

# **ANAEROBIC DIGESTATE TREATMENT**

# **Technology Introduction & Case Studies**





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# **1.Introduction**

This document gives a brief introduction to anaerobic digestion technology; the benefits and drawbacks. The focus will then shift to the production, handling and <u>treatment options</u> of the digestate slurry arising as a by-product of anaerobic digestion.

The final section will look at pilot work conducted by Esmil & Ekoton on digestate treatment thus far with reference to case studies of bench scale studies, pilot work or full scale operations.

# **1.1 Anaerobic Digestion**

Anaerobic Digestion (AD) is a natural process where plant and animal materials (biomass) are broken down by micro-organisms in the absence of air. The AD process begins when biomass is put inside a sealed tank or digester.

Naturally occurring micro-organisms digest the biomass, which releases a methane-rich gas (**biogas**) that can be used to generate renewable heat and power; helping to cut fossil fuel use and reduce greenhouse gas emissions. The remaining material (**digestate**) is rich in nutrients, so it can be used as a fertiliser.



Many forms of feedstock are suitable for AD; including food waste, slurry and manure, as well as crops and crop residues. However, woody biomass cannot be used in AD because the micro-organisms can't breakdown the lignin, the compound that gives wood its strength.

AD is not a new technology, it has been used in the UK since the late 1800s, but now an increasing number of AD plants are being built in the UK to generate clean renewable energy. AD is also used to treat the waste produced in homes, farms, supermarkets and industries across the UK. This helps divert waste from landfill.



# **1.2 Digestate**

Digestate is a nutrient-rich substance produced by anaerobic digestion that can be used as a fertiliser. It consists of left over indigestible material and dead micro-organisms - the volume of digestate will be around 90-95% of what was fed into the digester. Digestate is not compost, although it has some similar characteristics. Compost is produced by aerobic micro-organisms, meaning they require oxygen from the air.

By using digestate instead of synthetic fertilisers derived from natural gas, we can save energy, cut consumption of fossil fuels and reduce our carbon footprint.

All the nitrogen, phosphorous and potassium present in the feedstock will remain in the digestate as none is present in the biogas. However, the nutrients are considerably more available than in raw slurry, meaning it is easier for plants to make use of the nutrients.

The exact composition of digestate is determined by the AD plants diet. However, some typical values for nutrients are:

- Nitrogen: 2.3 4.2 kg/tonne
- Phosphorous: 0.2 1.5 kg/tonne
- Potassium: 1.3 5.2 kg/tonne

#### **1.2.1 Where can digestate be used?**

Before investing in AD it is important to consider where and how the digestate will be used. If it is not to be considered waste, digestate must meet the standards set out in the **Quality Protocol** and **PAS110** (England & Wales) and the **SEPA** position statement (Scotland).

The Quality Protocol sets out criteria for the production of quality outputs from anaerobic digestion of bio-waste. Producers and users are not obliged to comply with the Quality Protocol. If they do not, the digestate will be considered to be waste and waste management controls will apply to its handling, transport and application.

The Publicly Available Specification (called PAS110) for digestate, derived from the anaerobic digestion of source-segregated biodegradable materials creates an industry specification against which producers can verify that the digested materials are of consistent quality and fit for purpose. If an AD plant meets the standard, its digestate will be regarded as having been fully recovered and to have ceased to be waste, and it can be sold with the name "Bio-fertiliser". PAS110 is for the digestate product, the QP is about safeguards and process needed to achieve PAS110.

The Anaerobic Digestate Quality Protocol is not applicable in Scotland, however SEPA have published a Regulatory Position Statement which is to be followed and used in conjunction with PAS110 certification. Also note that in Scotland under the Zero Waste Plan digestate which is not PAS110 certified and produced in accordance with the SEPA position statement above, will not be counted towards recycling targets even if it is currently produced and used under an exemption.



The Northern Ireland Environment Agency (NIEA) published a Northern Ireland Regulatory Position Statement explaining how digestate will be regulated, in July 2010 and at the same time adopted the AD Quality Protocol.

The Biofertiliser Certification Scheme (BSC) provides assurance that biofertiliser (the BSC name for digestate) is safe and of good quality. Renewable Energy Assurance Ltd., a subsidiary of the Renewable Energy Association, administers the Scheme for England, Scotland, Wales and Northern Ireland.

#### **1.2.2 Digestate enhancement and treatment**

Digestate can be used whole, spread on land with tankers or umbilical pipe lines. Alternatively, it can be separated in to liquor and fibres, which have differing distributions of nutrients. The liquor should contain less than 6% dry matter. Separated liquor can be spread more easily to growing crops. Separated fibre can be used fresh as a soil conditioner or, after further aerobic composting to stabilise it, a material suitable for making into a compost product.

With a planned increase in the number and capacity of AD plants to treat a variety of organic waste streams in the UK, digestate enhancement technologies are gaining more attention. Digestate enhancement technologies could be assessed by an AD operator looking to provide any of the following options for an AD plant:

- increase the value of digestates;
- secure use of digestates;
- create new markets for digestate products; and
- decrease the operating costs (OPEX) of the facility.

Digestate's major drawback is that it is a pre-determined mix of nutrients that cannot be altered. There must be enough land in the vicinity of the digester that can accept the digestate within the restrictions of Nitrate Vulnerable Zones (NVZs); 62% of land in England and 4% in Wales falls within NVZs.

Furthermore, due to fertilizer application timings, digestate is typically stored for an extended duration before application to agricultural land requiring large storage tanks or lagoons. Also proximity of the farmland to the digesters has a significant impact on the transportation costs, both economic and environmental.

For these reasons among others digestate treatment/enhancement has gained some traction in this industry and is likely to become widely adopted with an increasing number of AD operators in the UK.

## **1.3 Water Recovery and Reuse**

A less considered drawback of AD plants is the water requirement. The feed to an anaerobic digester is required to be pumped as a slurry and hence some organic material requires dilution. Sometimes liquid waste is utilized but quite often, considerable volumes of freshwater are consumed to enable this process.



By installation of a digestate treatment solution, significant volumes of clean reusable water can be recovered. This can be used across the AD plant or any other processes occurring on site. The following gives some examples of how recovered water from digestate may be reused within the process:

- Anaerobic digester feed dilution;
- Plant washing/ cleaning operations;
- Boiler feed (may require additional polishing);
- Or simply discharge at a very low cost.



# **2. Digestate Treatment Solution(s)**

This section illustrates our digestate treatment solution(s) that have been tried and tested successfully at laboratory scale, piloting stages and full scale operation. The aim of digestate treatment/enhancement is to provide additional environmental and economic benefit for an AD operation. This can be achieved through reduction of digestate volume required for storage/transport and reduced need for feed dilution and other process water through water recovery and reuse.

## **2.1 Process Description**

Figure 1 demonstrates a simplified process diagram of a viable digestate treatment solution. As can be seen a number of treatment steps (units) are required to attain a high quality water stream suitable for reuse. The remaining two outlet streams include the solid (cake) fraction and the nutrient dense liquid concentrate.



#### Figure 1. Simplified Block Diagram of Digestate Treatment Solution

Each step is integral in producing a reusable water stream and concentrated solid and liquid fractions. These will be described in full in the following sections (2.2 - 2.4), but to summarise:

#### ✤ MDQ – Multidisc Screw Dehydrator

- Separates the solid and liquid fraction of the digestate, producing a very dry, stackable cake.

#### **VSEP – Vibratory Shear Enhanced Process**

- Proprietary membrane technology to remove majority of the contaminants.
   Uses vibration of membrane packs for improved performance and exceptional fouling resistance.
- RO Reverse Osmosis
  - Spiral-wound membrane technology for residual contaminant removal to produce high quality water.



# **2.2 Digestate Dewatering**

Traditionally in sludge dewatering applications, a centrifuge, filter press or screw press are used to separate the solids from the liquid fraction. Particularly in the anaerobic digestion sector centrifuges have been installed and soon after discontinued due to their high resource cost (water, energy, chemicals).

Instead a novel technology, manufactured by Ekoton, known as the multidisc screw press dehydrator can be utilised for digestate dewatering. This outperforms conventional dewatering technologies particularly in the typical operating flows of an anaerobic digestion plant. This infers a significant reduction in energy, chemical, flushing water and maintenance costs associated with digestate dewatering.

