de ontwaterbaarheid mee te voorspellen, namelijk de Modified Higgins centrifuge test. Deze Higgins test is relatief eenvoudig uit te voeren, waarbij wel speciale apparatuur nodig is (grote labcentrifuge en speciale centrifugebuizen). De methode geeft geen absolute waarde voor wat bij bepaalde instellingen haalbaar is, maar laat zien of er verbeteringen mogelijk zijn. Wel dienen er voldoende meetpunten per slibsoort aanwezig te zijn.

4. Is er een wijziging in PE-kwaliteit over het jaar en zo ja, is dit terug te zien in variaties in ontwateringsresultaten?

➔ Conclusie: Er is geen significant verschil in kwaliteit van het PE gemeten gedurende de twee testjaren. Al is de onnauwkeurigheid van de meetmethode groot (12%).

STOWA IN HET KORT

HOE WE WERKEN

STOWA is het kennis- en innovatiecentrum voor regionale waterbeheerders in Nederland; de waterschappen en provincies. We helpen ze met het verkrijgen van nieuwe kennis en inzichten die nodig zijn om de opgaven van de regionale waterbeheerders beter te kunnen uitvoeren. Dat doen we door kennisvragen te formuleren en te selecteren in programmacommissies. We zetten ons onderzoek uit bij een keur aan experts, adviesbureaus, instituten en universiteiten, die we begeleiden tijdens hun werk. We zorgen voor de beschikbaarstelling en verspreiding van de kennis, inzichten en antwoorden aan de gezamenlijke waterbeheerders. We stimuleren de uitwisseling van kennis en ervaringen, via bijeenkomsten, werkgroepen, excursies, conferenties en communities of practice. We werken samen met onder andere ministeries, Rijkswaterstaat, gemeenten, drinkwaterbedrijven.

WAT WE ONDERZOEKEN

Inhoudelijk richt Stowa zich op alle onderdelen van waterbeheer, van waterkering en stedelijk waterbeheer tot waterzuivering en watersystemen. Belangrijke thema’s s daarbij zijn klimaatadaptatie, waterveiligheid, waterkwaliteit en ecologie, energietransitie en circulaire economie.

De kennisvragen die Stowa beantwoordt liggen meestal op technisch, natuurwetenschappelijk, bestuurlijk-juridisch of sociaalwetenschappelijk gebied. Onze kennis is altijd gericht op de praktijk van regionale waterbeheerders. Dat is waar we voor staan, als Stichting Toegepast Onderzoek WAterbeheer.

WIE WE ZIJN

STOWA is als kennisorganisatie onafhankelijk, onpartijdig en transparant. De afnemers van onze kennis moeten erop kunnen vertrouwen dat de inhoud van onze rapporten objectief en representatief is. Alleen zo kan onze kennis worden ingezet voor beter waterbeheer en innovaties die antwoord geven op de uitdagingen van vandaag en morgen. Het is aan regionale waterbeheerders zelf te bepalen hoe ze de kennis van Stowa in de praktijk gebruiken. STOWA kan daarbij een rol spelen als adviseur, maar is geen uitvoerder of regisseur.

STOWA is een stichting die de richtlijnen volgt voor organisaties zonder winstoogmerk (RJ-640). In ons jaarverslag is daarom naast de cijfermatige jaarrekening onder meer ook een directieverslag over de stichting, haar activiteiten en kentallen opgenomen.

THE STOWA IN BRIEF

HOW WE WORK

STOWA is the knowledge and innovation hub for regional water managers in the Netherlands, serving the water authorities and provinces. We help them gain the insights and knowledge they need to tackle regional water management challenges more effectively. We do that by identifying and prioritizing key research questions through dedicated program committees. We commission our research from a range of experts, consultancies, institutions, and universities, and oversee them in their work.

Once the research is complete, we ensure that the findings are shared widely within the broader water management community. We also promote experience and knowledge sharing through events, working groups, excursions, conferences and communities of practice. We work together with ministries, Rijkswaterstaat, municipalities and water companies, among others.

OUR RESEARCH FOCUS

STOWA's research covers all facets of water management, from flood defense and urban water management to water treatment and systems. Key themes include climate adaptation, flood protection, water quality and ecology, energy transition and circular economy.

The research questions we address span technical disciplines, the natural sciences, governance, legal frameworks and the social sciences. All our work is geared towards providing practical solutions for regional water managers. This is central to our mission as the Foundation for Applied Water Research.

WHO WE ARE

As a knowledge organization, STOWA is independent, impartial and transparent. Users of our knowledge can trust that the findings in our reports are objective and balanced. This ensures that our knowledge provides a reliable foundation for improving water management and fostering innovations to meet today's and future challenges. The regional water managers themselves decide how to apply STOWA's insights in practice. While STOWA can serve as advisor, we do not play a role as implementors or directors.

STOWA is a non-profit foundation and follows the guidelines for non-profit organizations (RJ-640). Our annual report includes both financial statements and a detailed overview of the foundation, its activities and key statistics.

ONDERZOEK NAAR HET VOORSPELLEN VAN DE ONTWATERBAARHEID VAN SLIB EN PE GEBRUIK

INHOUD/CONTENT

INTRODUCTION

During biological treatment of wastewater, primary sludge (PS) and waste activated sludge (WAS) are produced. WAS combined with primary sludge (PS) are often digested to reduce the solid content, to produce biogas and to stabilize the resultant sludge. Also, in some wastewater treatment plants (WWTPs), both PS and WAS are not anaerobically digested and are directly dewatered. The final treatment step for sludge in the WWTPs is dewatering from which, the stabilised sludge is send to the final disposition (incineration in The Netherlands). Dewatering is a crucial step to reduce sludge volume, which affects the sludge transportation costs. WAS and PS contain a significant amount of water (more than 90%) (Dai et al. 2018), part of which needs to be removed before transporting the stabilised sludge from the WWTP. Sludge dewatering is the process of mechanically forcing the water out of the sludge with the aid of chemical agents (conditioners) such as polyelectrolytes (PE). After mechanical dewatering, the water content of sludge decreases to 60 to 80% (Dai et al. 2018; Mowla et al. 2013). The PE modifies the colloidal structure and releases part of the water that is entrapped in the sludge flocs. In full-scale different dewatering techniques are employed such as filtration and centrifugation (Cai et al. 2018; Dai et al. 2018).

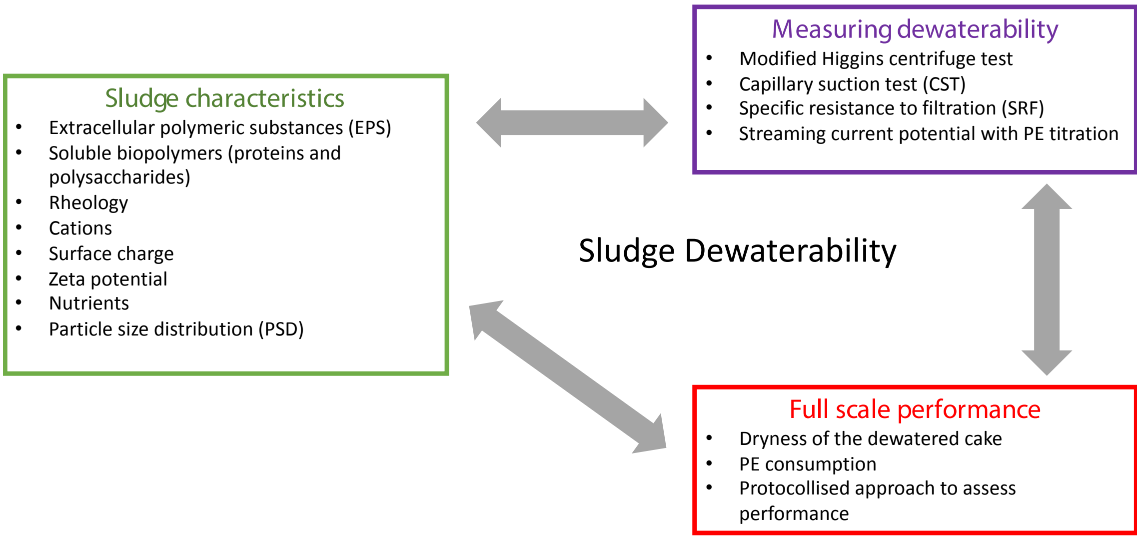
Annually, 1.3 million tons of dewatered sludge from the WWTPs are handled in the Netherlands. Furthermore, from 20 to 30% of the operational costs in WWTPs go to sludge handling and dewatering (Serrano et al. 2016; Wei et al. 2009). For instance, in The Netherlands, transportation of the dewatered sludge can cost 5-10 euros per ton of sludge (STOWA 2018-16), and the final disposal cost can be as high as 75 to 100 euros per ton of dewatered sludge. Moreover, the annual costs of polymers used in The Netherlands for conditioning of the sludge before dewatering can be as high as 22-27 million euros. Besides the high cost of PE, it also represents 10-15% of the total environmental impact of the treatment plants (bedrijfsvergelijking zuiveringsbeheer 2022). Considering that in the Netherlands PE addition is a ubiquitous technique during dewatering, PE reduction may reduce both operational costs and CO_2 footprint of WWTPs.

To reduce the costs and environmental impact of sludge handling processes in the WWTPs, it is critical to lower PE consumption and remove more water from sludge to decrease sludge transportation costs. This means that the dewaterability of sludge should be improved to obtain a higher total solids (TS) concentration of the dewatered sludge cake (%) and lower PE consumption (g active PE/kg TS sludge). To improve dewaterability, it is important to know which sludge characteristics or measurable parameters affect the dewatering performance. Dewaterability is often evaluated at laboratory scale by measuring parameters such as capillary suction time (CST), specific resistance to filtration (SRF), TS concentration of dewatered sludge cake, bond energy, and bound water content (Liu et al. 2021; Zhang et al. 2019; Higgins et al. 2017). These parameters, which are shown in Figure 1, have been linked to changes in sludge characteristics and eventually dewaterability in multiple studies (Zhang et al. 2015; Xu et al. 2018; Liang et al. 2020; Huang et al. 2020; To et al. 2016; Zhang et al. 2019b).

In practice operators of sludge dewatering installations experience variations in the performance of their installation resulting in varying cake dryness and PE consumption. The reasons for these variations come from an improper operation of the installation, changes in sludge composition or changes in PE quality. For the operators of these plants, it is often difficult to understand the cause of these variations because they do not have enough tools to identify the source of variations in dewatering performance. In earlier studies STOWA has developed tools to identify changes in PE quality (Raffa/STOWA 2017) and the preparation of the PE solution (Korving/STOWA 2014). However, a good practical methodology to validate changes in sludge dewaterability is not available for operators of sludge dewatering facilities. As formerly described scientific literature has suggested many different parameters but an evaluation of the correlation of these parameters with the full-scale dewatering results is missing.

In this study, an attempt is made to link the parameters that are often used to study dewaterability at laboratory scale, to full-scale data. All the mentioned parameters in the literature (Figure 1) can address a change in the sludge characteristics but the link with the full-scale dewaterability results is not yet clear. The ultimate goal is to increase the dryness of the dewatered sludge cake, decrease the PE consumption and keep the remaining water (centrate/filtrate) clear in full-scale. This research tries to connect fundamental knowledge found in the literature with practice in order to find the most appropriate laboratory techniques to estimate, monitor and improve sludge dewaterability in full-scale.

FIGURE 1 **STUDIED PARAMETERS IN THE RESEARCH**



The research questions for this study are set as below:

1. Do the data from lab-scale tests found in literature (shown in below) correlate with the full-scale sludge dewaterability data? The parameters and techniques to study are:
   a. Capillary suction time (CST)
   b. Specific resistance to filtration (SRF)
   c. PE consumption in the streaming current potential test
   d. Modified Higgins centrifuge test
2. Which sludge characteristics (shown below and Figure 1) can be linked to changes in full-scale dewaterability data?
   a. Organic content of sludge (Volatile solids (VS),

   b. Chemical oxygen demand (COD),
   c. Extracellular polymeric substances (EPS))
   d. Nutrients (Nitrogen and phosphorus)
   e. Cations (Monovalent and divalent)
   f. Particle size distribution (PSD)
   g. Viscosity/Rheology
   h. Surface charge (Zeta potential)
3. Which parameters can be used to predict dewaterability? Can this parameter be easily measured at laboratory scale?
4. Is there a change in PE quality in full-scale throughout the year(s) and if yes, can it be linked to changes in full-scale dewaterability data?

1

EXPERIMENTAL APPROACH AND METHODS

In this research, anaerobic digestate from two different WWTPs in the Netherlands was studied. Digestate samples were regularly taken before and after the dewatering equipment, during a timeframe of two years in different seasons of the year. The full-scale performance of the dewatering equipment also was studied, and samples of the sludge were studied in detail in a laboratory.

In this section, the WWTPs of which the sludge was studied (sampling locations) are introduced and described, and the sampling procedure is explained. The measured sludge characteristics and the reason why they are used in this study are detailed, as well as the employed methodology. Furthermore, the employed statistical tools in this study are described. These analyses were used to build a correlation matrix between different parameters. This correlation matrix was used to identify potential relations between sludge characteristics and dewaterability of sludge, both in lab-scale and full-scale.

1.1 STUDY CASES

For this study two WWTPs were selected. One of the selection criteria was that the two locations did (almost) not receive any external sludge. Therefore, there will be no external source that can have an impact on the dewaterability, which simplifies the data interpretation. Earlier studies have shown that the type of phosphate removal in the water line has an impact on sludge dewaterability. Compared to chemical phosphate removal, the use of enhanced biological phosphate removal has shown to lead to a less dry sludge cake and higher polymer use (Korving/STOWA 2012, Shimp 2013). Therefore, we selected two WWPT's with different phosphate removal methods.

Harnaschpolder WWTP and Bath WWTP were the study cases for this research. A general overview of the treatment processes in these plants is given in the section below. The treatment capacities for both locations are expressed in population equivalent (p.e.). (One p.e. is equal to 150 g of chemical oxygen demand (COD) per day (Driessen et al. 2020)).

HARNASCHPOLDER WWTP

Harnaschpolder municipal WWTP is one of the four treatment plants under supervision of Delfland waterboard and located in Den Hoorn, in the province of South Holland. It is the largest treatment plant in the Netherlands with a capacity of approximately 1.2 M p.e. Phosphorus is removed biologically, and nitrogen is removed from the wastewater using nitrification/denitrification.

The wastewater flows through a pre-clarifier in which primary sludge (PS) is separated (sedimentation). Because of the biological treatment of water, WAS is produced and later separated in secondary sedimentation tanks. WAS is mechanically thickened by addition of PE. The thickened PS and WAS are stabilized in mesophilic anaerobic digesters which produce biogas. Antifoam is dosed into the anaerobic digesters to reduce foaming. Also,

$FeCl_3$ is dosed into the anaerobic digesters to reduce H_2S in the biogas. After anaerobic digestion, the digested sludge is buffered in a sludge buffer tank in which $Mg(OH)_2$ is added to precipitate struvite prior to dewatering. After this, PE is added, and the sludge is dewatered in the centrifuges.

The PE used in Harnaschpolder WWTP is purchased in powder form. The used PE is Zetag 8185, SOLENIS which is a cationic PE. To prepare the PE solution, PE in powder form is mixed with water to reach a concentration of approximately 0.3% of active PE. After this, the sludge and PE solution are brought in contact together in in-line mixers before entering the centrifuges.

BATH WWTP

Bath municipal WWTP is one of the 17 treatment plants under supervision of Brabantse Delta waterboard. The treatment plant is located close to the village of Bath in the province of Zeeland, The Netherlands. It has a capacity of approximately 500,000 p.e. Phosphorus is removed from the wastewater by chemical precipitation with ferrous sulphate ($FeSO_4$). PS is settled in pre-settlers and later thickened. The WAS from the secondary treatment is decanted and sent to belt thickeners to be thickened prior to anaerobic digestion.
The thickened PS and WAS are anaerobically digested for solid reduction, stabilization, and biogas production. After anaerobic digestion, PE is added, and the sludge is transferred to belt filter presses to be dewatered.

The PE used in Bath is a cationic PE in liquid form. The PE brand name is MELFLOC MS4570. Before the PE mix with sludge, the liquid PE and water are mixed together to make a PE solution with an approximate concentration of 0.3% active PE. This PE solution is brought in contact with digested sludge with in-line mixers before the sludge enters the belt filter presses.

1.2 SAMPLING PLAN

In this research six sampling rounds and analysis campaigns were conducted at both studied WWTPs in order to find statistically valid relations between the measured parameters in lab and full-scale. From each season, at least one sample was taken to study the effect of seasonal changes on the dewaterability of sludge and its relation to the sludge characteristics. Table 1 shows an overview of the season and the specific dates of sampling. During each sampling round, the samples were taken from 4 different sampling points around the dewatering equipment (digested sludge, dewatered sludge cake, diluted PE and centrate). The diluted PE samples were taken to perform the lab scale dewaterability tests. This was done to ensure that there was no difference in the way the PE solution was prepared between lab and full scale. A potential disadvantage of this approach that some of the PE might hydrolyse if the time between sampling and use is too long (Saveyn 2013). The samples were sent on the day of sampling to the laboratories of Royal HaskoningDHV and TU Delft for characterisation. In addition, samples of the concentrated PE were taken regularly and sent to Intertek laboratory for PE analysis.

TABLE 1 **OVERVIEW OF SAMPLING ROUNDS CONDUCTED IN THE WWTPS BATH AND HARNASCHPOLDER**

Sampling round	Date	Season	Year	Day of the year number	Sampling points
1	March 21st	Winter	2022	80	Digested sludge (Inlet to the dewatering unit) Dewatered sludge cake PE Diluted PE Centrate
2	June 27th	Spring		178	
3	September 19th	Summer		262	
4	February 20th	Winter	2023	51	
5	July 10th	Summer		191	
6	October 9th	Fall		282	

1.3 SAMPLING PROCEDURE

Royal HaskoningDHV gave an optimization protocol to the operators of both WWTPs to ensure that the samples were taken while the dewatering equipment was working at its optimum performance. The steady and optimum full-scale operation during sampling rounds was necessary to ensure that the change in sludge dewaterability was due to changes in the sludge characteristics, and not to fluctuations in the dewatering equipment performance. This optimization protocol has separate sections for optimization of centrifuge and belt filter presses. The optimization protocol is in Dutch language and can be found in appendix A1.1. In practice, this protocol was used more frequently at Bath WWTP than in Harnaschpolder WWTP. Therefore, it is not sure that the operational parameters of the dewatering equipment did not have an effect on sludge dewaterability data of the full-scale.

It was also requested to both WWTPs to notify the parties when a significant change in dewaterability results in full-scale was observed (dryness of dewatered cake and/or PE consumption), so that samples could be taken on that day. In practice this did not happen and therefore all samples were distributed over the different seasons in a year.

1.3.1 SLUDGE DEWATERABILITY

MODIFIED HIGGINS CENTRIFUGE TEST

The dewaterability of the full-scale samples was measured quantitatively using a modified Higgins centrifuge test, which was adopted from Weij (2018) and To et al. (2016). This method was used to assess the dewaterability at laboratory scale in the same way as in full-scale, by looking at both dryness of dewatered sludge cake and PE consumption. The results from modified Higgins test were introduced in a correlation matrix to see how they are correlating to the full-scale sludge dewatering results and also the sludge characteristics.
The samples were compared based on the TS concentration (%) of the dewatered sludge cake and the required PE dosage in gr active PE/kg TS of sludge. A PE solution of 0.3% active w/v was used to form sludge flocs prior to the centrifugation. The PEs used for Higgins test were the same as the ones used in full-scale practice The tests were conducted using a lab-scale centrifuge model Roto Silenta 630 RS (Hettich, Germany). More details of the methodology are given in appendix A1.2.

STREAMING CURRENT POTENTIAL (SCP) AND POLYELECTROLYTE DEMAND

SCP measures the surface charge of sludge particles. Zeta potential and SCP are measured in different ways, but they are both describing how negatively sludge particles are charged (Product sheet Mütek™ PCD-06). The SCP value for sludge samples is negative due to negative surface charge of sludge particles. This measurement was performed using a Mütek™ PCD-06 streaming current detector. The samples were diluted 50 times with NaCl solution at

900 µS/cm. During SCP measurement, a standard cationic PE solution was added to the sludge samples up to the point where a SCP of 0 mV was reached (isoelectric point). The more PE is used the more negatively charged the sludge sample is. The SCP PE demand to reach the isoelectric point was recorded for each sample and introduced in a correlation matrix to see if it is correlating to the full-scale and lab-scale sludge dewatering results.

SPECIFIC RESISTANCE TO FILTRATION (SRF)

SRF is a commonly used methodology in sludge dewaterability research that evaluates the sludge filterability (To et al. 2016). SRF is a measurement methodology that is relatively independent of the TS concentration of a sample, therefore, it can be used for sludge samples with different TS concentrations (To et al. 2016). When the SRF value increases, the sludge filterability decreases, and it can be considered as deterioration of sludge dewaterability. The SRF was measured and introduced in a correlation matrix to see how it relates to the sludge dewatering results in full-scale and lab-scale.

SRF was measured by using the methodology described by Dereli, Grelot et al. (2014). Firstly, the sludge samples were diluted to 10 g TSS/L using demineralised water, and the temperature equalised to 20°C. The diluted sludge samples were placed in a 50 mL Amicon® filtration cell (Merck Millipore, Germany) and filtered by dead-end filtration at a trans-membrane pressure of 500 ± 5 mbar. The filtration speed was assessed by online recording the permeate weight using a balance model XS2002S (Mettler Toledo, Switzerland). The measurements were recorded every 6 seconds for 30 minutes (300 points in total). The permeate density was assumed to be equal to the density of water (ρ_{water}=1 g/mL). A Whatman glass microfiber filter Grade GF/C-1.2 µm (GE Healthcare Life Sciences, USA) was used to conduct the filtration. The dynamic viscosity of the permeate (η) was measured using a 50 mL Cannon-Fenske routine viscometer, where the viscosity of the water at 20°C was used as a reference (1 mPa·s). The SRF was calculated according to the equation below.

$$SRF\ [m/kg] = \frac{200 \cdot TMP \cdot A^2 \cdot b}{\eta \cdot C} \qquad \textit{Equation 1}$$

Where:
TMP= transmembrane pressure [mbar]
A= filtration area [m^2]
b= slope between t/V and V [s/m^6]
η: dynamic viscosity of the filtrate [Pa·s]
C: TSS concentration [kg/m^3].

The slope between t/V and V was measured in the linear range, considering 100 values. All samples were measured in triplicate.

CAPILLARY SUCTION TIME (CST)

CST is frequently measured in sludge samples to assess dewaterability, due to the fast implementation and operation (To et al. 2016). A sample with elevated CST value has lower filterability which means lower dewaterability (To et al. 2016). The CST was measured and normalized over the TS concentration of a sample to make it independent of TS concentration. The normalized CST was introduced in a correlation matrix to see how it is relating to the sludge dewatering results in full-scale and lab-scale.

CST was measured in a CST apparatus model 304M (Triton Electronics Ltd., United Kingdom) and standard CST paper from the same brand. To measure CST, 7 mL of sludge without polymer was added to a 1.8 cm stainless steel funnel and the CST recorded (Dereli, Grelot *et al.* 2014). All samples were measured in triplicate. Furthermore, to compare the different samples the CST values were normalised by dividing the CST value by the TS concentration in the sludge.

