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Efficient Management of Sewage Sludge from Urban Wastewaters with the Addition of Inorganic Waste: Focus on Rheological Properties

Andreia F. Santos *, Abel G. M. Ferreira and Margarida J. Quina

CIEPQPF, Department of Chemical Engineering, University of Coimbra, Rua Sílvio Lima, Pólo II–Pinhal de Marrocos, 3030-790 Coimbra, Portugal
* Correspondence: affs@eq.uc.pt

Abstract: Sewage sludge (SS) from urban wastewater treatment is still an environmental, economic, and social problem. Current SS management is not consensual, and more alternatives are required to recover some valuable compounds, such as nutrients and organic matter. This study investigates the use of green liquor dregs from the pulp and paper industry—GLDs—as an adjuvant of drying, to develop a product for agronomic applications, focusing on the rheological behavior. The rheological properties were assessed for anaerobically digested sludge (ADS). The limit viscosity of raw ADS was about 0.005 Pa·s in the case of 5% TSs (total solids) increasing to 0.51 Pa·s for 20% TSs. From the oscillatory tests, the ideal viscous flow below 10% TSs was observed, whereas a viscoelastic–solid behavior was detected for a higher concentration of TSs. The addition of GLDs to the ADS reduced the consistency index, reducing the shear resistance of the material. Rheological assays showed that GLDs may facilitate sludge handling (e.g., extrusion) from the dewatering unit to the dryer. Overall, the addition of GLDs to ADS showed to be a viable option for drying and subsequent soil application. Reusing both residues promote the transition from a linear to a circular economy in the wastewater treatment sector.

Keywords: sewage sludge; green liquor dregs; drying; rheology; viscosity; consistency index

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1. Introduction

Currently, the treatment of urban wastewater is a common practice in developed countries to return high-quality water to the natural environment, protecting both human health and the environment. However, wastewater treatment plants (WWTPs) produce large quantities of sewage sludge (SS) that needs proper management. In general, the high water content of SS (around 80% after mechanical dewatering), the presence of some pollutants (e.g., pathogenic microorganisms), and the potential odor emissions hamper transportation, storage, and post-treatment operations (e.g., land application, incineration, composting, and thermal drying) [1–4]. Indeed, this study is part of a project (Dry2Value), which aims to develop an industrial dryer to be used in WWTPs as part of a solution to facilitate the subsequent management phases, namely the application in soil. The drying process can solve the aforementioned problems, allowing to obtain a dry and sanitized product for later use in agricultural applications. Our previous studies explored the characterization of the process from the technical point of view (drying operation) and the prospects of the final product aiming at soil application [3–6]. However, the effect on rheological properties after the addition of inorganic drying adjuvants has not been addressed in our previous studies or in the literature. It is well known that the high viscosity of SS may hinder the transport and handling (spreading process), and thus information on the rheological properties can be crucial for the design of specific equipment, such as feeders, transport lines, extruders, and dryers [7,8]. In this context, the effect of inorganic additives in SS on rheological properties must also be addressed.

SS is a very complex matrix composed of microorganisms (from the biological treatment), colloids, long-chain polymers, and particles of different origins, which makes rheological characterization difficult [9]. According to the literature, SS behaves as a non-Newtonian fluid since the shear stress is not linearly proportional to the shear rate [10–12]. The apparent viscosity (ratio of shear stress to shear rate) concept has been adopted to illustrate each viscosity value obtained, and its behavior is well-described through Power Law detailed by the Otsward-de-Waele or Herschel–Bulkley models. In general, SS exhibits a shear-thinning behavior (pseudoplastic fluid) characterized by a decrease in viscosity, while the shear rate increases. At high shear rates, the samples behave as a Newtonian fluid, and the viscosity becomes almost constant, named "limit viscosity", which is an indicator of internal resistance [11,13]. At high shear conditions, the risk of molecule degradation is high, with a possibility of destroying the original chemical structure. Since the Power Law does not consider these upper and lower Newtonian plateaus observed for low and high shear rates, the empirical equation of Cross allows to overcome this drawback [14,15]. Due to the complex nature of SS and its non-Newtonian behavior, the limit viscosity value for this system can have large variability. Nevertheless, several researchers agree that the total solids content (TSs), bounded water, treatment processes in the WWTPs, and temperature can influence this parameter. In addition, the limit viscosity of SS increases with the total solids content (at a constant temperature) due to the strong interparticle interactions [8,16,17]. According to Pevere et al. (2006) [18], at a constant solids concentration, the viscosity increases with the decrease in particle size since the surface area of particles to interact is superior.

