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*Article*

# Ferric oxide containing waterworks sludge reduces emissions of hydrogen sulfide in biogas plants and the needs for virgin chemicals

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**Abstract:** Ferric oxide containing waterworks sludge can be used to reduce formation of hydrogen sulfide during anaerobic digestion. The ferric compound is reduced biochemically in the digester and forms insoluble pyrite in digester sludge. Often virgin ferric chloride is used to solve the hydrogen sulfide problem. Since 2013, Sydvatten AB has supplied a growing number of digestion plants in Sweden with ferric containing dewatered waterworks sludge from the drinking water treatment plant Ringsjöverket to limit the formation of hydrogen sulfide. At the waterworks, ferric chloride is added to enhance coagulation of organic matter from the source water. The sludge formed in this process is dewatered and landfilled, but also recycled in the biogas production in order to reduce the hydrogen sulfide concentration. In this study, the use of sludge, for hydrogen sulfide removal in digesters, was technically and economically evaluated from cases studies from 13 full-scale digesters in Sweden. Compared with use of fresh ferric chloride, the operational costs are reduced up to 50 % by using sludge. The quality of the sludge is high and its content on metals is low or very low, especially when compared with requirements from different certification standards on biosolid reuse applied in Sweden. An addition of waterworks sludge containing iron to a digester for removal of dissolved hydrogen sulfide is a technically and economic good alternative when producing biogas. It is also one step closer to a circular economy, when replacing use of virgin chemicals with the by-product waterworks sludge which saves energy, material and reduces the carbon foot-print of the waterworks.

**Keywords:** biogas digestion; hydrogen sulfide; ferric oxide; waterworks sludge)

## 1. Introduction

Biogas production is getting increased attention as a source for replacing fossil based fuels with renewable fuels in society. The biogas is typically produced in digestion plants where different substrates rich in organics are digested by methanogenic bacteria. Most substrates contain also sulfur which in anaerobic environment can be microbiologically reduced to hydrogen sulfide, which negatively affects the metabolic activity of the methanogens and eventually poison the digester. Hydrogen sulfide is also a technical issue in the plant and when the biogas is used. If not removed from the biogas, hydrogen sulfide damages pipes and generators in anaerobic digestion system and equipment downstream. It is also health hazard, being toxic to humans. Improving the

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quality and quantity of biogas usually requires pre-treatment to maximize methane yields and/or post-treatment to remove hydrogen sulfide. This requires considerable energy consumption and higher cost; hence there are needs for better and more efficient measures to control hydrogen sulfide production.

One way to remove hydrogen sulfide as a gas is to add ferric salts to the substrate or to the digester. Ferric salts can be reduced to ferrous iron and form pyrite ($FeS_2$) as a precipitate. Often, ferric chloride solution is dosed to the reactor to achieve this removal effect on hydrogen sulfide. Addition of virgin ferric salts has however an operational cost, and a water and carbon footprint. The profitability in for instance the Swedish biogas industry is relatively poor. Swedish and European climate ambitions state that the greenhouse gas emissions should be reduced to at least 55% below 1990 levels by 2030 and be climate-neutral by 2050. Policies to help transition of the society towards the circular economy further on suggest a reduction of waste generation and reuse and recycling of materials and energy, as expressed in the EU New Green Deal, the agenda for sustainable growth. The EU's transition to a circular economy will reduce pressure on natural resources and will create sustainable growth and jobs. It is also a prerequisite to achieve the EU's 2050 climate neutrality target and to halt biodiversity loss. Could other ferric materials with lower costs, less climate footprint and better material use replace the need for virgin ferric chloride? If so, the operational costs would decrease, the footprints become less and more biogas plants could achieve positive results contributing to the New Green Deal.

The segment that has the greatest untapped potential for biogas production in Sweden, but also the biggest economic challenges, is the agricultural sector. Reducing the hydrogen sulfide concentration during digestion is presently associated with significant costs and the handling of corrosive chemicals. For farm-based biogas plants, this is extra stressful because these plants are small, have small financial margins and limited resources for handling hazardous chemicals. In addition, manure (especially pig manure) is rich in sulfur and contains concentrations which can be converted to several thousand ppm hydrogen sulfide during the digestion process. How much hydrogen sulfide is formed during the digestion process depends on the sulfur content of the substrate as in the form of sulfate or as sulfur bound in amino acids [1,2]. High costs for removal of hydrogen sulfide can mean the difference between a positive or a negative financial result at the end of the year for a farm facility and thus also something that limits the expansion of biogas production in agriculture in Sweden. The removal of hydrogen sulfide is also a significant cost in co-digestion plants. Requirements are higher for the separation of hydrogen sulfide in these plants, since the biogas produced is in principle exclusively upgraded to vehicle gas quality. In the case of biogas generated from sewage sludge in wastewater treatment plants, primarily those based on biological phosphorus separation have issues with high hydrogen sulfide concentrations during digestion. Also for these plants, reduction of hydrogen sulfide is associated with costs that make the biogas business less profitable, which is why alternative solutions can be of interest. The sulfur hydrogen is corrosive and must be removed before the biogas is upgraded to vehicle fuel or used for power / heat production. Before upgrading biogas, all sulfur must be gone unless the upgrade is done with a water scrubber, or in some cases amine scrubber, as a few hundred ppm can be accepted. When the biogas is to be used for power / heat production, the requirements are usually around 50-200 ppm, but by lowering the concentration further, the service life can be increased and the need for maintenance on the engine / turbine used for power / heat production can be reduced.