1.3.2 SLUDGE CHARACTERIZATION

ANALYTICAL MEASUREMENTS

Total solids (TS) and volatile solids (VS) were measured to define the inorganic and organic content of sludge. Total suspended solids (TSS) and volatile suspended solids (VSS) were also measured in the centrate and filtrate samples. TS, VS, TSS, and VSS were measured according to the Standard Methods (APHA, AWWA, WEF, 2012). These parameters were later used in a correlation matrix to assess the correlation with the sludge dewaterability. The centrate and filtrate measurements were conducted to judge the dewaterability of the sludge. A dark centrate/filtrate means a poor dewaterability, see also appendix A1.1

TS and VS content of sludge were measured because it was previously observed that elevated VS and TS concentration showed a negative impact on sludge dewaterability measured as CST.

Chemical oxygen demand (COD) was determined using the test kit LCK 014 and LCK 514 kits (Hach-Lange, USA). Three different types of COD were measured to examine if they can be linked to changes in dewaterability of sludge as follows:

- Total COD (tCOD): measured in the whole sludge samples.
- Soluble COD (sCOD): Measured in the permeate of 0.45 µm filtered samples.
- Centrifugation COD: Measured in the permeate of centrifuged samples at 15,000 RCF for 15 min.
- Colloidal COD: Calculated as the difference between centrifugation COD and soluble COD.

Total phosphorous (total P) was measured in the whole sludge samples using the test kit LCK350 (Hach-Lange, USA), digesting the samples according to the manufacturer instructions. Total ammoniacal nitrogen (TAN) and orthophosphates ($PO^{3-}{}_4$-P) were measured in the soluble part of the sludge and in the reject waters. TAN and orthophosphates were measured spectrophotometrically using a discrete analyser model AQ400 (Seal Analytical, USA), following the methods EPA-350.1 (SEMI 1993) and EPA 365.1 (Agency 1993), respectively.

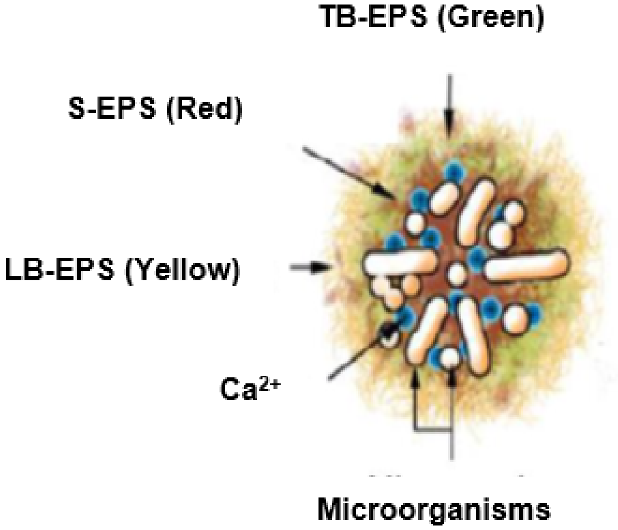
EXTRACELLULAR POLYMERIC SUBSTANCES (EPS) EXTRACTION AND QUANTIFICATION

EPS are negatively charged natural biopolymers that act as glue that binds cells together to form a sludge floc and interact with water molecules and cations. EPS can absorb water molecules forming a hydrogel structure (Mowla et al. 2013). EPS has a multi-layered structure (Figure 2). These layers can be expressed as soluble EPS (S-EPS), loosely bound EPS (LB-EPS) and tightly bound EPS (TB-EPS) (Guo et al. 2020) (Figure 2).

EPS fractions were extracted using a mild-harsh heat method described by Li and Yang (2007). Once the EPS fractions were extracted, total organic carbon (TOC) was measured in every fraction. TOC was measured using the combustion-infrared method with a TOC analyser model TOC-V CPH (Shimadzu, Japan).

EPS fractions and concentrations are measured in this study, because in previous studies presence and changes of EPS layers were linked to changes in sludge dewaterability (Zhang *et al.* 2019a; Xu et al. 2018a, Berkhof/STOWA 2016, Berkhof/STOWA 2018). However, their link to changes in dewaterability results in terms of dewatered sludge cake dryness and PE consumption is not (yet) clear. Therefore, in this study they are introduced in a correlation matrix to see if a relationship can be found between EPS and sludge dewaterability parameters.

FIGURE 2 DIFFERENT FRACTIONS OF EXTRACELLULAR SUBSTANCES (EPS) SURROUNDING MICROORGANISMS AND CATIONS (CA^{2+}). SOURCE: ADAPTED FROM GUO *ET AL.* (2020)



CATIONS MEASUREMENT

Cations affect sludge characteristics and especially flocculation and dewaterability since they connect the EPS fractions and play an important role in forming the matrix of the floc by entrapping cells, particles and organic and inorganic substances (Mirzaee 2021; Prodănescu 2017; Urbain et al. 1993).

In previous research, the presence of monovalent cations such as Na^+ and K^+ seemed to have a negative effect on dewaterability (Mirzaee 2021; Prodănescu 2017; Sobeck and Higgins 2002). Excess of Na^+ or K^+ cations caused the floc to break and led to higher solubilization of biopolymer and capillary suction time increased, indicating a deterioration of dewaterability.

Divalent cations such as Ca^{2+} and Mg^{2+} are known to have a positive impact on sludge dewaterability since they have a high affinity for charged proteins which may lead to a lower PE use (Berkhof/STOWA 2016). The addition of divalent cations decreases the floc surface charge, resulting in an improved dewaterability.

There are different opinions about the effect of trivalent cations such as Fe^{3+} and Al^{3+} on dewaterability (Li et al 2005; Wilén et al. 2003). Some researchers observed positive impact on dewaterability when Fe^{3+} was added since CST values decreased significantly (Novak et al. 2001). Also, previous research by STOWA (Berkhof/STOWA 2016 and Berkhof/STOWA 2018) showed positive effects of the addition of iron salts on sludge dewaterability. These positive effects are mainly reported for trivalent iron that is dosed just before the sludge dewatering. The effect of iron that is already present in the sludge is less evident although in general sludges from plants using iron to remove phosphate show a better dewaterability then plants using enhanced biological phosphorus removal. Most of the iron dosed in the water line will be reduced to divalent iron in the sludge digester and will bind with sulphides and phosphate (Wilfert 2018). Most of the iron in sludge is therefore probably not available for cation bridging in the EPS structure. Aluminium is mostly present as insoluble clay particles or as aluminium phosphates and is not very soluble and will therefore also not contribute significantly to cation bridging.

In this research, we decided to look mainly into monovalent-divalent ratio (M/D) since it is known to be related to changes in sludge dewaterability (Higgins and Novak 1997). Therefore, the cation concentrations were measured in this study and introduced in a correlation matrix in order to find a link between a change in cations content of the sludge and a change in full-scale and lab-scale dewatering results. Furthermore, the possible correlations with the iron content were checked separately.

Ca^{2+}, Mg^{2+}, Na^{+}, K^{+}, $Fe^{2+/3+}$ and Al^{3+} were measured in the sludge and reject water samples using inductively coupled plasma optical emission spectroscopy (ICP-OES). The analysed samples were previously mineralised using aqua regia and microwave digestion, following the method EPA 3051A (U.S. Environmental Protection Agency, 2007). The cation concentrations were measured using an ICP-OES Model 5800 (Agilent, USA).

Additionally, the M/D was calculated using the concentrations of cations measured in the reject waters. M/D was measured using the method proposed by Higgins and Novak (1997). M/D was calculated both with and without considering TAN concentration. M/D and M/D_{+TAN} were calculated according to Equation 2 and Equation 3, respectively.

$$\mathrm{M/D} = \frac{1\cdot[Na]+1\cdot[K]}{2\cdot[Ca]+2\cdot[Mg]} \qquad \textit{Equation 2,}$$

$$M/D_{+TAN} = \frac{1\cdot[Na]+1\cdot[K]+1\cdot[TAN]}{2\cdot[Ca]+2\cdot[Mg]} \qquad \textit{Equation 3,}$$

Where:
[X]= molar concentration of the X-th metal (X= [Na, K, TAN, Ca, Mgl]).

SOLUBLE BIOPOLYMERS

Soluble proteins and carbohydrates were measured in the soluble fraction of the sludge samples to assess if they can be linked to changes in the sludge dewaterability. Soluble carbohydrates were measured according to the phenol—sulphuric acid method (Dubois, Gilles et al. 1951). Soluble proteins and humic substances were measured based on the modified Lowry method, considering the interference of humic substances, according to Fr, Griebe *et al.* (1995).

PARTICLE SIZE DISTRIBUTION (PSD)

It was concluded in previous studies that particle size and particle size distribution (PSD) can have a significant impact on dewaterability. This was based on the finding that pre-treatment methods that changed the PSD improved dewaterability (Houghton and Stephenson 2002; Mowla et al. 2013). In a study by Shao et al. (2009), samples with larger particles showed higher CST values which was translated into deterioration of dewaterability. Therefore, PSD measurements were done and used in inserted in a correlation matrix to see how they are linked to sludge dewaterability results both in full-scale and lab-scale.

PSD was determined by a particle size analyser model Bluewave (Microtrac, USA) using demineralised water as a carrier, following the manufacturer instructions. The mean particle size value was reported in this study and used in the correlation matrix. The samples were measured in triplicate.

RHEOLOGY ANALYSIS

Rheology provides information about sludge structure strength which eventually can have an impact on the sludge dewaterability (Zhang et al. 2019a). Therefore, rheology measurements were done in this study and inserted in the correlation matrix to assess their relationship to sludge dewatering results both in full-scale and lab-scale.

Shear stress was measured at incremental shear rates at 35°C using a concentrical cylinders rheometer model Anton Paar MCR 302 (Anton Paar GmbH, Austria). The procedure was adapted from (Cao, Pan *et al.* 2021), in which the samples were pre-sheared at 1000 s^{-1} for 5 min and let it rest for 1 min to consider the sludge thixotropy. The Hershel-Bulkley model (Equation 4) was fitted to the rheograms measured, and the model parameters (τ_0, and n) used in the correlation matrix.

$$\tau = \tau_0 + k\dot{\gamma}^n$$ *Equation 2,*

where:
τ = Shear stress (Pa)
τ_0= Yield stress (Pa)
k = Consistency index (Pa sn)
$\dot{\gamma}$ = Shear rate (1/s)
n = Flow index (dimensionless)

ZETA POTENTIAL MEASUREMENT

Sludge suspensions are known to have a negative surface charge and this charge can be quantified by zeta potential measurements (Mowla et al. 2013). This negative charge stabilises the suspension since it can keep cells apart. Also, for sludge samples with more negative charge, more PE with positive charge must be used to be able to destabilise the suspension and to form flocs (To et al. 2016). Therefore, zeta potential measurement is studied in this research to see how it is correlating to sludge dewaterability and specially to PE consumption. The zeta potential of sludge samples and their reject waters was measured using a Zetasizer Nano ZS (Malvern Panalytical, UK). To measure zeta potential the samples were sonicated in a Branson 2510 Ultrasonic Cleaner (Branson Ultrasonics, USA), for 30 minutes to break the clumps and flocs in the sludge structure. After sonication, the sludge samples were diluted 16 times with a 450 µS/cm NaCl solution. The reject water samples were diluted twice with the same NaCl solution. After the dilution, 1.5 mL of NaCl-diluted samples were transferred to a DTS 1070 cell to measure the zeta potential according to the manufacturer’s instructions.

1.3.3 PE CHARACTERIZATION

The main aim of this study was to study possible correlations between sludge dewaterability and sludge parameters. However, changes in the PE used for the dewatering, both at full scale and lab scale could influence these correlations. Therefore, for this study the PE that used at the two WWTP’s was regularly analyzed to check if there were any significant changes to the PE quality that could have an impact on the understanding of the results.

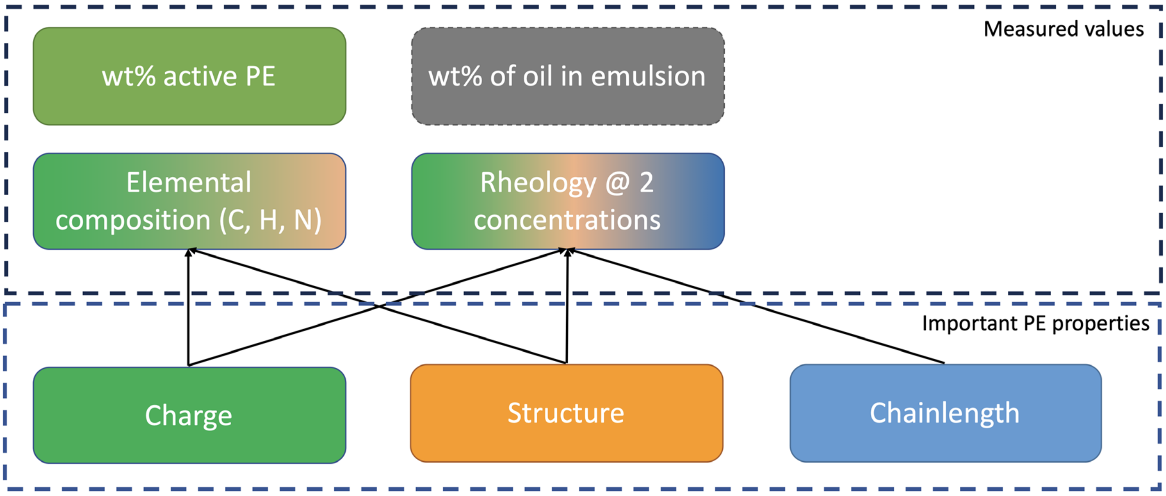
For the characterization of the PE a method was used that was first developed for STOWA (see Raffa/STOWA 2017) and has thereafter been further optimized and accredited by Intertek for the Vereniging van Zuiveringsbeheerders. In the last three years Dutch water authorities have regularly supplied PE samples for this characterization and a large dataset of the quality parameters for different PE sludges has been obtained. More information on this flanking project and the analysis strategy can be found in (Korving 2023). In this report we give a short summary.

The method determines:

- The quantity of active material in the PE emulsion. This is done via an extraction in acetone. In the case of the PE of Harnaschpolder this step is not relevant because a powder PE is used. This measurement has an uncertainty of +/- 10%.
- The oil quantity in the polymer sample by determining the oil extracted with the acetone with gas chromatography. This measurement still has a relatively large uncertainty of +/- 35%.
- The elemental composition (C, H, N) is then determined on the extracted PE using an elemental analysis which involves incineration of sample material and measuring the gas composition. This measurement of C, H and N percentage has an uncertainty of respectively 5, 15 and 10%.
- The rheology of a 0,1 and 1,0 wt% PE solution made using the extracted PE. The rheogram is fitted to the power law which gives three parameters for each concentration:
  - Maximum viscosity at low shear (mPa.s)
  - Flow consistency index k (Pa.s)
  - Flow behavior index n (-).

These parameters all have a measuring uncertainty of +/- 12%.

FIGURE 3 **OVERVIEW OF PARAMETERS MEASURED TO CHARACTERISE A PE SAMPLE AND HOW THESE PARAMETERS RELATE TO DIFFERENT IMPORTANT PE PROPERTIES (KORVING 2023)**



The main properties of a PE product that influence its performance are charge density, structure and chain length. All these three parameters influence the rheology of a PE solution and therefore rheology parameters are important indicators of PE quality. However, in theory two PE's with a different structure could show similar rheology at a certain concentration but it is unlikely that they are similar at two different concentrations since different PE properties (charge, structure, chain length) show different changes in rheology as a function of concentration. For this reason, the rheology is determined at two concentrations.

Together all these parameters give insight into the main properties of a PE product.

However, it has to be realized that the method should be considered a “fingerprint” method because the measured parameters do not have a direct correlation to the main properties of a PE product.

It has to be noted that also the preparation of the PE before use and the mixing into the sludge influence the quality of the PE (Korving/STOWA 2014). For this study it was assumed that the way of preparation would not change significantly over time and diluted PE samples were taken to ensure that in the lab-scale tests a PE solution was used that was the same as in full scale. A possible disadvantage of this approach can be that some of the PE hydrolyses if the sample is not used quick enough after sampling (Saveyn 2013).

1.4 DATA AND STATISTICAL ANALYSES

FULL-SCALE DATA

Full-scale operational data were received from both WWTPs, and the datasets were cleaned to only include datapoints that were based on actual measurements either in the laboratory or from online sensors. The time span of the data for both location is from January 1st until October 31st for years 2022 and 2023.

CORRELATION MATRIX

A correlation matrix between specific measured parameters was build using the Kendall rank correlation coefficient. The correlation matrix shows how parameters correlate to each other and how strong this correlation is. The Kendall correlation matrix shows monotonic correlations. The parameters should not necessarily correlate linearly. The Kendall non-parametric index is recommended for small datasets such as the one in our present study (Field 2013), which comprised only six points per studied WWTP. The plotted points only correspond to the correlation coefficients that were significantly different to zero at 95% of confidence.

1.5 PARAMETERS CATEGORISATION

The parameters studied in this research were categorized (Table 2), from ‘easy to measure and possible on site’ (in the laboratory of a common wastewater treatment plant) to ‘difficult to measure’ (specialised lab and personnel required). In this study we were looking for predictors of sludge dewaterability as ‘easy’ as possible.

TABLE 2 **CATEGORIES OF PARAMETERS**

Category	Test
Easy and possible on site	TS & VS
	CST
Easy via external analysis with regular lab	Total P
	TAN
	Cations
More difficult and could be done on site in practice	Modified Higgins test
	Rheology
	SRF
	SCP
	PSD
	TS/VS/TSS/VSS/COD/sCOD
Difficult (Requires skilled person) and requires specialised lab	Proteins and polysaccharides
	EPS extraction (TB-EPS/LB-EPS/S-EPS)
	TOC
	Zeta potential

2

RESULTS

In this section the main outcomes of the study are discussed. An important tool for the study was to identify important correlations between different parameters. For this reason, a Kendal correlation matrix was made showing the correlation between different parameters. This matrix can be found in appendix A2. The correlation matrix only shows significant correlation between two parameters at 95% of confidence. Kendall correlation coefficients range from -1 to 1, in which -1 means a perfect negative correlation and 1 a perfect positive correlation. A correlation coefficient close to 0, means a weak correlation.

The datasets from the two different WWTP's were merged and used in one correlation matrix to see if there were correlations that were not dependent on the location. In practice the results show that there are different correlations for each WWTP, which is logical because different types of dewatering machines are used (Bath: belt filter presses, Harnaschpolder: decanter centrifuge). Therefore, in this result section we focus on the correlation for each WWTP separately.

We first discuss the representativeness of the collected samples in section 3.1 In section 3.2 we then evaluate how the lab scale dewaterability tests correlate with the large-scale dewatering results. Then, in the next sections we take the lab-scale dewaterability tests as a reference point and then study interactions between dewaterability and sludge parameters that can possibly explain the dewaterability result. The lab-scale dewaterability results were used as a reference because since these tests were performed in a standardised method, whereas at full scale other influences on sludge dewatering might have played a role.

2.1 REPRESENTATIVENESS OF THE SAMPLES

Figure 4 and Figure 5 show the variation in cake dryness and PE consumption during the two sampling years and also show the moments where samples were taken. For WWTP Bath the data show that the sampling dates give a variability in cake dryness and PE consumption that fits with the normal variation in this data over the two years. For WWTP Harnaschpolder the yearly data shows that there were several situations where cake dryness and PE consumption was significantly higher than at the six sampling dates. In hindsight it would have been interesting if samples were taken during the periods of higher dry solid content and higher PE dosage

FIGURE 4 TH FULL-SCALE DATA FOR YEARS 2022 AND 2023 A) TS CONCENTRATION OF DEWATERED CAKE B) PE CONSUMPTION

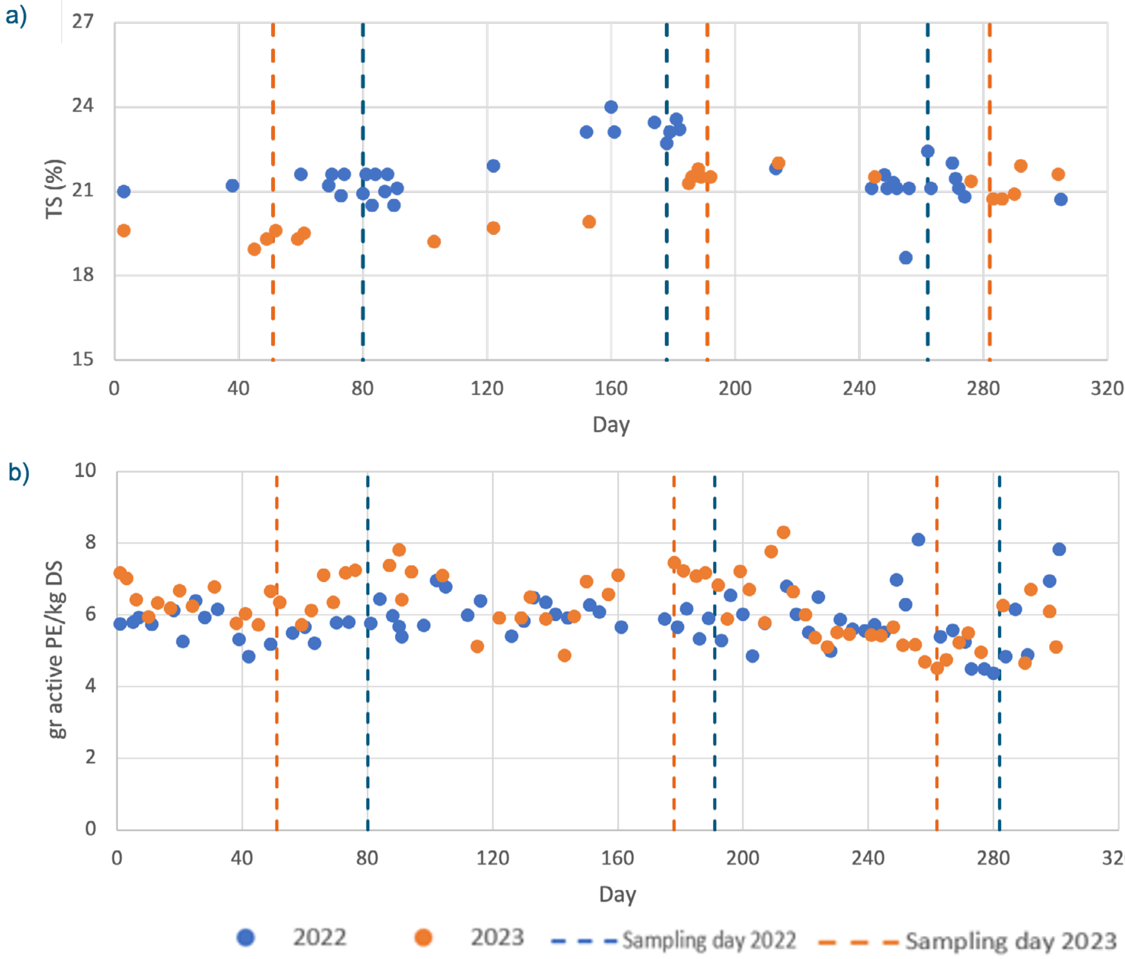
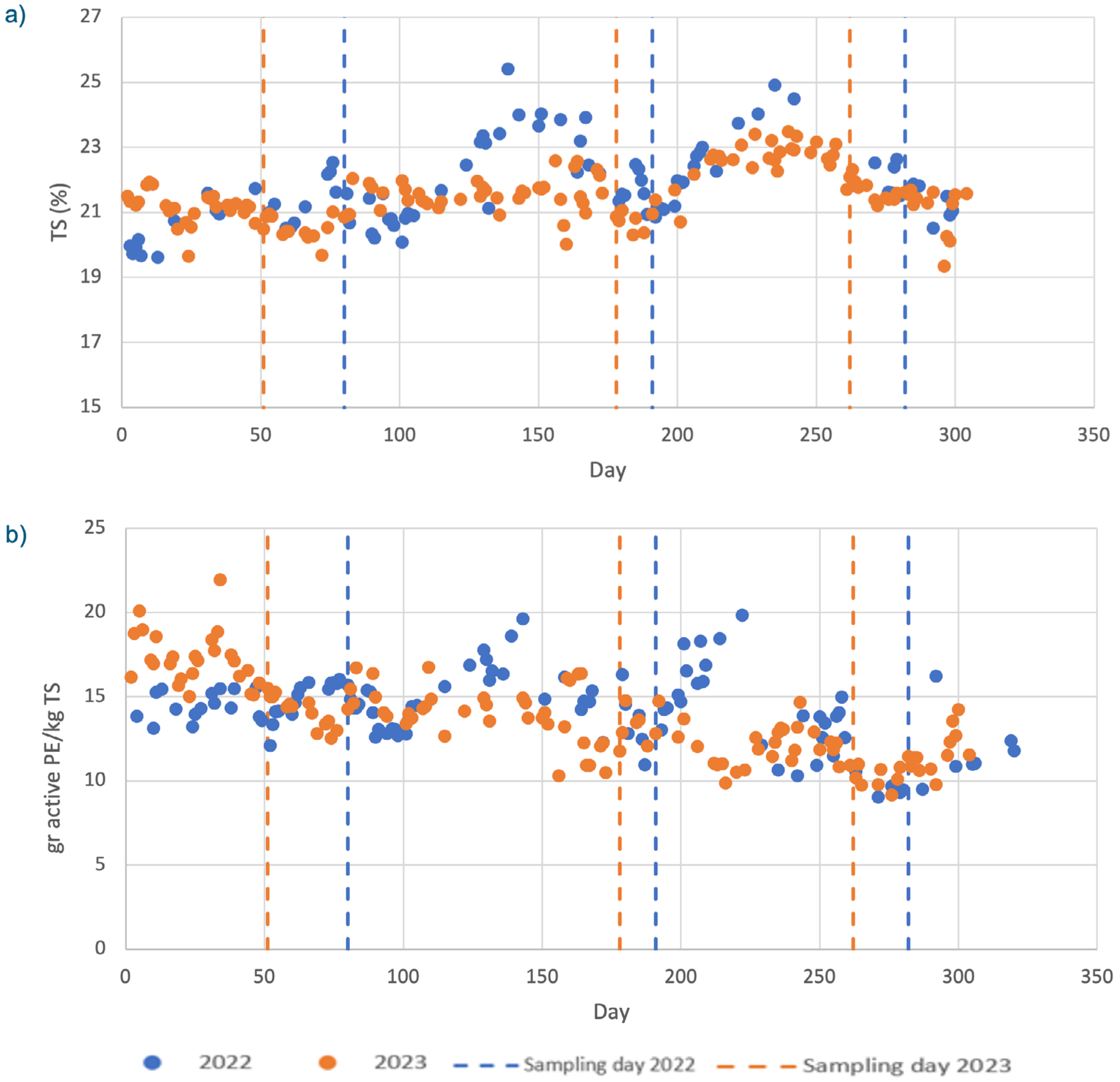


