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ORIGINAL PAPER

Hydrogeological Properties of Till Amended with Green Liquor Dregs (GLD): Recycling of an Industrial Residue for Reclamation of Acid Generating Mine Sites

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Abstract The oxidation of sulfide minerals in mine wastes is a possible threat to the environment as it might have potential to generate acid rock drainage (ARD). A common method to reduce ARD is to apply a dry cover on the mine waste deposit. Considering the massive amounts of mine waste produced in Sweden (104-million-ton in year 2018) there is a great need for suitable dry cover materials. Using non-hazardous industrial residues in the dry cover would be beneficial for both the mining industry and the providing industry as stricter waste management legislation incentivizes them to develop their waste management strategies. The objective of this study was to investigate if an addition of Green Liquor Dregs (GLD), a residue from pulp production, can decrease the hydraulic conductivity and increase the water retention capacity (WRC) of three different tills, with the purpose of improving the performance of a dry cover material on a mine waste deposit. Another objective was to investigate how the hydraulic conductivity and WRC are affected by the contents of fines and clays in the tills. The study concludes that the water retention capacity of the tills improves with GLD addition, however, the hydraulic conductivity did not decrease enough to reach the in Sweden required $< 10^{-8}$ m/s. Even though, GLD could still successfully be used in the reclamation of mine sites as the high WRC can be seen as the most important factor in deterring acid rock drainage by keeping the sealing layer close to saturation. This study further indicates that there are other factors than the particle size distribution of the materials that controls the hydraulic conductivity of the mixtures, such as initial water content, dry density, and compaction effort.

Keywords Green liquor dregs · Circular economy · Mine reclamation · Sealing layer · Hydraulic conductivity · Water retention capacity

Abbreviations

GLD	Green liquor dregs
ARD	Acid rock drainage
WRC	Water retention capacity
SWCC	Soil water characteristic curve
wt%	Weight percentage
SaT	Sandy till
SiT1	Silty till 1
SiT2	Silty till 2
TSC	Total solid content
PSD	Particle size distribution
PC	Proctor compaction
VSC	Volumetric shrinkage curve
N	Number of samples

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1 Introduction

Massive amounts of mine wastes are generated all over the world. Sweden alone generated 104-million-ton mine waste in 2018, accounting for 75% of all waste produced during that year (Avfall Sverige 2020). These wastes need to be managed in an environmentally-, technically- and economically sustainable way. About 70% of the mine waste in Sweden contain sulfide minerals (SGU and Swedish EPA 2017) which if left in contact with oxygen and humidity oxidize and may produce acid rock drainage (ARD). ARD is a major long-term threat to the environment as metals and metalloids may become mobile (Saria et al. 2006). The GARD guide (Global Acid Rock Drainage guide; Verburg et al. 2009) categorize different methods to prevent ARD after closure, into two main categories; engineered barriers and water covers. In the relatively humid climatic conditions in Sweden a dry cover, a form of engineered barrier, can be used to reduce water and oxygen flux to the underlying reactive wastes, and thus reduce ARD (Collin and Rasmuson 1990; Bussière et al. 2003; Dagenais et al. 2006). A dry cover in Sweden usually consists of a sealing layer placed on top of the mine waste, made of a fine-grained compacted material to prevent oxygen to diffuse to the waste underneath by keeping it close to saturation. Above the sealing layer the protective layer protects the underlaying layer from erosion, frost and/or root penetration. Because of the Swedish geology most sealing layers are usually made of till, ideally a clayey till. The availability of a clayey till nearby a mine is however often limited, and the till can either be improved or replaced with another material. Bentonite amendments to till is one solution to improve the sealing layer-qualities of the local till. However, bentonite is costly both economically and environmentally due to time- and resource consuming production. The use of an industrial residue as an amendment to a local till is therefore highly motivating. The European Union produces about 2.5 billion tons of waste each year (Avfall Sverige 2020) and Sweden produced 139 million tons waste year 2018, excluding mine waste, of which about 2.9 million tons was classified as a non-hazardous waste. Using a non-hazardous inert industrial residue in a mine remediation program is beneficial for the environment, for the industry providing the residue, and the mining industry. Some examples of industrial residues that previously have been used as a mine waste cover are sewage sludge, fly ash, desulfurized tailings, coal combustion by-products (CCB), and steel slag (Hallberg et al. 2005; Bäckström et al. 2009; Dobchuk et al. 2013; Lu et al. 2013; Nason et al. 2013; Park et al. 2014; de Almeida et al. 2015; Phanikumar and Shankar 2016). According to the Confederation of European Paper Industries (CEPI) 155 Mt of wood was consumed by the paper industry and 38 Mt of pulp was produced in year 2018 (CEPI Key Statistics 2018). Sweden, Finland, and Portugal are the top three countries accounting for 69% of the pulp production (Quina and Pinheiro 2020). In Sweden, about 200 000 tons of GLD were generated annually according to a survey made in 2003 (SGI 2003), and production has increased based on a survey made 2017 (unpublished data). Green Liquor Dregs (GLD) is an inert, alkaline, inorganic waste originating from the recycling process at sulfate pulp and paper mills which has the same grain size distribution as silt. It has properties suitable as sealing layer, as it is fine grained (d100 < 63 µm), commonly has a hydraulic conductivity in the range of 10^{-8} to 10^{-9} m/s, a higher water retention capacity (WRC) compared to materials with similar particle size, such as clayey/sandy silt (Mäkitalo et al. 2014) and a low oxygen diffusion coefficient (Virolainen et al 2020). GLD is regarded as an inert material (Mäkelä and Höynälä 2000), where the main solid compounds consist of $CaCO_3$, $Mg(OH)_2$, C, and metal sulfides, especially FeS (Sanchez and Tran 2005; Jia et al. 2014). The liquid phase of the GLD consists of Na_2CO_3 and NaOH, which generates its characteristic high pH. A scientific review of GLD made by Quina and Pinheiro (2020) suggest that the mineral phases of GLD consist of calcite (CaCO3), Dolomite ($CaMg(CO_3)_2$), Cesamite ($Ca_2Na_3(SO_4)_3(OH)$), Pirssonite ($Na_2Ca(CO_3)_22H_2O$) and Brucite ($Mg(OH)_2$). Manskinen et al 2011 and Novais et al 2018 indicate that Pirssonite is the dominant mineral in GLD, while Martins et al. (2007) suggest calcite is the most abundant phase. Other characteristics of GLD are as mentioned a high pH (10–11), relatively high porosity (73 – 82%), a bulk density of 0.44–0.67 g/m^3 and a compact density of 2.47 to 2.60 g/cm^3. Sequential extraction has been performed on GLD and indicates relatively low bioavailability of metals in general (Nurmesniemi et al. 2005). In contradiction to this and other studies (i.e., Mäkelä and Höynälä 2000), a study made by Bandarra et al.