In the literature, some studies evaluated the influence on the rheological behavior of high TSs (≥10%) of activated sludge [19–22] and anaerobically digested sludge [11,22,23]. On the contrary, limited information was found about strategies to reduce the viscosity of dewatered sludge through chemical conditioning with industrial residues. This can be a relevant issue since the dewatered sludge may have a viscosity above 0.104 Pa·s, which requires a large amount of energy for its movement [8]. Thus, any strategy to reduce the viscosity and induce fluidity may be of practical interest. From the background obtained in our previous studies, green liquor dregs (GLDs)—industrial waste from pulp mills—aided the drying process and enhanced the characteristics of the final product, namely reducing the microbiological contamination of SS [3,4]. GLDs are formed at a rate of 10–20 kg/ton of pulp during the clarification of green liquor and contain mainly insoluble material of the recovery boiler inorganic flux (smelt). The main phases are sodium and calcium carbonates, sodium hydroxide, sulfide, and small quantities of other metals. Currently, GLDs are mostly disposed of in industrial landfills, so their reuse is of interest according to the circular economy framework [24]. In addition to the positive impact of GLDs on the parameters mentioned before, it is intended to explore their possible improvement on the rheological properties of SS to facilitate the management operations.

In this context, for the first time in the literature, the present study addresses the effect of GLDs on the rheological properties of anaerobically digested sludge, comparing it with the rheological properties of raw sludge. Moreover, this work integrates the results of the effect of GLDs on drying and final product properties with rheology findings.

2. Materials and Methods

2.1. Sludges Characteristics and Conditioning Procedure

The present study involves two sewage sludge samples from different WWTPs with anaerobic digestion processes (referred to as ADS—anaerobically digested sludge). The raw sample ADS1 contains 17.9% total solids (TSs) and volatile solids of 51.4% TSs, and ADS2 contains about 23.2% TSs and volatile solids of 53.6% TSs. The raw samples were diluted (with distilled water) or dried (at 40 °C) to achieve 5, 10, 15, and 20% TSs to predict the rheological behavior as a function of total solids. Although this study aims to fill the lack of rheological information about sludges with a high total solids content (>10%), the

5% TSs test allows a comparison with the data in the literature. The samples stood for 48 h at 4 °C after dilution and were then heated to 25 °C using a water bath before analysis.

A sample of green liquor dregs (GLDs) from a Portuguese kraft pulp mill was collected, dried at room temperature, milled, and sieved through a 40-mesh (425 μm) sieve. Based on previous studies [3], 0.15 g GLDs/g SS_{wb} was mixed for 5 min with ADS1 (hereafter defined as ADS_GLDs) to obtain a final TSs content of 15%. The samples rested for 48 h before analysis at room temperature (~20 °C). The main characteristics of the GLDs used in this study are pH 10.3, electrical conductivity 15.6 mS/cm, and VS 9.8% TSs. The content of the main elements Na, Mg, and Ca reported as Na_2O (8.0% TS), MgO (16.8% TS), and $CaCO_3$ (52.9% TS) were determined by X-ray fluorescence (Nex CG Rigaku equipment). The main crystalline phases were identified in GLDs in our previous study by X-ray diffraction (PANalytical X'Pert PRO diffractometer, CuKα-radiation with a 10–80° 2θ range) and were calcite ($CaCO_3$), sodium sesquicarbonate ($Na_3H(CO_3)_2$), and brucite ($Mg(OH)_2$). This material has a high acid neutralization capacity (20 meq H+/g_{db}) [3].

2.2. Analytical Methods

TSs and volatile solids (VSs) were determined based on the EPA Method 1684. Nitrogen was determined according to the Kjeldahl method using VELP Scientifica equipment (Usmate Velate, Italy). Phosphorus was quantified as P_2O_5 by colorimetric analysis following the EPA method 365.3 using a UV-Vis spectrophotometer (T60 UV/VIS, PG Instruments Lda, Lutterworth, UK) at a fixed wavelength of 650 nm. Potassium was determined using flame atomic absorption spectroscopy (ContrAA 300 Atomic Absorption, Analytik Jena, Jena, Germany) after acid digestion with aqua-regia.

The drying rate and diffusion coefficient were obtained based on modeling the kinetic drying curves determined in the laboratory, according to our previous work [3–5]. The samples were dried on a cylindrical plate using a Moisture Analyzer (XM50 Moisture Analyzer, Precisa, Dietikon, Switzerland) at 100 °C, until constant weight. The acid neutralization capacity (ANC) of the samples was determined following the procedure described by Mäkitalo et al. (2014) [25].

The germination tests were carried out with *Lepidium sativum* L. (garden cress) in a 10 liquid/solid ratio. For each sample, 5 mL of extract and ten seeds were placed in a Petri dish over a Whatman filter paper for 48 h at 25 ± 1 °C in dark conditions. The germination index (GI) corresponds to the percentage of the germinated seeds and the root length compared to the control sample (distilled water). The biomass production corresponds to the number of roots, stems, and leaves of *Lepidium sativum* L. growth in pots. The pots were filled with ADS and ADS_GLDs and sandy soil and incubated in an incubator with white LED light, photoperiod of 12 h, temperature of 21 ± 0.1 °C, and relative humidity of 50% [5]. The *Escherichia coli* (*E. coli*) enumeration was performed based on ISO 16649-2:2001.