FIGURE 5 HARNASCHPOLDER FULL-SCALE DATA FOR YEARS 2022 AND 2023 A) TS CONCENTRATION OF DEWATERED CAKE B) PE CONSUMPTION



2.2 FULL-SCALE AND LAB-SCALE DEWATERABILITY

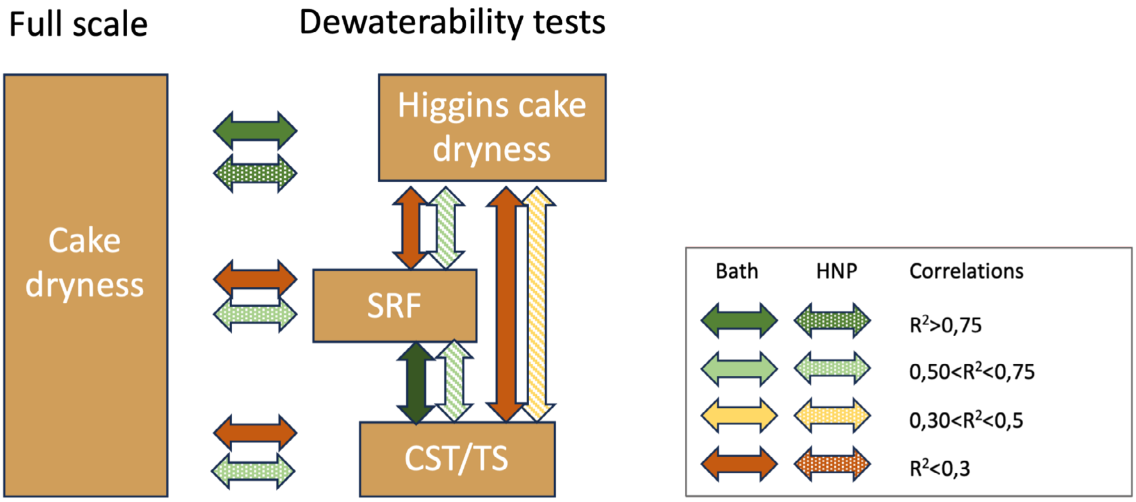
For this study we selected a number of labscale tests that are designed to mimic all or some aspects of the sludge dewatering process. In this section we first discuss the correlation with these tests and the full-scale results.

2.2.1 CORRELATION WITH FULL-SCALE CAKE DRYNESS

The Modified Higgins centrifuge test, the specific resistance to filtration (SRF) and the capillary suction test (CST) are all methods to evaluate how easily water can be removed from sludge and could therefore in theory have a correlation with the cake dryness obtained in a full-scale dewatering facility. Therefore, the correlation with full scale dewatering results and their mutual correlations were evaluated. For this evaluation the CST value was normalised for the dry matter content of the sludge (CST/TS).

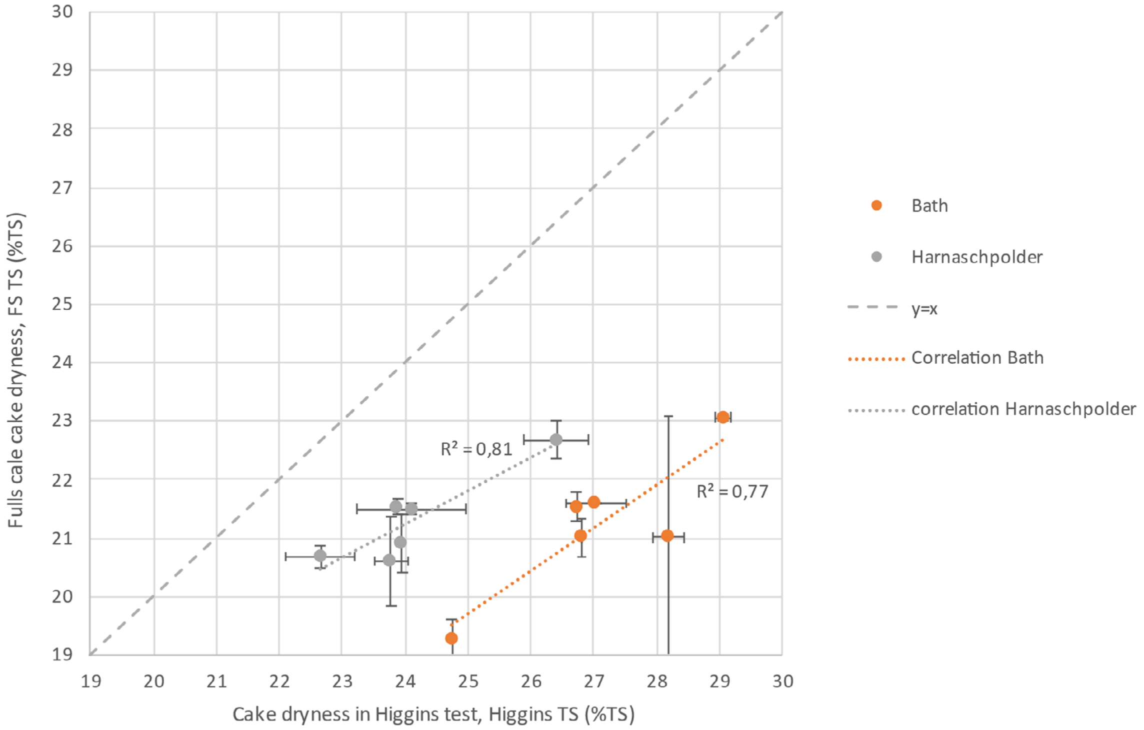
Figure 6 summarises the correlations between the different tests. The results show that the Higgins test shows the best correlation with the full-scale data. The SRF and CST tests also show correlations with the full-scale data for Harnaschpolder but not for Bath. For Harnaschpolder these correlations are weaker than for the Higgins test. Interestingly SRF and CST also show a reasonable correlation with each other, but less with the Higgins tests. This suggests that the Higgins test incorporates aspects that are relevant for dewatering but are not an element of the SRF and CST test.

FIGURE 6 **STRENGTH OF THE CORRELATIONS BETWEEN FULL SCALE CAKE DRYNESS AND LAB-SCALE DEWATERING TESTS**



Because the other correlations for SRF and CST with the full scale and Higgins cake dryness were relatively weak they will not be shown in this section. Figure 7 zooms in on the comparison of the Higgins cake dryness results and the full-scale cake dryness. The figures shows that data on cake dryness of the dewatered cake at full-scale correlates strongly with the cake dryness in the Higgins test. This shows that the Higgins dewaterability test is potentially useful to predict changes in full-scale cake solids concentration. It can also be seen that values from Higgins were always higher than those measured at full-scale. This suggests that with Higgins test a reference dewaterability of a sludge sample can be estimated but that in practice the dewaterability will be lower. This is due to the fact that the mixing and floc forming in the lab are more ideal than in the full-scale test.

FIGURE 7 TS CONCENTRATION OF DEWATERED CAKE: FULL-SCALE VS HIGGINS TEST



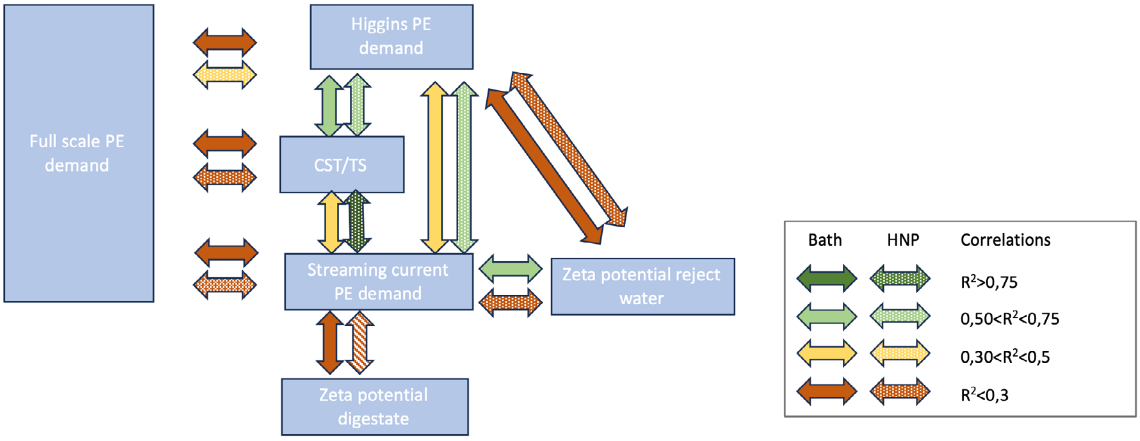
The difference between the results of the Higgins test and the results in practice will depend very much on the type of dewatering installation and may also vary from WWTP to WWTP. Therefore the correlation between the Higgins test and the results in practice will probably have to be evaluated for each WWTP separately. The difference in distance between the ideal correlation (y=x) is larger for Bath compared to Harnaschpolder. This is logical because the Bath WWTP uses a belt filter press for dewatering and Harnaschpolder uses dewatering centrifuges. From practice it is known that centrifuges can achieve higher cake dryness. Nevertheless, it is interesting to see that the Higgins centrifuge test also correlates with the full-scale results for Bath despite the different full scale dewatering approaches.

Based on the good correlation of Higgins with the full-scale cake dryness the Higgins test is used in the next sections as a reference to study correlations with sludge properties. Compared to using the full-scale dewatering results, the Higgins test has the advantage of being performed in a similar way for Bath and Harnaschpolder sludge and is therefore more suitable to find universal correlations that are valid for both sludges.

2.2.2 CORRELATION BETWEEN FULL-SCALE PE CONSUMPTION

The modified Higgins test (Higgins PE demand) and the streaming current PE demand are two tests that give a direct estimate of the optimal PE dosage for a good dewatering result. The streaming current potential measures the charge of the sludge and then PE is added until the charge is zero. The zeta potential also gives an indication of the charge of the sludge and could therefore also be a predictor for the PE consumption and was measured both directly on the digested sludge and on the reject water after dewatering. Finally, some publications mention the CST test to correlate with PE consumption. Therefore, the correlation with the full-scale PE consumption and the mutual correlations were analysed to understand how consistent these potential predictors for PE consumption were. Figure 8 gives a simplified overview of the main correlations.

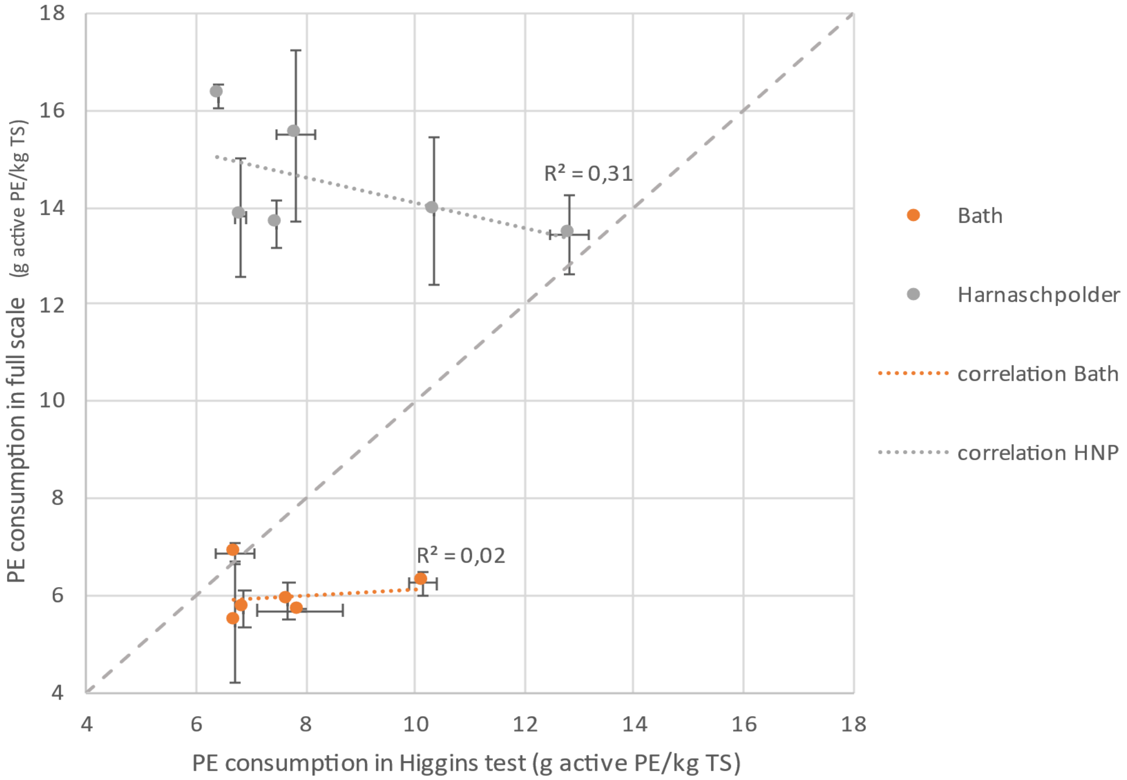
FIGURE 8 **STRENGTH OF THE CORRELATIONS BETWEEN FULL SCALE PE CONSUMPTION AND LAB-SCALE TESTS**



The figure shows that unfortunately none of the lab scale tests showed a correlation with the full-scale results. This is further illustrated by Figure 9 for the Higgins PE demand. This can be due to the following reasons:

- The optimization of the dewatering equipment was not implemented properly on the sampling day itself. Thus, the full-scale data can also have been affected by the operational parameters of the dewatering equipment and not only by changes in sludge characteristics.
- There was not a significant difference in the PE consumption and TS concentration of the dewatered sludge cake from full-scale data on the sampling days (shown in Figure 5 (Harnaschpolder) and Figure 4 (Bath)). When the values from full-scale data remain constant, only low correlations can be made.

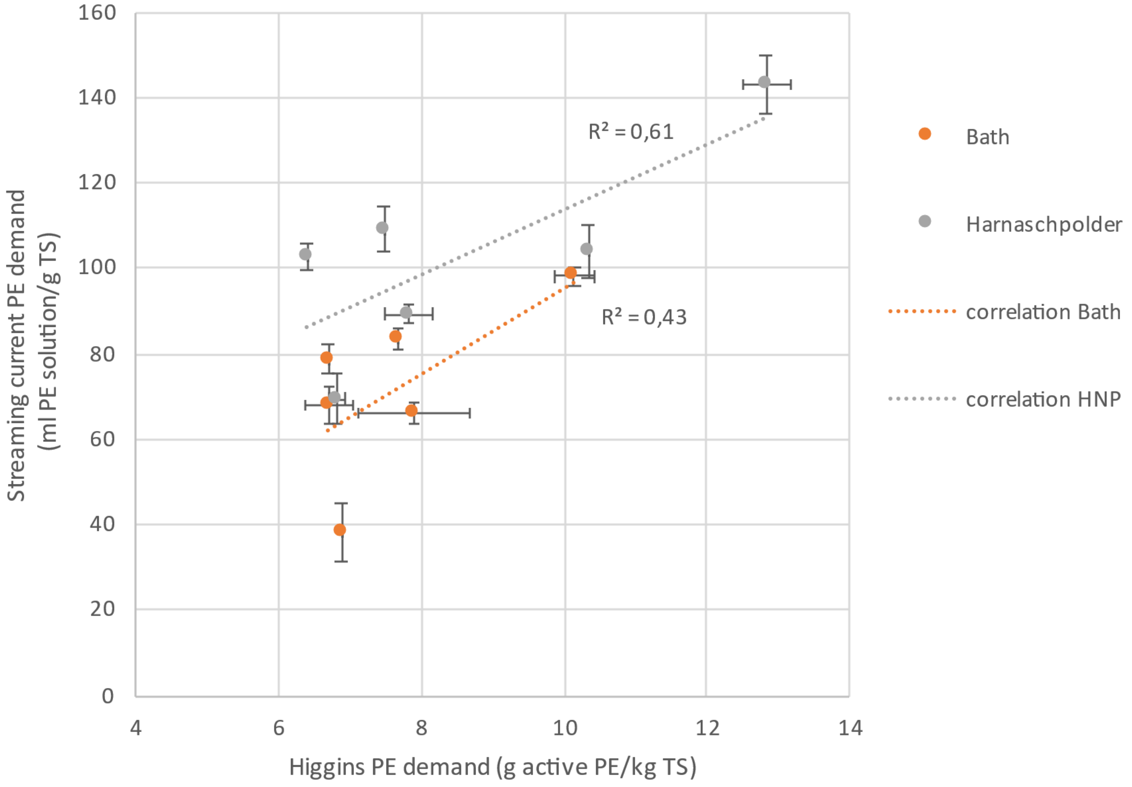
FIGURE 9 **PE CONSUMPTION FOR DEWATERING: FULL-SCALE RESULTS VS HIGGINS TEST**



Although there was no clear correlation with the full-scale results, the lab-scale experiments did show internal correlations. However, these were not so strong that one could state that these tests are fully measuring the same properties. For instance, Figure 10 shows the correlation between the Higgins PE demand and the streaming current PE demand. There is a correlation but not very strong, so both parameters could still be different predictors for the PE consumption.

SUMMARY

FIGURE 10 **PE CONSUMPTION IN THE HIGGINS TEST VERSUS STREAMING CURRENT PE DEMAND**



Since the full-scale PE dosage is most probably not optimised and not representative for “the” PE use of the sludge, it would still be useful to have a reference for the polymer use to evaluate how other sludge parameters could influence the PE use. The Higgins PE demand and the streaming current PE demand are logical candidates because these are both the most direct tests to determine the polymer consumption. However, it cannot be concluded which one is the best at this point. Therefore, both parameters will be used as a reference for the PE use in the next sections.

Finally, it is interesting to note that the CST result shows correlations with both Higgins PE demand and streaming current PE demand. This is nice because it confirms that these three parameters are evaluating similar sludge properties. Contrary, the zeta potential measurements do not show correlations with the other parameters. Only the zeta potential in the reject water correlates somewhat with the streaming current PE demand (but not with Higgins PE demand).

In summary these are the main conclusions from the evaluation of different tests that are available to test the dewaterability of sludge at lab scale.

CORRELATION BETWEEN CAKE DRYNESS AT FULL SCALE AND LAB DEWATERING TESTS

- Higgins cake dryness showed best correlation with full scale dewatering cake dryness for both WWTP's. For Harnaschpolder other tests (SRF, CST) also showed correlations but these were weaker than for Higgins cake dryness. For Bath only Higgins cake dryness showed a correlation.
- The correlation between Higgins TS and full-scale TS was different for the different WWTP (albeit with a similar slope). This can be explained by the use of different dewatering machines at full scale. Bath uses a belt filter press which will give a lower dry cake solids concentration compared to the use of a centrifuge at Harnaschpolder.
- For this reason, Higgins TS was used as a reference to check which sludge composition parameters have a correlation with cake dryness. This method will give comparable results for different sludges and will enable to make broader conclusions then when full scale TS would be used.

CORRELATION BETWEEN PE CONSUMPTION AT FULL SCALE AND LAB DEWATERING TESTS

- The PE consumption at full scale showed little variation whereas different lab tests that should have a relation to PE demand showed large variations. Therefore, none of the available lab tests showed a reasonable correlation to the full-scale PE demand. This could indicate that the full-scale results were therefore not optimal.
- The most direct estimates at lab scale for PE consumption are the Higgins PE demand and the streaming current PE demand. Both were therefore used as a reference for PE consumption of a sludge. The Higgins test showed the best and most correlations with other parameters (see next sections). Therefore, Higgins PE demand is considered the best lab scale parameter to evaluate sludge PE use.
- CST also showed a good correlation with Higgins PE demand, but it does not give a direct estimate of PE consumption and was therefore not used as a reference for PE use for a certain sludge.

