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DEWATERING OF SLUDGE USING SUPERCRITICAL CARBON DIOXIDE

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ABSTRACT

Traditional sludge disposal methods pose environmental concerns and are economically expensive due to high moisture content. Dewatering is an important step to address these concerns. Conventional dewatering technologies include thermal drying and mechanical compression of sludge. Recently, dewatering with supercritical CO_2 has started gaining attention as a potential low-cost alternative to these conventional technologies. While many published studies on supercritical CO_2 dewatering are usually in a small reactor volume (~ 0.1 L), sludge dewatering in a relatively large reactor volume (~ 1 L) is demonstrated here as a feasible alternative to existing methods. Influence of equilibrium/ sCO_2 residence time and wet-sludge loading rate on the dewatering performance was determined from three-staged experiments. Within the first stage of dewatering, 40-60% dewatering was noticed. Preliminary techno-economic analysis showed a 5-50% reduction in sludge dewatering cost with supercritical CO_2 in comparison to thermal drying.

KEY WORDS: Sludge dewatering, supercritical carbon dioxide, levelized cost of dewatering (LC)

1. INTRODUCTION

Sludge is a waste by-product from process industries and wastewater treatment plants. In the United States alone, annual dry sludge production is estimated to be about 10 MT, while, the same in European Union and China is estimated at 7.2 MT and 39 MT, respectively [1]. The volume of the sludge disposal is much higher in wet form. Traditional wet sludge disposal methods include incineration, landfilling, ocean dumping, etc. These disposal methods pose environmental challenges and economic constraints [2]. So, facilities must adopt processing methods to address these concerns. Sludge is also a valuable precursor for the agricultural and energy sector for producing energy, biofuels, and biochar [3]. To realize these value-added benefits from sludge, the moisture content of the sludge must be reduced to increase the heating value of the product.

Popular dewatering methods are either based on mechanical dewatering or adopt thermal drying techniques. Other methods like chemical and biological conditioning of sludge have also been explored, but are less common. Mechanical dewatering follows applying mechanical pressure on the porous material to remove water, typically free water, i.e., water outside pore interstices of the sludge [4]. Mechanical dewatering systems are usually centrifuges or screw/ belt filters. These heavy mechanical forces squeeze moisture and compress the sludge to a thick cake with less than 10-15% moisture content. Although the specific energy consumption (SEC) is lower than 100 kWh/m^3 [5], heavy mechanical equipment is needed, so this method is suitable for processing very large volumes due to large system footprints and high capital investment.

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Thermal drying, on the other hand involves heating the sludge to high temperatures to remove moisture. The SEC to remove a unit volume of water, is ~ 700-1400 kWh/m^3 with thermal drying, depending on the type of convective dryer used [6]. Usually, in thermal drying, the dryer surface is heated by hot gases from typical fossil/gas-burning processes. To reduce the fossil fuel consumption for sludge drying, solar-assisted drying methods have been explored, where the specific energy consumption could be only about 200 kWh/m^3. The sludge thermal drying rate varies between 0.2-30 kg/m^2-h [7]. Although thermal drying is a simple and high-efficiency method, it suffers from limitations like long drying times, bad odours, and gaseous emissions. Also, heating sludge to a high temperature poses a risk of cellular structure breakdown [1]. Another form of thermal dewatering is the freeze-thaw method, where dewatering occurs due to solid-water separation [8]. Recent advances in dewatering processes with supercritical carbon dioxide have opened up the possibility of low-cost dewatering of sludge and other porous materials.