Addition of iron source, may it be iron chloride, iron oxides or waterworks sludge rich in iron salts, binds the hydrogen sulfide in the slurry in the digestion chamber and reduces the problem that the hydrogen sulfide can otherwise inhibit biogas production [1]. The addition of ferric salts can also increase the availability of trace metals that the microorganisms need and thus increase the efficiency of the biogas process [3]. Addition of air and oxygen reduces the hydrogen sulfide concentration in the gas phase, but does not remove to the same extent the problem of hydrogen sulfide inhibiting the microorganisms in biogas production. In addition, the use of oxygen / air in methane streams is associated with some risks and it is important that biogas producers have a good margin to the lower explosion limit for biogas. It is not possible to use air if the biogas is to be upgraded to vehicle quality, but then the oxygen in the air must first be separated from the air nitrogen in an external process [4]. Ferric chloride and ferric oxide have similar properties when it comes to binding sulfide, with the difference that iron oxide is less reactive and less corrosive and thus easier to handle. Regardless of the method used, the reduction of hydrogen sulfide is associated with significant costs for the biogas producer, with the exception of those plants that use only air. A Swedish feasibility study for biogas production at farms [5] showed that the cost of hydrogen sulfide reduction was around €0.01-0.02 per $Nm^3$ biogas at farm biogas plants. The cost is higher for plants where the hydrogen sulfide level must be kept below 100 ppm in the produced biogas.

Since 1997, Sydvatten AB has utilized ferric chloride as a coagulant in the drinking water production at Ringsjöverket, the waterworks in Stehag, south Sweden. The coagulant forms a sludge which is gravimetrically removed from the sedimentation step in the waterworks. The waterworks sludge is dewatered in two steps and landfilled in an area previously used for peat extraction. Sydvatten AB has a sustainability plan decided by the board of directors in 2018 stating among other items that resources should be utilized as efficient as possible and that energy and material should be reused and recycled to largest possible extent [6]. The board of directors has stated that the company must be climate neutral by 2030 and the work on defining how climate neutrality can be reached and what measures must be taken in the organization to achieve climate neutrality been reviewed in the Climate Accounts Report 2020 [7].

Already in 2016, tests were performed to investigate if a reuse of dewatered waterworks sludge could be applied in anaerobic digesters in the biogas industry in Sweden [8]. A growing number of biogas plants with varying sulfur – containing substrates mean a growing need for efficient hydrogen sulfide management. To minimize the amounts of sludge deposited and to increase recycling of materials is beneficial for society and reduces the costs and carbon footprint in the digester. The sludge contains mostly ferric oxide in various forms that originate from the chemical precipitation with iron chloride in the waterworks. The purpose of this study is to technically and economically evaluate the use of the sludge for hydrogen sulfide reduction and to discuss to what extent reuse of ferric waterworks sludge can contribute to the objectives for the company to reach climate neutrality by 2030. A technical evaluation of the methods to add sludge to digesters and which specific dose of sludge should be added to digesters is also presented. We present some experiences from the field of how large amounts of ferric compounds are required to reduce the hydrogen sulfide concentration in different biogas plants.

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The residual solids from the biogas production should be of such a quality so it can be brought back to arable land as organic fertilizers also when using ferric waterworks sludge as a hydrogen sulfide measure in the digester. Efficient material use requires these measures in the sustainable society. In Sweden, two different certification standards are used, depending on the origin of the substrate to biogas plants. If the substrate comes from a wastewater treatment plant, REVAQ is applied [9]. It is national standard for quality control of residuals from wastewater treatment plants and has been used since 2008. If the substrate origins from other sources, such as manure or food waste, the SPCR 120 standard is used instead [10]. This certification standard has been developed by the solid waste industry in Sweden and has been used since 1999.

## 2. Materials and Methods

In substrates containing sulfur and being rich in organic material, the anaerobic microbial metabolism generates sulfide and hydrogen sulfide, depending on the pH. If iron is present, some iron is reduced microbiologically to ferrous iron. Pyrite ($FeS_2$) is a highly insoluble sulfide which can be formed in anaerobic conditions at the presence of sulfide ions. Waterworks sludge from drinking water treatment plants utilizing ferric salts for coagulation contains large amounts of ferric oxide. Mixing such sludge into the digester will cause the ferric ion ($Fe^{3+}$) to be reduced in the anaerobic environment to ferrous iron ($Fe^{2+}$) which binds sulfide ions to form pyrite. To dose ferric compounds to the digester is a method to facilitate the removal of hydrogen sulfide from the biogas. It has been reported that around 2%–4% of influent S enters the digesters, which could be removed sufficiently by a dosage of 1.1 mg/L of Fe into the raw wastewater. They also observed a higher dry matter content in of the dewatered cake as an additional secondary benefit when changing from alum dosage to iron dosage for phosphorous removal [11]. A drop in hydrogen sulfide emission from full-scale anaerobic digesters (ADs) at a largescale municipal wastewater treatment plant could be achieved when dosing ferric chloride. The ferric salt was applied at a range of 24-105 mg $FeCl_3$/L in the feeding line and the sludge thickener unit. The hydrogen sulfide emission was reduced with 4 mg/L in directly-dosed AD, but only 1.3 mg/L in non-dosed ADs. The formation of hydrogen sulfide could be correlated to the volatile primary sludge solid loading rates based on data for a 17-month study period [12].