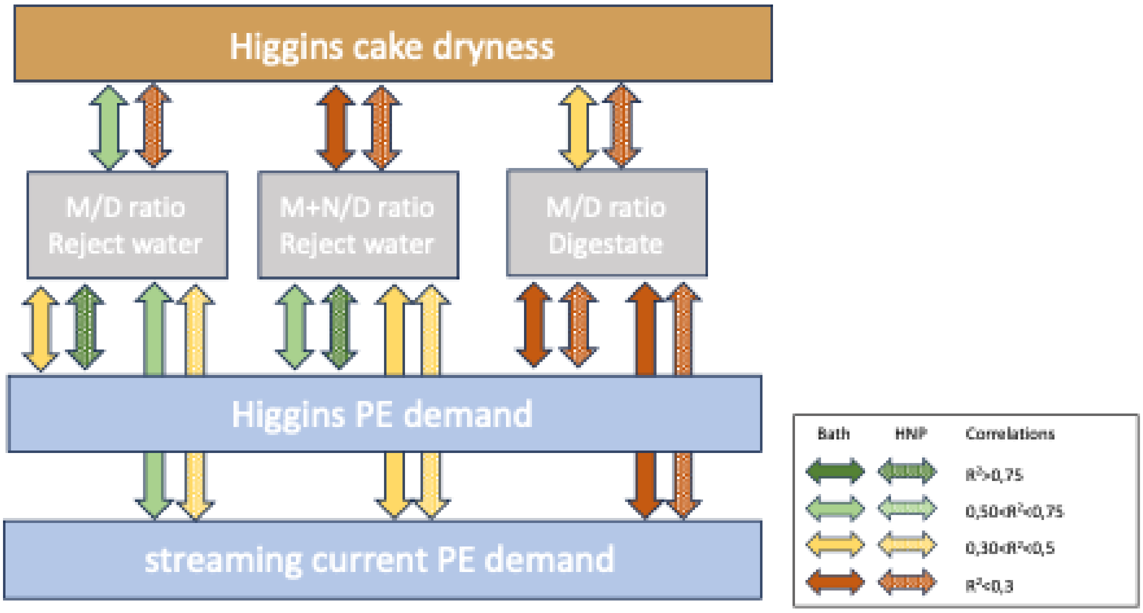
2.3 RELATION BETWEEN SLUDGE DEWATERABILITY AND SLUDGE CHARACTERISTICS

2.3.1 MONO-DIVALENT CATION RATIO

The mono to divalent cation ratio (M/D ratio) has been mentioned in literature as an important factor influencing the sludge dewaterability. The monovalent ions are considered to be sodium and potassium and the divalent ions magnesium and calcium. The ratio was determined for the reject water (the sludge soluble phase, RW) and for the total sludge composition (DIG). Ammonium is sometimes also included in the M/D ratio and was therefore also included in a separate M/D ratio (M+N/D).

For these M/D ratio's correlations were evaluated with the cake dryness and PE use (determined via to the Higgins centrifuge test). In addition, possible correlations with the SP demand were evaluated as a second indicator for the PE use. Figure 11 shows how strong the different correlations were for these parameters for each WWTP.

FIGURE 11 STRENGTH OF THE CORRELATIONS BETWEEN DIFFERENT MONO TO DIVALENT CATION RATIO'S (M/D RATIO) FOR THE REJECT WATER WITHOUT AND WITH AMMONIACAL NITROGEN (M+N/D) AND FOR THE DIGESTATE



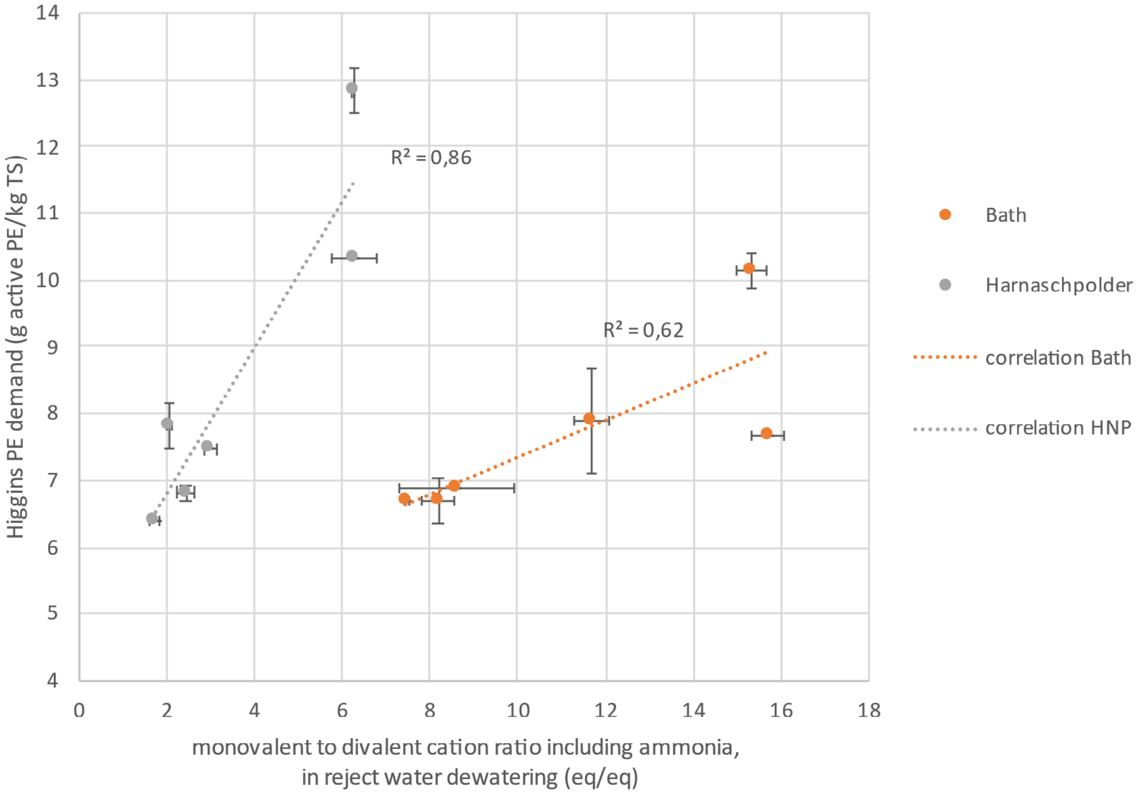
The results show very little correlation between the cake dryness and the different M/D ratios. Only a slight correlation was found for the sludge of Bath for the M/D ratio in the reject water (R^2=0,5). Better correlations were found with the PE demand determined via the Higgins test, especially if ammonium was included in the correlation, see Figure 12. This suggests that ammonium influences the PE demand. This influence is however only there in a connection to the other cations because ammonium alone did not show a correlation with PE demand (see section 2.3.2). The increasing trend in PE use with increasing monovalent ions is in line with the theory that these monovalent ions replace the divalent cations in the EPS matrix that serve as bridges between the EPS polymers. Due to their single charge monovalent ions cannot perform this bridging function.

If ammonium was excluded in the calculation of the M/D ratio in the reject water than the correlation stayed more or less equally strong for the Harnaschpolder sludge (correlation coefficient was 0,90 without ammonium) but became less for the Bath sludge (correlation coefficient reduced to 0,46) indicating that the role of ammonium was more relevant for Bath than for Harnaschpolder.

Interestingly the correlations with the M/D ratio were much less pronounced for the streaming current PE demand. This difference highlights that the determination of the PE demand via the Higgins approach may not describe fully the same sludge property as the streaming current PE demand.

The M/D ratio in the total sludge showed no relevant correlations with sludge dewatering properties. This is also more or less expected because according to the theory only the mobile divalent cations can contribute to the EPS structure and the total divalent cation concentration in the sludge will also include precipitated divalent cations that have no influence on the dewaterability.

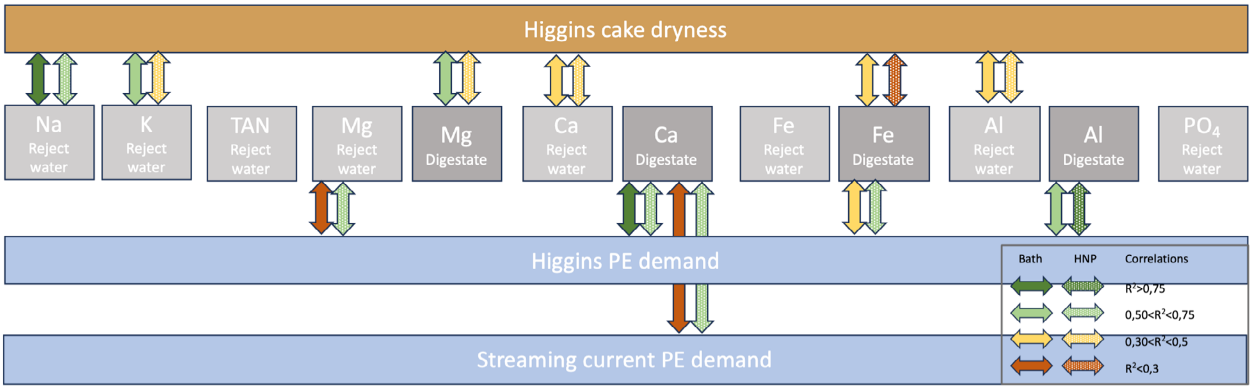
FIGURE 12 CORRELATIONS BETWEEN MONO AND DIVALENT RATIO IN THE REJECT WATER (INCLUDING AMMONIUM) AND THE PE CONSUMPTION IN THE HIGGINS DEWATERING TEST



1.1.1 CATIONS

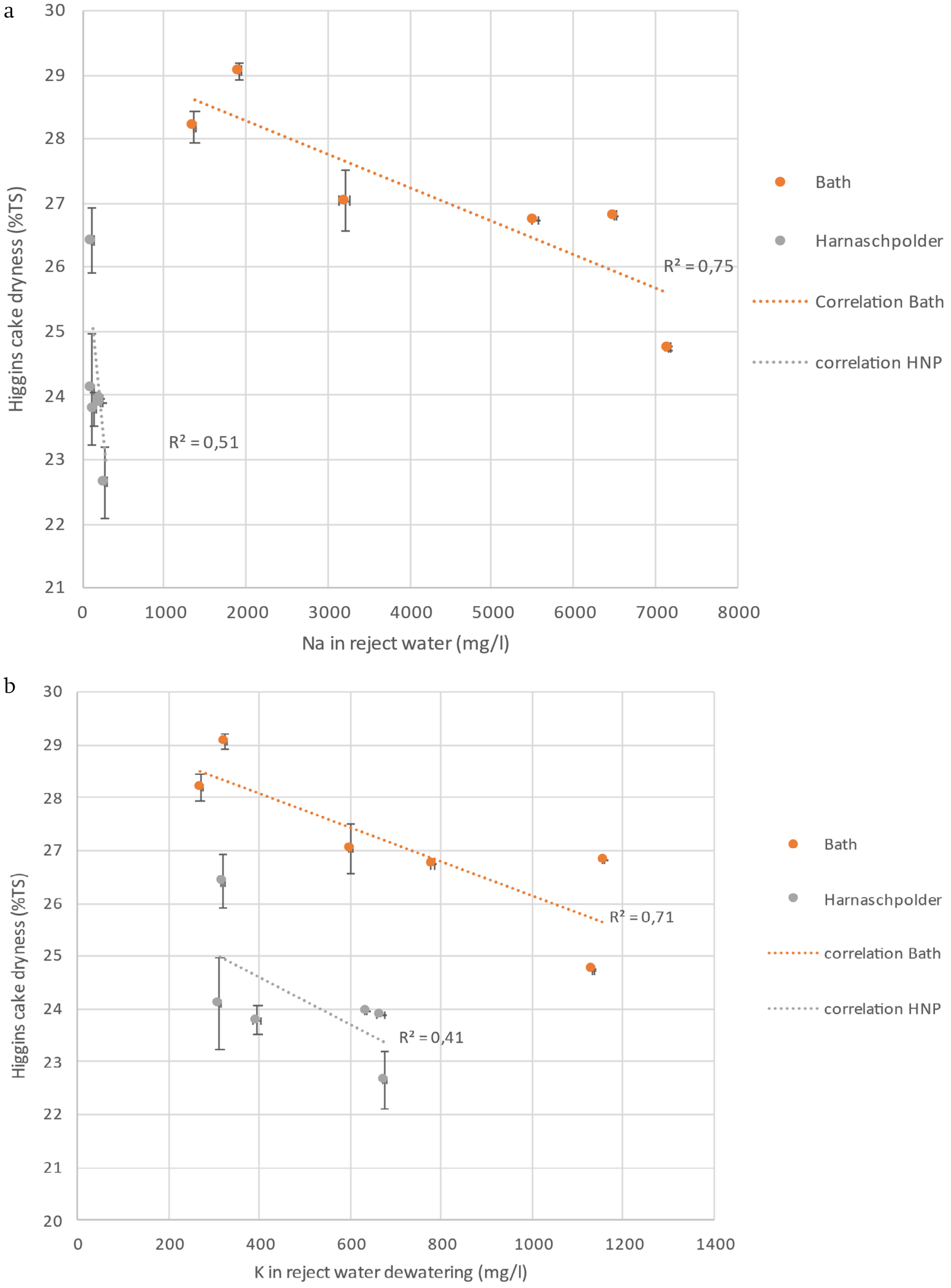
In addition to the monovalent-divalent cation ratio it is also interesting to evaluate if individual cations show strong correlations with sludge dewatering properties. Figure 13 shows the relevant correlations. In the previous paragraph we discussed that the M/D ratio (with and without ammonium) showed a correlation with the Higgins PE demand. Interestingly the individual cations that make up this M/D ratio do not show a strong correlation, suggesting that it is indeed the ratio that is important.

FIGURE 13 STRENGTH OF THE CORRELATIONS BETWEEN DIFFERENT IONS IN THE SLUDGE AND DEWATERING PROPERTIES (ONLY RELEVANT CORELATIONS INCLUDED TO SIMPLIFY THE PICTURE, CORRELATIONS NOT SHOWN HAD AN $R^2<0,3$)



Surprisingly relatively strong correlations were found for sodium and potassium concentrations with the cake dryness (Figure 14) with the strongest correlations found for the Bath sludge.

FIGURE 14A AND B CORRELATIONS OF NA AND K IN THE REJECT WATER WITH CAKE DRYNESS DETERMINED WITH THE HIGGINS TEST

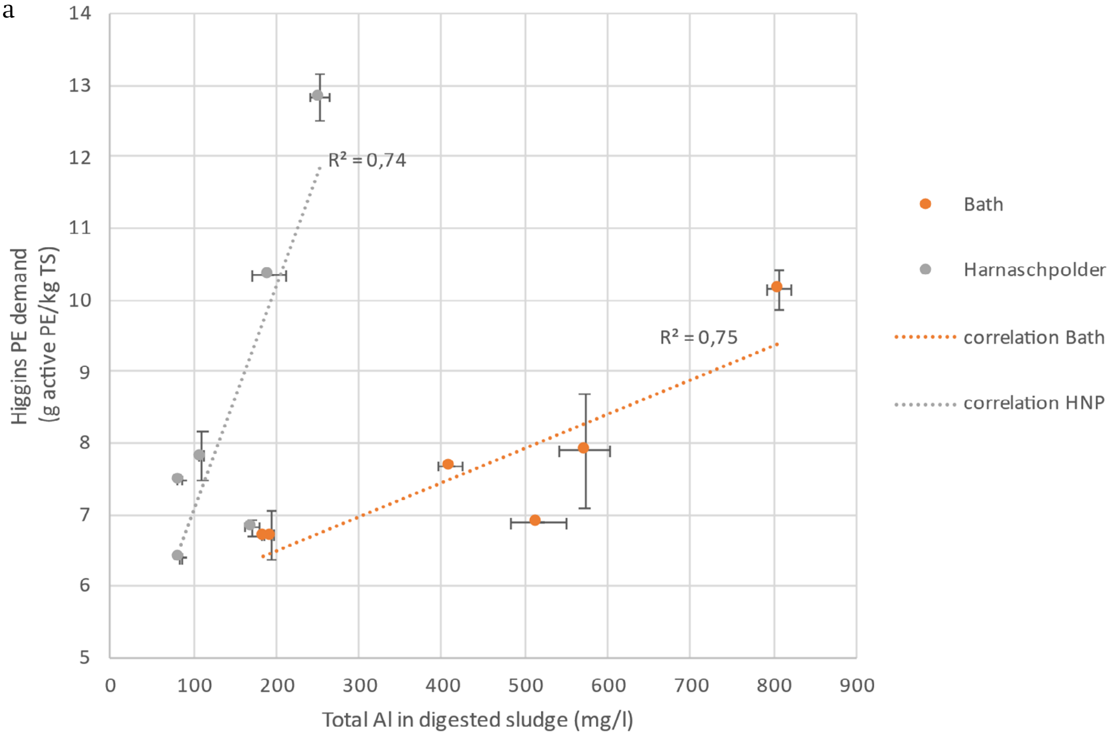


For the M/D ratio no relevant correlations were found for the total cation concentration in the sludge but interestingly the individual ions do show correlations with the Higgins PE demand. The strongest were for Ca and Al and to a lesser extent with Fe (Figure 15). The correlations were equally strong for both WWTP’s for Ca and Al. For Fe the correlation was strong for Harnaschpolder but less for Bath. Especially the relatively strong correlation for Al is surprising as literature did not before report a strong effect of the Al content in the sludge with the dewaterability.

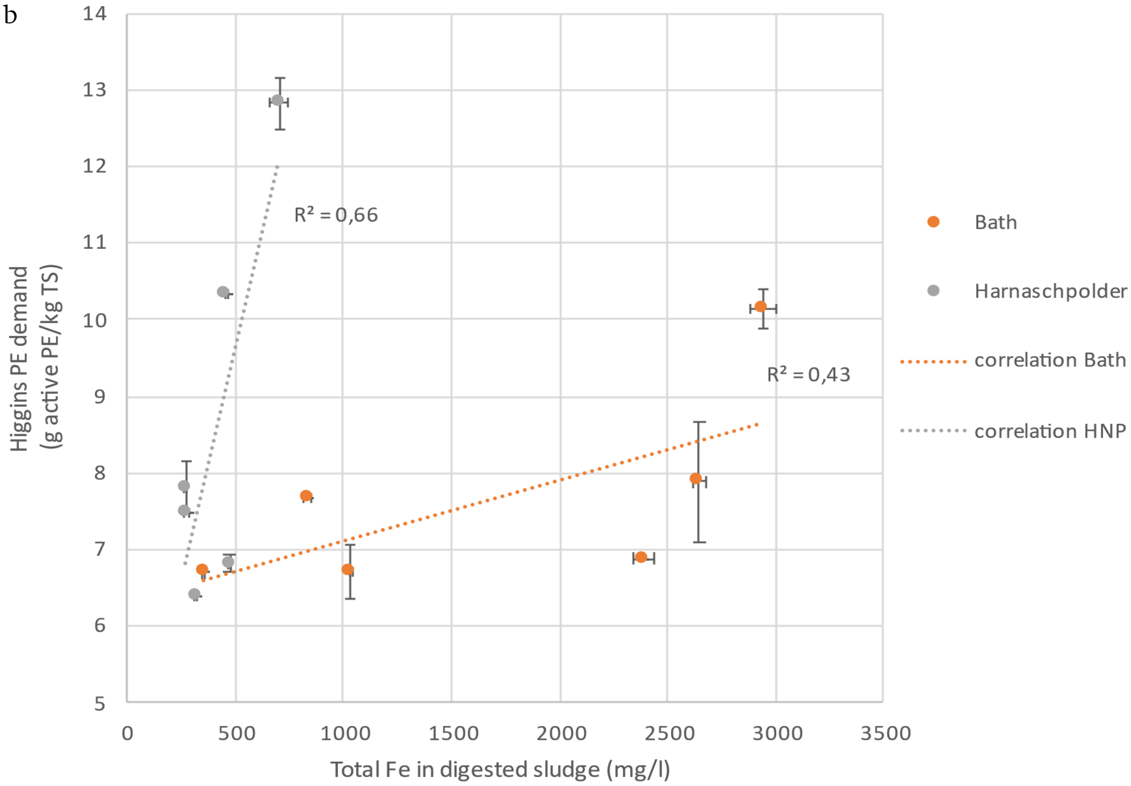
FIGURE 15 A, B AND C

CORRELATIONS FOR TOTAL AL, CA AND FE CONCENTRATIONS IN THE SLUDGE WITH PE CONSUMPTION DETERMINED WITH THE HIGGINS TEST

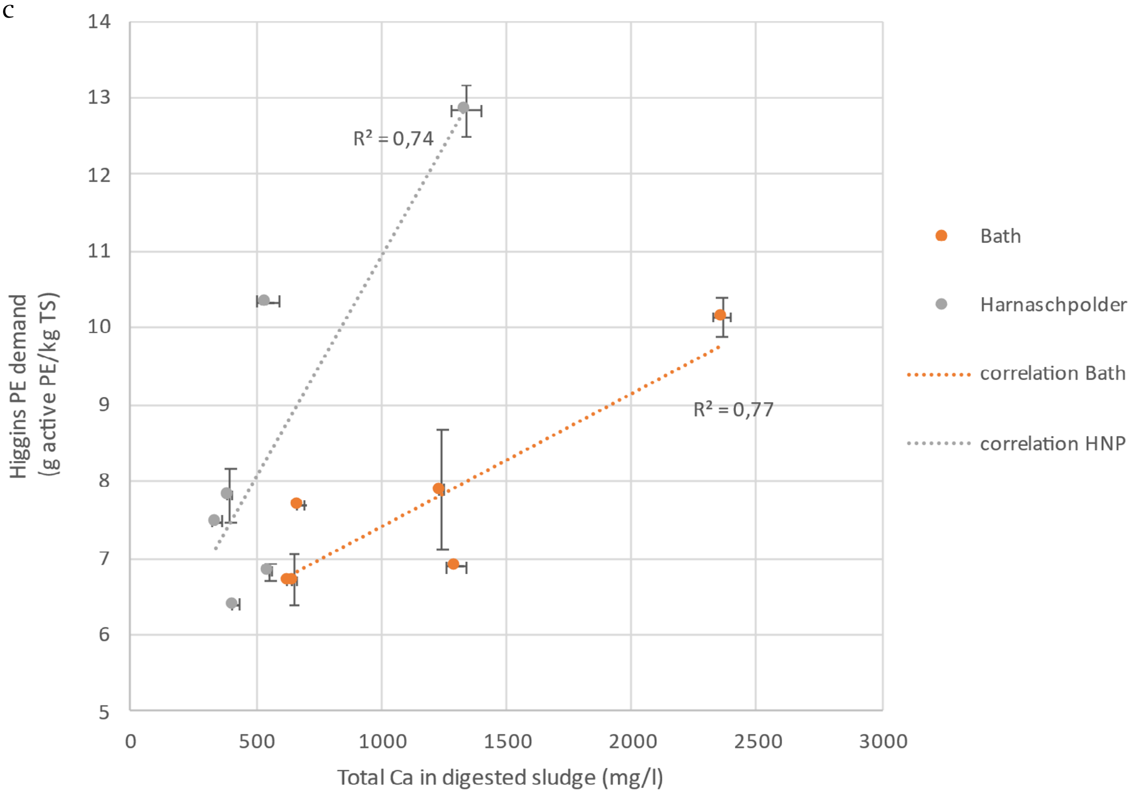
a



b



c



Some literature sources reported a correlation between the phosphate concentration and dewatering results, but for the two WWTP's in this study such a correlation was not observed (Shimp 2013). Often the studies reporting the correlation with phosphate are connected to struvite production via the addition of Mg. Possibly in these studies, it is not the removal of the phosphate that induces the better dewaterability but rather the addition of the Mg (Berkhof/STOWA 2016, Sobeck 2002).

Interestingly the correlations that were found for Higgins PE demand for the various cations were not present or weaker. Again, this shows that Higgins PE demand has better correlations with other sludge parameters than the streaming current PE demand and is therefore more useful than the streaming current PE demand.