(2019) denoted GLD as a "possible hazard" according to the chemical analysis and the biotests made in the study indicated high ecotoxic effects for three out of five organisms. However, in this study the purpose is to use the GLD as an amendment to till, requiring only a small amount of GLD in the mixtures (< 20 wt%). The material is also to be used in the sealing layer which purpose is to retain the water reaching it, not flushing it out, meaning that there should not be much chemical leachate from the GLD reaching out to the biosphere.

Reducing oxygen diffusion to the mine waste is the main important factor deterring the formation of ARD, and oxygen has two main ways of transport in a soil: through the water, or the air in the pores. Instead of measuring oxygen diffusion, hydraulic conductivity is usually used to evaluate the effectiveness of a sealing layer in mine waste remediation in Sweden. This as oxygen diffusion is more difficult to measure in field, mainly due to leakage of air through the instrumentation. The requirements for hydraulic conductivity in a sealing layer are site specific, but commonly the requirement in Sweden is below 10^{-8} m/s. An important factor influencing the hydraulic conductivity is the presence of fine-grained material. An increasing proportion of fines in the material decreases the hydraulic conductivity (Benson et al. 1994; Benson and Trast 1995; Leroueil et al. 2002), as the porosity of the material decreases. The water retention capacity (WRC) is another important feature of a sealing layer material, as a high WRC usually corresponds to a high water-saturation. In general, the oxygen flux rates are at a minimum when the degree of saturation is greater than 85–90%, this as the air-phase at a saturation greater than 85% becomes discontinuous (Corey 1957) and the oxygen is then transported through the water phase (Aubertin and Mbonimpa 2001; Aachib et al. 2004). One way to estimate the WRC is to use a soil–water characteristic curve (SWCC), which demonstrates the correlation between the matric suction (ψ) and the water content or the degree of saturation. The matric suction required to begin draining a fully saturated soil is called the air-entry suction (ψ_a). The residual water content (θ_r) is defined as the amount of water in the soil that cannot be removed even at great suction heads, due to adhesive forces between the water molecules and the soil particles, or due to entrapment in disconnected pores. Factors controlling the shape of the curve are mainly the type of the soil (Fredlund and Rahardjo 1993; Tinjum et al 1997; Sillers et al. 2001), but also the molding water content is an important factor (Vanapalli et al. 1996, 1999; Tinjum et al. 1997).

The long-term stability of the GLD has been studied by Mäkitalo et al. (2016) and suggest that the low shear strength of GLD increases over time, but not enough to ensure a long-term physical stability in slopes. Considering its lack of long-term physical stability, it is not reasonable to solely use GLD in the sealing layer from a geotechnical point of view. However, using GLD as an amendment to a local till is a possibility that have been studied by Hargelius (2008), Mäkitalo et al (2015a and b) and Virolainen et al (2020) and have shown promising results with decreasing hydraulic conductivity, increasing WRC, and increasing compaction degree. The properties of the till and GLD are however not homogeneous. The contents of fines and clays are one example of factors that varies in the materials. It has a great potential to affect the final hydraulic conductivity of the mixture and has not been studied previously in GLD-amended till. Another gap in this field of research is the effect of the till and its physical properties (i.e., fines- and clay-content) on the final hydraulic conductivity and water retention capacity of the mixtures in its own. Which makes it difficult to conclude if the amendment of GLD really improves the sealing layer qualities of the till. There is, therefore, a great need for further studies on how these factors control the final till-GLD mixtures and at which percentages the materials should be mixed with each other. A maximum of 20 weight percentage (wt%) of GLD addition was set as a limit in this study, as a mixture with more GLD becomes difficult to compact and handle, due to increased water content above the optimum molding water content and decreased shear strength. 5 to 20 wt% of GLD from Smurfit Kappa paper mill were mixed with three sieved tills (< 20 mm) with different contents of fines and clays, with the objectives to find out (i) if GLD can improve the hydraulic conductivity (below 10^{-8} m/s) and WRC of three tills with different particle size distributions so they can be used in a sealing layer on top of sulfidic mine waste, and (ii) how the contents of fines and clays in three different tills will affect the hydraulic conductivity and WRC in the till-GLD-mixtures.

Table 1 The total solid content (TSC), particle density, sand-, fines- and clay contents in the mixtures

	GLD addition(wt.%)	TSC (%)	Particle density (g/cm^3)	Sand (< 2 000 µm)	Fines (< 63 µm)	Clays (< 2 µm)
SaT	0	91.7 ± 0.4 (N = 9)	2.68	73%	14 ± 1% (N = 3)	0.7% (N = 1)
	5		2.67	70%	16%	0.9%
	10		2.66	68%	19%	1.2%
	15		2.65	65%	22%	1.4%
	20		2.64	63%	25%	1.6%
SiT1	0	91.5 ± 0.4 (N = 9)	2.71	49%	34 ± 5% (N = 9)	2.6% (N = 1)
	5		2.70	48%	33%	2.8%
	10		2.68	47%	35%	2.9%
	15		2.67	46%	37%	3.1%
	20		2.66	45%	39%	3.2%
SiT2	0	91.1 ± 0.8 (N = 6)	2.70	54%	35 ± 1% (N = 2)	4.3% (N = 1)
	5		2.69	52%	33%	4.3%
	10		2.68	51%	35%	4.4%
	15		2.67	49%	37%	4.5%
	20		2.66	48%	39%	4.5%
GLD		43 ± 4 (N = 12)	2.63	24 ± 29% (N = 3)	76 ± 29% (N = 3)	5.4 ± 4.1% (N = 3)