Waste iron powder produced by laser cutting machine in a steel and iron industry was mixed with dairy manure at a concentration between 2.0 and 20.0 g/L in digestion batch experiments and between 1.0 and 4.0 g/L in bench experiment. For batch experiments, the hydrogen sulfide concentration could be reduced up to 93% at a dosage of waste iron powder of 2.0 g/L. If the waste iron powder concentration was higher than 8.0 g/L the reduction was more than 99%. Waste iron powder did not have significant effect on methane yield in batch and bench experiments, but the hydrolysis rate constant was almost doubled and the lag-phase period halved in test digesters compared to control digester without iron dosage. In bench experiment, $H_2S$ concentration was reduced by 89% at 2.0 g/L, while 50% at 1.0 g/L without harming the digestion process [13].

The use of $Fe_2O_3$ and $TiO_2$ nanoparticles at four different concentrations in two different combinations, from 20 to 500 mg/L, where used for the mitigation of hydrogen sulfide emission during anaerobic digestion of cattle manure in a batch system. The $H_2S$ production was 2.13 - 2.64 times lower than a control. Additionally, biogas and $CH_4$ production were 1.09 - 1.191 times higher than those of the control [14]. Titanium is relatively costly and in another study, the researchers investigated whether directly adding waste iron powder and iron oxide nanoparticles to batch digesters could offer a more cost-efficient solution to the hydrogen sulfide generation. By adding iron in the form of microscale iron powder at concentrations of 100 mg/L to 1000 mg/L, the methane yield could be improved up to 57%. The equivalent dosages of iron nanoparticles improved the yield up to 21%. The highest iron powder dose (1000 mg/L) achieved the maximum improvement in the rate of hydrolysis which was 1.25 times higher than in control reactions. High dosage of iron powder also decreased the rate of hydrogen sulfide production with up to 77% compared with the reference. A direct mixing of microscale iron powder was proposed as a practical and economical means of supporting the production of biogas from dairy manure [15]

Addition of iron-rich drinking water sludge directly in the urban domestic wastewater system was tested to reduce dissolved sulfide in sewer systems and to aid phosphate removal in wastewater treatment and reduce hydrogen sulfide in the anaerobic digester. It was tested using two laboratory-scale urban wastewater systems, one as an experimental system and the other as a control, each comprising sewer reactors, a sequencing batch reactor (SBR) for wastewater treatment, sludge thickeners and anaerobic digestion reactors. The experimental system received in-sewer drinking water sludge corresponding to 10 mg Fe/L while the control had none. Addition of ferric sludge decreased the hydrogen sulfide concentration in the wastewater by 3.5 mg S/L as compared with the control. The phosphate concentration decreased by 3.6 mg P/L after biological wastewater treatment in the experimental SBR. In the experimental anaerobic digester, the sulfide concentration decreased by 16 mg S/L compared with the reference. The drinking water sludge dosing also enhanced the settleability of the mixed liquor suspended sludge and the dewaterability of the anaerobically digested sludge. The cake solids concentration increased from 16% to 19%. Also the COD and TSS concentrations in the wastewater were increased yet did not affect normal operation. The authors concluded that addition of iron-rich drinking water sludge could be used in the urban wastewater system achieving multiple benefits [16].

Just over 2.1 TWh of biogas was produced in Sweden in 2019 Swedish biogas production increased by 3.3 percent in 2019 to a total of 2111GWh (Table 1). Biogas production increased at all plant types except industrial plants and gasification plants in 2019. The largest increase was in digestion plants (+68 GWh), which also accounted for most of the increase in the last decade. A total of 49 percent of the biogas was produced in co-digestion plants and 35 percent at sewage treatment plants. There are a total of 280 biogas production facilities in Sweden [17]. The biogas is mainly produced from various types of waste and residual products such as sewage sludge, food waste, manure and waste from the food industry & slaughterhouse. More and more biogas is produced from manure. A total of 71 plants use fertilizer as a substrate and the amount of manure that is digested has increased by 9 percent to 1.1 million ton. In total, around 2.8 million ton of digestate (wet weight) was produced at Swedish biogas plants in 2019, of which 2.4 million ton (87 percent) were used as fertilizer in agriculture. It is at the same level as in 2018. From co-digestion plants and farm plants, all digestate (bio fertilizer) was used as fertilizer. From the sewage treatment plants, 41 percent of the digestate (digestate sludge) was used as fertilizer. Just under two thirds of the biogas is upgraded The long-term trend that an increasing amount of biogas is upgraded continues, after a temporary decline in 2018. The biogas is upgraded to be used as vehicle gas or fed into the gas network. Of the biogas produced, 64 percent was upgraded (1351 GWh) and 19 percent was used for heat production (Table 2). Direct electricity production

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continues to decline. The share of biogas that goes to flaring is a total of 11% of production, a certain increase towards 2018. Flaring has to be applied during start-up phases of digesters and occasionally when operational problems occur. In 2019, a large new digester was commissioned and the start-up issues took some time to solve [17].