2.3. Rheological Measurements

A rheometer HAAKED RheoStress1 (Thermo Fisher Scientific, Waltham, MA, USA) connected to a temperature-controlled water bath (Phoenix II, Thermo Fisher Scientific) was used in the present study, with a cup and bob geometry Z34 DIN (gap: 7.20 mm, TSs ≤ 10%) or parallel plate geometry (rotor PP20 with 20 mm diameter, gap: 1.00 mm, TSs > 10%). The shear rate was linearly increased from 0.001 to 1000 s^{-1} for 3 min (upward curve) and then linearly decreased to 0.001 s^{-1} in 3 min (downward curve) to obtain the flow curve [19]. The viscosity was calculated automatically from the shear rate and shear stress. In the oscillatory tests, the frequency (1 Hz) remained constant, while the shear stress varied linearly from 0.2 to 20 Pa (TSs ≤ 10%) or 1–100 Pa (TSs > 10%) for the stress sweep. Then, shear stress remained constant, while the frequency logarithmically varied from 10 to 0.01 Hz for frequency sweep. The temperature during the tests was kept at 25 ± 1 °C using a water bath. Each assay was performed in duplicate.

2.4. Mathematical Modeling

Although diverse models describe the rheological behavior of sludge, the Power Law detailed by Otsward-de-Waele (Equations (1) and (2)) is one of the simplest models used to characterize non-Newtonian fluids. However, the Herschel–Bulkley model (Equation (3)) can complement Equation (1) by representing the shear stress as a function of shear rate and considering the possibility of assessing the yield stress [26],

$$\tau = K\,\dot{\gamma}^{n} \tag{1}$$

$$\eta = K\dot{\gamma}^{(n-1)} \tag{2}$$

where τ is the shear stress in Pa; $\dot{\gamma}$ is the shear rate in s^{-1}; K is the consistency coefficient in Pa·s; n is the dimensionless rheological index (n = 1: Newtonian fluid, n > 1: dilatant fluid; n < 1: pseudoplastic fluid), and η is the viscosity in Pa·s.

$$\tau = \tau_y + K\,\dot{\gamma}^{n} \tag{3}$$

where τ_y is the yield stress in Pa.

Despite this, the Power Law does not predict the upper and lower Newtonian plateaus observed for low and high shear rates [14]. Thus, the empirical equation of Cross [15], Equation (4), was also used,

$$\eta = \eta_\infty + \frac{\eta_0 - \eta_\infty}{1 + \left(\frac{\dot{\gamma}}{\dot{\gamma}_c}\right)^{n}} \tag{4}$$

where η_0 and η_∞ are the viscosity in Pa·s for $\dot{\gamma} \to 0$ and $\dot{\gamma} \to \infty$ (limit viscosity), respectively, and $\dot{\gamma}_c$ (s^{-1}) is a characteristic shear rate corresponding to a balance between destruction and formation of interparticle links.

Flow curves can show different viscosity values on upward and downward curves for the same shear rate values, which may cause the formation of a hysteresis loop. The hysteresis area (Ha) was assessed by Equation (5) [26].

$$Ha = \int_0^t \tau\, d\dot{\gamma}(t) \tag{5}$$

where t is the time of applied stress.

3. Results and Discussion

3.1. Effective Management of ADS Using GLDs

As described before, the management of SS is a challenging operation to safeguard the environment and take advantage of the resources contained in SS (e.g., organic matter and nutrients). In this context, thermal drying seems a favorable alternative to mitigate some of the related issues (e.g., high water content with high transport costs and contamination by microorganism). Although thermal drying has gained relevance as a pretreatment before SS soil application to safeguard human health and the environment, it is an energy-intensive technology. The use of drying adjuvants to boost the mass transfer of moisture and reduce the drying time has been explored in the literature. Specific waste as a drying improver was explored in our previous studies. For the first time in the literature, GLDs were considered a viable option as a conditioning material. Since landfill is the usual management alternative for GLDs, this new approach may give it a renewed perspective within the product value chain that is in line with the European Circular Economy guidelines. Table 1 summarizes some of the parameters obtained from our previous research [3–5] to assess GLDs as a viable option to promote the effective management of SS using drying and obtain a soil improver. This table gathers parameters that characterize the process from the technical point of view (drying operation) and the final product perspective aiming at the soil application (general, technical, and environmental parameters). The results reported in Table 1 were obtained with the ADS1 sample.

Table 1. Summary of ADS and ADS_GLDs characteristics after a drying at 100 °C [3–5].

	ADS	ADS_GLDs
General parameters *		
pH	6.71	8.40
OM (%)	63.7	56.7
$N_{Kjeldahl}$ (%)	3.90	2.38
P_2O_5 (%)	3.83	3.35
K_2O (%)	0.22	0.20
Technical parameters		
Drying rate (gH_2O/min.kg_{wb})	16.81	17.78
Diffusion coefficient (m^2/s)	6.92×10^{-8}	6.49×10^{-8}
Acid neutralization capacity (g $CaCO_3$/g)	0.06	0.38
Environmental parameters		
Germination index (%) **	5.53	6.43
E. coli contamination (log CFU/g)	4.28	2.32
Biomass production (g) ***	0.38	0.39

* Parameters obtained before drying; ** liquid/solid ratio of 10 L/kg; *** biomass dry weight obtained in growth tests with garden cress using pots (300 cm^3) for an application rate of 24 t/ha; OM—organic matter and CFU—colony-forming unit.