N = number of analyzed samples, SaT-, SiT1 and SiT2-0 to 20 is the sandy till-, silty till- and silty till 2 with 5 to 20 wt% GLD added. For the till-GLD mixtures the particle density and particle size distribution (PSD) were calculated by combining data from the separate materials

Table 2 Chemical elements (mg/kg) in the GLD used in this study and the Swedish guidelines for contaminated soil (*; Swedish EPA 2016)

Ag	Al	As	Au	B	Ba	Be	Bi	Br	Ca	Cd
1 ± 0	7 100 ± 1 600	0.4 ± 0.1	< 0.01	11 ± 1	270 ± 50	0.2 ± 0.1	0.08 ± 0.01	2 ± 1	73 000 ± 6 000	3 ± 1
25*					300*					15*
Ce	Co	Cr	Cu	Cs	Dy	Er	Eu	Fe	Ga	Gd
3 ± 1	3 ± 0	34 ± 2	60 ± 3	0.3 ± 0.1	0.2 ± 0.1	0.1 ± 0.02	0.05 ± 0.01	2 700 ± 500	2 ± 1	0.2 ± 0.1
	15*	150*	200*							
Ge	Hf	Hg	Ho	I	Ir	K	La	Li	Lu	Mg
< 0.1	0.13 ± 0.06	< 0.01	0.04 ± 0.01	< 0.1	< 0.01	8 500 ± 1 700	2 ± 1	2 ± 1	0.02 ± 0.01	8 800 ± 600
		2.5*								
Mn	Mo	Na	Nb	Nd	Ni	Os	P	Pb	Pd	Pr
3 700 ± 200	0.8 ± 0.1	20 000 ± 600	0.6 ± 0.3	1	18 ± 1	< 0.01	1 200 ± 173	7 ± 1	< 0.02	0.3 ± 0.1
	500*				120*			400*		
Pt	Rb	Re	Rh	Ru	S	Sb	Sc	Se	Si	Sm
< 0.01	31 ± 6	< 0.01	< 0.01	< 0.01	6 630 ± 60	0.4 ± 0.1	0.6 ± 0.1	< 0.5	19 000 ± 6 000	0.2 ± 0.1
Sn	Sr	Ta	Tb	Te	Th	Ti	Tl	Tm	V	W
0.6 ± 0.2	117 ± 6	0.05 ± 0.03	0.03 ± 0.01	< 0.01	0.2 ± 0.1	210 ± 80	0.2 ± 0	0.01 ± 0.01	4 ± 2	0.2 ± 0
									200*	
Y	Zn	Zr								
1.3 ± 0.6	720 ± 50	6 ± 3								
	500*									

Table 3 The main chemical components (%TSC) in the GLD used in this study

TSC	SiO_2	Al_2O_3	CaO	Fe_2O_3	K_2O	MgO	MnO	Na_2O	P_2O_5	TiO_2	Sum
52	0.593	0.493	34.5	0.38	0.296	5.85	1.81	3.1	0.679	0.0148	47.7

2 Materials and Methods

2.1 Materials

Three different tills were used in this study, a sandy till (SaT) and two silty tills with different clay contents, where SiT1 had a lower clay content than SiT2 (Table 1). SiT1 was collected at a till quarry at the Brännkläppen facility in Boden, Sweden. SiT2 from another till quarry outside Boden and the sandy till from Luleå, Sweden. The GLD used in this study derived from Smurfit Kappa paper mill in Piteå, Sweden and was collected in sealed plastic containers to preserve the water content of the material (TSC 43%; Table 1).

The main elements of the GLD were Ca > Na > Si > Mg > K > Al > S > Mn (Table 2). Lime mud added in the process is the source of the high Ca concentrations, and the dominant component in the GLD was CaO, followed by $MgO > Na_2O > MnO > P_2O_2 > SiO_2 > Al_2O_3 > Fe_2O_3 > K_2O > TiO_2$ in significantly smaller proportions (Table 3). For elements that are included in the Swedish Environmental Protection Agencies general guidelines for contaminated soil (Swedish EPA 2016), only zinc shows values above these limits with 720 mg/kg to be compared to the limit of 500 mg/kg (Table 2). However, the material is not to be used in its own, but to be mixed with 80 to 95% till and the Zn values in these mixtures are then expected to be below the limits.