2.3.2 ORGANICS

The organic composition of the sludge greatly determines the water binding capacity, and it is therefore interesting to understand if different measurements related to the organic composition correlate with sludge dewatering parameters. The extracellular polymeric substance (EPS) is considered to contribute the most to the water binding properties and in this study we used an extraction approach that distributes the EPS over the soluble or bulk fraction, a loosely bound and an tightly bound fraction. The soluble organic matter is often considered to be important for the PE use because the charged PE will bind easily with this fraction before it can create further binding with the bulk of the EPS. For this reason, also the soluble organic fraction was characterised. Firstly, the COD in the centrate after centrifugation was determined as well as the COD in the liquid phase after filtration over a fine filter of 0,45 um. The difference between the two was considered to be "colloidal" COD. Furthermore, the soluble fraction was also characterised for the three most important classes of organics: carbohydrates, proteins and humics.

FIGURE 16 STRENGTH OF THE CORRELATIONS BETWEEN DIFFERENT ORGANIC RELATED PARAMETERS FOR THE SLUDGE AND DEWATERING PROPERTIES (ONLY RELEVANT CORELATIONS INCLUDED TO SIMPLIFY THE PICTURE, CORRELATIONS NOT SHOWN HAD AN R2<0,3)

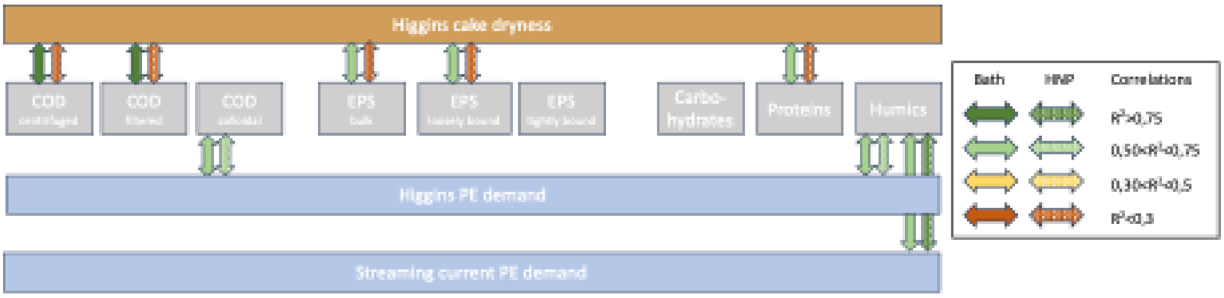
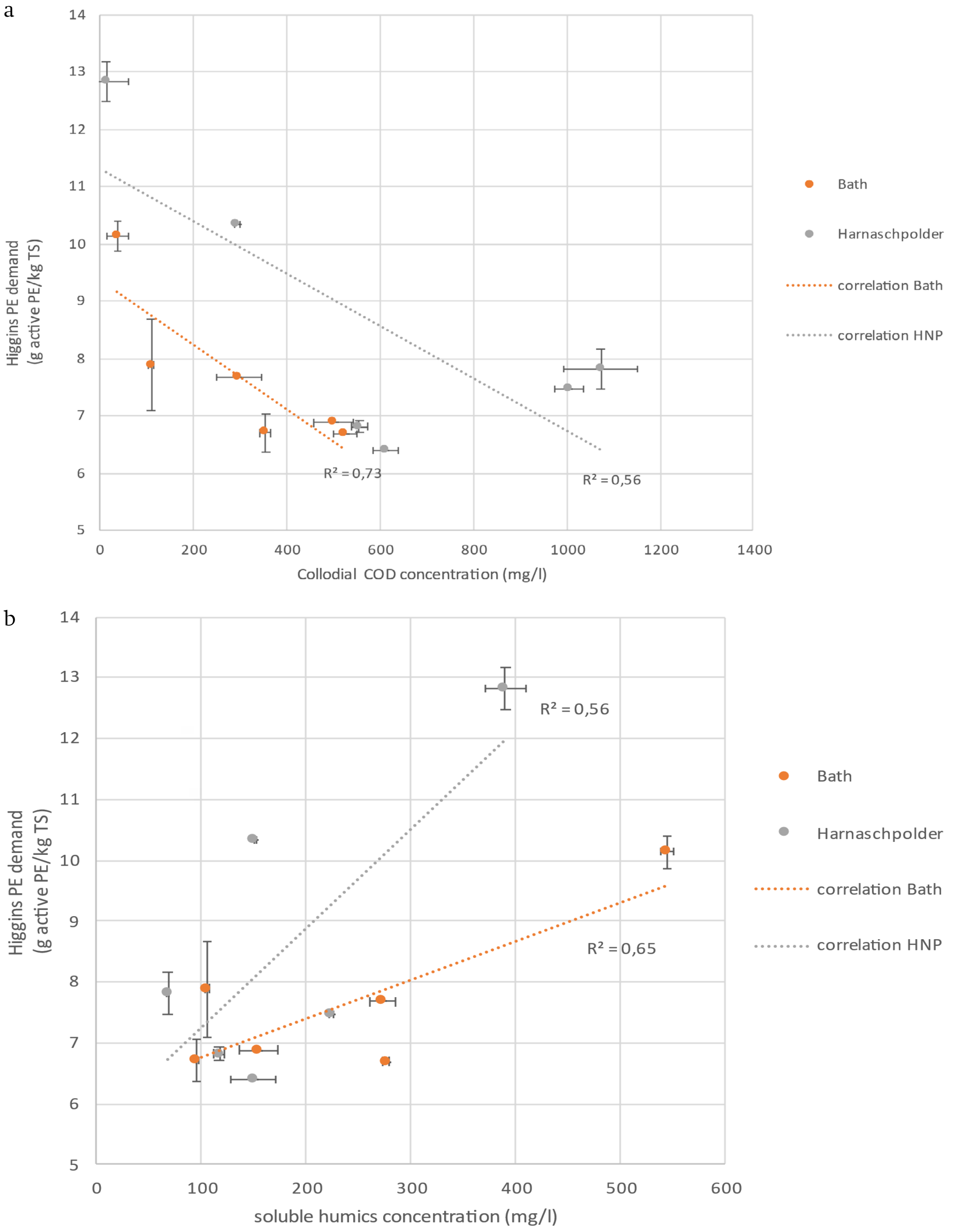


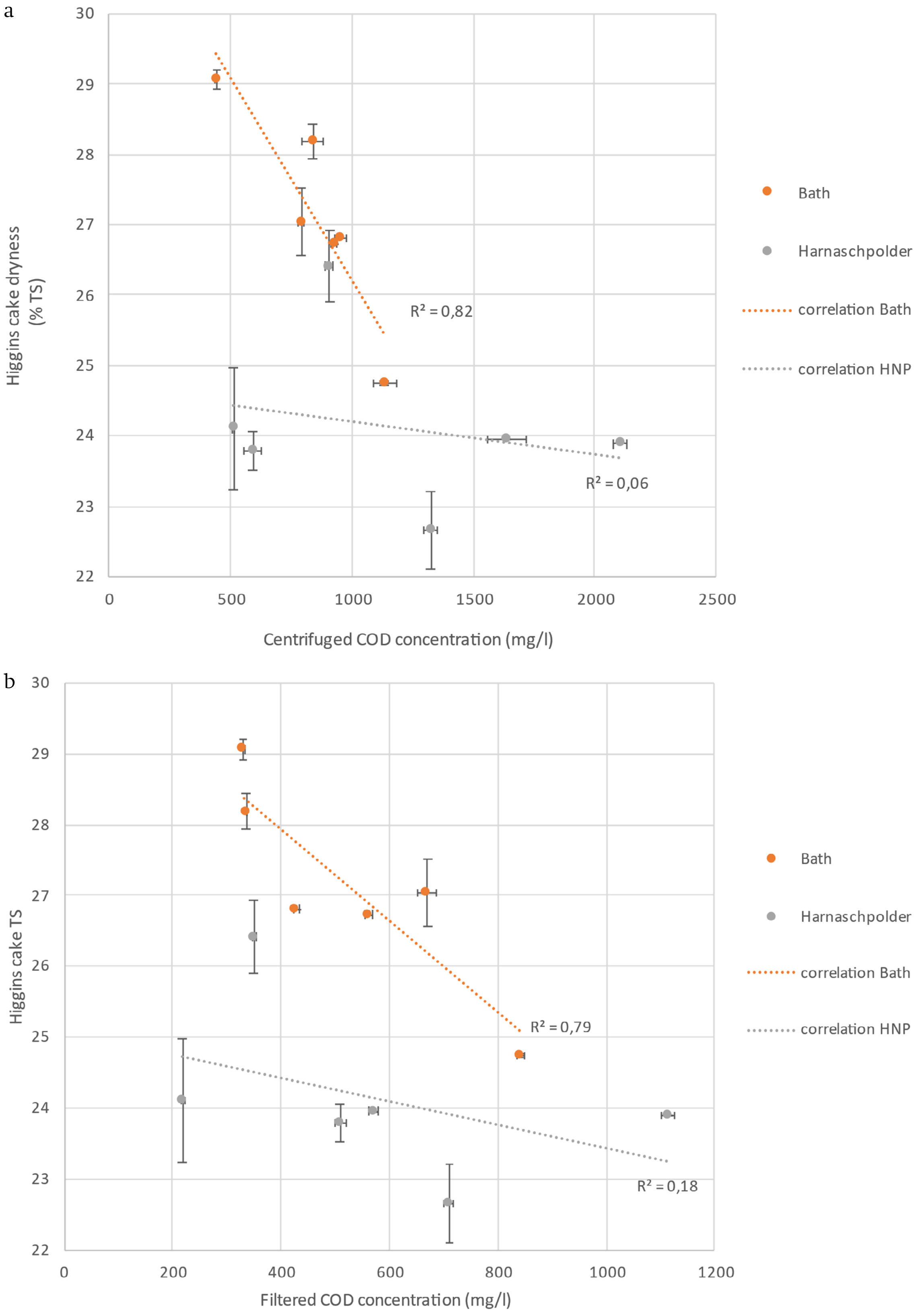
Figure 16 summarises relevant correlations that were found between these different organic parameters and sludge dewatering properties. There were only two parameters, colloidal COD and humics, that correlated with the PE demand determined via the Higgins test (Figure 17). That colloidal COD is a predictor for polymer use has been reported before in literature (Zhang et al 2019) but then always higher colloidal COD was connected to higher PE consumptions. In this study we find a surprising inverse correlation for both WWTP's. In contrast a higher soluble humics concentration correlated with higher PE demand. Humics have not often been mentioned in relation to the PE consumption in literature therefore this correlation is surprising. A higher soluble protein content has been associated in literature (Higgins, 2004b, Novak 2010) to higher polymer use because of their charged nature. Also, humics are charged molecules and may have a similar effect on the PE consumption. It should also be noted that higher concentrations of colloidal or undissolved matter can also be a result of a lower dewaterability because a lower dewaterability can influence the reject water composition.

FIGURE 17 A AND B CORRELATIONS FOR COLLOIDAL COD (A) AND SOLUBLE HUMICS (B) IN THE SLUDGE WITH PE CONSUMPTION DETERMINED WITH THE HIGGINS TEST



For cake dryness only correlations with organic parameters were found for the Bath sludge. For Harnaschpolder none of the parameters showed any correlation with the cake dryness. For Bath relatively strong correlations were found for centrifuged and filtered COD (figure). For Bath similar, but less strong correlations were found for bulk EPS (R^2=0,66), loosely bound EPS (R^2=0,67) and the soluble protein content (R^2=0,50). The way the bulk EPS concentration is measured has a lot of similarities to the way centrifuged COD is measured and therefore it is not surprising that these show similar correlations. The fact that loosely bound COD also shows a similar correlation suggests that this slightly bound fraction also contributes to the cake dryness. The correlation with the soluble protein content is lower and very dependent on one datapoint and is therefore much less certain.

FIGURE 18 A AND B CORRELATIONS FOR CENTRIFUGED COD (A) AND FILTERED COD (B) IN THE SLUDGE WITH CAKE DRYNESS DETERMINED WITH THE HIGGINS TEST



1.1.2 PARTICLE SIZE AND RHEOLOGY

This paragraph discusses possible correlations between the sludge particle size and sludge rheology data with the dewatering results. Rheology and particle size are not really connected to each other but are discussed together in this paragraph for practical reasons.

Particle size could especially have an influence on the PE consumption because a smaller particle could imply a large (charged) surface area of the sludge flocs and therefore a higher PE consumption. For this study the average particle size was used to check for correlations. Rheology data describe the viscosity of the sludge as a function of different shear conditions. Since shear force is applied to the sludge in the dewatering machine this data may have relations with the sludge dewatering properties. For this study the rheograms were fitted to the Hershel-Bulkley model which makes it possible to describe the rheogram with three parameters: the yield stress (in Pa), the flow consistency index K (in Pa.s^n) factor and the flow index n (dimensionless).

FIGURE 19 STRENGTH OF THE CORRELATIONS BETWEEN DIFFERENT PARTICLE SIZE AND RHEOLOGY PARAMETERS FOR THE SLUDGE AND DEWATERING PROPERTIES (ONLY RELEVANT CORELATIONS INCLUDED TO SIMPLIFY THE PICTURE, CORRELATIONS NOT SHOWN HAD AN $R^2<0,3$)

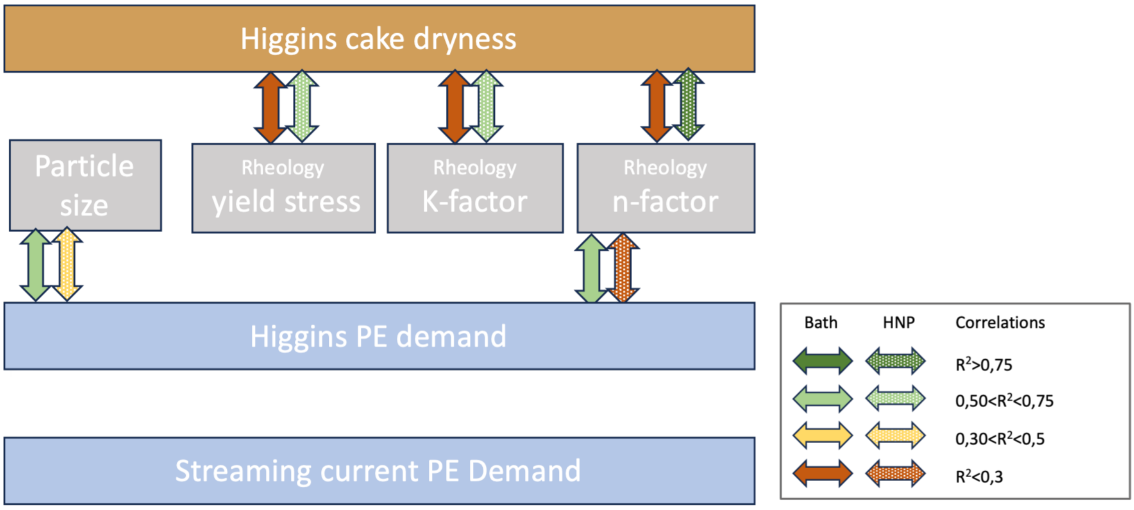
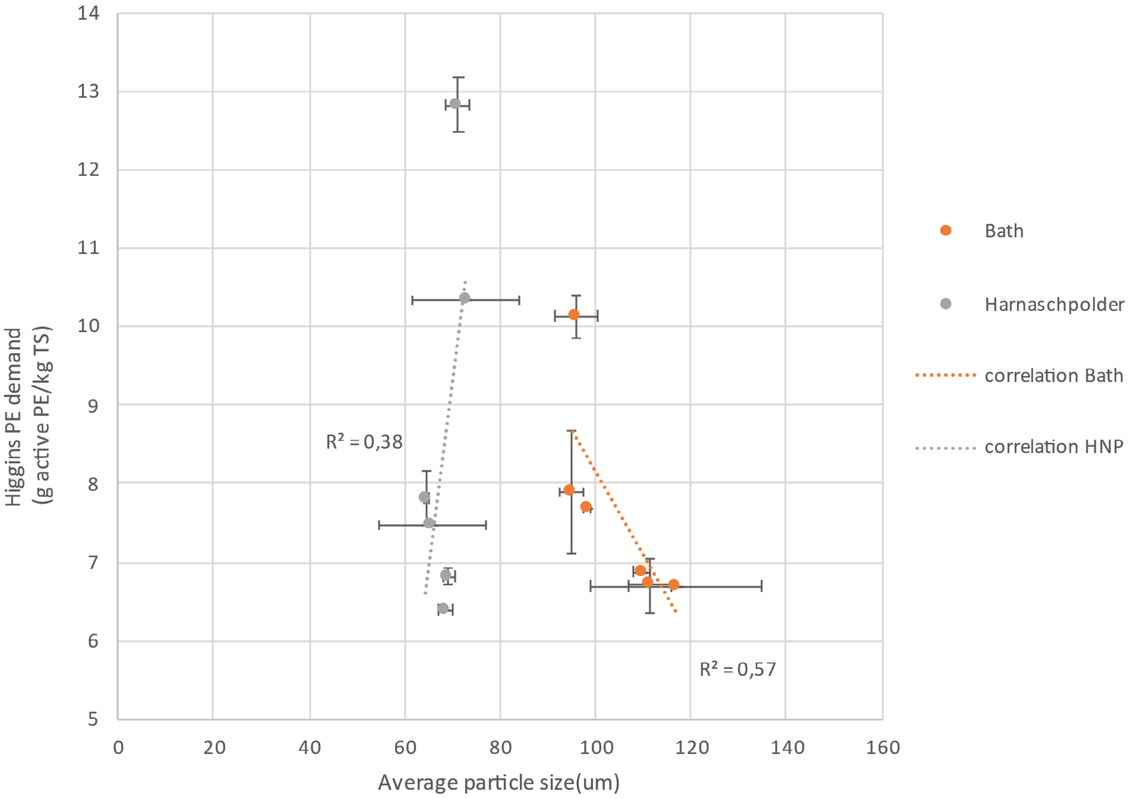


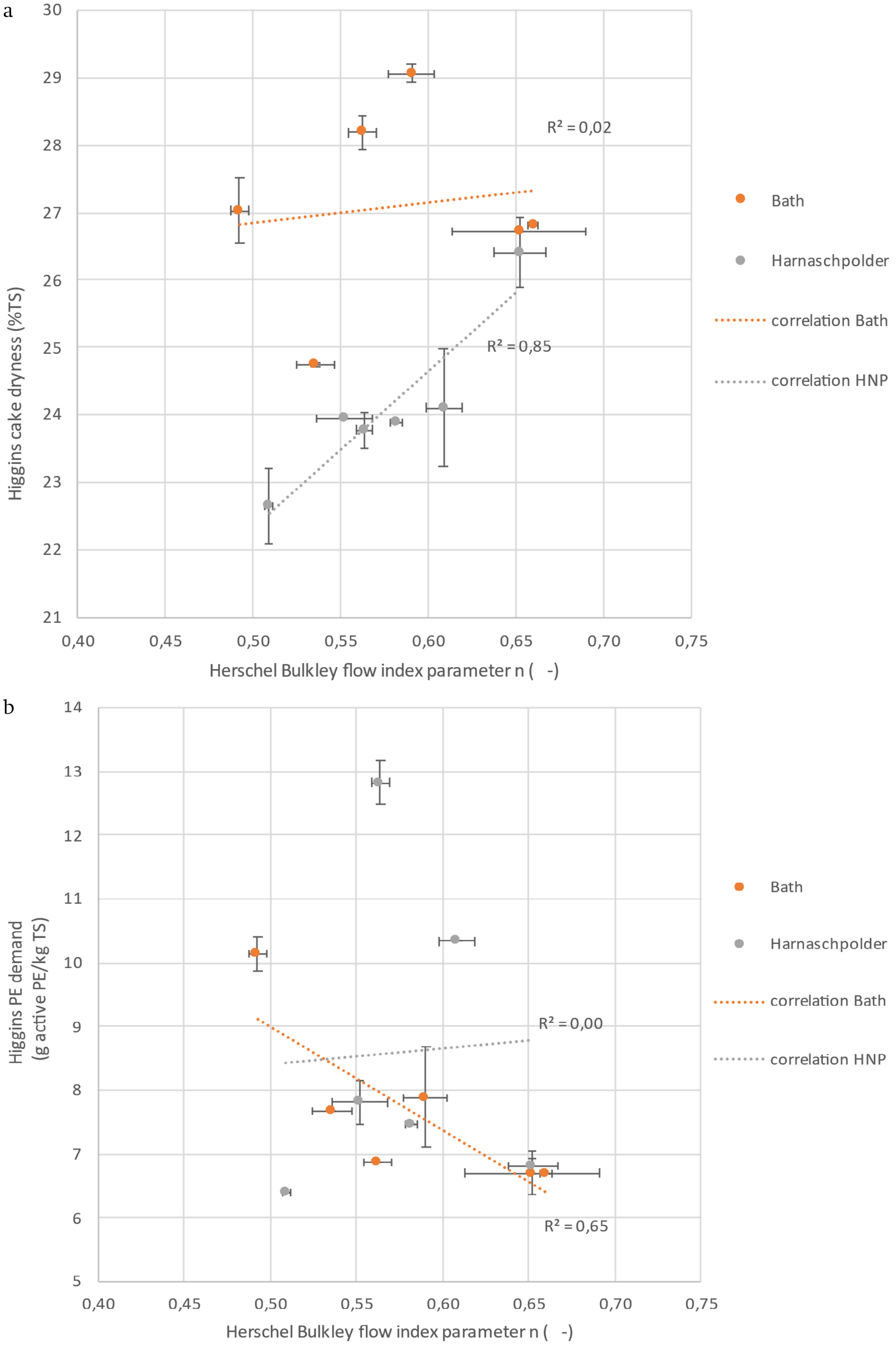
Figure 19 above shows that there were few strong correlations with the sludge dewatering parameters for the sludge particle size and the rheology parameters. For the particle size weak correlations were found for Bath and Harnaschpolder with the PE demand, but they are inverse for both WWTP's (Figure 20). For Bath sludge the PE demand decreases with increasing particle size as one would expect. In contrast, the PE consumption for Harnaschpolder increases but with a much less strong correlation compared to Bath. Based on this data it can be concluded that particle size is not a strong predictor for sludge dewatering parameters.

FIGURE 20 **CORRELATION FOR AVERAGE SLUDGE PARTICLE SIZE WITH PE DEMAND DETERMINED WITH THE HIGGINS TEST**



For the rheology data a strong correlation (R^2=0,84) was found between the flow index n and the cake dryness determined with the Higgins test for the Harnaschpolder sludge but not for the Bath sludge. However, for Bath there was a correlation with PE demand that was not there for Harnaschpolder. For cake dryness also correlations were found for the other two rheology parameters (K and T0) (Figure 21). These three parameters are strongly correlated with each other, but these results suggest that the flow index n is most important for dewatering performance. This flow index n describes the shear thinning behaviour of sludge as a function of increasing shear force. A classic Newtonian liquid has a value of 1 and the lower the value of n becomes the more a sludge shows shear thinning behaviour. The results in this study show that sludge with a low n value (so more deviation from a Newtonian liquid) gives a poorer dewatering performance. This is in line with expectations as a sludge with a lower n value would show a more “gelly” behaviour and can therefore maybe hold more water in the sludge.