From the technical point of view, it is possible to conclude that GLDs slightly improved the drying process, with neutral and positive effects on the drying rate and diffusion coefficient, respectively, compared with raw ADS. In addition, the incorporation of GLDs helped to raise the acid neutralization capacity of ADS, a relevant aspect for application in acidic soils.

From the perspectives of the final product and soil application, there are several conclusions that should be highlighted:

(i). a reduction in the OM and nitrogen content is observed after GLDs incorporation, while the phosphorus and potassium concentrations are not significantly changed.
(ii). the germination index increased slightly by mixing ADS with GLDs, while caution is recommended for both material applications during the seed germination phase.
(iii). GLDs allowed a significant reduction of *E. coli* contamination below the legal limit established by Portuguese legislation for applying SS on the soil (<3 log CFU/g). On the other hand, biomass production during a growth test is not influenced negatively by the application of GLDs.
(iv). the levels of potentially toxic metals in both materials are below the Portuguese legal limit (Portuguese Decree-Law No. 276/2009) established for the direct use of sludge in soil [27].

Overall, the use of GLDs for ADS management proved to be a viable option for drying and subsequent soil application as the final disposal route. In general, the addition of GLDs had no harmful effects on the parameters analyzed.

Regarding the practical application of these studies, they are part of a project to develop an industrial-scale dryer for SS. The dryer is designed to be placed in the WWTPs, and the SS is fed by extrusion. In this context, the rheology of SS and mixtures with drying adjuvants is a relevant aspect that has not yet been explored, so, it will be considered in the next section. Indeed, rheology is a relevant aspect to evaluate when managing since the viscosity can influence the transport, handling, and design of specific equipment, such as transport lines and extruders [7,8].

3.2. Rheological Characteristics of Raw and Conditioned ADS

3.2.1. Rheology of Raw ADS

Figure 1a,b show the viscosity as a function of shear rate of ADS samples collected in the two WWTPs containing 5, 10, 15, and 20% TSs. As expected, the increase in TSs leads to an increase in limit viscosity since an intensification in the interaction between particles occurs [18]. Regarding the apparent viscosity curve, both ADS samples show shear-thinning behavior as the viscosity decreases continuously as the shear rate increases. The viscosity results presented in Figure 1a,b were obtained from the shear stress versus shear rate curves (not shown) that allowed to draw some relevant conclusions about the two ADS samples: (i) for TSs < 10%, both samples behave as pseudoplastic and dilatant fluids in different zones of the curves (Figure 1a); (ii) for TSs > 10%, the samples exhibit a pseudoplastic behavior with yield stress. These samples with yield stress will start to flow only when external forces acting on the materials are superior to their structural ones, which needs attention in the sludge operation equipment [28]. It is important to note that the yield stress is dependent on the speed resolution of the rheometer used, not being an intrinsic material constant, and it is possible to obtain only a rough estimate by the applied methods [29]. Since the two samples exhibit similar behavior, the following tests (including the chemical conditioning) and the mathematical modeling were carried out only with the ADS1 sample (hereafter defined as ADS).