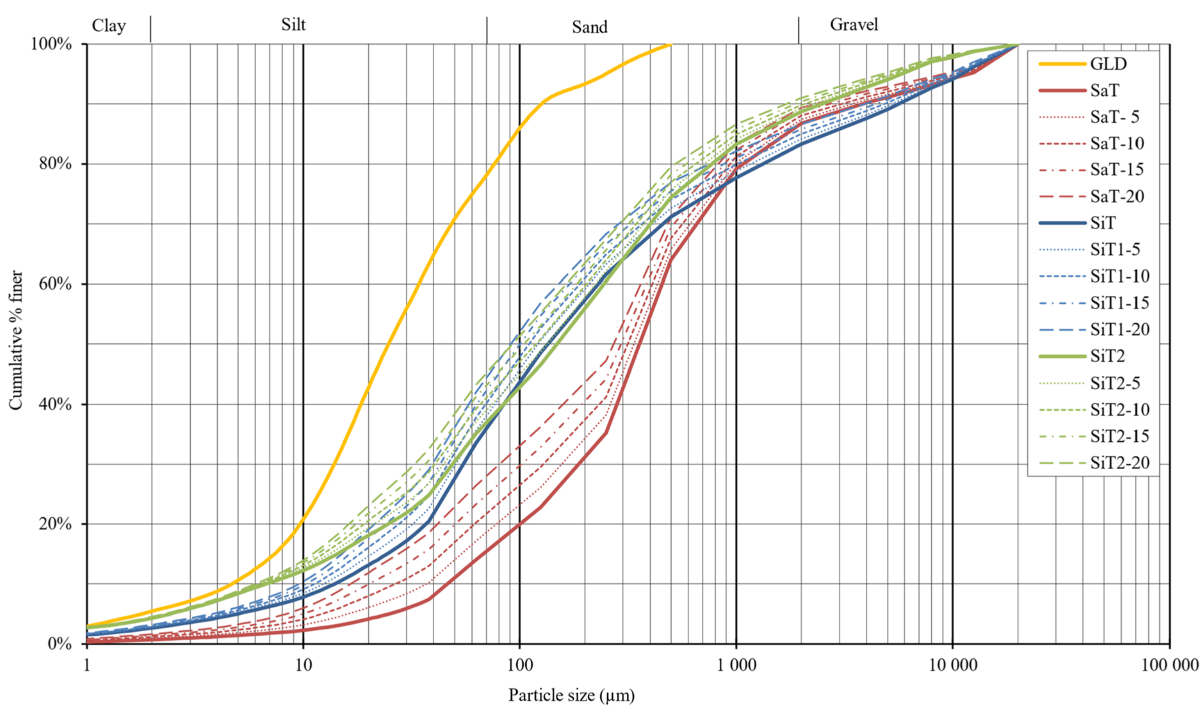

Fig. 1 Particle size distribution (PSD) curves of the three different tills (SiT1, SiT2 and SaT), the GLD and their mixtures (5–20 wt% addition of GLD)

2.2 Mixing of Till and GLD

The tills were air dried until TSC of 100% and sieved through a 20-mm sieve. Particles above 20 mm were removed, which is praxis in a material that is to be used in a sealing layer as they might affect negatively on the effectiveness of the sealing layer. The sieved tills were then mixed with 5-, 10-, 15- and 20 weight percentage (wt%) of GLD. The wt% was calculated towards a dry till and a naturally moist GLD. The GLD was kept naturally moist (TSC 43%; Table 1) as previous unpublished studies has shown that the material might be difficult to rewet without affecting its physical properties. The mixing was carried out by hand with a small shovel until the mixture was estimated as homogenized.

2.3 Particle Size Distribution (PSD)

The tills were washed and dry sieved according to SS-EN 933–1:2012 to obtain the weight percentage of fines. A mechanical sieve tower (Retsch AS 200) with an amplitude of 2.2 mm/"g" was used. The cut-off sizes were 12.5-, 10-, 8-, 5-, 4-, 2-, 1-, 0.5-, 0.25-, 0.125- and 0.063 mm.

Particle size distribution (PSD) for the fines (< 0.063 mm) in the tills and GLD were done by laser diffraction analysis on triplicate samples of each material by a CILAS Granulometer 1064 (CILAS, Orléans, France). The PSD was then calculated using the CILAS software. The PSD for the mixtures of the materials were calculated by combining the CILAS-data from the pure till and GLD (Fig. 1; Table 1).

2.4 Particle Density

The particle density of the materials was determined using an AccuPyc II 1340 Pycnometer. For the mixtures of till and GLD, calculations were done by combining the data from the pure till and GLD (Table 1).

2.5 *Total Solid Content*

The total solid content (TSC) of the materials were performed according to the SIS standard SS-EN 14,346:2007 (Table 1).

2.6 Proctor Compaction

Proctor compaction tests were carried out according to standard SS-EN 13,286–2:2010, to obtain the maximum dry density and optimal water content of the till-GLD mixtures.

2.7 Hydraulic Conductivity

Prior to hydraulic conductivity-analysis, water was added to the tills until they reached a TSC (total solid content) of 91% (± 1%, N = 24). 5, 10, 15 and 20 wt% of GLD was then added to the till. The constant head-method was used (Sandoval et al. 2017) in air-tight cylinders with a volume of 996 cm^3. The walls of the cylinders were coated with a thin layer of bentonite to prevent preferential wall flow. The mixtures inside the cylinder were compacted with standard proctor compaction (PC) method.

Water was directed to the bottom of the cylinder with a hydraulic gradient of 8.7 for SiT1 and SaT. The hydraulic gradient for SiT2 was 13 due to a much lower hydraulic conductivity. The water passing through the cylinders were collected in plastic bottles, sealed from the top to prohibit evaporation. The plastic bottles were weighed regularly, and the time was noted to measure the velocity of the water passing through the sample. The hydraulic conductivity was calculated using Darcy's law.

2.8 Water Retention Capacity

Water retention capacity was measured with a pressure plate test using a 1500F1 Pressure Plate Extractor. The air-dried till used in the mixtures to be tested was wetted to reach a TSC of approximately 91%. Specimens were compacted by hand with 25 blows in five layers using a metallic compactor (2 kg). The goal was to reach an even compaction between the samples and simulate standard proctor compaction. When compaction was done, the samples were levelled off with a knife and weighed.

The samples were placed on saturated ceramic plates with 15 bar air entry value, which base was connected to the atmosphere. Gas pressure was applied in 12 increasing steps, up to a pressure of 145 mH_2O to the samples. One sample was taken out after 48 h for each pressure step and characterized