FIGURE 21 A AND B CORRELATION FOR THE FLOW INDEX PARAMETER N (ACCORDING TO HERSCHEL BULKLEY MODE) AND CAKE DRYNESS (A) AND PE DEMAND (B) DETERMINED WITH THE HIGGINS TEST



2.3.3 SUMMARY: EXPLAINING VARIATIONS IN SLUDGE VARIABILITY

In order to be able to predict and understand variations in sludge dewaterability it is useful to understand which sludge characteristics have an important influence on the dewaterability. For this reason, inorganic ions, the organic composition and macroscopic parameters such as particle size and viscosity were measured and correlation checked between these parameters and sludge dewaterability. In this study we found the following relevant correlations (see also Figure 22).

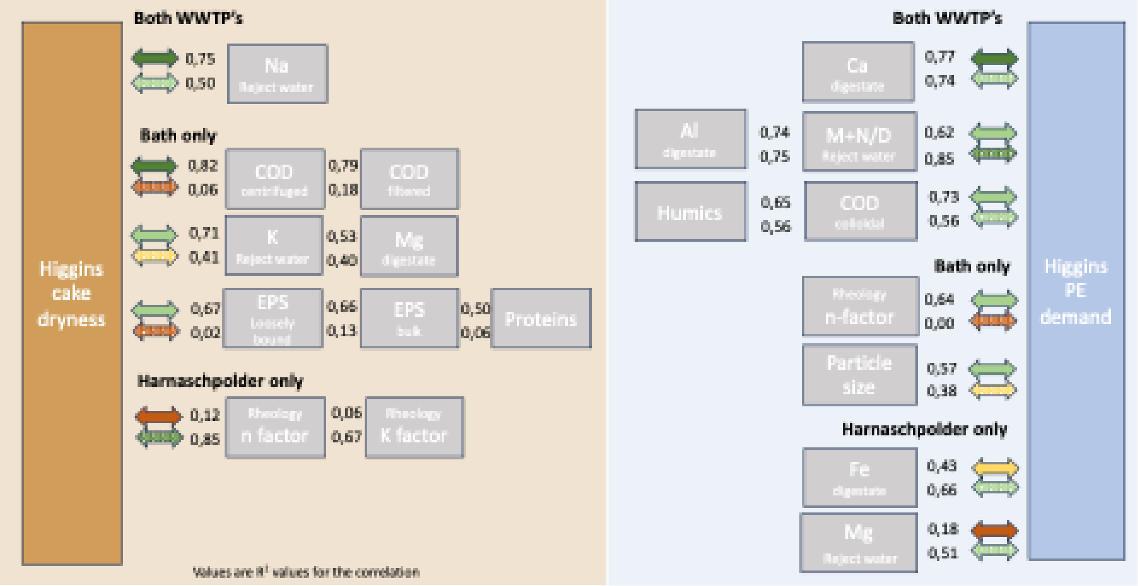
EXPLAINING CAKE DRYNESS

- Only the Na content of the reject water had a correlation with the cake dryness determined with the Higgins test. This correlation was strong (but different) for both WWTP's.
- For the Bath sludge the strongest correlation was found with the COD content of the liquid phase of the sludge. Also a correlation was found to bulk and loosely bound EPS but this was a significant weaker correlation. Also K in the liquid phase and Mg in the total sludge showed good correlations with the cake dryness of the sludge. In a way this is interesting because these parameters are linked to biological phosphate removal which is normally not applied in Bath.
- For Harnaschpolder there was no clear correlation between sludge composition and cake dryness. However, there was a clear correlation with sludge viscosity parameters.

EXPLAINING PE CONSUMPTION

- Surprisingly the Ca and Al of the total sludge showed a strong correlation with the PE consumption in the Higgins test for both WWTP's.
- A similar strong correlation was found for the ratio of mono-valent to divalent ions (if ammonium was included as a monovalent ion) in the liquid phase of the sludge. Interestingly the individual ions did not have a strong correlation, so it is the ratio that makes the correlation.
- Colloidal COD and the humic content also showed a strong correlation to the PE consumption. The strong correlation with the humic content is surprising because a strong effect of the humics content on the PE consumption was not reported before.
- For Harnaschpolder also the total Fe content of the sludge and the Mg concentration in the liquid phase showed correlation with the PE use. This is interesting because both Fe and Mg are actively dosed at this plant to control respectively H_2S emissions and dewaterability.
- For Bath also correlations were found with the particle size of the flocs and sludge viscosity.

FIGURE 22 SUMMARY OF THE PARAMETERS WITH THE STRONGEST CORRELATION TO THE SLUDGE DEWATERING PARAMETERS DETERMINED VIA THE HIGGINS DEWATERING TEST. VALUES IN THE FIGURES ARE THE R^2 VALUES FOR THE CORRELATION



2.4 POLYMER CHARACTERISTICS

2.4.1 PE DATA FOR WWTP BATH

Table 3 shows the results of the PE quality measurements for the PE used at Bath WWTP. During the two years of the project in total eleven PE samples were taken and analysed. The table shows the result of the six samples that were taken closest to the date on which the sludge samples were taken.

TABLE 3 RESULTS PE QUALITY MEASUREMENTS WWTP BATH FOR SAMPLES CLOSEST TO THE SLUDGE SAMPLING DAYS. ETA IS THE MAXIMUM VISCOSITY AT LOW SHEAR, K IS THE FLOW CONSISTENCY INDEX AND N IS THE FLOW BEHAVIOUR INDEX OF PE SOLUTIONS WITH A 0,1 AND 1 WT% SOLUTION. C, H, N GIVE THE ELEMENTAL COMPOSITION OF THE PE.

	TS (wt%)	Oil (wt%)	Eta0,1% (mPa.s)	Eta1% (mPa.s)	k0,1% (Pa.s)	k1% (Pa,s)	n0,1% (-)	n1% (-)	C (wt%)	H (wt%)	N (wt%)
5-apr-22	48	9	2500	60300	0,43	8,8	0,25	0,16	48,0	8,6	7,7
12-jul-22	50	23	2600	53600	0,46	7,8	0,25	0,15	48,0	10,0	8,1
6-sep-22	50	10	2400	54000	0,45	7,9	0,27	0,16	48,0	9,0	8,1
21-mrt-23	48	8	2700	55100	0,45	7,8	0,25	0,15	49,0	9,3	8,2
16-may-23	50	8	2400	59600	0,43	8,6	0,26	0,14	50,0	9,0	7,5
19-sep-23	48	4	2500	55300	0,45	8,3	0,27	0,18	48,0	8,6	7,9
Average 2022-2023 (n=11)	49	8	2400	56400	0,44	8,2	0,27	0,16	48,4	8,8	8,0
St dev 2022-2023	0,9	5	500	3300	0,04	0,4	0,04	0,01	0,7	0,6	0,3

The table makes a comparison to the average composition for all PE samples and parameters that have a value that is outside the range of two times the standard deviation are considered outliers. These outlying samples could indicate that the PE quality was different compared to the other samples. For the interpretation of the results, it is important that all individual parameters have measurement uncertainties that should be considered (see also 1.3.3).

The table shows that the PE content (dm%) of the different samples can deviate at 5% from one sample to the other sample and such deviations would not have had a huge impact on the interpretation of the PE consumptions determined at full scale or via the lab scale tests.

The table shows that the sample of July 12 in 2022 shows a relatively high oil content and a relatively high hydrogen content. From the experience gained with the method so far it is known that the oil measurement sometimes has issues and the measurement itself also has a relatively high measurement error. The hydrogen content is higher than for the other samples which could indicate a slightly different structure although the carbon and nitrogen content show normal values. Normally the nitrogen content is considered the most relevant element because it is linked to the charge of the PE which is an important property of the PE. The sample of May 16 also shows a slightly higher carbon content which could also indicate structural differences. However, for both samples the viscosity parameters do not show deviations from the other samples and in the end these parameters are most closely linked to the properties of the PE (chain length, structure, charge) and therefore the performance of the PE.

Therefore, these results give no strong indication that the PE quality was different for the six dates when the sludge was sampled.

2.4.2 PE DATA FOR WWTP HARNASCHPOLDER

Table 4 shows the results of the PE quality measurements for the powder PE used at Harnaschpolder WWTP. During the two years of the project in total thirteen PE samples were taken and analysed. The table shows the result of the six samples that were taken closest to the date on which the sludge samples were taken.

TABLE 4 **RESULTS PE QUALITY MEASUREMENTS WWTP HARNASCHPOLDER FOR SAMPLES CLOSEST TO THE SLUDGE SAMPLING DAYS. ETA IS THE MAXIMUM VISCOSITY AT LOW SHEAR, K IS THE FLOW CONSISTENCY INDEX AND N IS THE FLOW BEHAVIOUR INDEX OF PE SOLUTIONS WITH A 0,1 AND 1 WT% SOLUTION. C, H, N GIVE THE ELEMENTAL COMPOSITION OF THE PE**

	Eta0,1% (mPa.s)	Eta1% (mPa.s)	k0,1% (Pa.s)	k1% (Pa,s)	n0,1% (-)	n1% (-)	C (wt%)	H (wt%)	N (wt%)
1-apr-22	3000	64800	0,50	9,2	0,24	0,17	47,0	9,3	9,3
30-jun-22	2300	66800	0,48	9,5	0,28	0,17	45,0	8,7	9,0
2-aug-22	2900	67600	0,50	9,5	0,24	0,15	46,0	8,1	9,1
8-mrt-23	3600	76000	0,55	10,4	0,22	0,14	48,0	7,4	9,2
17-jul-23	**300**	**22500**	**0,18**	**3,8**	**0,41**	**0,26**	**42,0**	**9,0**	**11,0**
9-oct-23	2400	45500	0,42	6,8	0,27	0,20	45,0	7,8	9,8
Average 2022-2023 (n=13)	2800	64300	0,47	9,1	0,27	0,16	46,5	8,3	9,4
St dev 2022-2023	700	13700	0,08	1,7	0,04	0,03	1,6	0,5	0,5

The PE used at Harnaschpolder is a powder PE and therefore active content and oil content were not regularly measured for these samples. Some active content measurements were done showing that the content was >98,5 wt% and the oil content was in the range of 0-0,3 wt%.

Again, a comparison is made to the average composition for all PE samples and parameters that have a value that is outside the range of two times the standard deviation are considered outliers. In this case the composition of the PE sample taken on May 16, 2023 is clearly an outlier and has a very different composition compared to the other samples. In 2022 and 2023 WWTP Harnaschpolder also submitted samples for other two other PE types used for thickening or as an alternative to the regular PE. The sample of May 16 shows more similarities with one these PE types and probably this sample was booked under a wrong PE type.

All other PE samples shows similar results for the composition, albeit that some results for the sample of 9 October are a bit on the low side. Nevertheless, they still are within a two times standard deviation bandwidth. So overall there are no significant indications that the PE quality changed in such a way that it may have influenced the dewatering results.

3

DISCUSSION

3.1 NO CORRELATION BETWEEN FULL SCALE PE CONSUMPTION AND DEWATERING PARAMETERS

In the correlation matrix presented in Appendix 2, it was observed that the consumption of polymeric flocculant (PE) did not exhibit correlations with dewatering parameters such as the Higgins centrifugation test, Specific Resistance to Filtration (SRF), or Capillary Suction Time (CST). In an optimized system, PE consumption is anticipated to reflect changes in sludge composition, aiming to achieve a targeted solids concentration. Consequently, alterations in sludge composition should influence both the dewaterability indices and PE consumption.

Several factors may account for the absence of correlation, including:

- Non-optimized PE dose calculation: Examination of the dataset reveals minimal variation in PE consumption throughout the experiment. Consequently, changes in sludge composition may not have been accurately captured in the PE calculation.
- Non-optimized settings of the dewatering equipment. A positive or negative effect of a change in sludge quality can be nullified by not optimal settings of the dewatering equipment.
- Limitations of lab-scale tests: Tests such as CST and SRF, while assessing dewaterability, may not fully represent the behaviour of full-scale equipment. This lack of reproducibility can be attributed to the failure of CST and SRF to consider sludge compression in full-scale equipment. Moreover, adjustments to the Higgins centrifugation test are necessary to align with specific conditions of the full-scale equipment, involving modification of centrifuge RPM, filtration porous medium, and centrifugation time.
- Unmeasured underlying parameters: Certain parameters influencing the dewatering process, not addressed in this report, may exhibit correlations with lab-scale dewaterability indicators and PE consumption. However, since these parameters were not measured, the variables examined do not exhibit an evident correlation.

3.2 SIGNIFICANCE OF THE CORRELATIONS

In this study we were only able to obtain 6 datapoints per WWTP and the correlations that were found could have been stronger. But on the other hand, if more than one parameter influence for instance cake dryness one cannot expect a strong correlation with each parameter individually because the data does not represent a controlled experiment. So even weak correlations could indicate there is an influence because maybe this influence is overshadowed by other influences etc.

The correlation matrix revealed a monotonic relationship among the parameters. However, it is important to highlight that correlation does not necessarily imply causation, particularly considering the limited measurement of only 6 data points per wastewater treatment plant (WWTP). The low number of data points introduces random fluctuations that may erroneously appear as correlated.

Given this constraint and recognizing that various parameters collectively may impact factors such as cake dryness, anticipating a robust correlation with each parameter individually is impractical in the absence of controlled experiments. To comprehensively understand the influence of unmeasured underlying parameters or their combinations, mechanistic models elucidating the dewaterability process are imperative in the dewatering field. This study underscores the need for such models. Considering the data collected, weak correlations may still indicate influence, suggesting the possible interactions of other (not measured) factors with the observed relationships.

3.3 DIFFERENT CORRELATIONS FOR DIFFERENT SLUDGES

In this study no universal correlation were found for different sludges because the correlations are different for each sludge. Depending operating conditions one parameter of the dewatering process may have more influence than the rest on the sludge final solids content or may affect differently the process. For instance, the PE dose in g of active polymer per grams of sludge TS may work differently if the charge density in the polymers is different, which would not be reflected if only the variable "PE dose' is measured and introduces in the correlation matrix. Also, non-foreseen fluctuations in the operational parameters, sludge and polymer characteristics are not considered in these studies, which may make it difficult to compare different WWTPs. The best way to approach the problem in the future would be monitoring one plant, trying to measure as much parameters as possible to make strong correlations.

3.4 HIGGINS PE-DEMAND VERSUS STREAMING CURRENT PE-DEMAND

Although both Higgins PE demand and streaming current PE demand quantify PE consumption, they do so in distinct ways. Higgins PE demand measures the actual dewaterability of sludge, encompassing its compression, whereas streaming current PE demand solely measures the quantity of polymer required for charge neutralisation. Charge neutralisation, while an important phenomenon during sludge dewatering, is not the sole enough to characterise the whole process. Therefore, the analysis should also encompass compression and expression phenomena. This can be achieved, for instance, through the application of the Higgins test or a filter press.

3.5 BATH: CORRELATION WITH MG AND K

Higgins cake dryness (TS) showed a positive correlation with Mg content in the sludge (solid phase). It's important to stress that Mg in the sludge may not necessarily form part of microbial cells; instead, it can be present in the form of precipitates like struvite, which contributed to the TS. According to Bergmans et al. (2014), struvite precipitation in the sludge can enhance dewaterability.

An elevated concentration of K in reject water may be detrimental to dewaterability, similar to other monovalent cations. Increased concentrations of monovalent cations lead to ion exchange with the divalent cations in the extracellular polymeric substance (EPS) matrix of the sludge, consequently reducing dewaterability. The ratio of monovalent to divalent cations has been previously investigated by Higgins and Novak (1997).

3.6 HARNASCHPOLDER: CORRELATION WITH FE AND MG

In the Harnaschpolder plant, Fe and Mg are introduced for the precipitation of S^{2-} and PO_4^{3-}, respectively. The presence of S^{2-} comes from the reduction of SO_4^{2-} and necessitates removal to prevent corrosion in the biogas upgrading unit and the combined heat and power (CHP) unit. Mg is dosed to facilitate the precipitation of struvite and prevent uncontrolled precipitation. Under anaerobic conditions, both Fe and Mg exhibit an oxidation state of +2, a characteristic noted by Higgins and Novak (1997) to be conducive to improved dewaterability.

3.7 POSSIBLE IMPORTANCE OF HUMICS

Humic substances comprise a diverse set of compounds generated through microbial decomposition. Within AD, these substances are form as a by-products of organic material breakdown. Broadly, humic substances exhibit highly polymerized, aromatic structures with variable composition. A distinguishing feature of humic substances is the presence of carboxyl and hydroxyl groups, imparting a negative charge to their structure.

The negatively charged nature of humic substances may pose challenges in the application of cationic polymeric flocculants, potentially increasing PE dosage. Typically, the soluble concentration of humic substances is not a significant concern during conventional anaerobic digestion, as it does not reach concentrations comparable to solids concentration. However, in scenarios involving pre-treatments like thermal hydrolysis processes, the concentration of humic substances can increase to levels where it may impact the efficacy of PEs or need alternative PEs. In full-scale installations employing thermal hydrolysis processes, adjustments to PE dosages and type are imperative to accommodate the altered substrate conditions.

4

CONCLUSIONS AND RECOMMENDATIONS

4.1 CONCLUSIONS

It is of interest for a water authority to always operate a sewage sludge dewatering installation at the optimal settings. In practice operators of sewage sludge dewatering installations experience significant differences in dewaterability and it is not always clear if these differences are the result of suboptimal dewatering parameters, changes in PE quality or changes in sludge composition.

For the operator of a dewatering installation, it would be of interest to understand if changes in sludge dewaterability can be explained by changes in sludge composition because this could help to know if there is potential for further optimisation of the dewatering results either via other settings of the dewatering installation or by learning more on how changes in the way the water line is operated or chemicals are dosed can influence dewaterability.

In literature a lot of different sludge parameters have been reported to be a predictor for variations in sludge dewaterability. Also, various dewatering tests are used to simulate the dewatering at full scale. The aim of this study was split into four questions.

1. Do the data from lab-scale tests found in literature correlate with the full-scale sludge dewaterability data?
2. Which sludge characteristics can be linked to changes in full-scale dewaterability data?
3. Which parameters can be used to predict dewaterability? Can this parameter be easily measured at laboratory scale?
4. Is there a change in PE quality in full-scale throughout the year(s) and if yes, can it be linked to changes in full-scale dewaterability data?

To see firstly which of these proposed parameters have a good correlation with full scale results and secondly propose a practical mix of parameters that water authorities could measure to best understand and analyse changes in sludge dewaterability.

To achieve this the sludge composition and dewaterability was measured over a period of 2 years, taking in total 6 sludge samples for each WWTP.

COMPARISON WITH FULL SCALE DEWATERING RESULTS

First of all, a comparison was made between full scale dewatering results and lab scale dewaterability tests. This comparison showed that the lab scale tests showed reasonable correlation with the cake dryness obtained at full scale. Of the different tests, the Higgins centrifuge test showed the best and consistent correlation with full scale cake dryness results.

Disappointingly, there was no clear correlation between the PE consumption at full scale and the lab tests that intend to predict PE consumption for a certain sludge. This study cannot explain this discrepancy. One reason might be that the PE consumption at full scale was not always optimal because the consumption data showed much less variation in PE consumption compared to the lab scale tests. In the design of the study an effort was made to protocolise the full-scale dewatering settings to avoid suboptimal full scale dewatering settings, but this protocol may not have been followed always in practice leading to possibly suboptimal PE dosing settings at full scale. In any follow-up research, it is therefore advisable to carry out tests with the full-scale dewatering equipment after optimizing the dewatering and then to take samples for lab tests of the optimally functioning equipment.

The full-scale PE consumption data also showed little to no correlation with other lab scale parameters that are suggested by literature to influence the PE consumption. In contrast, the lab scale Higgins PE consumption test did show important better correlations with other sludge parameters. The Higgins test also showed more and better correlations than other tests that could be indicators for PE demand (such as streaming current PE demand, CST and zeta potential). For this reason, this study concludes that the Higgins PE demand is the best indicator for the PE demand of a certain sludge.

Based on the results of this study it is concluded that the Higgins test is the best lab scale test to evaluate sludge dewaterability (cake dryness and PE consumption). Compared to other lab scale tests it also has the advantage that it gives both an estimate of the cake dryness as well as the PE consumption. These estimates may not be the try cake dryness and PE consumption in practice but can serve as reference values to which practical results can be compared. To execute a Higgins test a large laboratory centrifuge, some special centrifuge tubes and a skilled employee are necessary. Question 1 and 3 of the aim of this study have been answered with this.

SECOND QUESTION: RELATIONS BETWEEN SLUDGE DEWATERABILITY AND SLUDGE CHARACTERISTICS

Based on the previous conclusion the Higgins test was used to find other sludge parameters that had significant correlations with the dewaterability as determined via the Higgins test. The advantage of using the Higgins test compared to full scale data is that the Higgins test is always performed under controlled conditions and also makes it possible to compare the dewaterability of Bath and Harnaschpolder. Since both WWTP's use different dewatering machines a comparison of full scale dewaterability data between the two sites is less useful.

The results show that the cation composition is an important parameter that correlates to the sludge dewatering parameters in different ways. Correlations with the PE consumption determined with the Higgins tests were found for both sludges. The most relevant correlations were with the monovalent/divalent ratio in the liquid phase and total Ca and Al in the sludge. For the cake dryness only the soluble sodium content correlated for both sludges. For Bath also potassium and magnesium showed correlations with cake dryness whereas Harnaschpolder sludge showed a strong correlation with total iron and soluble Mg concentrations and the PE consumption.

Soluble and colloidal COD of the sludge also correlated importantly with sludge dewatering parameters. The colloidal COD content correlated strongly with the PE consumption for both sludges. For the cake dryness the soluble COD content (centrifuged or filtered) only correlated strongly with cake dryness for Bath but not for Harnaschpolder. The colloidal COD content correlated in its turn with the soluble humics concentration suggesting that it is mainly colloidal humic matter that is responsible for the PE consumption. In soluble COD content (centrifuges or filtered) correlated in its turn with the bulk and loosely bound EPS (for Bath only).

DIFFERENT CORRELATIONS FOR BATH AND HARNASCHPOLDER

As discussed above some parameters show similar correlations for both WWTP's. However, these correlations were never universal in the way that the correlation was similar for both WWTP's: the slope and intercept of the correlation was always significantly different. Also, some correlations were found for one WWTP but not for the other. This shows that it might be necessary to develop such correlations for each sludge separately.

One of the reasons that the correlations were not similar is that dosing regimes for cations like iron and magnesium were different for both sludges and also different during the two years for one sludge. The results show that the cation concentrations influence sludge dewaterability and differences in dosing might have hidden underlying correlations.
As an answer on question 2 of the aim of the study there are some correlations found between sludge dewaterability and sludge characteristics, but there is no relation yet that can predict the needed amount of polymer and the dewaterability of the sludge to be achieved. With a lot of measuring results under optimized conditions and advanced programs it is maybe possible to find a relation between multiple characteristics and the dewaterability.

POLYMER QUALITY

Last question of the aim of the study, does the quality of polymer change over the year and does this influence the dewaterability. This is a short answer. Within the uncertainty of the measuring method there are no large variations in polymer quality. Because of this, it could not be determined whether the quality of PE influenced the results.

RELEVANCE FOR UNDERSTANDING SLUDGE DEWATERABILITY

One of the challenges in understanding sludge dewaterability is that there are many parameters that may have an influence. Therefore, this requires a multivariate data analysis or machine learning approaches to be able to come to good predictions of changes in sludge dewaterability. Such a multivariate data analysis requires a bigger data set then was provided by this study. This study has shown that both the cation composition and the soluble and colloidal organics have significant correlations with sludge dewaterability. These parameters can be collected relatively easy via the regular sampling and analysis campaigns that water authorities already perform. Regular measurement of these parameters could provide a data set that can provide a solid training set for machine learning approaches. However, to be able to do that the dataset should also contain good reference data of the theoretical dewaterability. This study shows that the Higgins centrifuge test could be such a reference. Alternatively, a water authority could also regularly perform a protocolised optimisation of the dewatering machine to get this reference.