Table 1

Volume of biogas production and number of plants in Sweden in 2019 per plant type and change since 2018 [17]

| Plant type | Number of plants | Production (GWh) | Share | Change since 2018 |
|---|---|---|---|---|
| Sludge from wastewater treatment plants | 135 | 738 | 35 | +2 |
| Co-digesters | 36 | 1031 | 49 | +7 |
| Farm units | 50 | 58 | 3 | +4 |
| Industrial plants | 7 | 142 | 6 | -1 |
| Landfill gas plants | 52 | 142 | 7 | +1 |
| Gasification plants | 0 | 0 | 0 | -100 |
| Sum | 280 | 2111 | 100 | +3.3 |

Table 2

Use of produced biogas in Sweden 2019 with change since 2018. [17]

| Area | Use (GWh) | Share (%) | Change since 2018 (%) |
|---|---|---|---|
| Upgrading | 1351 | 64 | 4 |
| Heat | 397 | 19 | -1 |
| Electricity | 38 | 2 | -10 |
| Industrial use | 52 | 2 | 0 |
| Other uses | 23 | 1 | -15 |
| Flaring | 235 | 11 | 11 |
| Losses and lack of data | 15 | 1 | 2 |
| Sum | 2111 | 100 | 3.3 |

Of the upgraded biogas, 539 GWh was injected directly into the gas distribution net in south-west Sweden and in the regional net in Stockholm. In 2019, the total biogas use increased with 7 percent, and the import was estimated to around 1,8 TWh, giving a total biogas use in Sweden in 2019 to 4 TWh. The biogas market grows in Sweden. Since 2015, it has doubled, but the Swedish production has only grown with in total 9 procent during the same period [17]. Profitability in the Swedish biogas industry is relatively poor and many biogas producers are struggling to achieve positive results. The segment that has the greatest untapped potential for biogas production in Sweden, but also the biggest economic challenges, is the agricultural sector. In order for there to be growth in this segment, it is necessary to be able to report profitability for the business. Reducing the hydrogen sulfide concentration during digestion is today associated with significant costs and the handling of corrosive chemicals. For farm-based biogas plants, this is extra stressful because these plants are small, have small financial margins and limited resources for handling

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hazardous chemicals. In addition, manure (especially pig manure) contains sulfur which can be converted to several thousand ppm hydrogen sulfide during the digestion process. How much hydrogen sulfide is formed during the digestion process depends on the sulfur content of the substrate as in the form of sulfate or as sulfur bound in amino acids [1,2]. High costs for hydrogen sulfide reduction can mean the difference between a positive or a negative financial result at the end of the year for a farm facility and thus also something that limits the expansion of biogas production in agriculture in Sweden today.

The reduction of hydrogen sulfide is also a significant cost for co-digestion plants. Here, the requirements for the separation of hydrogen sulfide are higher than at farm-based biogas plants that produce power / heat, since the biogas produced at digestion plants is in principle exclusively upgraded to vehicle gas quality (see Table 2). In the case of wastewater treatment plants, it is primarily plants that have biological phosphorus separation that experience high hydrogen sulfide concentrations during digestion. Also for these plants, reduction of hydrogen sulfide is associated with costs that make the biogas business less profitable, which is why alternative solutions can be of interest. The sulfur hydrogen is corrosive and must be removed before the biogas is upgraded to vehicle fuel or used for power / heat production. Before upgrading biogas, all sulfur must be gone unless the upgrade is done with a water scrubber, or in some cases amine scrubber, as a few hundred ppm can be accepted. When the biogas is to be used for power / heat production, the requirements are usually around 50-200 ppm, but by lowering the concentration further, the service life can be increased and the need for maintenance on the engine / turbine used for power / heat production can be reduced. Addition of iron chloride, iron oxides or waterworks sludge from iron coagulation steps binds the hydrogen sulfide in the slurry in the digestion chamber and reduces the problem that the hydrogen sulfide can otherwise inhibit biogas production [1]. The addition of iron can also increase the availability of trace metals that the microorganisms need and thus increase the efficiency of the biogas process [3]. Addition of air and oxygen reduces the hydrogen sulfide concentration in the gas phase, but does not remove to the same extent the problem of hydrogen sulfide inhibiting the microorganisms in biogas production. In addition, the use of oxygen / air is associated with some risks and it is important that biogas producers have a good margin to the lower explosion limit for biogas. It is not possible to use air if the biogas is to be upgraded to vehicle quality. The oxygen in the air must first be separated from the air nitrogen in an external process [4]. Iron chloride and iron oxide have similar properties when it comes to binding sulfide, but with the difference that iron oxide is less reactive and less corrosive and thus easier to handle. Regardless of the method used, the reduction of hydrogen sulfide is associated with significant costs for the biogas producer. One study for Swedish farm-based digesters indicated a cost of hydrogen sulfide reduction to be around €0.01-0.02 per $Nm^3$ biogas at farm biogas plants. The cost was higher for plants where the hydrogen sulfide level had to be kept below 100 ppm in the produced biogas [5].

Since 2013, iron-containing sludge from drinking water production at Sydvatten's waterworks in Stehag has been offered biogas production plants in southern Sweden for hydrogen sulfide control. Sydvatten's interest is to minimize and eventually avoiding landfilling of waterworks sludge and find pathways for how to reuse the sludge for other applications. A survey of the properties of the waterworks sludge and how it has been used for counteracting hydrogen sulfide formation during biogas production has previously been reported [8]. The sludge contains mostly iron in various

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forms that originate from the chemical precipitation with iron chloride in the waterworks. The purpose of this study is to technically and economically evaluate the use of the sludge for hydrogen sulfide removal and study parameters such as how large amounts are required to reduce the hydrogen sulfide concentration in different biogas plants and how the quality of digestate is affected by the sludge and whether plants can be used, certified by SPCR 120 or Revaq.