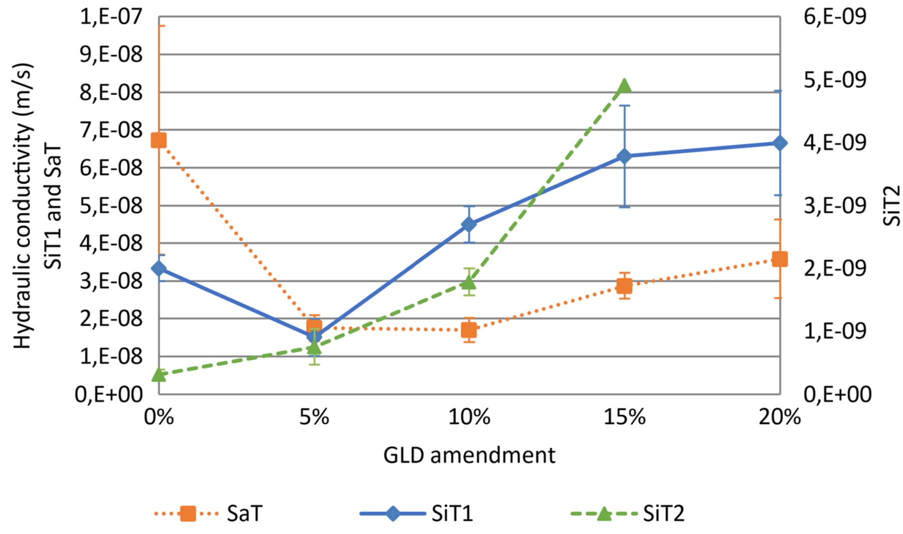

Fig. 2 The hydraulic conductivity in the different mixtures of till and GLD, where the silty till 1 (SiT1)- and the sandy till-mixtures (SaT) are on the left y-axis and the silty till 2-mixture (SiT2) on the right y-axis. The amount of GLD in the mixtures are presented as weight percentage

Table 4 A summary of the data from the hydraulic conductivity and compaction studies with hydraulic conductivity (m/s), dry density (ρ_d; g/cm^3), compaction degree (R_D; %), water content (w; %), porosity (%), maximum dry density ($\rho_{d;}$; g/cm^3), optimum molding water content (omw; %)

Materials		Hydraulic conductivity					Compaction properties	
	GLD (wt. %)	Hydr. cond. (m/s)	ρ_d (g/cm^3)	R_D (%)	w (%)	Porosity (%)	Max. ρ_d (g/cm^3)	omw (%)
SiT1	0	$3\times10^{-8}\pm3\times10^{-9}$ (N=3)	2.01 ± 0.05 (N=3)	97	9 ± 0.5 (N=3)	26	2.08 ± 0.01 (N=3)	6
	5	$2\times10^{-8}\pm5\times10^{-9}$ (N=3)	1.97 ± 0.02 (N=3)	96	10 ± 0.6 (N=3)	31	2.05 ± 0.01 (N=2)	8
	10	$4\times10^{-8}\pm5\times10^{-9}$(N=3)	1.88 ± 0.01 (N=3)	95	14 ± 1.3 (N=3)	30	1.99 ± 0.01 (N=2)	9
	15	$6\times10^{-8}\pm1\times10^{-8}$(N=3)	1.74 ± 0.05 (N=3)	90	18 ± 0.4 N=3)	35	1.95 ± 0.00 (N=2)	10
	20	$7\times10^{-8}\pm1\times10^{-8}$(N=3)	1.65 ± 0.02 (N=3)		20 ± 0.7 (N=3)	38		
SiT2	0	$3\times10^{-10}\pm8\times10^{-11}$ (N=3)	2.12 ± 0.01 (N=3)		10 ± 0.1 (N=3)	22		
	5	$8\times10^{-10}\pm3\times10^{-10}$ (N=3)	1.95 ± 0.01 (N=3)		14 ± 0.5 (N=3)	27		
	10	$2\times10^{-9}\pm2\times10^{-10}$(N=3)	1.87 ± 0.03 (N=3)		15 ± 1.0 (N=3)	30		
	15	5×10^{-9}	1.67 (N=1)		21	35		
	20							
SaT	0	$7\times10^{-8}\pm3\times10^{-8}$ (N=3)	2.04 ± 0.02 (N=3)	100	9 ± 0.6 (N=3)	25	2.03 ± 0.01 (N=3)	8 ± 0,005 (N=3)
	5	$2\times10^{-8}\pm3\times10^{-9}$ (N=3)	1.99 ± 0.03 (N=3)	99	11 ± 1 (N=3)	25	2.01 ± 0.00 (N=2)	9 ± 0.002 (N=2)
	10	$2\times10^{-8}\pm3\times10^{-9}$(N=3)	1.92 ± 0.02 (N=3)	97	14 ± 0.3 (N=3)	28	1.98 ± 0.02 (N=2)	11 ± 0.013 (N=2)
	15	$3\times10^{-8}\pm3\times10^{-9}$(N=3)	1.78 ± 0.05 (N=3)	92	18 ± 1 (N=3)	33	1.93 ± 0.02 (N=2)	12
	20	$4\times10^{-8}\pm1\times10^{-8}$(N=3)	1.64 ± 0.04 (N=3)		21 ± 0.4 (N=3)	38		

The amount of GLD in the mixtures are presented as weight percentage (wt. %)

for its water content according to standard SS-EN 14,346:2007. Water retention curves were produced using the van Genuchten equation (Van Genuchten 1980).

3 Results and Discussion

3.1 The Possibility for GLD to Reduce the Hydraulic Conductivity and Increase the WRC of Sandy-

and Silty Tills

The hypothesis was that the addition of GLD would reduce the hydraulic conductivity of the tills that alone would not reach the for sealing layers on top of sulfidic mine waste in Sweden required hydraulic conductivity of $< 10^{-8}$ m/s. Previous studies have shown promising results for a GLD amended till to be used as a sealing layer, with decreased hydraulic conductivity, increased WRC and increased compaction properties (Hargelius 2008; Mäkitalo et al. 2015a and b). After a certain limit of GLD addition (10–15 wt%) the high water-content and low shear strength of the GLD was expected to decrease the compaction degree and therefore increase the hydraulic conductivity of the mixtures. The hydraulic conductivity of silty till 1- (SiT1) and the sandy till (SaT) mixtures did improve (decrease), but not enough to reach below the in Sweden commonly required 10^{-8} m/s (Fig. 2; Table 4).