Supercritical carbon dioxide (sCO_2) as a dewatering agent has recently gained attention. CO_2 becomes supercritical at low temperatures and moderate pressure around 31.1 °C and 73.8 bar. Intermittent gas-liquid density and low viscosity make sCO_2 favourable for efficient dewatering of porous materials like sludge with low specific energy consumption and cost. The mechanism of sCO_2-based dewatering of porous materials involves the dissolution of bound porous water and forced mechanical displacement of free water & bound water [9]. From Darcy's law of porous media flow, it can be explained that the large difference in the viscosity of the intruding species and the bound species leads to viscous fingering. The intruding species, as a result, forces out the bound fluid from the pores [10]. Recent investigation with porous materials like coal, paper, wastewater sludge, wood, black liquor, etc. has shown tremendous potential for sCO_2-based dewatering in place of traditionally adopted dewatering technologies. Banerjee et al., [10] performed dewatering experiments on bituminous & lignite coal with sCO_2 at 75 °C and 138 bar. They determined that dewatering lignite coal, which has a high moisture content, was beneficial over bituminous coal. Iwai et al., [11] noted an improvement in coal dewatering performance by adding methanol as an entrainer. System operating temperature and pressure play an important role in the dewatering performance of the material with sCO_2. Adjaye et al., [12] performed dewatering experiments on black liquor and lignin, where dewatering efficiency was positively correlated to treatment temperature. They also noted a positive correlation in dewatering performance against the initial moisture content of the material. Adjaye et al., [9] performed dewatering experiments on alum sludge, wastewater sludge, and papermill sludge. At a given temperature, they noted that higher pressure tends to negatively affect the dewatering performance because of the increasing viscosity of the fluids. Recently, sludge dewatered with sCO_2 was found to have improvements in the heating value of the material [13]. Additionally, sCO_2 dewatering can also yield value-added benefits like compound extraction [14]. These findings have garnered interest in investigating and implementing sCO_2-based dewatering. However, the evidence for sCO_2-based dewatering in the above-mentioned literature has been through experiments in a very small reactor volume, i.e., ~ 0.12 L. Also, information on the feasibility of implementing sCO_2-based dewatering for a particular material of interest is scarcely available. This manuscript contributes to the scientific and engineering community in two ways: 1. In this manuscript, dewatering experiments were performed on sludge, an important biomass feedstock of interest, obtained from a recycling facility in a relatively larger reactor volume (~ 10 x larger than above-mentioned works)of 1 liter; 2. From the results obtained, the feasibility of implementing sCO2-dewatering technology for sludge pre-drying is presented by determining the specific energy consumption and the *levelized cost of dewatering* (*LC*). From the analysis, it was determined that sCO_2 dewatering of sludge is competitive, if not, economically beneficial in comparison to thermal drying.

2. EXPERIMENTAL SYSTEM AND PROCEDURE

2.1 Description of sludge

Sludge for dewatering experiments was obtained from KW Plastics, a plastic recycling company. The sludge procured was mostly comprised of bio-degradable food waste. Figure 1 shows a picture of the as received wet sludge from the recycling company and the dry sludge. The as-received sludge had excess free water. So, for experiments, excess water was first drained off by taking sludge in a container with a porous bottom and weighed. The wet sludge was then considered to be saturated with water. Then, the moisture content of the sludge was determined by thermally drying the sludge at 80-100 °C to remove water completely. The dry sludge was then weighed again. The difference in the mass of sludge is the amount of water that was present in the sludge. The moisture content by mass basis is the ratio of the total mass of water in saturated sludge divided by the mass of wet sludge. The moisture content of sludge was determined to be 63±5% for 5 trials.

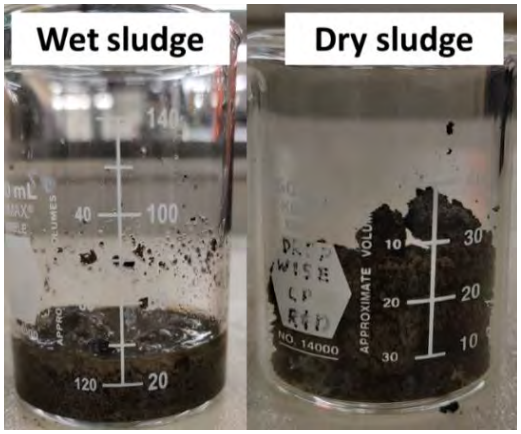

Figure 1. Photograph of as-received wet sludge from the recycling facility and fully dried sludge (after thermal drying several samples and collecting them in the same vessel)

2.2 Description of the experimental System

A lab-scale supercritical fluid (sCO_2) extraction system with a 1 L pressure vessel assembled for dewatering experiments is shown in Figure 2. The sCO_2 extraction unit consisted of a CO_2 pump, a liquid CO_2 cylinder, a refrigeration chiller, a flow distribution chamber, a control unit, and a 1 L pressure vessel. Before the start of the experiments, the pressure vessel was first pre-heated to the desired temperature. The pressure vessel, flow distribution & control unit, and pressure vessel were procured from Applied Separations, Inc. Liquid CO_2 cylinders with CO_2 stored at 60 bar at atmospheric temperature were purchased as the CO_2 source from a provider. The cold liquid CO_2 was then pumped at the desired operating pressure. CO_2 becomes superfluid as it enters the pressure vessel.