Dewatered waterworks sludge was collected three times in 2016 and analyzed with reference to metal content in an accredited lab, AlControl AB. Sludge was collected from three different dewatering batches and mixed prior to analysis. 13 biogas producers from different sites in south Sweden which use the waterworks sludge in full scale for hydrogen sulfide removal were asked to share their experiences from these facilities have been collected and compiled below under different categories. Experiences from waterworks sludge transportation, transport cost, operational and maintenance costs for storage, dosing and cleaning of the equipment used in the handling of waterworks sludge at the biogas plant, the practical dosage and use of waterworks sludge in the digester, effects of storage conditions due to storage time and ambient temperature, and general operational observations on conditions when the waterworks sludge was dosed into the digester and mixed with substrate were recorded in the interview series. All interviews were done through direct visits to the plants and through interviews with plant operators and managers.

## 3. Results

At Sydvatten's waterworks Ringsjöverket in Stehag, approximately 9000 tons of sludge with 15% total solids (TS) are produced annually (see Table 3). The sludge is formed in the chemical precipitate which is the first part of the waterworks process. The raw water comes from Lake Bolmen in Småland and the organic content of Bolmen's water is virtually inert, i.e. it will not contribute to the biogas production in the digestion chamber. Today, ferric chloride is used as a coagulation chemical to coagulate organic material in the water. The water pH is corrected with lye before the addition of the coagulant. The sludge settles in lamella sedimentation and is then thickened in a gravity thickener after the addition of iron chloride, lime water and polymer to be able to reach a dry content of about 2%. The sludge is then pumped to a sludge handling plant where the sludge is pressed in sieve belt presses after the addition of additional polymer and iron chloride to a dry content of about 15%. The sludge is finally landfilled in a closed peat extraction area. The overall generation of sludge and the fraction used in biogas plants are presented in table 3.

Table 3

Waterworks sludge generation and fraction used for sulfide removal in biogas plants (Sydvatten, internal statistics).

| Year | Total waterworks sludge generation (ton) | Fraction to biogas plants for hydrogen sulfide removal |
|---|---|---|

| | wet weight | total solids | [%] |
|---|---|---|---|
| 2012 | 10388 | 1558 | 0% |
| 2013 | 10709 | 1606 | 5% |
| 2014 | 9378 | 1407 | 10% |
| 2015 | 9521 | 1428 | 16% |
| 2016 | 7907 | 1186 | 27% |
| 2017 | 6739 | 1011 | 40% |
| 2018 | 8730 | 1310 | 34% |
| 2019 | 7682 | 1152 | 56% |
| 2020 | 9584 | 1438 | 56% |

There are operating situations at the waterworks when no iron-containing sludge is produced. This occurs especially if there is a landslide in the tunnel that runs between Lake Bolmen in Småland and Ringsjöverket. This has historically occurred on three occasions over the past 30 years and then the tunnel has had to be drained and renovated. On these occasions, water from Ringsjön is used at the waterworks and then an aluminum-based precipitation chemical is used instead of iron chloride. The sludge formed then cannot be used in the biogas industry. These malfunctions usually take between one and two years to rectify. It is therefore important for biogas producers who choose to use the waterworks sludge that they can quickly switch to an alternative solution, such as ferric chloride, during such a period.

The waterworks sludge from Ringsjöverket has a black-brown appearance (see Figure 1) and if it has been in contact with air for a while, small black iron crystals form on the surface. The sludge is water-soluble and has a slight iron odor. Density is around 1.1 kg / $dm^3$ and the sludge is a bit acidic with a pH value around 4.2. The sludge consists mostly of iron compounds (about 30% iron and> 40% iron oxides) and various organic compounds (about 25% TOC). In the waterworks, before the sludge is separated, only iron chloride and lye are added. Then a small amount of the drinking water-grade polymer Magnafloc LT22S-DWI is added together with additional iron chloride in both the thickener and the screen belt press to facilitate dewatering [8].

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Figure 1: Waterworks sludge (left) and manure storage (right). Photo: Annika Nyberg.

A detailed metal analysis has been done on the dewatered sludge from Ringsjöverket on three different occasions. Results for the most relevant metals are reported in Table 4. Table 4 compares the analysis results with the limit values that exist within Avfall Sverige's certification rules for biofertilizers, SPCR 120 [10] and the Swedish Environmental Protection Agency's general guideline values for contaminated land for sensitive land use published in 2009 [18]. It is clear that the metal content of the waterworks sludge is by a very good margin below these levels for virtually all metals. Only the limit value for Arsenic is in the same order of magnitude in the sludge as the Swedish Environmental Protection Agency's guideline value for contaminated soil in sensitive land use. Revaq is a certification system with the aim of reducing the flow of hazardous substances to treatment plants and creating a sustainable return of plant nutrients by spreading sludge from wastewater treatment plants on arable land [9]. The heavy metals that have the greatest focus in Revaq and that require the most frequent analysis intervals within Revaq are Lead, Cadmium, Copper, Chromium, Nickel,

Zinc, Mercury, Silver and Tin and analysis results from all of these are included in Table 4. Even in this case, the concentrations are low compared to the guideline values that exist. Table 4 also shows that the iron content is relatively constant during the year with only small variations. This is important for biogas producers to be able to use a similar dosage for different sludge deliveries.