In summary, it means that in practice more data must be collected from optimized equipment. This can be done by collecting all data surrounding sludge dewatering in accordance with the method mentioned in Appendix A1.1. If the dewatering equipment is functioning optimally, sufficient samples must be taken to be able to carry out analyses, like cation composition and COD fractions. To check whether the dewatering equipment performs optimally, the maximum dewaterability can be tested with the modified Higgins test.

4.2 RECOMMENDATIONS

Based on the outcome of this study we recommend water authorities that want to invest in a better understanding and prediction of their sludge dewaterability to take the following steps:

1. Build up a historical data set that records the following data on sludge dewaterability:
   a. sludge dewaterability (via the Higgins test or a protocolised optimisation of the full-scale installation)
   b. cation measurements for the liquid phase of the sludge for Ca, Mg, Na, K and TAN and the total sludge for Ca, Mg, Fe and Al.
   c. determination of the COD in the liquid phase of the sludge via both centrifugation and filtration to determine the COD after centrifugation and the colloidal COD.

2. Invest in capacity to analyse this data and ensure that a learning and optimisation process is intitiated using the above data set. This should be a combination of the following:
   a. Ensure there is process engineering capacity to analyse the data
   b. Invest in machine learning tools in order to be able to make multi-variate learning from the data possible.

5

GLOSSARY OF TERMS AND ABBREVIATIONS

5.1 TERMS

Cake dryness	The dryness of the sludge cake that is obtained after mechanical dewatering of the sludge. The dryness is measured by thermal drying the sludge and measuring how much weight is lost by evaporation of the remaining water in the cake (TS content).
Dewatering	A process to remove the maximum amount of water from a sludge by mechanical means. Often centrifugal or pressing forces are used to achieve this. Coagulants and flocculants can be added to assist the dewatering. As opposed to thickening the term dewatering is used to obtain an as dry as possible solid residue.
Dewaterability	A way to describe how easy a sludge is to dewater. A good dewaterability is characterised by an as dry as possible solid residue, a water phase with as little solids as possible (high separation efficiency) and little use of poly-electrolyte or other chemicals.
Digestate	This term is used in this study to describe the total sludge sample before dewatering.
EPS or extracellular polymeric substance	This is the polymeric substance that is excreted by bacteria. This material is important in the building of flocs that make up the organic matter of sewage sludge. It has a high water binding capacity.
Flow consistency index (k)	A term from rheology to characterise polymeric substances that show a shear thinning behaviour (viscosity reduces with increasing shear). This is the constant k that is used in the power law fit of the viscosity curve. See paragraph 1.3.3 for a detailed explanation
Flow behavior index (n)	A term from rheology to characterise polymeric substances that show a shear thinning behaviour (viscosity reduces with increasing shear). This is the constant n that is used in the power law fit of the viscosity curve. The constant is an indication how different the viscosity behaviour deviates from normal Newtonian behaviour (n=1). See paragraph 1.3.3 for a detailed explanation
Flocculant	This is a substance that is added to a water stream to ensure that sludge particles agglomerate so that they are easier to dewater. A flocculant is often an poly-electrolyte (PE).
Higgins cake dryness	The dryness of the sludge cake (TS) obtained by applying the protocol of the Modified Higgins centrifuge test.
Higgins PE demand	The demand of PE (poly-electrolyte) that is needed to get an optimal dewatering result when applying the protocol of the Modified Higgins centrifuge test.

K-factor	See flow consistency index
Maximum viscosity at low shear	A term from rheology to characterise polymeric substances that show a shear thinning behaviour (viscosity reduces with increasing shear). This is the highest viscosity measured at the lowest possible shear rate. See paragraph 1.3.3 for a detailed explanation.
Modified Higgins centrifuge test	A structured approach to use a lab scale filtering centrifuge to simulate and predict full scale dewatering in a decanter centrifuge. See paragraph 1.3.1 for a detailed explanation
Mono to divalent cation-ratio (M/D ratio)	The ratio of the sum of monovalent cations (Na, K, optionally ammonium) to the sum of divalent cations (Ca, Mg) expressed in charge equivalents per liter. A charge equivalent is the molar concentration times the charge of the ion. Divalent cations are known to be able to bridge negative charges in the EPS and can improve dewaterability.
n-factor	See flow consistency index
PE or poly-electrolyte	A poly-electrolyte is a charged polymer that is used as a flocculant for sludge dewatering. For sludge dewatering mostly positively charged polymers are used because they can interact with the negatively charged organic substance (EPS) that make up the sludge flocs.
Reject water	This is the water that is separated from the sludge during mechanical dewatering. In this study we use this water as a way to get an impression of the overall ionic composition of the water phase in the sludge.
Rheology	Rheology is used in this study to describe the changes of the viscosity of a polymeric solution as a function of the shear forces applied. The viscosity of polymeric solutions changes depending on the amount of shear and show non-Newtonian behavior. In this study we use rheology to characterise the sludge solution and it is also used to characterise the PE flocculants used for sludge dewatering.
Streaming current PE demand	The PE demand determined at lab scale that is needed to neutralise the streaming current potential of a sludge to zero. In other words: the amount of PE needed to neutralise the surface charge of sludge flocs. See paragraph 1.3.1 for a detailed explanation.
Surface charge	The electric charge on the surface of a small particle in water. Positive or negatively charged ionic groups making up the structure of the particle are responsible for this charge. The charge may depend on the pH of the solution.
Thickening	A process to remove the bulk of the water from a sludge that is easiest to remove. Often gravity-based methods are used for this, sometimes assisted with a simple level of filtration. Coagulations and flocculants may be added to assist the thickening. As opposed to dewatering the focus with thickening is not to get an as dry as possible solid residue but more to cheaply remove the bulk of the water. Thickening is often a process that is done before dewatering.

Yield stress	A term from rheology to characterise polymeric substances that show a shear thinning behaviour. This is the highest stress that can be imposed on the solution when applying a low shear rate. See paragraph 1.3.1 for a detailed explanation.
Zeta potential	A specific way to measure and define the surface charge. The zeta potential is the charge at the plane at the interface between the bulk of the solution and the liquid that is attached to a particle.

5.2 ABBREVATIONS

Below is a list of the most frequently used abbreviations. See also appendix A2.1 for a more extensive list.

COD	Chemical oxygen demand
CST and CST/TS	Capillary suction test, see paragraph 1.3.1 for a detailed explanation. CST is often divided by the total solids content to take into account large differences in solid content (CST/TS)
EPS	Extracellular polymeric substance. See also glossary of terms.
M/D ratio	Ratio of monovalent (Na, K) to divalent cations (Mg, Ca). See also glossary of terms.
M+N/D ratio	Ratio of monovalent to divalent cations in which also ammonium is included as a monovalent cation. See also glossary of terms.
PE	Poly-electrolyte or flocculant used for sludge dewatering. See also glossary of terms.
PS	Primary sludge, sludge produced in the primary settler
PSD	Particle size distribution
SCP	Streaming current potential, see paragraph 1.3.1 for a detailed explanation
SRF	Specific resistance to filtration, see paragraph 1.3.1 for a detailed explanation
TOC	Total organic content
TAN	Total ammoniacal nitrogen.
TS and TSS	TS stands for Total Solids and TSS for total suspended solids. The difference is that for TSS the sample is filtered and that dissolved salts are therefore not taken into account for the TSS content.
VS and VSS	VS stands for Total Volatile Solids and VSS for total volatile suspended solids. The difference is that for TSS the sample is filtered and that dissolved salts are therefore not taken into account for the TSS content. The VS content is obtained from the TS content by glowing the sample at a temperature of 500 C and measuring the weight loss.
WAS	Waste activated sludge, the surplus sludge production of a biological activated sludge treatment plant.
WWTP	Waste water treatment plant. In the context of this study this is a sewage treatment plant.

6

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APPENDICE A

METHODOLOGY

A1.1 PROTOCOL OPTIMALISATIE SLIBONTWATERING

INLEIDING

Als onderdeel van het STOWA project slibontwatering, vinden er periodiek monsternames plaats rondom de slibontwatering van de RWZI's Bath en Harnaschpolder. Voorafgaand aan deze monsternames wordt de slibontwatering door Royal HaskoningDHV (RHDHV) beoordeeld en indien nodig in samenwerking met de bedrijfsvoerders geoptimaliseerd. Om dit op een gestructureerde en repeteerbare wijze uit te voeren, is door RHDHV een protocol opgesteld.

Het protocol zal handvaten bieden voor optimalisatie daar waar het functioneren van slibontwateringsinstallatie afwijkend is aan de reguliere bedrijfsvoering en het ontwateringsresultaat afwijkt van de bekende prestatie.

PROTOCOL SLIBONTWATERING

Het protocol bestaat uit meerdere stappen. De stappen 1, 2, 3 en 4 zijn generiek, stap 5 richt zich op de specifieke ontwateringstechniek en stap 6 is weer generiek voor beide onderzoek locaties. Bij de specifieke ontwateringstechnieken gaat het om centrifuges zoals deze op rwzi Harnaschpolder wordt toegepast en zeefbandpersen zoals deze worden toegepast op rwzi Bath.

1. CONTROLEREN ONLINE METINGEN (INDIEN AANWEZIG)

Bij ontwatering wordt in de meeste gevallen zowel het toevoerdebiet als de PE-dosering gerelateerd aan de hoeveelheid ingaande droge stof (DS). Het is voor de aansturing dus van belang dat de online DS-meter van het toevoerslib overeenkomt met de werkelijkheid. Daarom dient van het toevoerslib het DS-gehalte bepaald te worden ((NEN 6499:2014).
De monstername hiervoor dient één dag voor uitvoering van de optimalisatie plaats te vinden. Zodoende kan het vastgestelde DS-gehalte tijdens de optimalisatiedag vergeleken worden met de online DS-meter. Indien hier een afwijking aanwezig is van meer dan 5% (relatief) wordt geadviseerd de online DS-meter te kalibreren.

Bovenstaande geldt ook voor eventuele online DS-metingen op het centraat/filtraat en/of slibkoek. In de meeste gevallen wordt op deze data niet (automatisch) gestuurd, maar het is tijdens de optimalisatiedag wel praktisch om hier ook betrouwbare input van te verkrijgen.

2. STUREN OP CONSTANTE TOEVOER (VRACHT/DEBIET)

Na het controleren van de online metingen is het zaak om de toevoer (vracht/debiet) naar de ontwateringsinstallatie te stabiliseren indien dit nodig blijkt. Dit omdat de ontwatering is gebaat bij een constante toevoer. Afhankelijk van de bedrijfsvoering kan de toevoer als vracht (debiet x DS-gehalte) of als debiet worden ingesteld. Om een constante toevoer te kunnen realiseren moet rekening gehouden worden met de beschikbare slibbuffercapaciteit vóór en na de ontwateringsinstallatie.

Over het algemeen is de toevoer een handmatige instelling of een automatisch setpoint op basis van het slibniveau in de toevoerbuffer. De optimale toevoerinstelling dient indien nodig in overleg met de bedrijfsvoerders te worden vastgesteld.

3. MONSTERNAME ACTUELE SITUATIE (NA DOORVOEREN WIJZIGING)

Na stap 1 en 2 dienen er monsters genomen te worden van de actuele situatie na wijziging van instellingen ter optimalisatie. Het gaat hierbij om monstername van de toevoer, het centraat/filtraat, de slibkoek en het aangemaakte PE. De monsters dienen, met uitzondering van het centraat/filtraat, op DS-gehalte geanalyseerd te worden. Voor het centraat/filtraat moet de zwevende stof (NEN-EN 872:2005) worden vastgesteld.

4. OPTIMALISEREN PE-DOSERING

4.1 CHECK VAN DE PE-AANMAAK EN PE-DOSEERINSTALLATIE

Een goed ontwateringsresultaat wordt bereikt door

a. Een goede menging van aanmaakwater en ruw PE in de PE aanmaakunit. Hiervoor dient de inhoud in de aanmaakunit voldoende te worden opgewoeld, zonder drijflaag en randeffecten.
b. Een goede vlokking van het slib en voldoend slib/waterscheiding. Dit kan worden beoordeeld door een monster van het slib na opmenging met aangemaakt PE te nemen en visueel te beoordelen.

Zoals aangegeven onder paraaf 3 wordt een DS bepaling uitgevoerd op het aangemaakte PE na wijziging van instellingen en vergeleken met de DS als gevolg van de instellingen hiervoor. De beschikbare rijpingstijd wordt daarbij vastgesteld bij de ingestelde PE concentratie tov het normale verbruik.

4.2 OPTIMALISATIE PE DOSERING

Bij het optimaliseren van de ontwatering speelt de PE-dosering een belangrijke rol. Voor PE-dosering geldt dat gestuurd dient te worden op een lichte overdosering. Zowel een te hoge als een te lage PE-dosering heeft een negatief effect op het ontwateringsresultaat. Daarnaast zorgt een te hoge PE-dosering voor onnodig hoge operationele kosten.

In principe wordt ervan uitgegaan dat de ontwateringsinstallaties inclusieve PE dosering naar behoren functioneren. Daarom wordt op de optimalisatiedag voornamelijk gekeken of het ontwateringsresultaat voldoet. Dit kan worden beoordeeld door het drogestofgehalte van de slibkoek, de helderheid centraat/filtraat en het PE verbruik op het moment te vergelijken met de behaalde resultaten in voorgaande periodes.

Indien optimalisatie nodig is omdat de prestatie van het moment afwijkt kunnen onderstaande stappen worden doorlopen om de PE-dosering te optimaliseren:

a. Centraat/filtraat monster visueel beoordelen

Hierbij zijn onderstaande bevindingen over het algemeen het gevolg van een te hoge PE-overdosering:

i. *Melkachtig centraat/filtraat;*
ii. *Overmatig schuimen;*
iii. *Drijflaag met zichtbare slibvlokken.*

De volgende bevindingen wijzen over het algemeen juist op PE-onderdosering:

i. *Donker centraat/filtraat;*
ii. *Geen duidelijke afscheiding tussen slib en centraat/filtraat;*
iii. *Aanwezigheid kleine slibdeeltjes (slechte vlokvorming).*

b. Toevoerslib mengen met centraat/filtraat

Om een beeld te krijgen van de mate van PE-dosering is het een mogelijkheid om wat toevoerslib te mengen met een centraat/filtraat monster. Het gaat hierbij om een hoeveelheid van 50 tot 100 gram toevoerslib bij ongeveer 1 liter centraat/filtraat. Indien het toegevoegde slib (na mengen) snel en volledig 'uitvlokt', is dit een signaal van een te hoge PE-overdosering.

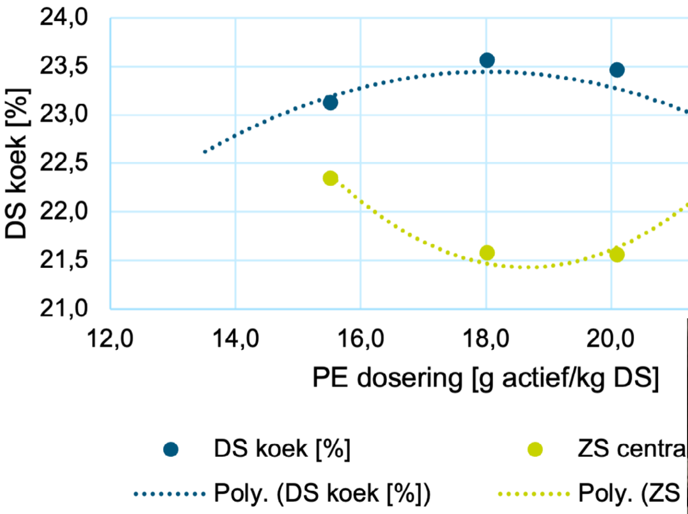
Indien het toegevoegde slib (na mengen) niet of nauwelijks 'uitvlokt', betekent dit dat er mogelijk sprake is van PE-onderdosering.

c. PE-dosering aanpassen

Afhankelijk van de conclusies bij stap 4a en 4b kan er besloten worden om de PE-dosering te verhogen of te verlagen. Dit dient te gebeuren in stappen van maximaal 10% (relatief, t.o.v. de huidige PE-dosering). Bij elke aanpassing van de PE-dosering dient er minimaal 45-60 minuten gewacht te worden, alvorens een nieuwe monsternameronde wordt uitgevoerd.

Als het ontwateringsresultaat afwijkt van voorafgaande resultaten kan de PE dosering worden geoptimaliseerd. Hiervoor dienen een aantal monsternamerondes uitgevoerd te worden bij verschillende PE doseerhoeveelheden. Op deze manier is het mogelijk om (na uitvoering van de laboratorium analyses) vast te stellen wat de optimale PE-dosering is. Dit kan gevisualiseerd worden in een PE-curve zoals weergeven in onderstaande voorbeeld.

FIGUUR 1 PE DOSERING – DROGESTOF - CURVE



5. OPTIMALISEREN INSTELLINGEN ONTWATERINGSINSTALLATIE

In principe wordt ook bij deze stap ervan uitgegaan dat de ontwateringsinstallaties naar behoren functioneren. Daarom wordt op de optimalisatiedag voornamelijk gekeken of het ontwateringsresultaat voldoet. Dit kan worden beoordeeld door het drogestofgehalte van de slibkoek, de helderheid centraat/filtraat en het PE verbruik op het moment te vergelijken met de behaalde resultaten in voorgaande periodes.

Onderstaande beschrijft grofstoffelijk hoe de ontwateringsinstallaties kunnen worden geoptimaliseerd. De beschrijving richt zich op 2 verschillende types ontwateringsinstallaties. In onderdeel a wordt de centrifuge optimalisatie besproken. De zeefbandpers optimalisatie komt in onderdeel b aan bod.

a. Centrifuge

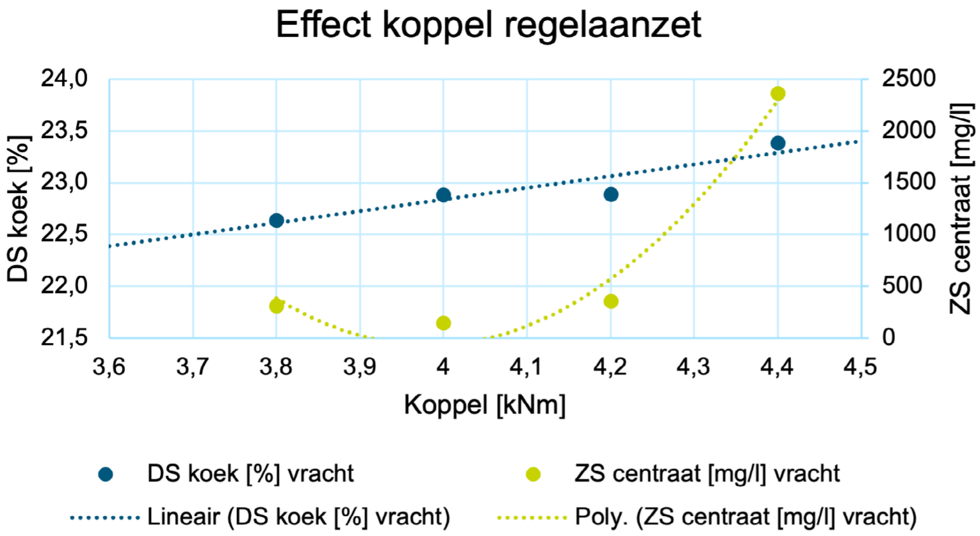
Om het ontwateringsresultaat te verbeteren kunnen de instellingen worden aangepast:

1. Verlagen/verhogen slibtoevoerdebiet,
2. Wijzigen van koppel door veranderen basis verschiltoerental (Δrpm) of veranderen koppel regelaanzet (% of kNm),
3. Toerental centrifuge (G kracht),
4. Overstorthoogte centraat,
5. Optimalisatie menging slib met aangemaakt PE oplossing.

Bij elke aanpassing van de instellingen dient er minimaal 45-60 minuten gewacht te worden, alvorens een nieuwe monsternameronde wordt uitgevoerd.

Om een volledig beeld te krijgen dient een aantal monsternamerondes te worden uitgevoerd bij verschillende instellingen.
Zie onderstaand beeld van een koppel-drogestof-curve zoals weergegeven in Figuur 2.

FIGUUR 2 KOPPEL – DROGESTOF - CURVE



b. Zeefbandpers

Om het ontwateringsresultaat van een zeefbandpers te verbeteren, kunnen de volgende instellingen worden aangepast:

1. Verlagen/verhogen slibtoevoerdebiet,
2. Verhogen/verlagen bandsnelheid,
3. Plaatsen drempel aan begin of einde van de voorontwatering,
4. Optimalisatie menging slib met aangemaakt PE oplossing.

6. MONSTERNAME AFRONDING FULL SCALE OPTIMALISATIE

Na voltooide optimalisatie van de full scale installatie dienen er monsters genomen te worden. Afhankelijk van de voortgang van de full scale optimalisatie kan de monstername aan het einde van de dag of op de eerstvolgende weekdag worden uitgevoerd. Doel is dat deze bemonsteringsronde een representatief beeld moet geven van de bedrijfsvoering na evt. wijzing in de bedrijfsvoering.

Bij deze monsternameronde worden monsters genomen van de toevoer ontwatering, het centraat/filtraat, de slibkoek en het aangemaakte PE ten behoeve van het STOWA ontwateringsonderzoek. De monsters worden verzameld door bedrijfsvoering en RHDHV en vervolgens ter voorbehandeling en analyses doorgezet naar TUDelft en RHDHV. Een monster van het ruw PE van die dag wordt via de op de locatie gebruikelijke route doorgezet naar Intertek.

A1.2 SLUDGE DEWATERABILITY

MODIFIED HIGGINS TEST

The centrifugation method was adapted from Weij (2018) and To et al. (2016) to be used with the bench-scale centrifuge in the Royal HaskoningDHV laboratory. The centrifuge force was chosen based on TS of dried cake obtained with different g forces to make it comparable with full-scale dewatering results.

Firstly, a PE solution (0.3% active PE, w/v %) was added to 150 ml of sludge in a beaker and poured back and forth between two beakers until a clear layer of water was formed. The required amount of PE solution was recorded for calculating the PE consumption per kg of TS (or VS) of the sample. The sludge floc was separated from the liquid and put in a plastic bag and further pressed in a belt filter. After this, the sample was placed in three layers of Dispolab, glasfibre GF/C filters, and put in a gauze. The whole package was put in the centrifuge tube with a holder inside to keep the package at a distance from the bottom of the tube. Then, the package was centrifuged for 5 min at 1,040 × g. The centrifuge tube was taken out and the liquid was decanted, and the package and centrifuge tube were put back in in the centrifuge and was centrifuged for another 15 min at 1,040 × g. The TS content of the dewatered sludge cake was measured by placing the sample in an oven at 105 °C for at least 24 hours and reported in percentage of TS.