Figure 2. Supercritical fluid (CO_2) extraction system *[15]*

Equilibrium three-staged sequential experiments were performed to determine the sludge dewatering performance with sCO_2. The term equilibrium here means sCO_2 residence/ holding time. After dewatering in the first stage, the same batch of dewatered sludge was again dewatered in the subsequent stage. This process was then repeated for the third time. The system operating conditions for sludge dewatering with sCO_2 is highlighted in Table 1.

Table 1. System operating conditions

Operating parameter	Value
Pressure	93 Bar [15]
Temperature	70 °C [15]
Wet sludge loading rate	100 g, 135 g, and 35 g
sCO_2 residence time	1 min, 5 min, 10 min

Two performance parameters were varied during the experiments:

1. Equilibrium/ residence time: dewatering experiments were performed for 1min, 5 min, and 10 min equilibrium time. The sludge loading rate was 100±6 g. This parameter was studied because the equilibrium time is related to the number of batches of sludge (total amount of sludge) that can be dewatered in an hour.

2. Mass of sludge: For 5 mins of equilibrium time, the dewatering experiments were performed for wet sludge loading mass of 35 g, 100 g, and 135 g. The mass loading rate is another parameter that influences the dewatering performance and the feasibility of the process.

3. EXPERIMENTAL RESULTS

3.1 Influence of equilibrium time

Equilibrium three-staged sludge dewatering experiments were performed with sCO_2. The mass of wet sludge in the vessel was 100±6 g. The mass of sCO_2 was then calculated by subtracting the volume of sludge from the vessel with an average sludge density of 1200 kg/m^3 [16]. The mass ratio, i.e., the ratio of sCO_2 to dry sludge was found to be ~ 4.5±1.

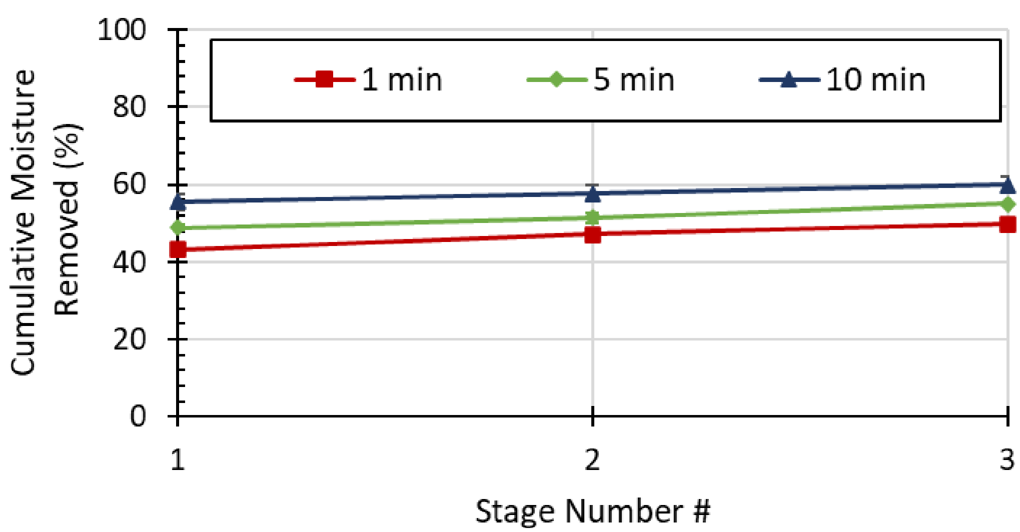

Figure 3. Influence of equilibrium time on sludge dewatering with sCO_2

Figure 3 shows the three-staged sequential sludge dewatering performance with 1 min, 5 min, and 10 min equilibrium time. The cumulative moisture removal rate is the total percentage of moisture removed from the sludge. When the equilibrium time was 1 min, 27.3 grams of water were removed. This corresponds to 43.3% of water present in the sludge. With the two subsequent stages, only 4 more grams of water were removed. The total moisture removed by the end of the third stage was 49.7%. With 5 mins and 10 mins of equilibrium time, 48.9% and 55.7% of water were removed in the first stage. In subsequent stages, the total amount of water removed was 6.1% and 4.3% only. These tests have been repeated for reproducibility and the error bar is provided.