Table 4. Composition of waterworks sludge from Ringsjöverket compared with guideline values from Swedish EPA [18] and the Swedish Waste Association standard SPCR 120 [10] for permissible metal content for bio solids reuse.

| Metal | Guideline value from Swedish EPA for contaminated land | SPCR 120 | 2015-10-12 | 2016-04-11 | 2016-06-27 | Unit |
|---|---|---|---|---|---|---|
| Antimony | 12 | | <2.1 | <2.1 | <2.1 | mg/kg TS |
| Arsenic | 10 | | 7.2 | 5.1 | 8.6 | mg/kg TS |
| Barium | 200 | | 40 | <23 | 45 | mg/kg TS |
| Cadmium | 1.5 | 1 | <0.11 | <0.11 | 0.13 | mg/kg TS |
| Chromium | 80 | 100 | 12 | 18 | 15 | mg/kg TS |
| Cobalt | 15 | | 4.3 | 5 | 4.7 | mg/kg TS |
| Copper | 80 | 600 | 11 | 17 | 15 | mg/kg TS |
| Iron | | | 30 | 29 | 28 | % of TS |
| Lead | 50 | 100 | 6.5 | 5.3 | 11 | mg/kg TS |
| Mercury | 0.25 | 1 | <0.051 | <0.051 | 0.052 | mg/kg TS |
| Molybdenum | 40 | | 4.1 | 4.5 | 4.1 | mg/kg TS |
| Nickel | 40 | 50 | 5.5 | 5.3 | 6.7 | mg/kg TS |
| Silver | | | <0.51 | <0.51 | <0.52 | mg/kg TS |
| Tin | | | <0.51 | 0.7 | 0.82 | mg/kg TS |
| Vanadium | 100 | | 47 | 47 | 64 | mg/kg TS |
| Zinc | 250 | 800 | 49 | 50 | 65 | mg/kg TS |

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In 2016, the waterworks sludge was accepted as an approved additive to the digestion process within the certification system SPCR 120. It is therefore now possible for the biogas plants that have this certification to use the waterworks sludge to reduce the hydrogen sulfide concentration in the biogas. When it comes to certification according to Revaq, the use of the waterworks sludge should only affect the cadmium (Cd) / phosphorus (P) ratio marginally because the cadmium content is very low. However, the phosphorus content is also low, which means that even a low cadmium concentration can have a negative effect on the Cd / P ratio. The size of the sludge feed is also affected because this is regulated not only by the metal concentration but also by the amount of a certain metal that may be laid per unit area. This applies in particular to the metals lead, cadmium, chromium, nickel, zinc and mercury. However, the content of these metals is very low and should only have a minor impact. The rules for the Revaq certification system state, however, that “The certificate holder shall not receive such material that is deemed to adversely affect the quality of sludge, through low nutrient content or high content of contaminants. The 60 trace elements must always be analyzed before receiving a new type of material”. This can be a problem for the use of the waterworks sludge in Revaq-certified treatment plants, but it is not really different from using ferric chloride to reduce the hydrogen sulfide concentration as it also contains some other heavy metals.

During the project, 13 biogas producers which use the waterworks sludge in full scale have been contacted. The experiences from these facilities have been collected and compiled below under different categories. Some of the results have already been published in a report in Swedish [8].

### Transport cost

The transport cost varies between different biogas producers depending on the distance and how the transport is performed. Some use trucks with trailers and others without. Some drive a few kilometers while others drive up to 600 kilometers. In addition to the actual transport of the sludge, the transporter must also spend time cleaning the platform afterwards. The cost reported by the biogas producers in different parts of the country varies between € 15 and 40 / ton of waterworks sludge with 15% TS.

#### Other costs

In addition to transport, biogas producers have costs for storage, dosing and cleaning of the equipment used. For most users of waterworks sludge for hydrogen sulfide control, these costs are considered to be marginal and estimated at somewhere between € 2 and 10 / ton wet waterworks sludge.

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Most of the biogas producers who today use waterworks sludge have previously used iron chloride that they bought from a chemical supplier. The cost they had to reduce the hydrogen sulfide concentration with iron chloride has for some plants been comparable to what they pay for the transport of the waterworks sludge today, while others have reduced the costs by up to about 50%. According to Broberg [5], the cost of hydrogen sulfide reduction with iron chloride and iron oxide is around €0.01-0.02 per Nm3 on farm biogas plants. The cost of using waterworks sludge ends up in the same order of magnitude if the cost of handling and transport is estimated at approximately € 40 per ton. However, several biogas producers have reported substantially lower transport and handling costs and in addition they reduce the hydrogen sulfide concentration to lower levels when using the sludge compared to when they used ferric chloride. This supports the conclusion that cost savings of up to 50% are possible but that this depends on the transport cost. The reason why they reduce the hydrogen sulfide concentration more with sludge is that they think they can afford to add a surplus of iron to the digester, since the marginal material cost is lower and that they thus can then control the dosage towards a lower hydrogen sulfide concentration in the biogas generated. A lower residual hydrogen sulfide concentration in the biogas increases the life of the power / heating unit and reduces its maintenance needs and costs. If the substrate used has lower sulfate content, the amount of sludge needed per volume of biogas produced is lower. Other benefits which the operators of the biogas production plants have observed is an easier handling of waterworks sludge when dosing compared to the corrosive ferric chloride solution and the corrosive damage to the equipment that this can lead to. Some of the farm biogas producers have previously used only aeration or aeration in combination with iron additive. Then the economic gain with the transition to waterworks sludge is smaller because the addition of air is associated with very marginal costs.

**Use of waterworks sludge in the digester**

The number of plants which utilize waterworks sludge from Ringsjöverket has increased since the test started in 2013. In 2020, a total of 24 different plants collect iron-containing waterworks sludge for hydrogen sulfide removal in the digesters. Based on information from seven biogas plants where only manure is used as substrate, one ton of wet waterworks sludge (with a TS of 15%) is sufficient to produce an average of 2700 $Nm^3$ of biogas if the hydrogen sulfide concentration in the biogas is to be reduced below 100 ppm. This corresponds to about 0.2-0.5% of the amount of substrate added, expressed as dry matter. The stoichiometric relation between sulfur and iron could be observed in an eight biogas plant where the residual hydrogen sulfide content was allowed to be higher. This plant was

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designed to produce a biogas with residual hydrogen sulfide concentration below 300 ppm. An addition of one ton of waterworks sludge to the digester was enough to produce 8000 $Nm^3$ of biogas with <300 ppm $H_2S$.