The Multidisc Screw Press dehydrator is manufactured from stainless steel AISI 304 with a variety of available dimensions. The dehydrators can operate as a standalone solution or part of larger mechanical dewatering systems. The standard system can include: the dehydrator, an external pump for initial sludge supply, a station of flocculent solution preparation, the flocculent solution dosing pump and the transporter of dewatered sludge.

Optionally, the dehydrators with 200 mm screws can be fitted with a rake type fine screen which is installed straight into the initial sludge inlet line in the technological chamber. This screen allows sludge filtration, which removes large and fibrous inclusions of the sludge before entering the inner pumps and dewatering drums of dehydrator. Respectively, the initial sludge can be fed directly into the dehydrator from the clarifiers and bioreactors, without the need for an additional external sludge storage chamber.

## **2.2.1 Operating Principle**

The dehydrator operates in continuous mode. The initial sludge is intermittently injected from the external storage tank into the dehydrator process tank. The mixing tank that is located in the process tank prevents stratification of the sludge.

The sludge is then supplied by the pump to the dosing tank, and then through the calibrated overflow to the flocculent tank. Sludge excess from the dosing tank returns into the process tank through the control overflow union.

The polyelectrolyte solution is supplied into the flocculent tank. The solution is mixed with initial sludge by means of an agitator to form floccules. Then the initial sludge enters into the dewatering drum(s) and moves within the screw to the zone of filter cake discharging, as a result of the screw rotation. As it moves along the drum, the sludge is dewatered and filtrate is drained through clearances between the rings.





Figure 2 Operating Principle of Multidisc Screw Dehydrator

The main operating sub system of the dehydrator is the dewatering drum, which features both a set of movable and fixed rings assembled on the supporting frame. A screw with a varying pitch is located inside the drum. In the drum mouth (condensation zone), the clearances between screw flights are larger than in the next zone (dehydration zone). The screw pitch gradually decreases to the wringing zone. This screw is driven by a geared motor at a low speed. The tray for filtrate receiving and draining is located under the dewatering drum.

The drum exit is partially overlapped by the movable divider. By changing distance between the drum exit and the divider, the counter pressure on the sludge moving inside the drum can be regulated, which influences the degree of sludge dewatering. The dewatered sludge is gravity fed into the receiving hopper of the transporter. The dewatered sludge produced by this dehydrator can reach as low as 75-82% residual moisture at an average flocculent dose of 1.5-3.5 kg/t of dry sludge matter.



#### 2.2.2 Energy

\* Comparison between dehydrating units only (Not including the related equipments.)

- Since dehydrator itself is operated at low-speed rotation energy demand is low.
- This means that there is no vibration and almost no noise arising from this unit during operation.



#### **2.2.3 Flushing Water**



- Self-cleaning mechanism reduces need for flushing water
- ✤ No more than 0.5% of treated sludge volume required as wash water.
- Wash cycle typically lasts 10 seconds once every 10 minutes.

#### **2.2.4 Maintenance**

Operation	Unit and Devices	Maintenance Period
Periodical check and	Oil Quality Check (Submersible Pump)	Annually
replacement if necessary	Replacement of oil and mechanical sealing (submersible pump)	Every 2 years
	Submersible Pump	Every 4 years
Replacement	Whole Drive	Once in a 4-6 year period
	Movable and stationary rings, bearings	

- Equipment does not need much time for daily inspection and service (only visual inspection and washing of chambers by tap water once per two-three days).
- Service and maintenance are not complicated and can be done by local staff.
- Maintenance is by far the cheapest in comparison to other dewatering technologies.



#### **2.2.5 Benefit Summary**

Figure 4 below details the CAPEX and OPEX cost comparison between conventional dewatering technologies and the Multidisc screw press. This has been evaluated based on a small scale municipal WWTP. For medium sized the cost becomes less attractive and large scale the technology is not feasible.



#### Figure 3. Cost Comparison of Sludge Dewatering Technologies (Small WWTP)

Other Benefits of Multidisc Screw Press include:

- Ability to control odour emission and improve sanitation due to closed construction.
- Dewatering of FOG sludge in a wide range of concentrations
- Able to treat low concentration sludge if necessary in a range of 0.3 10% DS content.
- Continuous automatic operation.



## **2.3 Vibratory Shear Enhanced Processing (VSEP)**

While membrane-based separations of liquids from solids have enjoyed increasing popularity over the last 20 years, the technology has an inherent Achilles heel that affects all membrane devices: fouling. This long-term loss in throughput capacity is due primarily to the formation of a boundary layer that builds up naturally on the membranes surface during the filtration process. In addition to cutting down on the flux performance of the membrane, this boundary or gel layer acts as a secondary membrane reducing the native design selectivity of the membrane in use. This inability to handle the build-up of solids has also limited the use of membranes to low-solids feed streams. <a href="http://www.vsep.com/industries/biogas.html">http://www.vsep.com/industries/biogas.html</a>



Figure 4. Cross Flow Membrane Filtration

To help minimize this boundary layer build-up, membrane designers have used a method known as tangential-flow or cross-flow filtration that relies on high velocity fluid flow pumped across the membranes surface as a means of reducing the boundary layer effect (Figure 5). In this method, membrane elements are placed in a plate-and-frame, tubular, or spiral-wound cartridge assembly, through which the substance to be filtered (the feed stream), is pumped rapidly.

In cross-flow designs, it is not economic to create shear forces measuring more than 10-15 thousand inverse seconds, thus limiting the use of cross-flow to low-viscosity (watery) fluids. In addition, increased cross-flow velocities result in a significant pressure drop from the inlet (high pressure) to the outlet (lower pressure) end of the device, which leads to premature fouling of the membrane that creeps up the device until permeate rates drop to unacceptably low levels.

## **2.3.1 Operating Principle**

New Logic, however, has developed an alternative method for producing intense shear waves on the face of a membrane. The technique is called Vibratory Shear Enhanced Processing (VSEP). In a VSEP System, the feed slurry remains nearly stationary, moving in a leisurely, meandering flow between parallel membrane leaf elements. Shear cleaning action is created by vigorously vibrating the leaf elements in a direction tangent to the faces of the membranes (Figure 6).





Figure 5. Vibratory Shear Enhance Processing

The shear waves produced by the membrane's vibration cause solids and foulants to be lifted off the membrane surface and remixed with the bulk material flowing through the membrane stack. This high shear processing exposes the membrane pores for maximum throughput that is typically between 3 and 10 times the throughput of conventional cross-flow systems.

The VSEP membrane filter pack consists of leaf elements arrayed as parallel discs and separated by gaskets. The disc stack resembles records on a record changer with membrane on each side.



Figure 6. VSEP Membrane Stack

The disk stack is oscillated above a torsion spring that moves the stack back and forth approximately 7/8 inches (2.22 centimetres). This motion is analogous to the agitator of a washing machine but occurs at a speed faster than that which can be perceived by the human eye.

The oscillation produces a shear at the membrane surface of about 150,000 inverse seconds (equivalent to over 200 G's of force), which is approximately ten times the shear rate of the best conventional cross-flow systems. More importantly, the shear in a VSEP System is focused at the membrane surface where it is cost effective and most useful in preventing fouling, while the bulk fluid between the membrane disks moves very little.



V**SEP** Resonating Drive System



Figure 7. VSEP Resonating Drive System

Because VSEP does not depend on feed flow induced shearing forces, the feed slurry can become extremely viscous and still be successfully dewatered. The concentrate is essentially extruded between the vibrating disc elements and exits the machine once it reaches the desired concentration level. Thus, VSEP Systems can be run in a single pass through the system, eliminating the need for costly working tanks, ancillary equipment and associated valving.

#### **2.3.2 VSEP System Operation**

At start up, the VSEP system is fed with slurry and the concentrate valve is closed. Permeate is produced and suspended solids in the feed are collected inside the VSEP filter pack. After a programmed time interval, valve one is opened to release the accumulated concentrated solids. The valve is then closed to allow the concentration of additional feed material. This cycle repeats indefinitely.