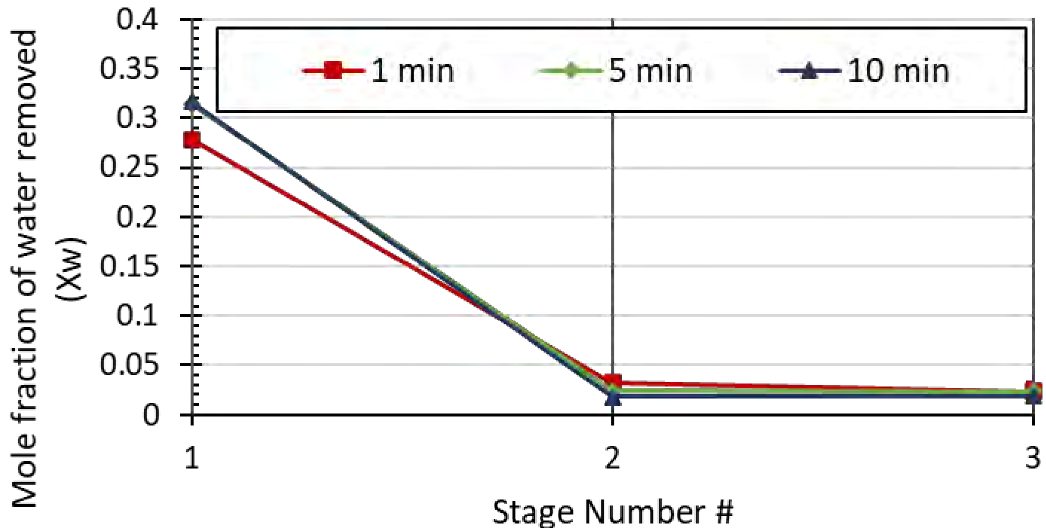

Figure 4. Mole fraction of water removed from sludge by sCO_2

The mole fraction of water removed from equilibrium staged experiments is shown in Figure 4. The mole fraction of water removed by sCO_2 is calculated as:

$$x_w = \frac{\frac{m_w}{MW_w}}{\frac{m_w}{MW_w}+\frac{m_{sCO_2}}{MW_{sCO_2}}} \tag{1}$$

Where, 'm' is the species mass and 'MW' is the species molar mass. In the first stage, the mole fraction of water removed was between 0.28-0.3. At a similar temperature and at a slightly higher pressure of 100 bar, the solubility limit is 0.012. This indicates free water and surface bound water is easily removed by mechanical displacement and potentially aided by the dissolution. In the subsequent stages, water removal rate was significantly lower with mole fraction of 0.015-0.025, which is near the solubility limit. However, this is inconsequential compared to the first stage, which is of significance to determine the feasibility of sCO_2 dewatering. It can be concluded that single staged dewatering of sludge with sCO_2 is sufficient.

3.2 Influence of wet sludge mass loading

The influence of mass loading rate was determined with initial wet sludge loading mass of 100 g, 135 g, and at a lower mass of 35 g. The equilibrium time for the experiments was 5 mins.

Figure 5 shows the influence of wet sludge loading rate on the sCO_2 dewatering performance. The dewatering performance with 135 g loading is slightly better than 100 g loading with a water removal percentage of 56.5% and 49.7%. Similar to three-staged equilibrium dewatering experiments, the majority of the water is removed in the first stage, while, subsequent stages were found to be inconsequential. The water removal percentage with 35 g sludge loading in the first stage was only 19%. In this case, the mass ratio was 15.06, while, with a higher sludge loading mass, the mass ratio was lower than 4.5. Possibly, this shows that there is an optimum sCO_2 to dry sludge mass ratio, but a lower ratio might be preferred for higher dewatering efficiency.