The decrease in hydraulic conductivity with addition of GLD to two of the tills is smaller than expected, and even an increase in hydraulic conductivity with GLD addition was seen in one of the tills (SiT2; Fig. 2; Table 4). One reason for the unexpectedly low impact of hydraulic conductivity with addition of GLD might be the method of compaction that was chosen. In this study a standardized proctor compaction method was selected to limit the differences between the samples and to simulate compaction in field. One disadvantage with using proctor compaction, is that a small margin on the sides of the cylinder is not properly compacted as the weight does not reach all the way to the sides. This can lead to a preferential flow along the edges of the cylinder and a higher hydraulic conductivity than in the field. In the study by Mäkitalo et al (2015a and b) the compaction was made by hand. However, if and how much the compaction method affects hydraulic conductivity is further investigated in a study by Nigéus et al. (2023a). The study by Mäkitalo et al. (2015a and b) conducted no hydraulic conductivity measurements on pure till, which makes comparison between the studies difficult, and it cannot be said if the hydraulic conductivity improved with GLD addition to the till used in the study. Another reason for the unexpectedly low decrease of hydraulic conductivity when adding GLD to the tills might be the heterogeneity in the mixtures due to GLD aggregation during mixing. This leads to that the GLD does not fill up the pores of the till, as expected, and an increase in porosity is seen instead of a decrease the more GLD is added to the till (Table 4). This aggregation of the GLD in till was also observed and discussed in a study by Virolainen et al. (2020).

A decreasing trend in hydraulic conductivity followed by an increase is seen in this study. This was expected due to the high water content of the GLD. First the hydraulic conductivity of the silty till-1 (SiT1) decreased from 3.5- to 1.5×10^{-8} m/s with an addition of 5 wt% GLD, to then increase up to 7×10^{-8} m/s with up to 20 wt% GLD in the mixtures (Fig. 2; Table 4). In the sandy till the hydraulic conductivity decreased from 7 to 2×10^{-8} m/s when adding 5 to 10 wt% of GLD, to then increase to 4×10^{-8} m/s when adding up to 20 wt% of GLD. However, only the hydraulic conductivity in silty till-2 (SiT2) meets the common requirements of a sealing layer in Sweden, i.e., a hydraulic conductivity below 10^{-8} m/s. However, the hydraulic conductivity increased (instead of decreased as expected) from 3×10^{-10} to 5×10^{-9} m/s with up to 20 wt% of GLD addition (Fig. 2; Table 4). The increase in hydraulic conductivity is unexpected and can partly be explained by the formation of aggregates discussed in the previous section. It might also be explained by that the porosity of this clayey silty till (SiT2) is already low and an addition of GLD will not decrease it any further as there is very little pores in the right size to be filled. Evidence of this can be seen in Table 4, where the porosity is increasing the more GLD is added to the tills. An addition of GLD could then only lead to an increase in the water content above the optimum molding water content causing the hydraulic conductivity to increase. This is further discussed in Sect. 3.2 and an article by Nigéus et al. (2023a).

Considering WRC, the hypothesis was that the addition of GLD would improve the WRC of the tills that alone would not reach the requirement of a sealing layer (hydraulic conductivity $< 10^{-8}$ m/s). This due to the high initial WRC of the GLD (Mäkitalo et al. 2014). The SWCC for the different till-GLD mixtures shows that WRC does improve with GLD addition (Fig. 3; Table 5). At 85% S_r the matric suction (ψ_{85}) increased from 3.2 to 6 mH_2O with 15 wt% GLD addition to the SiT1 (Fig. 3A–D; Table 5), 3.3 to 26 mH_2O in the SaT mixtures