The operating pressure is created by the feed pump. VSEP machines can routinely operate at pressures as high as 1,000 psig (68.95 BAR). While higher pressures often produce increased permeate flow rates, they also use more energy. Therefore, an operating pressure is used that optimizes the balance between flow rates and energy consumption.

In most cases, the filtration rate can be further improved by increasing the operating temperature. The temperature limit on a standard VSEP system is 175° F (79°C), significantly higher than competitive membrane technology. Even higher temperature constructions are also available.

The vibration amplitude and corresponding shear rate can also be varied which directly affects filtration rates. Shearing is produced by the torsion oscillation of the filter stack. Typically the stack oscillates with an amplitude of 3/4 to 1 1/4 inches (1.9 to 3.2 cm) peak to peak displacement at the rim of the stack. The oscillation frequency is approximately 53 Hz and produces a shear intensity of about 150,000 inverse seconds.

Feed residence time is set by the frequency of the opening and closing of the exit valve (valve one). The solids level in the feed increases as the feed material remains in the



machine. Occasionally, a cleaner is added to the membrane stack and continued oscillation helps clean the membrane in minutes. This process can be automated and only consumes approximately 50 gallons (189 litres) of cleaning solution thus reducing cleaner disposal problems inherent with other membrane systems.

#### **2.3.3 Membrane Selection**

Membrane selection is the single most important parameter that affects the quality of the separation. Other important parameters that affect system performance are pressure, temperature, vibration amplitude, and residence time. All of these elements are optimized during testing and entered into the programmable logic controller (PLC) which controls the system.

The VSEP system comprises of flat sheet membranes stacked to form the membrane pack. The most effective membranes to install for the first stage of digestate treatment is reverse osmosis (RO) membranes. The RO membrane is able to rejects suspended solids, free & emulsified oil, fats, proteins, high molecular weight organics; as well as smaller and more difficult to remove contaminants such as soluble solids, ions and even some ammonia.

		< Electro	n microscope	•	Optical microsc	ope	•	Visible with the eyes
	lonic range	Molecular ra	nge Ma	acromolecular range	Mic	ro particles		Macro particles
Micrometer	0.001	0.01	0,1	1	0	10	3	100 1
Angström Measures	1 2 3 5 8 10	20 30 50 80100	1000	) 11	р <sup>4</sup>	10 <sup>5</sup>		10 <sup>6</sup> 1
Approximate molecular weight	100 200	1.000 10.000 20.000	100.0	000 500	.000			
	Salts in H <sub>2</sub> O		Carbon black	Textile colou	r pigments		Human	hair
		Pyrogene				Yeast cells		Sea sand
Relative size	Metal ions		Viruses		Bacteria	0		Fog
of different materials					Tobacco fume	s Coal d	ust	
That of the o				Asb	estos dust	Red		
	Sug	ar	Colloidal silicate			cells	Pollen	
	Atomic radius	Oil emuls	ions	Solid state	e bodies			
Filtrations	Reverse osmosis			Micro filtration				
methods		Nano/ul	tra filtration			CI	assic filtration	

Spectrum of filtration processes

Figure 8. Membrane Separation Chart



# **2.4 Reverse Osmosis**

Another stage of reverse osmosis membrane separation is employed to further improve the treated water quality of the digestate treatment; ensuring the water is suitable for process reuse.

As the digestate has already undergone dewatering and VSEP treatment, a high recovery can be expected from this membrane system. The concentrate would also be suitable to recycle to the front end of the treatment system or even the digester itself! Meaning there is no problematic waste stream requiring disposal or further treatment.

#### **2.4.1 Operating Principle**

Osmosis is defined as the process of molecules passing through a semi-permeable membrane from a less-concentrated solution into a more-concentrated solution. An example of osmosis from nature is the roots of plants drawing water from the soil.





#### Figure 9. Principle of Osmosis and Reverse Osmosis

Molecules are forced through a semi-permeable membrane by an external pressure source (i.e. a high pressure pump) to form a less concentrated solution on the permeate side of the membrane. Essentially, the membrane acts like a type of filter as it has extremely tiny pores that help remove microscopic contaminants from the water.

In the case of reverse osmosis systems, the semi-permeable membrane only lets water molecules through while other contaminants are collected and flushed away.

The highly concentrated solution remains on the concentrate side of the membrane. The concentration factor or water recovery is therefore limited by the osmotic pressure of the system. Meaning that as the solution becomes more concentrated the osmotic pressure of the system increases and hence the external pressure applied also has to increase until a point at which it is no longer feasible.





Figure 10. Reverse Osmosis Membrane Filtration

The RO membrane system is able to retain almost all the soluble impurities from the feed. The very low molecular weight organic compounds however are poorly rejected. The Reverse Osmosis membrane is a Spiral Wound, Polyamide Thin Film Composite element. As the name indicates, these membranes are made by forming a thin, dense, solute rejecting surface film on top of a porous substructure. RO can remove very small ions based on their ionic charge. This process will successfully remove high percentages of ions as small as Sodium Chloride and organic molecules. RO is very widely used as a purification method in water treatment and is also used in Desalination Plants to produce drinking water in some parts of the world.



Figure 11. Spiral Wound Reverse Osmosis Membrane

## 2.4.2 RO System Design

A RO system can comprise of multiple 'stages' in series, with each stage consisting of membrane housings and a recirculation pump (if high shear cross-flow filtration is required). The feed enters the first stage where pressure vessels are connected in parallel. The permeate and concentrate exits from the tail end of each pressure vessel. The permeate from each pressure vessel is combined in the stage-1 permeate header. The concentrate



from each pressure vessel is combined in the stage-1 concentrate header. This is illustrated in figure 13 in a Christmas tree configuration RO system.

#### **2.4.1 RO System Operation**

The pre-filtered water is pumped by a high pressure pump into the system, which comprises multiple stages of pressure vessels in series. The feed enters the pressure vessels, and permeate and concentrate exit from the tail end of the vessels. The permeate is collected from each stage, combined and sent to the treated water tank.

The concentrate flow is controlled using a manual control valve. The concentrate is removed from the Esmil treatment process and collected either in the concentrate tank, allowed to pass into the site open drain system or recycled to front end of the effluent treatment system.

The RO Feed tank collects the VSEP permeate which is pumped through a micron rated cartridge filter by the feed pump to provide the sufficient suction pressure for the high pressure pump. The cartridge filter protects the membranes in the very unlikely event of VSEP membrane pack failure resulting in the suspended solids contaminating the RO feed. The cartridge filter works by trapping particles either on the surface of the media or within the depth of the media. A 'Differential Pressure Indicating' switch is provided to detect the fouling of the cartridges. The high pressure pump is provided with a variable frequency drive to adjust the desired flow rate.

A part of combined concentrate is fed forward to the second stage and the rest is recirculated and mixed with the feed before it is fed to the stage-1 recirculation pump. The second and third stages operate in the same fashion as above. The concentrate from the third stage is very high in contaminants and bled off from the system via the concentrate control valve and sent to the feed tank. The permeate from each stage is combined in a common permeate header. The combined permeate is then finally sent to the treated water tank.

The RO recovery (i.e. percentage of feed recovered as permeate) is set by controlling the concentrate flow rate. The feed and treated water conductivity is monitored to gauge the performance of the RO system.

The RO feed is dosed with antiscalents to inhibit precipitation and deposition of the concentrated species on the membrane surface. The dosing rate is set manually. In addition, pH balancing of the feed using acid and caustic dosing can be incorporated if necessary. The treated water may be dosed with Caustic to maintain pH in the range 6-9 or as required. The pH balancing is done automatically using feedback from the respective pH controllers.

The RO membrane tends to foul over the period of time and this can be seen as an increased pressure drop across the membrane system. The RO plant is taken off line periodically for a 'Clean in Place' (CIP), to remove these accumulated foulants from the membrane. A more detailed explanation of CIP can be found in the following section (section 2.5)



# **2.5 Clean in Place (CIP)**

The VSEP and RO membranes are subject to fouling by the deposition and precipitation of components in the feed, and this can be seen as an increased feed pressure or the pressure drop across the membranes. As such, it is necessary to take the membrane plant off line periodically for a 'Clean In Place' (CIP), to remove these accumulated foulants from the membranes.