A1.3 POLYMER CHARACTERIZATION

For more information about PE characterization, see references Korving 2023 and Raffa/ STOWA 2017

APPENDICE B

KENDALL CORRELATION MATRIX

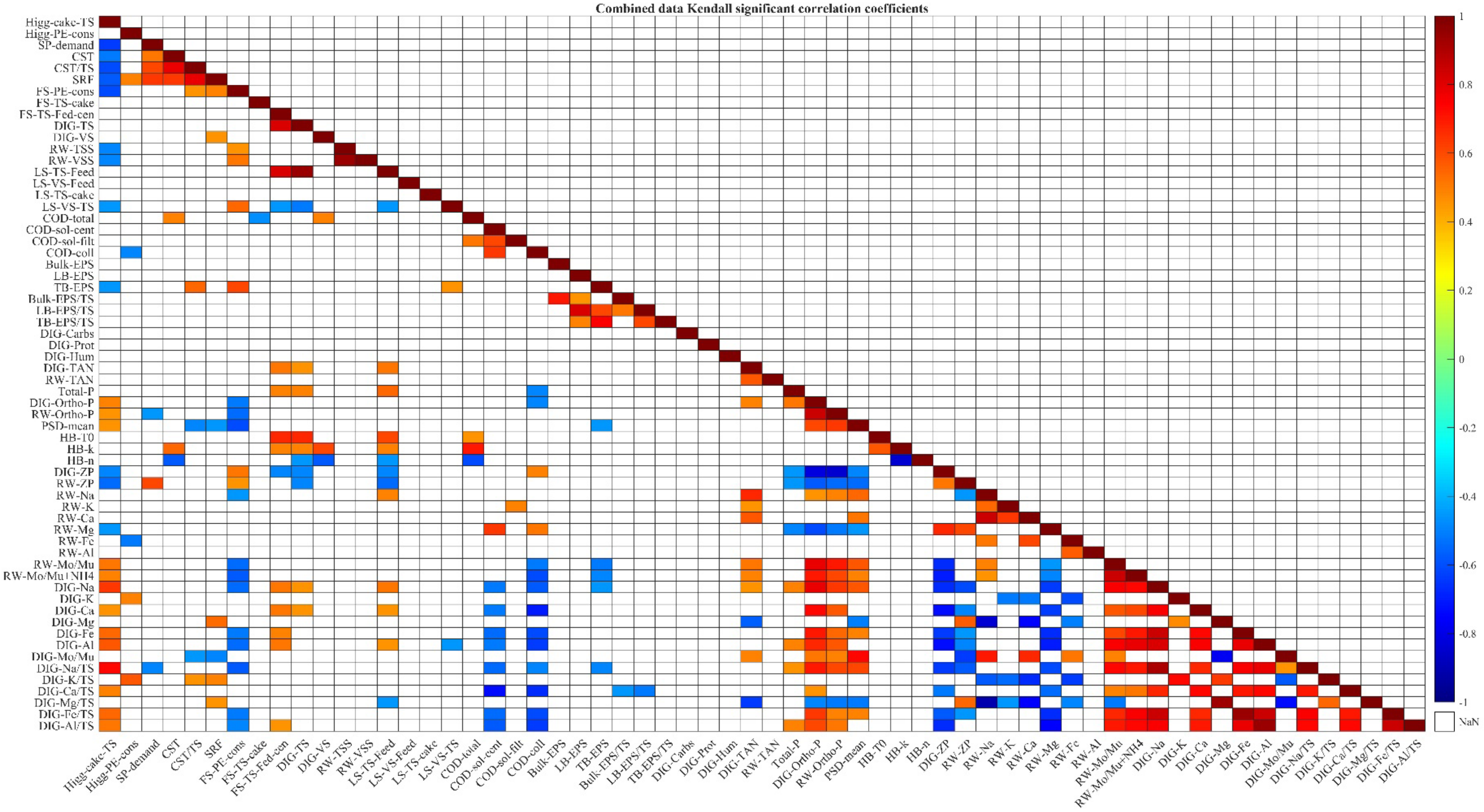
B.1.1 NOMENCLATURE

Abbreviation	Unit	Description
Hig-cake-TS	[%]	Higgins tests digestate cake concentration
Hig-PE-cons	[g PE/kg TS]	Higgins tests polyelectrolyte concentration per mass of total solids
SP-demand	[mL/gTS]	Streaming potential polyelectrolyte demand
CST	[s]	Capillary suction time
CST/TS	[s/gTS/L]	Capillary suction time per total solids concentration
SRF	[Tm/kg]	Specific resistance to the filtration of the digestate
FS-PE-cons	[g active PE/kg TS]	Full-scale polyelectrolyte dose
FS-TS-cake	[%]	Full-scale solids concentration in the centrifuge pellet
FS-TS- fed-cen.	[%]	Full-scale solids concentration fed to the centrifuge
DIG-TS	[g/L]	Total solids in the digestate
DIG-VS	[g/L]	Volatile solids in the digestate
RW-TSS	[g/L]	Reject water total suspended solids
RW-VSS	[g/L]	Reject water volatile suspended solids
LS-TS-Feed	[%]	Lab-scale total solids concentration in the digestate
LS-VS-Feed	[%]	Lab-scale volatile solids concentration in the digestate
LS-TS-cake	[%]	Lab-scale total solids concentration in the cake
LS-VS/TS	[%]	Lab-scale volatile solids concentration in the cake
COD-total	[mg COD/L]	Total digestate chemical oxygen demand
COD-sol-cent	[mg COD/L]	Soluble chemical oxygen demand measured after centrifugation (15 minutes at 15000 RCF) (1)
COD-sol-filt	[mg COD/L]	Soluble chemical oxygen demand measure after filtration (0.45 µm) (2)
COD-coll	[mg COD/L]	Colloidal chemical oxygen demand (1)-(2)
Bulk-EPS	[mg C/L]	Total organic carbon concentration of the soluble extracellular polymeric substances
LB-EPS	[mg C/L]	Total organic carbon conc. of the loosely bound extracellular polymeric substances
TB-EPS	[mg C/L]	Total organic carbon conc. of the tightly bound extracellular polymeric substances
Bulk-EPS/TS	[mg C/gTS]	Total organic carbon of the soluble extracellular polymeric substances per mass of total solids
LB-EPS/TS	[mg C/gTS]	Total organic carbon of the loosely bound extracellular polymeric substances per mass of total solids
TB-EPS/TS	[mg C/gTS]	Total organic carbon of the tightly bound extracellular polymeric substances per mass of total solids
DIG-Carbs	[mg Glucose/L]	Soluble concentration of carbohydrates in the digestate
DIG-Prot	[mg BSA/L]	Soluble concentration of proteins in the digestate
DIG-Hum	[mg HA/L]	Soluble concentration of humic substances in the digestate
DIG-TAN	[mg TAN/L]	Total ammoniacal nitrogen concentration in the digestate
RW-TAN	[mg TAN/L]	Total ammoniacal nitrogen concentration in the reject water*
Total-P	[mg P/L]	Total phosphorous in the digestate
DIG-Ortho-P	[mg PO_4^{3-}-P/L]	Orthophosphate concentration in the digestate
RW-Ortho-P	[mg PO_4^{3-}-P/L]	Orthophosphate concentration in the reject water*
PSD-mean	[meq/meq]	Average of the particle size distribution
HB-T0	[Pa]	Yield stress in the Herschel-Bulkley model

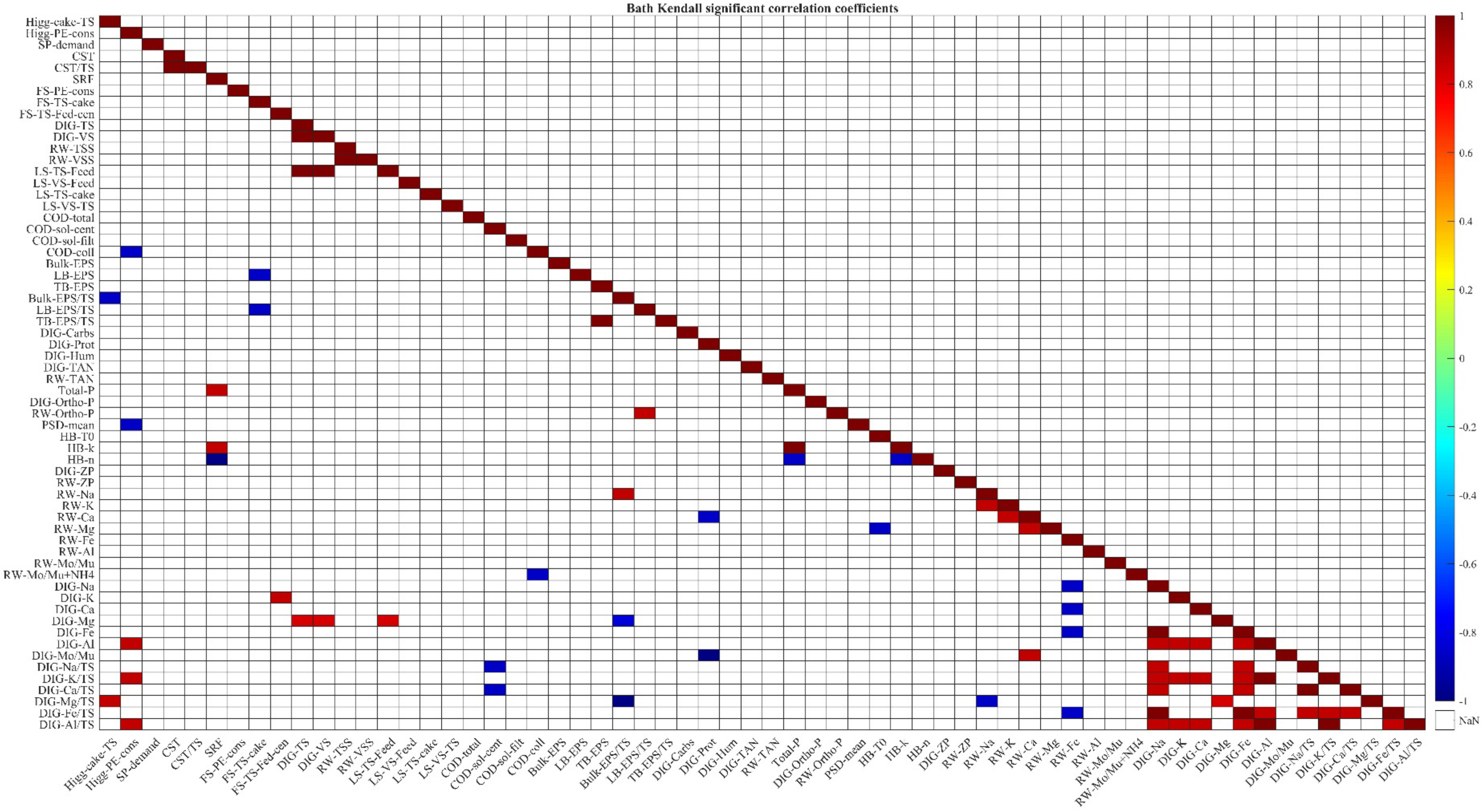
Abbreviation	**Unit**	**Description**
HB-k	[Pa*s^n]	Consistency index in the Herschel-Bulkley model
HB-n	[dimensionless]	Flow index in the Herschel-Bulkley model
DIG-ZP	[mV]	Zeta potential in the digestate
RW-ZP	[mV]	Zeta potential in the reject water
RW-Na	[mg/L]	Total sodium concentration in the reject water
RW-K	[mg/L]	Total potassium concentration in the reject water*
RW-Ca	[mg/L]	Total calcium concentration in the reject water*
RW-Mg	[mg/L]	Total magnesium concentration in the reject water*
RW-Fe	[mg/L]	Total iron concentration in the reject water*
RW-Al	[mg/L]	Total aluminium concentration in the reject water*
RW-Mo/Mu	[meq/meq]	Monovalent over multivalent cations ratio in the reject water
RW-Mo/Mu+NH4+	[meq/meq]	Monovalent over multivalent cations ratio in the reject water including TAN concentration
DIG-Na	[mg/L]	Total sodium concentration in the digestate
DIG-K	[mg/L]	Total potassium concentration in the digestate
DIG-Ca	[mg/L]	Total calcium concentration in the digestate
DIG-Mg	[mg/L]	Total magnesium concentration in the digestate
DIG-Fe	[mg/L]	Total iron concentration in the digestate
DIG-Al	[mg/L]	Total aluminium concentration in the digestate
DIG-Mo/Mu	[meq/meq]	Monovalent over multivalent cations ratio in the digestate
DIG-Na/TS	[mg/gTS]	Total sodium in the digestate per mass of total solids
DIG-K/TS	[mg/gTS]	Total potassium in the digestate per mass of total solids
DIG-Ca/TS	[mg/gTS]	Total calcium in the digestate per mass of total solids
DIG-Mg/TS	[mg/gTS]	Total magnesium in the digestate per mass of total solids
DIG-Fe/TS	[mg/gTS]	Total iron in the digestate per mass of total solids
DIG-Al/TS	[mg/gTS]	Total aluminium in the digestate per mass of total solids

* times 3.8 in Bath due to the dilution of the reject water from the belt filter with belt cleaning water.

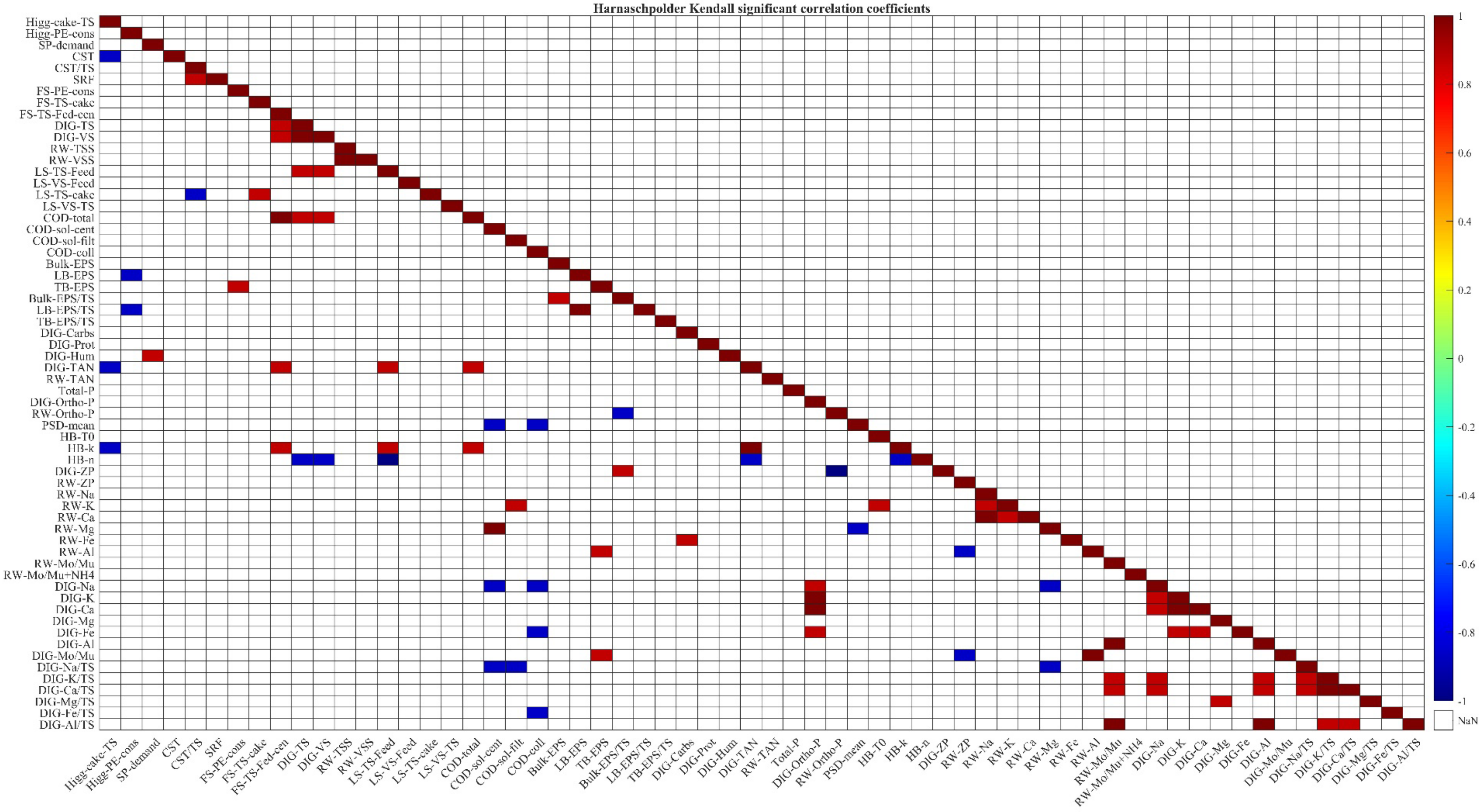
B2.2 KENDALL CORRELATION MATRIX – MERGE OF TWO DATASETS FROM TWO WWTPS



B2.3 KENDALL CORRELATION MATRIX – BATH WWTP



B2.4 KENDALL CORRELATION MATRIX – HARNASCHPOLDER

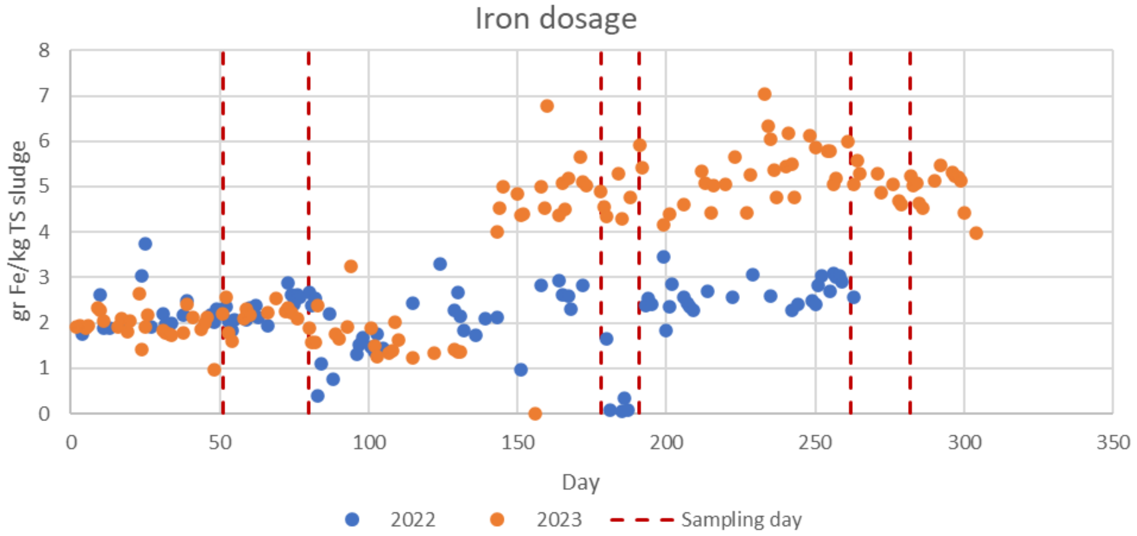


APPENDICE C

KRESULTS FULL-SCALE

C1.1 FULL-SCALE DATA HARNASCHPOLDER

FIGURE C1.1 CHEMICAL DOSAGE OF HARNASCHPOLDER WWTP IN SLUDGE LINE FOR YEAR 2022 AND 2023



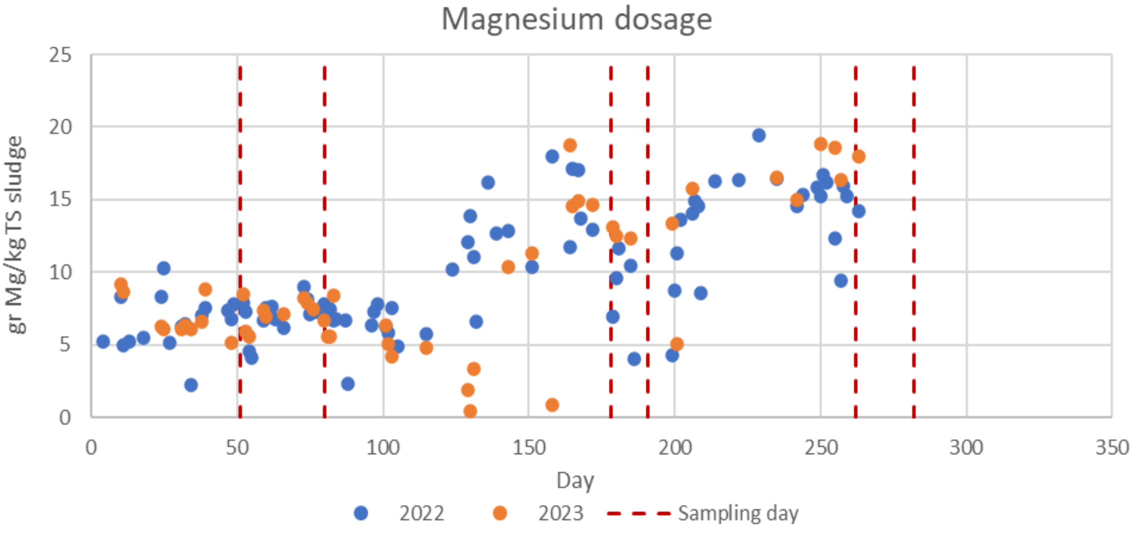
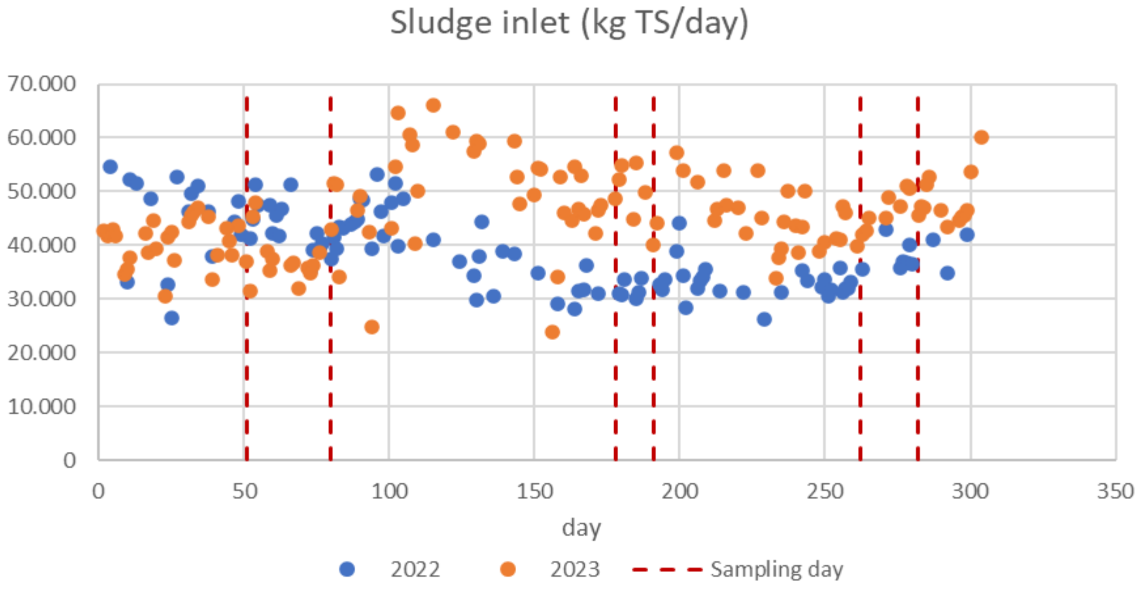


FIGURE C1.2 SLUDGE INLET TO DEWATERING CENTRIFUGES HARNASCHPOLDER WWTP FOR YEAR 2022 AND 2023



C2.1 FULL-SCALE DATA BATH

FIGURE C1.3 SLUDGE INLET TO DEWATERING CENTRIFUGES BATH WWTP FOR YEAR 2022 AND 2023