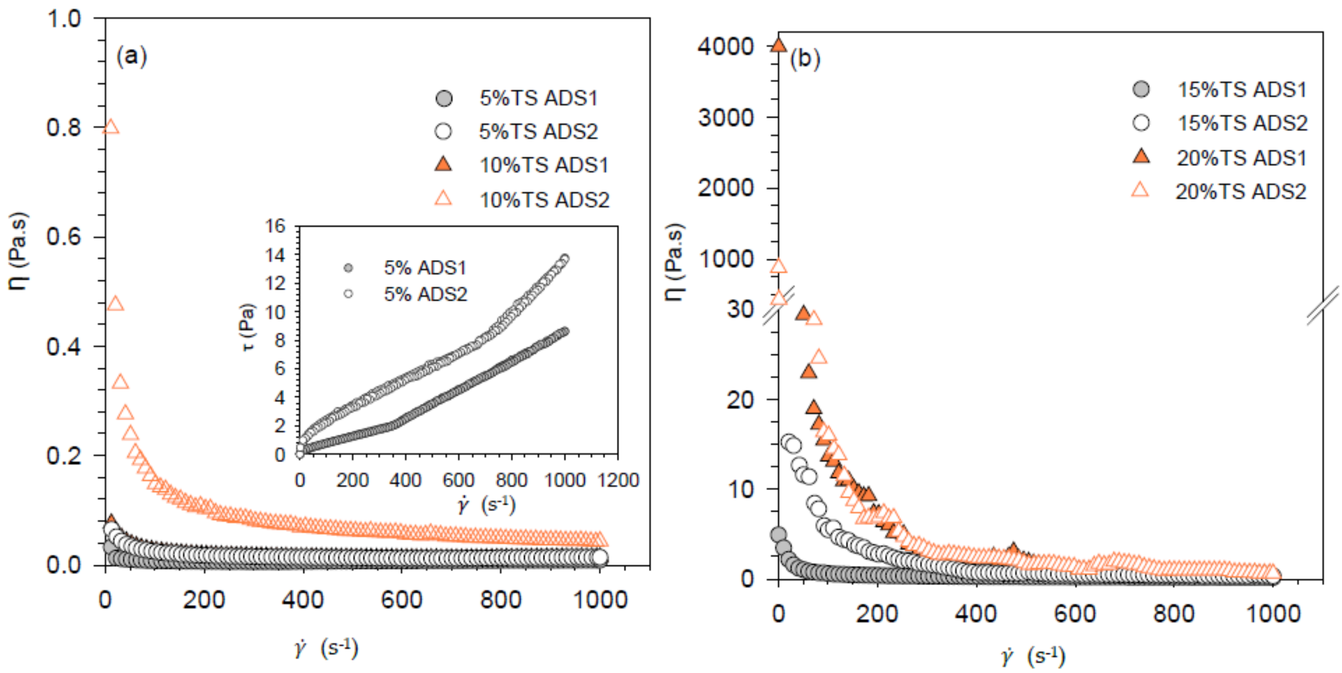

Figure 1. Viscosity as a function of shear rate: (**a**) 5 and 10% TSs and (**b**) 15 and 20% TSs.

It is important to note that the apparent viscosity may depend not only on the shear rate but also on the time of shearing, and thus a hysteresis loop was also considered. By increasing the shear rate (upward curve), the internal structure of the sludge floc can break. If it exceeds the deformation limit, a decreasing shear rate ramp (downward curve) leads to a reduction in the viscosity compared to the values obtained in the upward curve. In this scope, thixotropy describes the structural recoverability after destruction by a hysteresis loop method [17]. Higher hysteresis areas mean higher time-dependent behaviors, and the material must be sheared for a long period to achieve a steady-state [9]. According to Equation (5), the area enclosed by the hysteresis loop of ADS varies from about 1.24 Pa·s at 5% TSs to 43.1 Pa·s for 20% TSs, which indicates that the thixotropic loop area is directly proportional to the total solids content. This phenomenon may be due to an intensification of friction and collision between particles [30,31]. According to the literature, anaerobic digestion proves to be an effective process for reducing sludge thixotropy. Nevertheless, the shape and enclosed area of the hysteresis loop also depend on the duration of the shear, the shear rate range, and the past kinematic history of the sample [14].

Oscillatory tests were also carried out, and the results are represented in Figure 2. In this case, viscoelastic properties of the samples are present, demonstrating a combination of fluid and solid behavior [8,12]. In this scope, it is commonly defined he complex shear modulus (G^*) and phase angle (δ) or, alternatively, the storage modulus (G') and the loss modulus (G''). In viscoelastic materials, G' represents the elastic portion (solid behavior), and G'' represents the viscous property (fluid behavior). The phase angle between shear stress and strain reflects the response of the samples. If $\delta = 0°$, the material has ideal elastic behavior, and G' is dominant. On the contrary, if $\delta = 90°$, the material is ideally viscous, and G'' is dominant [12]. Viscoelastic materials exhibit behavior between these two ideal states. Although the studies occur in a low-deformation region, the liquid/solid behavior is already well-defined. Figure 2 shows that the ADS sample behaves similar at 5 and 10% TSs since G' tends to zero, and $\delta = 90°$ (Figure 2a,b), and, consequently, the sample reveals ideally viscous flow behavior. On the other hand, for samples containing 15 and 20% TSs, a similar behavior is observed. Indeed, the elastic behavior dominates the viscous one ($G' > G''$) (Figure 2c,d), and the samples are viscoelastic solids since $0° < \delta < 45°$. Between 10 and 15% TSs, there can be a substantial modification in the internal structure of the sample, possibly linked to a transition from a solid to a liquid property [23,32].