The cleaning may be either a flush cycle using hot water, or a more rigorous chemical clean using one or more different specialist cleaning chemicals. Generally low and high pH cleaners are required for the membrane cleaning. The cleaning chemical is prepared in CIP tank by the addition of hot water. The cleaning chemical is mixed thoroughly before CIP operation by re-circulating to CIP tank until correct pH and temperature is reached.

The CIP Cleaning Tank serves as a reservoir for the cleaning solutions or flushing water. During the recirculation stages the cleaning solutions and flushing water are fed to the membrane system being cleaned using CIP pump, then returned to the CIP Tank as part of a CIP recirculation loop.

The cleaning chemical leaving the CIP Pump are passed through the Cartridge Filter (On the membrane system) to remove any particulate matter that may have been picked up in the cleaning solutions/water as it is re-circulated around the Membrane Package being cleaned.

The Treated Water (Permeate) outlet from the membrane package is fitted with a manual valve to enable treated water to be diverted to the CIP tank should it be required.

The temperature of the water for CIP should be between 35°C and 45°C.



# **2.6 Operating Costs**

MDQ - VSEP - RO OPEX (15 m³/h)	Description	Value	Units	Cost /day	Cost /m³			
Power	Pumps, Instruments, Mixers, Control Panels, etc.	1491.2	kWh/day	£164.03	£0.46			
Water	Service Water, CIP, MDQ 10.3 m3/day £8.2				£0.02			
Chemicals	Polymer	144.0	kg/day as 100%	£475.2	£1.32			
	Antiscalent	1.0	kg/day as 100%	£3.93	£0.01			
	Acid	Piloting Required	kg/day as 100%	TBA	ТВА			
	Cleaning	41.3	kg/day as 100%	£165.19	£0.46			
	VSEP Membrane Pack	2	years	£345.21	£0.96			
	RO Membranes	2	years	£7.40	£0.02			
Replacements	Cartridge Filter (RO)	30	days	£2.40	£0.01			
	Cartridge Filter (CIP)	30	days	£0.60	£0.00			
	TOTALS							

 Table 1. Calculated OPEX cost for a 15 m3/h Digestate Treatment Plant.

The above table details the calculated OPEX costs of running a digestate treatment plant for a typical 15 m<sup>3</sup>/hr capacity food waste anaerobic digestion plant.

These calculations are based on a series of lab, pilot and chemical dosing trials. Also data and know-how of a full scale operation assisted in improving the economy and design of such a plant.

# **2.7 Capital Cost**

As an indication for a typical digestate treatment plant for a 15 m<sup>3</sup>/hr system as described previously, CAPEX in the range of:

- CAPEX £ 1.6 Million
  - MDQ £ 235,000
  - VSEP £ 1.25 Million
  - RO £ 115,000

Although a good indication, this is based on previous lab and pilot trials results for similar application and the resulting design calculations and accompanying assumptions.



# **3. Pilot Testing & Case Studies**

In this section we will explore the data and results obtained from employing the treatment schemes as above. This will include results of bench scale feasibility studies right the way through to full scale operation data.

# **3.1 Mixed Waste AD (Lab & Site Trial)**

Esmil was invited by the client to review their effluent treatment process, with a view to address:

- i. Suitable treatment solution to handle high COD and ammonia digestate.
- ii. With the hope in treating the highly contaminated water to a standard high enough for reuse within the process or as a boiler feed.

Attempts made by the client to treat the digestate effluent include:

- Centrifuge
- Belt Press
- Coagulation, flocculation

At present the anaerobic digestion (AD) plant is producing 2000 tonnes/week of digestate fluid (approx. 15 m<sup>3</sup>/hr) with the following initial analysis taken on the digestate before and after screening in tank A and tank B respectively.

Particle Distribution	% weight (per kg sample)		
>1.4 mm	0.64		
> 0.85 mm	0.33		
>0.25mm	0.83		
<0.25 mm	2.71		
DM (g/kg)	45.2		
рН	8		
NH4 - N (ppm)	7000		
CI (ppm)	6161		

#### Table 2. Tank 'A' pre-screened digestate

Parameter	Value
DM (g/kg)	45.2
COD (mg/l)	33,665
Dry Solids (%)	6.15
Volatile Solids (%)	63.45
рН	8.23
NH <sub>4</sub> - N (ppm)	8661
CI (ppm)	6071
Fos:Tac	0.35

Table 3. Tank 'B' post-screened digestate

Notes:

- 1. Digestate is screened through a 2.5 mm mesh to remove large particles
- 2. % of volatile solids refers to portion of total dry solids.

It has been made apparent that obtaining clear filtrate from the digestate has been notoriously difficult in the past hence; novel pre-treatment solutions shall be explored.



#### **3.1.1 Dewatering Site Pilot**

Dewatering of digestate will allow significant reduction of the waste volume as well as the recycling of filtrate (after post-treatment) and potential concentration of nutrients. Pilot tests were carried out to figure out its efficiency on this particular effluent and estimate operating expenses. Average designed sludge flowrate is 15 m3/h with 6 % DS content.

The multi disc screw press pilot unit (EKOTON-Tsurumi MDQ-201) was situated at site. Sludge was fed directly to internal sludge tank of a test unit. The flocculant solution preparation station was situated nearby, where different flocculant solutions were prepared and tried for the dewatering process. During studies, different dewatering parameters were tried and fitted to obtain the optimal solution.



Figure 12. MDQ Screw Press Pilot Unit

Samples of sludge, dewatered cake and filtrate were collected to analyze DS content in laboratory. Additionally filtrate was collected for exploration of further treatment methods such as membrane technologies to further concentrate nutrients (mostly ammonia) and to purify water to make it suitable for reuse within the plant.

As a result of the test study, information about sludge treatment ability with different flocculants and dewatering efficiency of multi disc screw press MDQ-201 was obtained. The press capacity was about 20 kg DS/h and dewatering cake contained up to **41 % DS** in it.





Figure 13. Dewatered Digestate Cake

Additionally, filtrate and sludge samples were tried for coagulant treatment in EKOTON laboratory.

- The cake up to 41% DS content was produced as against the target 20%.
- The filtrate was measured DS content 1 1.5% as against expected < 0.2% which mainly due to inadequate flocculation achieved using available stock of the chemicals.
- Increasing screw rotation speed can rise equipment productivity but with reduction of cake dryness from max 41 % to 29 %.
- Sludge volume can be reduced by 6-10 times.
- The choice of polyelectrolyte can influence the filtrate clarity, cake dryness and equipment capacity.
- **Chemical trials** to be performed prior to dewatering. Ensuring optimal dosage rates and effectiveness of flocculation.

#### **3.1.2 Laboratory Membrane Study**

Filtrate obtained from the preliminary dewatering trials was brought to Esmil to conduct a series of membrane bench scale trials carried out at the Esmil Lab in High Wycombe.

For this trial, the filtrate was fed through a 100 micron bag filter to remove any large size suspended solids which could potentially damage the pump and membranes. This was followed by a range of sulphuric acid dosages of the feed (0, 3.15, 6.25 ml/L) to determine the impact of downstream membrane performance, ammonia removal.

The pre-treated filtrate underwent VSEP Nano-filtration (NF); the permeate from VSEP was further treated using a reverse osmosis (RO) spiral wound membrane. This allowed for performance evaluation of the membrane technologies. In order to assess this suitability the following parameters were investigated and tested.



- Organic Matter (COD) separation.
- Membrane flux
- System recovery
- Membrane fouling tendency
- pH adjustment
- Ammonia removal

The feed to the membrane system is separated into a permeate stream (passing through the membrane) and a concentrate stream (retained by the membrane). The contaminants are expected to be rejected by the membrane and retained in the concentrate stream.



Figure 14. Membrane Pilot Trials (Batch Mode)

For both systems, permeate is continuously removed and concentrate recycled back to the feed tank as illustrated in figure 1. By this method, the concentration of rejected species in the feed gradually increases with time. This 'multiple stage' operating procedure simulates the progressive stages of a 'single pass', full scale plant.