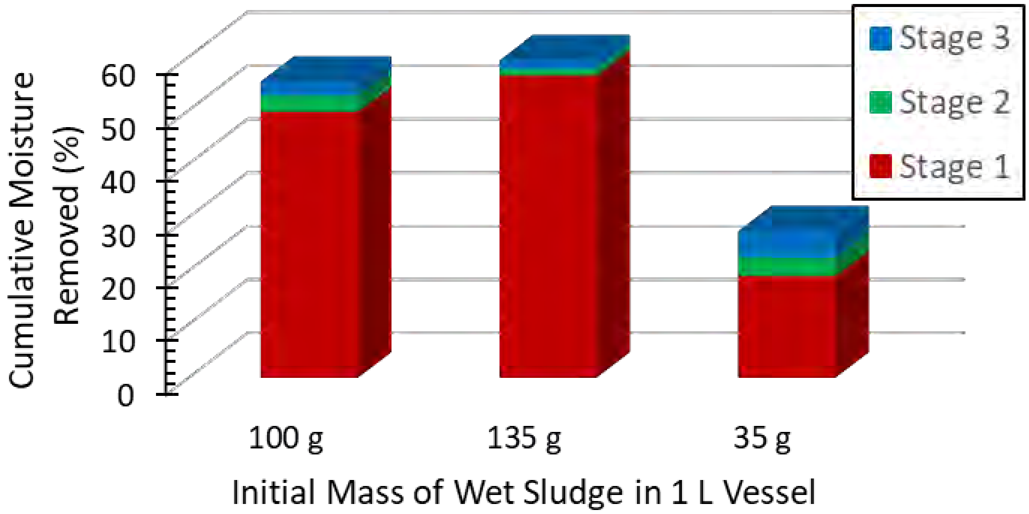

Figure 5. Influence of sludge mass loading on sCO_2 dewatering

3.3 Uncertainty measurement

The instrument error and measurement standard deviation for batch wet sludge loading and dry sludge collected after the experiments is tabulated in Table 2. The instrument error was not provided by the supplier but there was error in the measurement during the experiments. These values were input in programmatic software Engineering Equation Solver to calculate error propagation due to uncertainty. The percentage error in dewatering, was calculated to be 6.32%. The mass of sCO_2 in the pressure vessel due to these uncertainties was 166.7 g with an uncertainty of 9.0 g which resulted in the mole fraction uncertainty of 0.263 with an uncertainty of 0.0464.

Table 2. Instrument and measurement errors during sludge dewatering studies

Parameter	Value	Error (+/-)
Pressure	93 (bar)	3 (bar)
Temperature	70 °C	1 °C
Initial Moisture content	63%	5%
Mass of wet sludge	100 (g)	2.4 (g)
Mass of dry sludge collected	38.3 (g)	2.3 (g)

3.3 Specific Energy Consumption of the sCO_2 dewatering process

The specific energy consumption of the sCO_2 dewatering process was determined by calculating the total energy required to dewater the sludge and is calculated as:

$$SEC = \frac{\Sigma \dot{E}}{vol_w} \quad (2)$$

Where, $\dot{E}$ is the total energy consumption and is the summation of sCO_2 pumping power, chiller power, and thermal heating of sCO_2, and vol_w is the volume of water removed from the material.