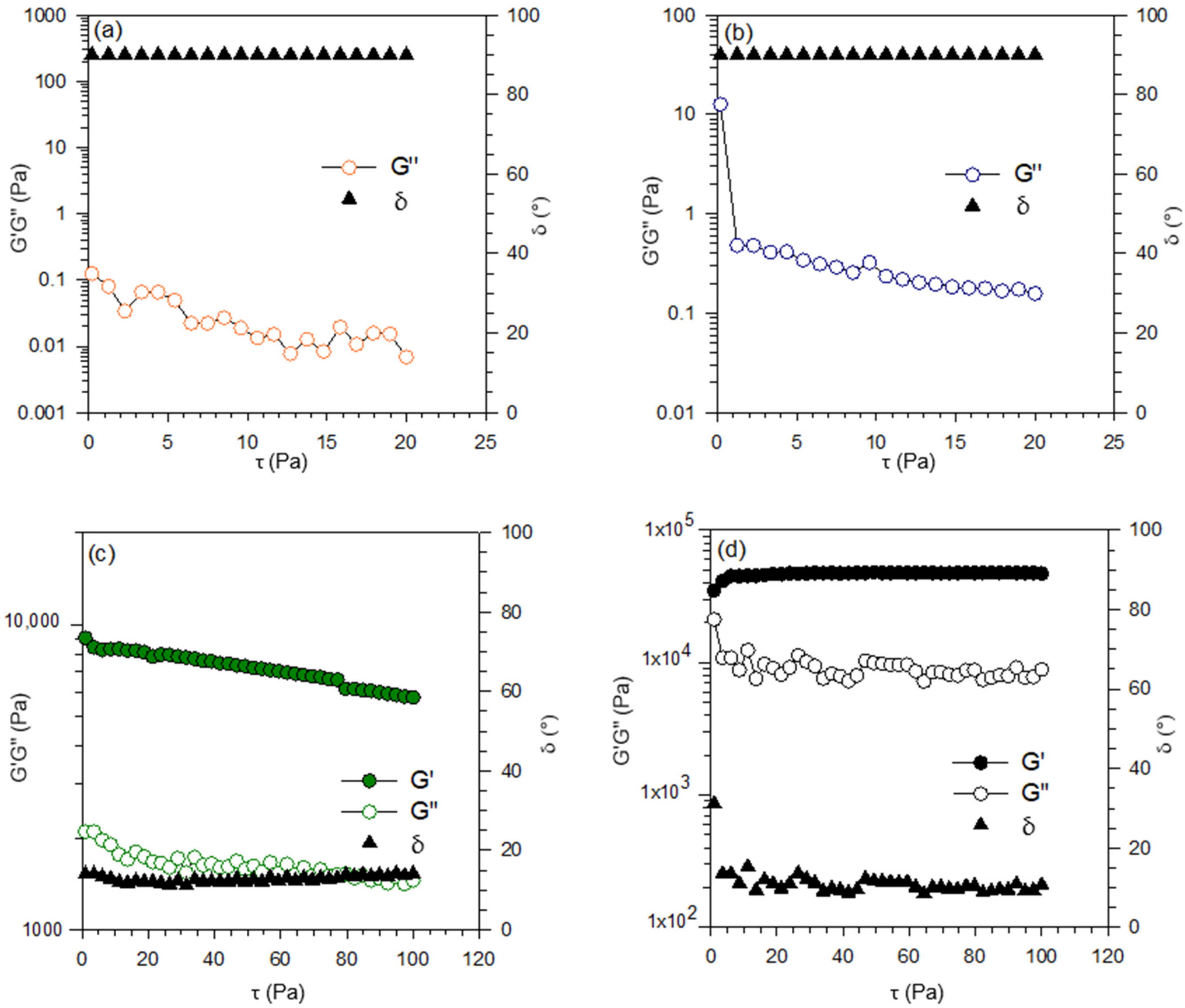

Figure 2. Oscillatory tests: (**a**) stress sweep at 5% TSs; (**b**) stress sweep at 10% TSs; (**c**) stress sweep at 15% TSs; and (**d**) stress sweep at 20% TSs.

Through the frequency sweep (Figure 3a), it is possible to conclude that with a high TSs content (>10%), both G' and G'' exhibit a plateau in the tested frequency range. Thus, it may indicate a well-structured system with strongly bonded particles, and the material properties do not change over the experiment (stable internal structure) [8]. For 5 and 10% TSs, G'' increases as the frequency increases, while G' is practically inexistent. At low frequencies, G'' was always superior to G', and the sample demonstrates a liquid behavior with an almost non associated particulate dispersion. Figure 3b presents the complex viscosity (η^*) obtained by the rheometer software, a frequency-dependent viscosity function determined for a viscoelastic fluid that represents the viscoelastic flow resistance of the sample. Low values of η^* ($0.001 < \eta^* < 0.1$ Pa·s) were observed for samples with 5 and 10% of TSs, while high values (>10 Pa·s) were determined for 15 and 20% of TSs.