Each run is carried out in batch mode for both NF VSEP and RO. The feed is pumped from a conical bottomed tank, by a high pressure, positive displacement pump, to the membrane system. The driving pressure on the feed side of the membrane forces the passage of water, gases and other small contaminants within the process feed into a permeate stream and a the majority of contaminants and solids remain within the concentrate stream. A needle valve on the concentrate outlet is adjusted to provide sufficient system pressure for permeation.

Permeate and concentrate flow rates are measured and recorded periodically. After each test run, volumes of final permeate and concentrate collected are measured. The samples are collected for fresh feed, final permeate and final concentrate.



#### Table 4. Filtrate (Membrane Feed) Characteristics.

Source	AD digestate slurry (dewatered filtrate)					
Appearance	Dark brown; some suspended solids still visible					
Temperature	Ambient					
Pre-treatment	Screened through 100 micron bag filter					
Test No.	1 2 3					
Sulphuric Acid Dose (ml acid/ L sample)	6.25 0 3.15					

#### 3.1.3 Results

Varying pH adjustment was carried out for all VSEP trials, however as our interest was only to see the effect of pH adjustment on ammonia removal, RO trials was only conducted on one VSEP permeate (high acid dose permeate).

For each sample the system was first started up using a NF membrane in VSEP machine in the recirculation mode and set to optimum pressure. The system was run for 30 minutes to verify flux stability. When equilibrium was reached, the permeate line was diverted to a separate vessel, and the batch mode separation test began. The sample was pumped through the Nanofiltration membrane at 24 barg pressure, collecting permeate and recycling the concentrate back through the membrane.

The permeate flow rate was measured at periodic time intervals to determine the flow rate/ flux produced by the system at various levels of recovery (% vol. of permeate/start up feed vol.). The pH and temperature are also recorded at these time intervals on both permeate and the concentrate. The test is run until sufficient permeate recovery has been achieved or until permeate flow is to low or stopped altogether.

#### 3.1.3.1 Run Summary

The following table presents a summary of the results of the membrane trials including the high dose NF VSEP trial (Run I Pass I) and the subsequent RO trial (Run I Pass II).

Run No.	Pass No	System Recovery (%)
Ι	I	73.3
I	II	80



#### 3.1.3.2 Cleaning Test

At the end of the separation test, a cleaning study was conducted to determine if the flux was dropped and recoverable. The system was underwent a clean water flush (CWF) first with cold water and then hot water if necessary and the flux was recorded. If the flux not recovered by just hot water, chemical cleaning would be carried out. However, the flux regain after was found satisfactory for all of the trials.

Membranes	Cold Flush	Hot Flush	Chemical Cleaning	New Membrane CWF (Imh)	Post Run CWF (Imh)	Pre-run MgSO₄/ NaCl Rejection-%	Post-run MgSO₄/ NaCl Rejection-%
NF	Yes	Yes	No	94.2	76	-	-
RO	Yes	No	No	51.7	49.4	99.5	99.5

No change in the salt rejection of the RO membrane was recorded before and after the trail and suggesting that the membrane retained its structural integrity. This suggests that the feed does not disintegrate or damage the membrane. The table above summarises the findings of the CWF tests for each membrane.

#### **3.1.3.3 Sample Images**

The following images show the different streams being fed or exiting both the NF and RO membranes from the trails described above. From left to right the images show:

**Sample-1** VSEP feed with 100% acid dosing. This is the filtrate from the site trial and dosed with sulphuric acid to lower pH to 6 from 9.3. In 16 l of sample, 100 ml of 98% sulphuric acid was dosed.

**Sample-2** VSEP Permeate with 100% acid dosing. This is the permeate from VSEP system using Nano-filtration membrane. The feed was as per sample-1

**Sample-3** VSEP Concentrate with 100% acid dosing. This is the concentrate from VSEP system using Nano-filtration membrane. The feed was as per sample-1

**Sample-4** VSEP Permeate with 0% acid dosing. For this run, a fresh sample was used and no acid dosing was carried out.

**Sample-5** VSEP Permeate with 50% acid dosing. For this run, a fresh sample was used with 50% acid dosing of original batch was carried out.

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**Sample-6** RO Permeate. Here RO was fed with VSEP permeate from the feed which was acid dosed to pH below 6

**Sample-7** RO Concentrate. From same feed as sample no. 6

There is a noticeable difference between the differing level of pH adjustment on the NF VSEP permeates. Particularly the high acid dose permeate (6 pH) had very little colour in comparison to the other levels of pH adjustment. As previously mentioned, precipitation of solid material occurred during acid dosing which may be removed during pre-treatment if dosed earlier on in the process. Also a reduction of pH favours the formation of ammonium sulphate, which unlike ammonia (gas) is easily rejected by the membrane hence, the improvement in permeate clarity is visible with an increase in acid dosing.







#### **3.1.3.4 Analytical Results**

The following table details Run-I of the trial which includes NF VSEP (Pass-I) and RO membrane separation (Pass-II) on the feed with 100% acid dose.

Deremetere	Unito	VSEP (Pass-I)			RO (Pass-II)		
Parameters	Units	Feed	Conc.	Perm.	Feed	Conc.	Perm.
Total COD	mg/l	13080	12800	7990	7990	31960	600
рН	-	5.68	5.6	5.7	5.7	6.1	5.9
Ammonia	mg/l	5150	11300	1760	1760	7425	100
Conductivity	us/cm	40400	-	15400	15400	73100	1389

As can be seen completing both passes of the membrane treatment run produces very high quality treated water with a COD and Conductivity reduction of >95% and <u>ammonia removal</u> of more than 98%.

The table below illustrates the effect that acid dosing has on the ammonia concentration after the feed has passed through the NF VSEP system.

Acid Dose	100%	50 %	0%
Feed Ammonia	5150	5150	5150
Feed pH	6	7.8	9.6
Ex-VSEP Ammonia (mg/l)	1300	2250	2750

This clearly demonstrates the ability of acid dosing in reducing the ammonia content of the treated water stream. This occurs due to the fact that as the pH tends to favour the RHS of the ammonia  $NH_3$  (g)  $\leftrightarrow$  ammonium  $NH_4^+$  balance. The ammonium ion forms a salt in the presence of sulphur which is easily rejected by NF and RO, hence the greater reduction of ammonia in the treated water stream is observed with increasing levels of acid dosing.

#### **3.1.3.5 Conclusions and Recommendations**

- 1. The two pass membrane system, NF VSEP followed by RO membrane technology can treat filtrate (dewatered AD digestate) to produce high quality water suitable for process re-use.
- 2. During pH adjustment sulphuric acid was dosed in various concentrations. During this process solid material clearly precipitated out of the feed therefore if pH adjustment is desirable it is suggested to dose earlier on in the pre-treatment process, reducing the load on the membranes in turn improving their performance.



- 3. Clear improvement in NF VSEP permeate was visible using a sample that has been adjusted to reduce the pH from 9.6 to 6. At reduced pH the ammonia in the feed converts to ammonium sulphate which is easily rejected by the membranes. At high pH ammonia remains in its soluble gaseous state which membranes find difficult to reject.
- 4. Results should be viewed as a baseline on which improvements can be made, particularly in terms of the pre-treatment which is expected to produce a much clearer filtrate. Nonetheless, the membranes produced extremely clear water.
- 5. Analysis of the water quality carried out showed that acid dosing significantly impacted ammonia removal. Lowering the pH to 6 more than halved the concentration of ammonia in comparison to no pH adjustment.
- 6. The flux profile was as expected for both membranes. No chemical cleaning was required which indicate membranes were not irreversibly fouled.
- 7. The membrane rejection remained intact suggesting that the feed material didn't deform or disintegrate the membrane and module structure.
- 8. Significant improvements on membrane performance are likely once a suitable pretreatment chemical solution is found.



# **4. Distillery AD (Pilot Trial)**

Esmil have recently completed a long term pilot study for an AD operator in the UK. This took place on site and extended almost 2 months. In this case the client already has a centrifuge in place for dewatering. Hence our trial only considered the VSEP (RO) followed by a spiral RO system.

Client was looking for a system utilising membrane technology to treat the digestate effluent from their anaerobic digestion (AD) plant. Digestate is currently dewatered using a decanter on site. Client interested in treating the water of the remaining liquid fraction to a quality high enough for process reuse and reducing the waste volume produced on site that is currently transported off site at high costs.