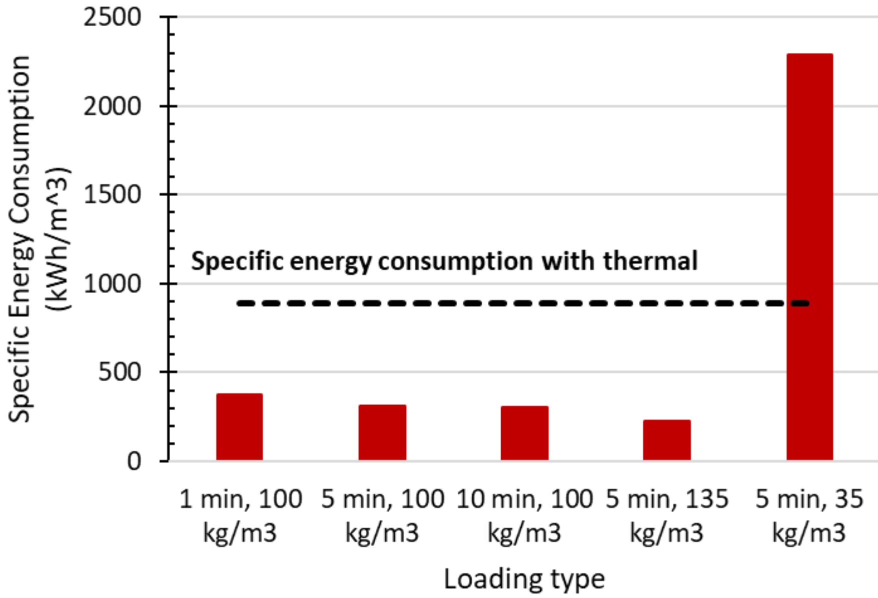

Figure 6. Specific energy consumption for sludge dewatering with sCO_2

Figure 6 shows the specific energy consumption for sludge dewatering system with sCO_2. Due to the variations in the initial moisture content, the mass of water removed, and some variations in pump pressure, the specific energy consumption varies between the lowest value of 227 kWh/ m^3 for a sludge loading mass of 135 g to an undesirable value of 2290 kWh/m^3 for sludge loading rate of 35 g. For 100 g sludge loading mass, the average specific energy consumption is ~ 310 kWh/m^3. This is approximately 3 times lower than the specific energy consumption with thermal drying at ~900 kWh/m^3. SEC of thermal drying was computed by considering average latent heat of vaporization of 2500 kJ/kg at flue-gas heat transfer efficiency of 78%.

4. PRELIMINARY TECHNO-ECONOMIC ANALYSIS

A preliminary economic analysis was performed using the levelized cost approach based on the net present value of the system and accounting for life-time system cost. The levelized cost of dewatering is calculated as:

$$LC = \frac{CAPEX + PVF * OPEX}{Vol_w * PVF * CF} \quad (3)$$

Where, $CAPEX$, $OPEX$, PVF, Vol_w, and CF is the total capital investment (in $), annual operational costs (in $/yr), present value function (in yr). and capacity factor. The cost parameters for the sCO_2 system are described in Table 3. The *CAPEX* accounts for all direct and indirect capital cost of the system. Direct capital cost pertains to the pressure vessel cost and the pump cost. The reactor walls must be sufficiently thick to sustain the high pressure of the system. From literature, it was determined that a thick stainless steel vessel for larger volume pressure vessels will be expensive and thus unfeasible. A 50/50 pre-stressed concrete/ stainless steel vessel was found to be an inexpensive alternative to the expensive stainless-steel vessel [17]. The normalized anticipated cost of this composite vessel was $ 6000/ m^3. The capital cost of the sCO_2 pump was obtained from cost correlations presented by [18]. The indirect capital cost was assumed to be 40% of the system cost [15]. Indirect cost is cost associated with engineering, planning and overhead expenses like insurance while procuring the system. The only operating cost considered was the cost of electricity for running the sCO_2 pump. Nominal capacity factor of 0.8, system discount rate of 5%, and system life of 30 years was used for computing LC.

Table 3. Cost parameters to determine levelized cost of dewatering

Supercritical CO_2 system		
Capital Cost		Notes/ Reference
Pressure vessel: 50/50 pre-stressed novel steel/ concrete composite vessel (SCCV)	$ 6000/ m^3	[17]
Supercritical CO_2 pump	$ (451.37*(KW) – 1769.7)	[18]
Indirect capital cost	40% total capital equipment	[15]
OPEX	$ 0.1/ kWh	Electrical power to run the system
Thermal Drying- Flue gas dryer		
Capital cost	$ 1480/ m^2	[19]
Other cost and system parameters		
OPEX	$ 0.1/ kWh	Electrical power required for pump
Capacity factor	0.8	
Discount rate	5%	
System life	30 years	