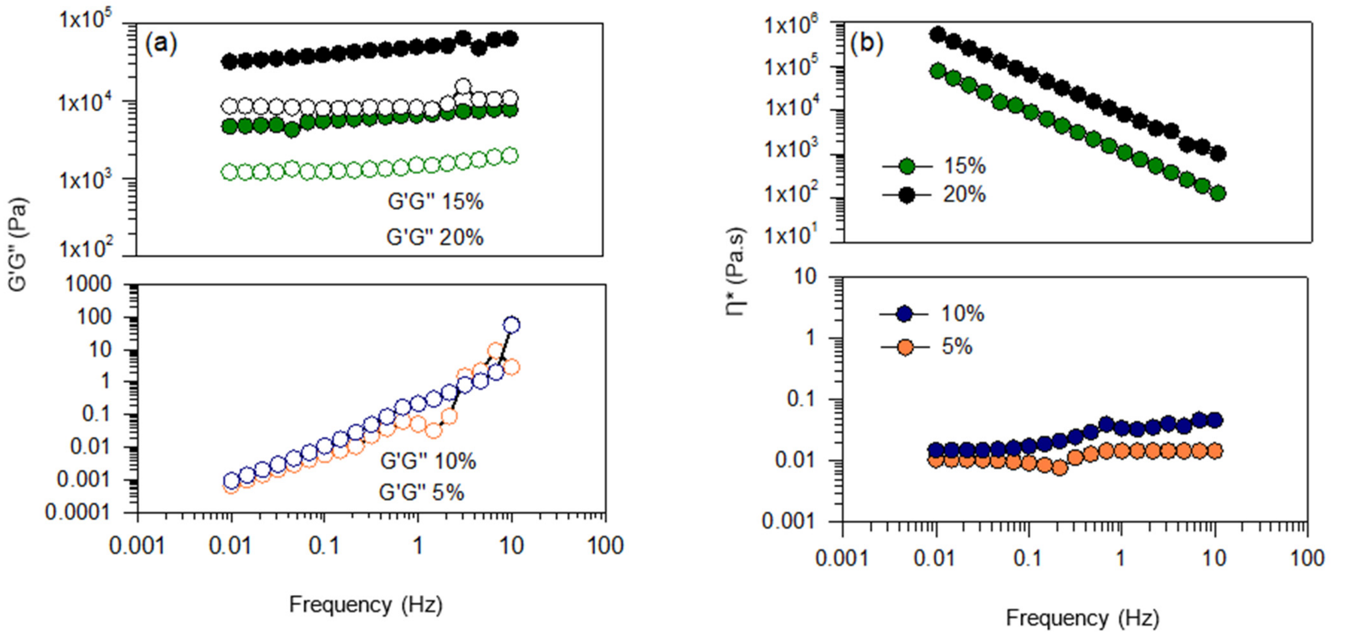

Figure 3. Oscillatory tests: (**a**) frequency sweep and (**b**) complex viscosity as a function of frequency.

This first assessment of rheological properties will make it possible to later compare the SS without and with chemical treatment (by adding GLDs).

3.2.2. Rheology of ADS Mixed with GLDs

As observed in the previous sections, sludge from WWTPs may exhibit high viscosity values for low shear rates, typically used in stirring and pumping equipment. Nevertheless, some studies indicate that chemical conditioning may decrease sludge shear resistance. Consequently, it reduces the load on subsequent equipment. In this context, Figure 4 presents the rotational and oscillatory assays conducted to evaluate the effect of GLDs (an alkaline residue from pulp mills) on ADS with 15% TSs. This content of total solids was selected since it is the typical value obtained after mechanical dewatering of SS.

Alkaline treatment did not change the shear-thinning observed previously in the raw sludge, but the η-$\dot{\gamma}$ curves were shifted downwards. Once again, regardless of the addition of GLDs, the elastic behavior is dominant compared to the viscous one, which is characteristic of a viscoelastic system.