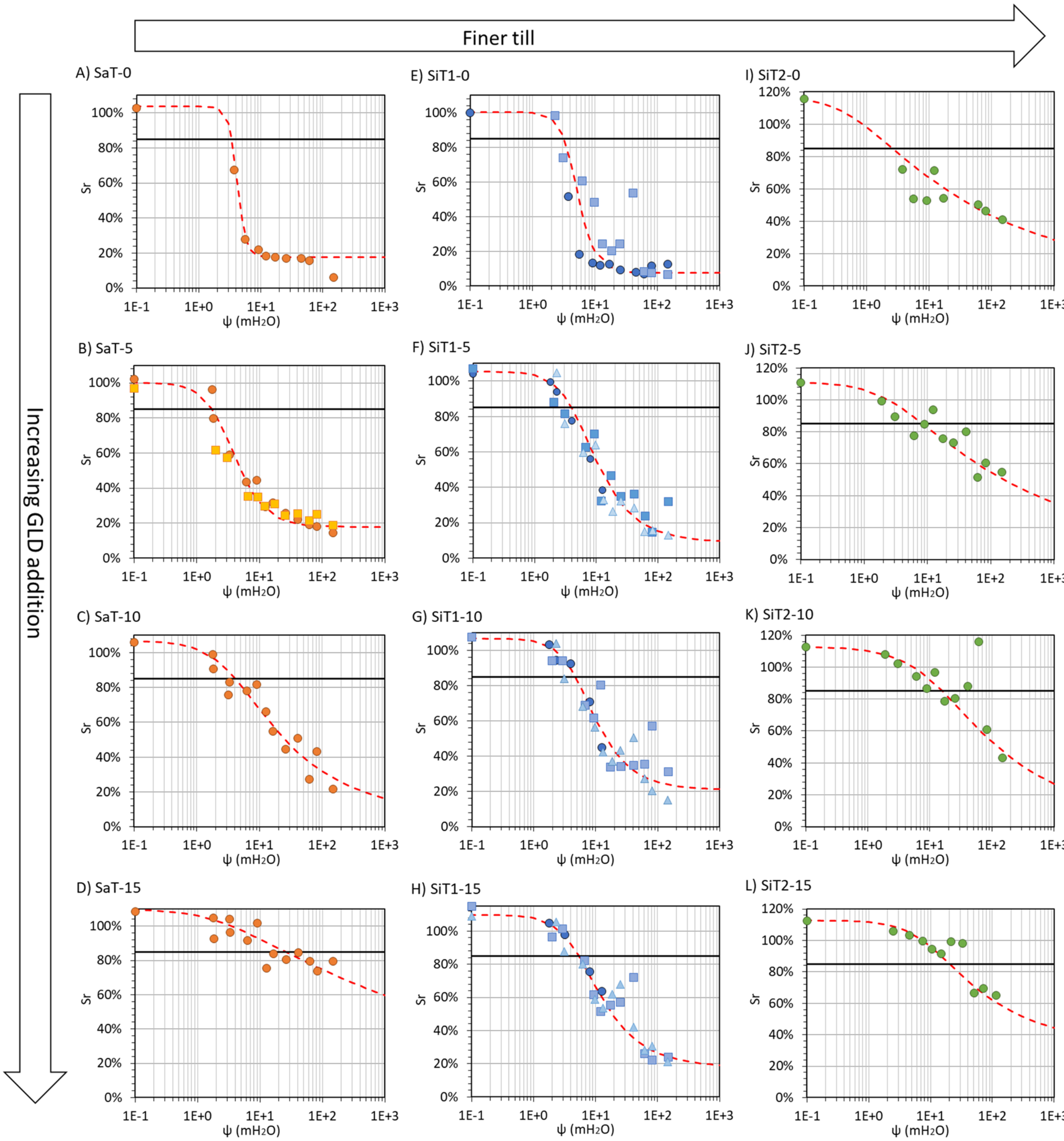

Fig. 3 WRC in the mixtures of GLD and silty till 1 (SiT1), sandy till (SaT), and silty till 2 (SiT2). On the x-axis is the suction in meter water column and on the y-axis the water saturation. Each mixture has been run 1–3 times and each run is marked with a different geometric figure in the graph. The red line is from the van Genuchten equation, best fit, and the black line represent the 85% water saturation. 0 m suction is in the graphs 0.1 m suction due to the logarithmic scale

(Fig. 3E–H; Table 5) and from 3.6 to 50 mH_2O in the SiT2 mixtures (Fig. 3 I–L; Table 5). The more GLD that was added the higher WRC was achieved in the mixtures. Studies has shown that to minimize oxygen diffusion the sealing layer should be kept at a saturation degree of at least 85% (Corey 1957,

Table 5 Key parameters in the SWCC

Materials		$\psi_{85\%}$	ψ_a	ψ_r	θ_r	θ_s	n	m	α	ρ_d	w	R_D
SaT	0	3.4	2.8	6.3	0.06	0.35	6.5	0.85	0.25	1.77	0.07	87
	5	1.7	1.2	14	0.06	0.338	1.9	0.80	0.3	1.83	0.12	91
	10	4.1	1.5		0.02	0.36	1.4	0.29	0.3	1.81	0.16	91
	15	25	0.9		0.005	0.37	1.1	0.09	0.5	1.77	0.19	92
SiT1	0	3.2	2.5	10	0.025	0.34	3.5	0.71	0.22	1.8	0.09	87
	5	4	2	45	0.03	0.36	1.9	0.47	0.2	1.95	0.12	95
	10	4.7	2.3	40	0.07	0.36	2	0.50	0.19	1.83	0.15	92
	15	5.3	2.5	50	0.06	0.37	1.8	0.44	0.19	1.78	0.19	91
SiT2	0	2.6			0.09	0.34	1.5	0.33	0.9	1.92	0.07	
	5	8.4			0.01	0.34	1.28	0.22	0.1	1.89	0.13	
	10	15.7			0.00001	0.34	1.37	0.27	0.04	1.86	0.17	
	15	20			0.005	0.34	3	0.67	0.012	1.75	0.2	

Van Genuchten parameters (m; n; α), matric suction for 85% saturation ($\psi_{85\%}$; mH_2O), the air entry suction (ψ_a; mH_2O), the residual suction (ψ_r; mH_2O), residual water content (θ_r; mH_2O), saturated water content (θ_s; mH_2O), dry density (ρ_d; g/cm^3), water content (w; %) and compaction degree (R_D; %). SaT/SiT1/SiT2-0 to 15 is the sandy till and the silty till 1 and 2 with 0–15 weight percentage of GLD added

Aubertin and Mbonimpa 2001; Achib et al. 2004; Virolainen et al. 2020) and with 15 wt% GLD addition to the tills a high saturation was kept even at high suction (Fig. 3).

For many mixtures the saturation is greater than 100%. The same overestimation in saturation of till-GLD-samples could be seen in a study by Virolainen et al. (2020) indicating that measurement errors on the specimen volume or water content during the setup or dismantling of the diffusion cells is an unlikely explanation. A more likely explanation might be the GLDs tendencies to shrink when drying (Gapak et al. 2017). The result from a volumetric shrinkage curve (VSC) might therefore be included in the SWCC in future studies (Fredlund 2002; Wijaya et al. 2015), where the VSC present the relationship between the volume of the specimen and the corresponding water content. The hysteretic effect might also be a parameter that impacts the interpretation of the SWCC, and the actual WRC of the mixtures studied. The hysteretic effect will give higher matric suction for drying than for wetting for the same water content (Tinjum et al. 1997), and the ψ_{85} will thus be higher. This could lead to an underestimation of the WRC in laboratory experiments compared to actual values in field.

To summarize, the hydraulic conductivity and the WRC for tills that does not meet the required hydraulic conductivity for a sealing layer material ($<10^{-8}$ m/s) improves with GLD addition. However, the improvement in hydraulic conductivity is not enough to reach the requirements. The WRC on the other hand shows promising results, especially for 15 wt% GLD addition and the WRC can be seen as the most important parameter minimizing the oxygen diffusion through a sealing layer, by keeping the layer saturated. The WRC measurements results show an increase the more GLD is added. However, due to compaction difficulties more than 20 wt% is not recommended if the GLD is not dried beforehand. According to a study conducted by Jia et al. (2019) indicates that GLD could be dried without affecting its physical properties after rewetting, regarding hydraulic conductivity and WRC. It is however worth considering that another previous study (Mäkitalo et al. 2016) has shown that GLD could lack long term stability in slopes due to its low shear strength, suggesting that there is still a limit for how much dried or wet GLD can be added to the till, and thus needs further investigations. In future studies it would also be of interest to connect WRC (i.e., ψ_{85}) with actual results from oxygen diffusion, to see what WRC is required to get minimum oxygen diffusion through the material.