To qualify for process reuse the water requires total solids removal and a significant reduction of the ammonia content. To achieve these requirements, Esmil have proposed a scheme which involves a first pass of VSEP containing RO membranes, to be followed by a second pass of standard spiral wound RO membranes. This scheme is tried and tested in the area of anaerobic digestate treatment leading to significant cost and fresh water use reduction.

A long term pilot study has been carried out at site between 16/04/2019 - 12/06/2019. The following parameters were investigated and tested.

- 1. Membrane flux and system recovery
- 2. Membrane fouling tendency
- **3.** Suitable cleaning regime
- 4. Solids and ammonia removal
- 5. Organic matter (COD) separation

## **4.1 Nomenclature**

Abbreviation	Description	Units
VSEP	Vibratory Shear Enhanced Process	-
RO	Reverse Osmosis	-
CIP	Clean in Place	-
AD	Anaerobic Digestion	-
COD	Chemical Oxygen Demand	mg/l
TSS	Total Suspended Solids	mg/l
DS	Dry solids	w/w%
NH <sub>4</sub> -N	Ammonium as Nitrogen	mg/l
Flux	Permeate flow rate per unit membrane area	l/h/m² (lmh)
CWF	Clean Water Flux	l/h/m² (lmh)
Normalised Flux	Flux normalized to 25°C	lmh @ 25°C

## **4.2 Process Descriptions**

As above for previous trail (section 3.1); detailed description of the process in section 2 for VSEP and RO units.



#### 4.2.1 Pilot Unit Set-up



VSEP Feed Set Up



VSEP Unit & CIP Tank



Spiral RO Feed Set Up



#### **4.2.2 Feed Characteristics**

The following table qualitatively describes the feed as received along with description of the pre-treatment performed prior to membrane testing

Source	AD digestate slurry (post-centrifuge)
Appearance	Dark brown; some suspended solids still visible
Temperature	Variable (13 – 35°C)
Pre-treatment	Screened through 100 micron bag filter 5ppm Antiscalent Dosing. Some runs were carried out without pre-treatment.

## 4.3 Results

#### 4.3.1 VSEP

Extensive testing has been carried out using the VSEP pilot unit containing an RO membrane pack. As the VSEP has to handle the majority of solids, ammonia, COD etc. hence the success of the VSEP is critical for operation of the entire process.

#### 4.3.1.1 Run Summary

Throughout the trail period, a total of 15 runs were carried out for the VSEP. The following table provides a summary of these runs.

Data	Dunne	Feed	Deservorre	Norm. F	lux @ 25 ⁰C	
Date	Run.no	Source	Recovery	Range	Avg. Flux	
dd/mm/yyyy	-	Site	%	lmh	lmh	
17/04/2019	1	A	64	NDA	NDA	
18/04/2019	2	A	68	NDA	NDA	
23/04/2019	3	Α	63	NDA	NDA	
24/04/2019	4	A	65	NDA	NDA	
25/04/2019	5	A	68	NDA	NDA	
29/04/2019	6	A	70	NDA	NDA	
30/04/2019	7	A	71	NDA	NDA	
07/05/2019	8	В	50	NDA	NDA	
09/05/2019	9	В	65	NDA	NDA	
13/05/2019	10	В	63	NDA	NDA	
14/05/2019	11	В	65	NDA	NDA	
15/05/2019	12	В	60	NDA	NDA	
16/05/2019	13	В	61	NDA	NDA	
23/05/2019	14	В	66	NDA	NDA	
30/05/2019	15	В	66	NDA	NDA	

## 4.3.1.2 VSEP Stream Sample Analysis

												VSEP – RO								
Data	Run	Run			F	eed						Permeate					C	Concen	trate	
Date	No.	(mins)	рН	Temp (°C)	NH4-N (mg/l)	TSS (mg/l)	%DS	COD (mg/l)	рН	NH4-N (mg/l)	NH4-N Reduction (%)	Permeate Recovery (%)	NH4-N Recovery (%)	TSS (mg/l)	COD (mg/l)	рН	NH4-N (mg/l)	%DS	TSS (mg/l)	COD (mg/l)
16/04					3,310	3,660				445	-86.56%			12	12		5,340		3,530	3,530
17/04	1		8.00		3,250	3,340		2,500	8.80	395	-87.85%	64		15	55	8.60	6,420		13,760	5,475
18/04	2		7.90		3,490	4,200		2,750	8.60	536	-84.64%	68		18	65	8.50	7,200		25,900	6,890
19/04					3,368	3,987		2,845		567	-83.17%			21	80		6,851		23,784	6,790
23/04	3		8.00		3,200	21,940		2,485	8.80	800	-75.00%	63		30	235	8.70	7,500		42,760	7,950
24/04	4		7.90		3,540	14,800		2,410	8.60	839	-76.30%	65		41	215	8.60	8,000		34,940	8,370
25/04	5		8.00		3,300	17,160		2,435	8.60	866	-73.76%	70%	51.6%	20	250	8.60	7,500		41,940	8,935
29/04	6		7.82		3,025	5,620	1.4%	3,895	8.02	835	-72.40%	70%	50.7%	15	315	7.84	5,450	4.0%	31,340	5,830
30/04	7		7.86		2,745	11,640	1.2%	2,741	7.89	975	-64.48%	70%	45.1%	17	251	7.89	5,285	4.1%	26,240	5,240
07/05	8		8.1	13.4	6,510	28,520	3.6%		8.61	1,140	-82.49%	50.0%	41.2%	48	265	8.23		4.6%	39,300	
09/05	9		7.97	34.5	3,300	16,840	2.4%	3,515	8.31	1,100	-66.67%	67.8%	45.2%	31	315	7.92	7,770	6.5%	-	10,330
09/05	9		8.05	32.1	3,180	16,440	2.4%	3,760	8.34	960	-69.81%	64.3%	44.9%	44.9% 18 345		7.95	7,470	6.5%		9,750
09/05	9		8.08	30.8	3,255		2.3%	4,070	8.39	880	-72.96%	60.2%	43.9%	15	335	7.96	6,735	6.2%		9,610
13/05	10		8.22	13.5	3,182	9,940	1.3%	3,480	8.48	790	-75.17%	61%	45.6%	10	10 235		7,413	4.40%		8,995
13/05	10		8.17	17.7	3,110	11,840	1.5%	3,965	8.54	800	-74.28%	64.74%	48.1%	16	235	8.03	7,190	4.80%	33,380	9,110
13/05	10		8.2	19.8	3,095		1.5%	3,510	8.54	780	-74.80%	63.80%	47.7%	25	255	7.99		4.90%		9,405
13/05	10		8.2	21.8	3,120		1.5%	3,875	8.54	870	-72.12%	63.34%	45.7%	18	315	7.98		4.90%		10,040
14/05	11		8.28	18.2	3,070	11,690	1.4%		8.45	750	-75.57%	67.94%	51.3%	24		8.04	8,270	4.4%	32,620	
14/05	11		8.21	23.6	3,025	16,120	1.7%		8.51	800	-73.55%	65.75%	48.4%	26		7.98	7,710	5.1%		
15/05	12		8.07	25.8	4,230	24,420	2.80%	4,810	8.37	1,400	-66.90%	59.40%	39.7%	36	505	7.91		7.5%	60,060	
16/05	13	22	7.92	31.7	3,200	17,100	2.20%	3705	8.24	1,050	-67.19%	68.56%	46.1%	26	330	7.8		6.60%	46,440	
16/05	13	150	8.05	31.4	3,210	16,980	2%	3695	8.44	1,100	-65.73%	65.47%	43.0%	76	300	7.84		6.40%	46,800	
16/05	13	270	7.98	30.5	3,220		2.10%	3750	8.32	970	-69.88%	61.47%	43.0%	88	275	7.86		5.50%		
16/05	13	690	8.18	29.3	2,840	14,880	1.80%	3,950	8.43	850	-70.07%	55.38%	38.8%	14	315	8.03	5,805	5.00%	39,590	9,347
16/05	13	990	8.07	29.9	2,975	16,680	2.20%		8.35	870	-70.76%	60.95%	43.1%	12		7.94		6.40%		
16/05	13	1170	8.05	29.7	3,240	17,620	1.90%	3,885	8.45	970	-70.06%	63.84%	44.7%	5	365	7.85	7785	5.90%	49,660	10,180
17/05	13	1440	8.16	26.3	3,030	17,420	2.20%	3,985	8.54	850	-71.95%	60.70%	43.7%	11	335	7.95	6,850	5.40%	47,140	6,850
17/05	13	1620	8.25	25.5	3,140	17,240	2.10%		8.66	740	-76.43%	60.05%	45.9%	10		7.99	6,970	5.40%	46,580	
23/05	14		7.95	38.6	2,890	15,100	2%	3,480	8.31	950	-67.13%	63.47%	42.6%	15	365	7.83	6,970	5.80%	50,520	13,452
30/05	15		8.22	35.9	3,012	14,674	1.8%	3,741	8.49	970	-67.80%	68.79%	46.64%	21	398	7.96	6,720	6.70%	51,230	10,475