In co-digestion plants where a mixture of manure, starch, food waste and slaughterhouse is applied, one ton of wet waterworks sludge was sufficient to reduce the hydrogen sulfide concentration to a significantly larger volume of biogas, since the mix of substrate contains less sulfur than pure manure. Five co-digestion plants surveyed in this study dosed less than half the amount of waterworks sludge to the substrate compared with the manure-based biogas plants and could still generate a biogas with less than 100 ppm hydrogen sulfide. If the proportion of manure dominates in the substrate, the need for more waterworks sludge addition still remains high since that kind substrate is similar to pure manure. Iron in waterworks sludge is less available and ample compared with an addition of pure ferric chloride solutions to control the hydrogen sulfide concentration. In two biogas plants, comparisons of these operational conditions showed that the total amount of iron added to the substrate had to be increased 2.5 to 3 times to achieve a similar effect on hydrogen sulfide removal.

**Impact of temperature and storage of waterworks sludge**

Out of 13 surveyed plants, one had experienced a slight loss of efficiency from waterworks sludge dosing in the summer. When stored for a long time in the summer, the waterworks sludge lost some of its function since iron crystals was form on the sludge surface when the dried in the sun. The iron in the crystals was less available for the microorganisms in the digester. Only one of the producers surveyed identified it as problematic. Storage of sludge in a shaded environment and modest addition of moisture to the sludge could mediate this issue.

**Observed operational conditions when mixing waterworks sludge with substrate and feeding the mix into digesters**

The mixing and feeding of waterworks sludge to the digester was generally a carefree process. Very few problems have been experienced in connection with the handling of the waterworks sludge in the biogas plants. The exception was for biogas plants utilizing solid substrates. In these plants, the substrate is mixed with waterworks sludge and fed it into the digestion chamber with a screw. The screw is designed for handling dry materials. If the substrate becomes too wet after the mixing with the sludge, it slides backwards and stops following the screw. With less feed to the digester, the production decreases. The solution to the problem has been to mix the sludge with drier materials and preferably also with longer straws. In one of these plants it was observed that parts of the waterworks sludge remained at the bottom of the digestion chamber when it was opened. No action has been

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taken, but it can be seen as is an indication that the dissolution of the sludge is slow when it is fed together with solid material.

In most of the plants that use liquid substrates, the waterworks sludge is scooped into a mixing well. In these wells the pH is often quite low and the stirring is vigorous. A low pH facilitates the dissolution of the sludge as the solubility of the iron increases with decreasing pH. Those who use this type of mixture have not experienced any problems with dissolving the sludge, nor have they seen any residues of undissolved sludge in the mixing well when it has been drained. For wastewater treatment plants, operators have expressed concerns that the addition of waterworks sludge could affect the drainage property of the digestate. Literature data indicates the opposite effect that the drainage of bio solids after addition of waterworks sludge is improved [11,16].

**Transportation of waterworks sludge to the digestion plant**

The waterworks sludge is slightly adhesive and may get stuck on the flatbed when transported. For this reason, various measures have been taken to make handling and cleaning easier. Many people have added straw or sawdust on the flatbed before loading the sludge to make cleaning easier. Another possibility is to spray the flatbed with rapeseed oil or similar prior loading. There is also a risk in cold climates that the sludge gets stuck on the platform due to freezing. In wintertime, it may be necessary to transport the sludge in closed containers.

## 4. Discussion

Assuming that the waterworks sludge is virtually inert and does not degrade in the digestion chamber, the entire dry content of the added waterworks sludge will pass through the digestion chamber and be present in the residual bio solids. Based on the information collected from the examined biogas plants included in the study, the waterworks sludge contributes to an increased amount of digestate that is about 1-3% based on dry matter, ie. about 1-3% of TS. The digestate in most plants has a TS content of around 5% while the waterworks sludge has a TS content of around 15%. The volume increase of the digestate to be handled due to addition of waterworks sludge is maximum 1%; the TS content in the digestion increases about 0.1-0.3% while the total amount of metals in the digestate increases by approximately 1-3%, see calculation in table 5. In table 5 is also referred to the quality requirements according to the biofertilizer certification system SPCR 120 [10]. This standard states that the proportion of each of the metals lead, cadmium, copper, chromium, mercury and zinc, may not exceed 15% of the total amount of the metal in the certified biofertilizer. In addition, the nickel content in the biofertilizer from the waterworks sludge must not exceed 6 mg / kg biofertilizer (wet weight). In 2019, around 2.8 million ton of digestate (wet weight) was produced in Sweden, of which 87 percent was

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used as fertilizer in agriculture [17]. From farm plants and co-digestion plants, 2.13 million ton, virtually all digestate (biofertilizer) was used as fertilizer. From the wastewater treatment plants, 0.25 million ton (41 percent of all the sludge) was used as fertilizer certified in the Revaq-system. The remaining amount was used mainly as construction material or for final coverage of landfills [17]. No biosolids are generated in the landfill gas plants.