3.2 The Effect the Contents of Fines- and Clay in Till Have on Hydraulic Conductivity and WRC in the Till-GLD Mixtures

PSD of the GLD showed that the majority was finer than 63 µm (Fig. 1 and Table 1). The silty till-1 (SiT1) consisted of 35% fines of which 2.6% was in the clay fraction. Silty till-2 (SiT2) had the same percentage of fines as the silty till-1, but a higher percentage of these were in the clay fraction, 4.3%. The sandy till (SaT) had a lower percentage of fines and clays (14% fines and 0.7% clay; Fig. 1 and Table 1).

A decrease in hydraulic conductivity is seen for SiT1 and SaT with addition of GLD up to a wt% of 5–10 (Fig. 2; Table 4). This was expected as the GLD increases the fine-grained material of the mixtures which results in decreasing hydraulic conductivity (Benson et al. 1994; Benson and Trast 1995; Leroueil et al. 2002). The hydraulic conductivity of the SiT 2 does not decrease with the addition of GLD, likely due to that the tills initial clay content leaves no space for the GLD in its micropores. Evidence of this can be seen in Table 4, where the porosity increases, not decreases (as expected) with increasing GLD-addition to the till. Therefore, the addition of GLD to the SiT2 is only deteriorating the compaction degree of the mixture due to an increase in water content, leading to a higher hydraulic conductivity. The same mechanism increases the hydraulic conductivity when more than 5 and 10 wt% of GLD is added for the SiT1 and the SaT. This is further investigated in an article by Nigéus et al (2023a) that follow up the results from this study.

The porosity of the till as a heterogenic material and a dominant part of the mixture, was expected to have a significant impact on especially the hydraulic conductivity of the mixtures. The finer the till is, the lower the hydraulic conductivity in the mixture was expected. The higher clay content in SiT2 is likely the reason for the much lower hydraulic conductivity compared to the silty till 1 (Fig. 2; Table 4). In a study conducted by Leroueil et al. (2002) the hydraulic conductivity decreased several orders of magnitude when the clay size fraction increased from 2 to 12%. Also, a study conducted by Benson et al. (1994) showed a strong relationship between clay content and hydraulic conductivity and a weak relationship between fines and hydraulic conductivity when studying clay.

For the WRC, an increase in air entry suction (ψ_a) and a decrease in porosity (*n*) was expected, with increasing GLD addition. This as materials with smaller pores generally has a higher ψ_a (Fredlund and Rahardjo 1993; Tinjum et al. 1997; Sillers et al. 2001) and a flatter slope of the SWCC in the desaturation zone (Sillers et al. 2001). This is a general trend in these experiments as well (Table 5; Fig. 3). However, comparing the tills with no GLD added, the SiT should have the highest ψ_a. This is not the case. The tills have similar ψ_a, 2–3 mH_2O (Table 4). In fine-grained soils compacted dry of optimum water content aggregation has shown to impact the ψ_a and the slope of the SWCC (Vanapalli et al. 1999). The soil will then act more like a coarse-grained soil due to its highly aggregated macrostructure, with steeper SWCC and lower ψ_a (Vanapalli et al. 1999). The SiT2 is in fact highly aggregated, but due to shortage on material there is no data on the optimal water content for this material. The dry density is another factor that has shown to have significant impact on the SWCC due to a decrease in the number of voids (i.e., decreased porosity) with increasing dry density (Table 5), leading to faster saturation (Vanapalli et al. 1999). However, the differences in dry density between mixtures is not great (Table 5) and cannot explain the lack of a higher WRC of the SiT2, compared to the SiT1 and the SaT. Neither can it explain the higher WRC in the SaT-15-mixture compared to the SiT1-15-mixture. Mineralogy is yet another factor that is known to affect the SWCC of a soil (Mitchell 1993; Tinjum et al. 1997) and needs further investigation in future studies.

In summary this study indicates that there are other factors than the PSD of the materials that controls the hydraulic conductivity of the mixtures, such as initial water content, dry density and WRC. Of which the first two parameters are studied further in an article by Nigéus et al (2023a). The high WRC in the till-GLD mixtures with 15 wt% GLD addition show promising results for GLD to be used in a sealing layer, even if the hydraulic conductivity did not reach the requirements. A high saturated sealing layer is the key factor of deterring oxygen diffusion to the mine waste, which can be achieved with a high WRC in the sealing layer material.

4 Conclusions

Adding GLD to tills that on its own does not reach the requirements for a sealing layer on top of sulfidic mine waste (i.e., $< 10^{-8}$ m/s) improves its sealing layer properties, i.e., decreases hydraulic conductivity and increases water retention capacities. However, the improvement in hydraulic conductivity was not enough to reach the in Sweden required $< 10^{-8}$ m/s. Despite the lack of sufficient decreases in hydraulic conductivity, the water retention capacity of the mixtures can be seen as the most important factor controlling the formation of ARD by keeping the sealing layer close to saturation. This study shows that GLD can beneficially be used in a sealing layer in mine site remediation as the till-GLD mixtures can keep a high saturation (< 80%) even with high matric suction (up to 25 mH_2O; SaT-20; Table 5) and that the water retention capacity increases with increasing amount of GLD added to the tills. Recycling of GLD in mine remediation is to strive for as it is beneficial for not only the mining company, but also the pulp and paper industry as it minimizes the waste landfilled. The use of an industrial waste as a product is however challenging as there is a great variability in its chemical compounds and physical properties. GLD varies both between industries, but also between batches, within the same industry (Nigéus et al. 2023b). This creates a challenge as it is difficult to predict its chemical and physical properties which of course affects its effectiveness as a product. This study further indicates that there are other factors than the PSD of the materials that controls the hydraulic conductivity of the mixtures, as initial water content, dry density and WRC. future studies, should investigate how dry density and initial water content of the tills and GLD affects the hydraulic conductivity. It is also of interest to study how the compaction method affects the hydraulic conductivity of a till and GLD-mixture.

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Data availability The datasets generated during and/or analyzed during the current study are not publicly available yet, but are available from the corresponding author on reasonable request.

Declarations

Conflict of interest Author Nigéus S, Maurice C and Lindblom J declare they have no financial interests.

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