Figure 15. VSEP Feed / Conc. / Permeate Samples



#### **4.3.2 VSEP Cleaning Tests**

Dates	Run No	Cold Flush	Hot Flush	Chemical Cleaning	New Membrane CWF (Imh)	Post Cleaning CWF (Imh)	New Membrane Rejection-%	Post Cleaning Rejection-%
17 <sup>th</sup> April	1	-	-	Yes	118.4	76	98.16	-
18 <sup>th</sup> April	2	-	-	Yes	118.4	75	98.16	-
23 <sup>rd</sup> April	3	-	-	Yes	118.4	71	98.16	-
24 <sup>th</sup> April	4	-	-	Yes	118.4	65	98.16	-
25 <sup>th</sup> April	5	-	-	Yes	118.4	<b>118.4</b> 60		-
29 <sup>th</sup> April	6	-	-	Yes	118.4	61	98.16	-
30 <sup>th</sup> April	7	-	-	Yes	118.4	61	98.16	-
7 <sup>th</sup> May	8	-	-	Yes	118.4	60	98.16	-
9 <sup>th</sup> May	9	-	-	Yes	118.4	53	98.16	-
13 <sup>th</sup> May	10	-	-	Yes	118.4	56	98.16	-
14 <sup>th</sup> May	11	-	-	Yes	118.4	53	98.16	-
15 <sup>th</sup> May	12	Yes	-	-	118.4	43	98.16	-
16 <sup>th</sup> / 22 <sup>nd</sup> May	13 <sup>1</sup>	-	-	Yes	118.4	51/80	98.16	98.01 / 92.36
23 <sup>rd</sup> May	14	Yes	-	-	118.4	80	98.16	92.36
30 <sup>th</sup> May / 11 <sup>th</sup> June	15 <sup>2</sup>	-	-	Yes	118.4	68 / 85	98.16	93.64

- Run carried out on 16<sup>th</sup> May followed by chemical cleaning and extensive cleaning was carried out on 22<sup>nd</sup> May by Esmil.
- 2) Run carried out on 30<sup>th</sup> May followed by chemical cleaning and extensive cleaning carried out on 11<sup>th</sup> June by Esmil.

The cleaning study shows, at the end of the pilot study, the cleaners were able to regain the flux within 20% range and the rejection characteristics within 5% of the new membrane which is a good sign.



#### 4.3.3 Spiral RO

The VSEP system is the critical unit ensuring successful process operation. The VSEP effectively acts as a pre-treatment for the spiral RO system, and the process reliability of Spiral RO membrane separation is highly dependent of the correct conditioning of the feed.

Nonetheless, the permeate arising from the VSEP system still contains a relatively high number of contaminants (COD, ammonia, salts) when compared to 'fresh water' applications. Hence 4 trails feeding the spiral RO membranes with the VSEP permeate were completed to confirm the successful pre-treatment of the feed and expected performance of the Spiral RO system.

#### 4.3.3.1 Run 1-2 Operating Parameters

The following table summarises the operating parameters:

Date	Feed Source	Run.no	Recovery
dd/mm/yy		-	%
18/04/2019	VSEP Permeate	1	94
23/04/2019	VSEP Permeate	2	94

The feed to the spiral RO is pre-treated using VSEP RO which poses very little challenge to the spiral RO membranes. The recovery achieved was as expected at greater than 90% for first two runs. No data is available for run-3 and 4 however the expected recoveries for these runs are also 90% or greater. This is based on the previous two runs and the consistent permeate quality achieved by the VSEP.





Figure 16. Spiral RO Feed / Conc. / Permeate Samples

#### 4.3.3.2 Run 1-4 Analytical Results Summary

The table below shows the analytical results of Run-I samples taken for the feed, combined permeate and combined concentrate. The samples were analysed at the laboratory on site for the parameters listed.

#### Run-1

Feed (Run-2 VS	EP Perm.)		Concentrate			
NH4-N <sup>*</sup> (mg/l)	TSS (mg/l)	NH4-N (mg/l)	NH4-N reduction (%)	TSS (mg/l)	NH4-N (mg/l)	TSS (mg/l)
536	18	110	-79.48%	7	4,140	36



\* Ammonium as Nitrogen (NH<sub>4</sub>-N) is tested for at the onsite laboratory. As this is not online measurement, the sample is likely to come to equilibrium at room temperature. Therefore the assumption will be made that the sample is tested at standard conditions (25°C and 1 bar).



Figure 17. pH effect on Ammonia - Ammonium concentrations at Standard Conditions

The dewatered digestate for Pass-I (Run-2 VSEP feed) originally contained 3490 mg/l NH<sub>4</sub>-N which is reduced to 536 mg/l in the VSEP permeate (Spiral RO feed). This shows an 85% decrease in ammonium nitrogen. Pass-I Run-I shows a 80% reduction of NH<sub>4</sub>-N from 536 mg/l to 110 mg/l. This gives an overall reduction of ammonium as nitrogen for both passes of **96.8%** (VSEP & Spiral RO).

The remaining concentrate has an ammonium as nitrogen concentration of 4,410 mg/l and TSS of 36 mg/l. The NH<sub>4</sub>-N concentration is in fact lower than the raw effluent and TSS concentration is considerably less. Therefore, this stream does not require disposal and can be recycled to the beginning of the process. This has many advantages including a slight dilution of the solids concentration of the raw effluent, improving overall system recovery and reduction of the total waste volume.

Reduction of ammonia may be improved on further still by reducing the pH of the system. The most effective way to approach this is by dosing the spiral RO feed with acid. The reason that VSEP feed would not be recommended for dosing is that a lot of alkalinity is likely in the VSEP feed increasing the buffering capacity. As the alkalinity is removed or reduced by the VSEP this reduces the buffering capacity of the permeate and hence also reduces the concentration and volume of acid needed to reduce the pH to the desired level for effective ammonia removal.

As can be seen in figure 5 reducing the pH to 7 or below will shift the  $NH_3 \leftrightarrow NH_4^+$  equilibrium to the right; increasing the ammonium concentration. Ammonium dosed with acid (sulphuric) would produce an ammonium salt (ammonium sulphate), which is easily rejected



by a reverse osmosis membrane, whereas ammonia as a gas is poorly rejected. It should be noted that the equilibrium is also temperature dependant; an increase in temperature would be seen as a reduction in the pKa value (figure 5) and vice versa.

#### Run-2

The table below shows the analytical results of Run-2 samples taken for the feed, combined permeate and combined concentrate. The samples were analysed at the laboratory on site for the parameters listed.

Feed (Run-3 VS	EP Perm.)		Concentrate			
NH4-N (mg/l)	TSS (mg/l)	NH4-N (mg/l)	NH4-N reduction (%)	TSS (mg/l)	NH4-N (mg/l)	TSS (mg/l)
800	30	177	-77.88%	5	4,860	68

The NH<sub>4</sub>-N concentration of the feed for Run-II is considerably higher than that of Run-I, however this saw little effect on the system recovery (93.75% or greater) or NH<sub>4</sub>-N reduction capacity (78%) of the spiral RO system. The raw feed to the VSEP had a NH<sub>4</sub>-N concentration of 3,200 mg/I, giving an overall reduction of ammonium as nitrogen for both passes of <u>94.5%</u> (VSEP & Spiral RO).