The biogas production from the other plants which also generate biosolids corresponds to a total of 1969 GWh [17]. These plants simultaneously produced 2.8 million ton of biosolids, or around 700 kWh / ton of biofertilizer. Assuming that 1 $Nm^3$ $CH_4$ corresponds to 9.81 kWh [19] and that the biogas contains 65% methane [20], this corresponds to a production of 110 $Nm^3$ biogas per ton biofertilizer. According to values for the 13 plants using waterworks sludge from Ringsjöverket surveyed above, around 1 ton of waterworks sludge is added per 8750 $Nm^3$ of biogas that has been produced. This corresponds to adding 1 ton of waterworks sludge per 108 ton of biofertilizer produced in the plant excluding the added waterworks sludge, or 9.2 kg of waterworks sludge per ton of biofertilizer produced. Table 5 shows how adding of waterworks sludge affects the concentration of metals in the biofertilizer. The starting point for the calculation is the average value of the metal contents in the biofertilizer for the 18 plants that were certified within SPCR 120 in 2014 [21]. Nickel is not included as there is no risk that the content of nickel can reach up to 6 mg / kg wet weight with the concentration of nickel present in the waterworks sludge. Among other heavy metals, lead and chromium make up the largest part of the total metal concentration with their 7 and 6%, respectively. However, this is by a margin below the limit of 15% specified according to SPCR 120. Therefore, this is also not considered to be a problem for the use of the waterworks sludge. With the addition of the waterworks sludge, which is assumed to be inert, the S content of the biofertilizer increases. Assuming the same waterworks dose as above, the TS in the biofertilizer increases from 3.9 to 4.0%. An effect of this is that the concentration of metals stated as mg / kg TS decreases for e.g. copper and zinc, while the concentration increases slightly for lead and chromium (see Table 5).

Table 5: Average concentration of heavy metals in biofertilizer for the facilities that were certified in 2014 [21] before and after an estimated addition of waterworks sludge. TS in the biofertilizer is 3.9% before the addition of waterworks sludge. Concentration in the sludge is an average of analyzed data from according to Table 3.

| | Unit | Pb | Cd | Cu | Cr | Hg | Zn |
|---|---|---|---|---|---|---|---|
| Concentration in bio ferti- | mg/kg TS | 3.6 | 0.4 | 89 | 8.3 | 0.06 | 292 |

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| | | | | | | | |
|---|---|---|---|---|---|---|---|
| lizer | | | | | | | |
| Concentration in bio ferti-lizer | mg/kg DS | 0.14 | 0.016 | 3.47 | 0.32 | 0.002 | 11.4 |
| Concentration in waterworks sludge | mg/kg | 1.15 | 0.019 | 2.15 | 2.25 | 0.01 | 8.27 |
| Content in 9.2 gram water-works sludge | mg | 0.011 | 0.0002 | 0.020 | 0.041 | 0.00007 | 0.150 |
| Total amount in 1 kg bio ferilizer + 9.2 g waterworks sludge | mg | 0.15 | 0.016 | 3.49 | 0.34 | 0.002 | 11.5 |
| Fraction of metals from waterworks sludge | % | 7.0 | 1.1 | 0.6 | 6.0 | 3.0 | 0.7 |
| Total conc in bio fertilizer produced with water-works sludge | mg/kg TS | 3.7 | 0.4 | 86 | 8.4 | 0.06 | 281 |

Since about half of the waterworks sludge consists of various organic compounds from the lake source water, the waterworks sludge contributes to increased organic content in soil where the digestate is spread as fertilizer. In many Swedish soils, the organic content is low, which is why this is a welcome contribution to improving the soil's properties. The organic content in soils improves physical, chemical and biological properties of the soil such as its water holding capacity, nutrient content, buffer capacity and the activity of soil organisms.

To reuse material is beneficial for climate and society and reduces the carbon footprint. According to the evaluation in Sydvatten's Climate Account Report 2020, production of virgin iron chloride generates about 0,395 kg $CO_2$ emisson per kg $FeCl_3$. Since Sydvatten used 3132 ton $FeCl_3$ 2020 for the drinking water treatment, the reuse of 60% sludge by replacing virgin ferric chloride with waterworks sludge would eliminate 740 ton carbon dioxide, which is about 17% of all carbon dioxide emitted by the company in 2020 [7], well in accordance with EU:s Circular Economy Plan [22]

## 5. Conclusions

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Waterworks sludge which contains iron works very effectively as an additive to reduce the hydrogen sulfide concentration in biogas production. This has been demonstrated in 13 full-scale biogas plants surveyed in this project. According to the costs reported for transport and handling of the sludge, there is potential to save up to 50% compared to if these plants would use virgin ferric chloride instead. In the manure-based biogas plants, one ton of waterworks sludge with 15% TS is sufficient to reduce the hydrogen sulfide concentration to below 100 ppm in 2-3000 $Nm^3$ biogas. In the digestion plants that participated in the study, the same amount of sludge was enough for more than twice as much gas, about 8000 $Nm^3$. In both cases, the exact figure depends on the substrate composition and the level at which the hydrogen sulfide concentration is reduced. The content of heavy metals in the waterworks sludge is well below the concentrations used in Avfall Sverige's certification system, SPCR 120. This has been crucial since SPCR 120 is a requisite for use of sludge in certified co-digestion plants. An additional positive impact is improved material utilization. Reuse of 56% sludge by replacing virgin ferric chloride with waterworks sludge saves 740 ton carbon dioxide corresponding to about 17% of all carbon dioxide emitted by Sydvatten in 2020. Nothing hinders an increased use of waterworks sludge for biogas production in the future.

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