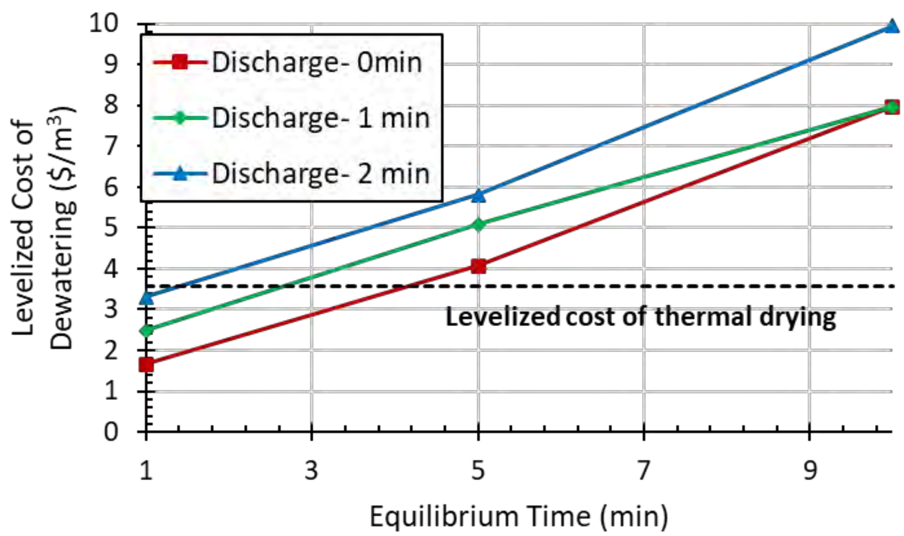

Figure 7. Levelized cost of sludge dewatering with sCO_2

Figure 7 shows the levelized cost of dewatering sludge with sCO_2 considering the first staged experimental results for varying sCO_2 equilibrium time of 1 min, 5 min, and 10 min. The discharge time, i.e., the time for depressurizing the pressure vessel was considered as a parameter of interest in this calculation. So, one dewatering cycle is essentially equilibrium time plus discharge time. Together make up one cycle. The levelized cost must then account for the number of cycles in an hour. Ideally, when the discharge time is zero, the levelized cost of dewatering is the lowest at $ 1.67/m^3. The levelized cost of thermal drying is $ 3.6/m^3. It was observed that an equilibrium time of 1 min realizes levelized cost of dewatering lower than thermal drying by 5% to 50% for the chosen operating conditions: 93 bar, 70 °C, and 100 g sludge loading mass/1 L vessel. From the analysis, it was determined that sCO_2 dewatering is potentially economical for dewatering sludge in comparison to conventional thermal drying.

5. CONCLUSIONS

Effective dewatering of sludge in a lab-scale 1 L reactor volume was demonstrated using supercritical CO_2 as the dewatering agent. Three-staged equilibrium experiments were performed to capture the influence of successive dewatering on the same batch of sludge. Two parameters of interest- equilibrium (residence) time, and sludge loading rate were investigated since they are associated with total amount of dewatering achieved (sludge processed) in a given hour. Experiments were performed at 93±3 bar and 70 °C. Following inferences were drawn from the experiments and economic analysis:

- The first stage of dewatering resulted in between 40-60% of total dewatering depending on the residence time. Subsequent stages removed very less water compared to the first stage. So, one stage of dewatering per batch is sufficient.
- Dewatering performance was marginally higher with a 135 g sludge loading rate in comparison to a 100 g sludge loading rate. The sCO_2 to dry sludge mass ratio was less than 4.5 for both cases. However, with only 35 g of sludge loading, dewatering efficiency was significantly reduced. Here, the sCO_2 to dry sludge mass ratio was 15. Possibly, a lower sCO_2 to dry sludge mass ratio is preferred. More experiments must be performed to determine the optimal loading ratio.
- Preliminary economic analysis based on the levelized cost method showed that sCO_2-based dewatering can be between 5-50% more economically beneficial compared to thermal drying.

From the above analysis, we determine that sCO_2 can be successfully used for the dewatering of porous materials like sludge. However, the experimental results presented are for one particular type of sludge at constant operating pressure and temperature. It is noted that the operating pressure and temperature are important parameters to be optimized along with material loading rate. Also, the optimal operating condition is a strong function of sCO_2 to material loading rate, which is easily influenced by the available moisture content. Determining performance correlations against operating parameters for a material of interest can be used to determine the feasibility of using sCO_2-dewatering technology in comparison to other competing technologies.

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NOMENCLATURE

CAPEX	Capital cost	(\$)	MW	Molar mass	(Kg/mol)
LC	Levelized cost of dewatering	(\$/m^3)	OPEX	Operating cost	(\$/yr)
m	Mass	(Kg)	x	Mole fraction	

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