The increased concentration of  $NH_4$ -N in the spiral RO feed had no significant effect on the  $NH_4$ -N reduction capability of this pass, attesting to the fact that the VSEP acts as a pretreatment system for the spiral RO. Therefore, as long as there are no major disruptions upstream, the spiral RO system will perform as expected – with high  $NH_4$ -N reduction and other contaminant removal to produce a high quality water stream and a low concentrate volume which can be recycled to the beginning of the process.

#### Run- 3 & 4

A further 2 trials were conducted during the later stages of the trial using the Spiral RO membrane. It appears no run data was collected for these trials however; the analysis of each stream has been completed and is presented in the table below.

Run		Feed			Perme	ate	Concentrate				
No.	NH4-N (mg/l)	TSS (mg/l)	COD (mg/l)	NH4-N (mg/l)	NH4-N reduction (%)	TSS (mg/l)	COD (mg/l)	NH4-N (mg/l)	TSS (mg/l)	COD (mg/l)	
3	890	21	250	185	-79.21%	2	20	6810	75	2440	
4	970	25	315	189	-80.52%	2	21	6792	51	2598	

Similar results to the previous runs (I & II) are clearly visible. A high NH<sub>4</sub>-N, TSS and COD reduction in the permeate is achieved producing a high quality water stream that is suitable for reuse.



In terms of the concentrate, again the quality is fairly similar to that of the raw feed (post centrifuge), aside from an increase in the  $NH_4$ -N. However, as the concentrate stream is of low volume relative to the feed flow this will have very little if any negative impact on the performance of the system. System recovery and reduced waste volume will still be achieved using this strategy of Spiral RO concentrate recycle.

#### 4.3.4 Spiral RO Cleaning Tests

The spiral RO is not expected to require cleaning very often as it is treating RO permeate (from the VSEP). The CWF remained almost unchanged after first run even after operating at high recovery value – 94%. It was not deemed necessary to check the CWF after every run as due to the fact that the feed was RO permeate.

For full scale system the provision shall be made for hot/cold water flush and chemical CIP of the spiral RO membrane system whenever necessary or as a part of the routine maintenance cleaning.

Dates	Run No	Cold Flush	Hot Flush	Chemical Cleaning	New Membrane CWF – (Imh)	Post Cleaning CWF- (Imh)	New Membrane Rejection-%	Post Cleaning Rejection- %
18 <sup>th</sup> April	1	Yes	-	=	38	36	99.37	-
-	2	-	-	-	38		99.37	
-	3	-	-	-	38	-	99.37	-
-	4	-	-	-	38	-	99.37	-



Figure 18. Sample of each stream from VSEP and Spiral RO





#### 4.3.5 Conclusions & Recommendations

- The double pass membrane system of VSEP RO and Spiral RO is capable of treating the AD digestate to produce a high quality treated water stream suitable for process reuse as illustrated in the image below.
- An overall system hydraulic recovery of between 60-65% was achieved, considering both VSEP and Spiral Membrane systems.
- The VSEP unit is critical in ensuring the successful operation of the process and has shown its capability in handling feed variation, different feed sources as well as upstream disturbances.
- Concentrate of Spiral RO can be recycled to front end of the system (VSEP feed) to dilute the feed, reduce the waste volume and improve overall system recovery.
- To further improve Ammoniacal nitrogen reduction, it is envisaged that acid dosing upfront of the Spiral RO system will convert ammonia to an ammonium salt which is easily rejected by the membrane.
- Antiscalent dosing is highly recommended for long term trouble free operation of VSEP system.
- The flux profile for both units was as expected. The chemical cleaning was required for the VSEP unit as this handles the majority of solids and other contaminants. The Spiral RO system chemical cleaning frequency is expected to be very low, perhaps once in a quarter.
- Esmil suggests arranging a meeting to discuss the results of the trial and how this forms a solid basis on which to construct a design for a full scale system.
- Esmil can now prepare a firm offer based on the outcome of this trial as the results have allowed the design to be fine-tuned, giving a detailed estimate of the operational costs and plant performance.



# **5. Bioethanol Plant Waste AD (Full Scale)**

The biomass recycling system consists of the following parts:

- 1. Mechanical separation system using centrifuge (liquid phase and compost);
- 2. Liquid Phase Filtration on VSEP (Partially Purified Water and Biomass Concentrate Remains);
- 3. Final filtration of partially purified water in two-stage reverse osmosis (RO) (clean water, suitable for bioethanol production and saline concentrate. Salt concentrate in intermediate container mixed with liquid phase from centrifuges and returned for recycling to VSEP plants).

# **5.1 Plant Description**

- 1. Particulate matter undergoes mechanical separation. The resulting liquid phase is directed to the VSEP feed tanks. The solid fraction is transported to the tractor trailers and transported to the storage area.
- 2. Liquid phase filtration in the VSEP modules is performed by batch method. When starting a batch, one of the feed tanks is filled and filtering is started by activating VSEP devices, which takes place until the required concentration is reached. Filtration separates the partially purified water (permeate) collected in the partially intermediate buffer tank. The other part of the flow (biomass concentrate) is returned to the feed tank until it is concentrated to the required concentration (30-50% by volume). The concentrated biomass from the feed tank is directed to the biomass concentrate. The biomass concentrate is then transported to the 10,000m3 reservoir (lagoon) as slurry. This is applied to the soil during the fertilization period.
- 3. Partially purified water (permeate) is directed to reverse osmosis units for final cleaning. During operation the water passes through the membranes and produces clean water that is directed to its storage tank. The resulting water is suitable for use in bioethanol production.

The biggest problem is poor water quality after centrifuges, very high amount of suspended particles and high salinity.

Otherwise the plant works well and as expected for process water recovery and nutrient removal.

The VSEP, RO system has been operational since 2013 and continues to provide an economic benefit for the process.





Figure 19. Full Scale Digestate Treatment Plant PFD

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													480mg/L	TSS		
500 m3/day													0.72%	TDS		
from digesters	6												32.6	mS/cm		
								Ro1 concer	ntrate retu	rn to VSEP	P filtration	<u> </u>	8.56	рН		
3.50%	DM															
30.000 mg/L	TSS							250	) m3	_						
1.10%	TDS							200 mg/L	TSS							
28-29 r	mS/cm						Permiate	0.15%	TDS					]		
8.0	рН		14000 mg/L TSS					9.0	рН		KO spira	le-1				
			1.80% TDS	9500	TSS			12000	μS/cm		Recover	155%				
			8.0 pH	0.8	TDS											
			28-29 mS/cm	8.6	рН			4		_						
			2.00% DM	25-26	mS/cm	ΝΑΝΟ										
				1.7	DM											
						FILINAI										
Bufor	tank		Feed		L											
Dulei			/ SWECO	Bufer	<b>★</b>	Becover	~ 50%		50mg/L	TSS						
T-17	0	Centrifuges	screen	tank		Recovery	~ 50/8		0.06%	TDS		*			First stage	
1 1/	•		75-150 μ						7000/9000	) μS/cm		Permeat	е		Second stage	;
									9.5	рН					Third stage	
									RO2 cor	ncentrate		0.06%	TDS			
									return t	o 2 stage		3000/500	0 μS/cm			
												9.5	рН			
						2	250 m3									
			Cake			•										
			23% DM			concen	trate			_					Clean water to bioeth:	anol
			about 6% from biomass		2	20220mg/L	TSS				RO spiral	-2				
						3.05%	DM				Recovery	70%			Permeate	
						1.00%	TDS								0.02% TDS	
						36.00	mS/cm								1000/2000 μS/cm	
						8.2	рН								10 pH	

Figure 20. Digestate Membrane Filtration Mass Balance

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# **6.Esmil Approach to Digestate Treatment**

- Chemical Trials (if applicable) For Optimum
   Polymer Performance and Economy
- The Laboratory Scale Test Evaluation.
- The Pilot Plant Site Trials for New or Challenging Applications
- The Design Team and Engineering Team Review
- The Project Team
- The Installation and Commissioning Team