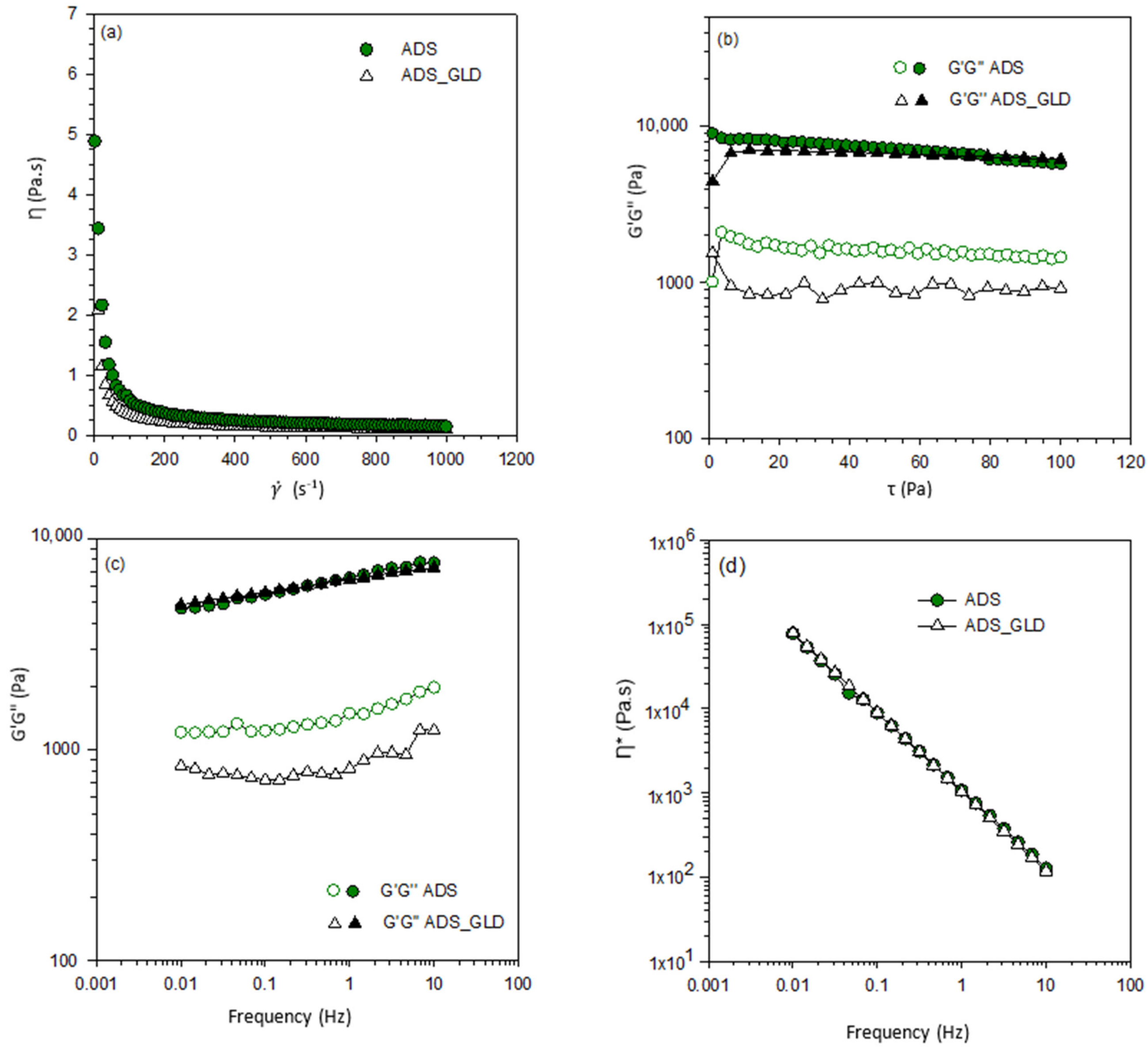

Figure 4. Rheological tests for ADS with GLDs with 15% TSs: (**a**) viscosity as a function of shear rate; (**b**) stress sweep; (**c**) frequency sweep; and (**d**) complex viscosity.

The changes in the sludge flocs during chemical conditioning may explain these results. The raw sludge flocs have a complex structure with a powerful interparticle interaction that promotes high apparent viscosity values. Studies show that one of the main substances of sludge flocs is extracellular polymeric substances (EPSs), an essential microbial cell structure [26,33]. However, these substances influence crucial processes in WWTPs (e.g., settling and dewatering). The dewaterability of sludge can be improved after EPSs are broken and disintegrated. Thus, the macromolecular organic matter decomposes into low-molecular forms, and, consequently, the apparent viscosity decreases. Several disintegration methods have been investigated, such as physical (thermal and ultrasonic treatments) and chemical (alkali and acid treatments). Alkali treatment is the most widely used method due to its simple operation and the powerful breakdown effects of sludge flocs. In this scope, this study considers chemical conditioning with GLDs as an alkaline treatment (using a waste) since the pH of GLDs and ADS_GLDs is close to 10. An increase in pH may reduce the viscosity due to changes in the surface charge density of the particles via hydroxyl anions (OH^-). In addition, the high electrostatic repulsion on the surface causes desorption of some parts of the extracellular polymers, contributing to the same end [33–35]. Indeed, GLDs are mainly $CaCO_3$ (about 53%) [3], and the calcium ions can play a crucial role in the increase in apparent viscosity. Neyens et al. (2003) [36] reported that calcium ions added to sludge decrease the bound water content, which increases floc density and strength. Ca^{2+} may create a tighter bound network that increases the apparent viscosity of the samples. Wang et al. (2016) [8] also observed the bridging action of divalent Ca^{2+} ions. However, this phenomenon was not significant in our study since the dosage of GLDs is relatively low compared to that used in the studies mentioned before. Liu et al. (2013) [33] used low dosages of CaO in the sludge (0–0.20 g/g of dry solids) that reduced the apparent viscosity with the different treatments compared to raw sludge. Thus, at low dosages, that effect can be neglected, contrary to the positive pH effect. Another effect to highlight is the organic matter reduction with the addition of GLDs (an inorganic material) [37]. The viscosity reduces with the organic fraction of the sample reduction, which is one of the advantages of using GLDs.

3.2.3. Mathematical Modeling

Table 2 summarizes the parameters obtained through Equations (3) and (4) for ADS. The model parameters confirm the results discussed above. Indeed, for 5 and 10% TSs in ADS, the value of n is superior to 1 (dilatant fluid). The remaining assays reveal a pseudoplastic behavior ($n < 1$). In addition, the yield stress increases for a higher total solids content [38]. As discussed before, the ADS sample with 5 and 10% TSs does not behave exclusively as a pseudoplastic fluid. Therefore, the model of Cross, Equation (4), does not adjust the experimental data properly. Cao et al. (2018) [17] found that a fresh sample of activated sludge with 10% TSs has a limit viscosity of about 0.175 Pa·s at 20 °C, while in another study, the same authors reported a value equal to 0.412 Pa·s [30]. Indeed, the samples under analysis are complex and variable, and the treatment in the WWTPs may have a strong influence on the results. Wang et al. (2016) [8] obtained at 100 s^{-1} a viscosity near 1 Pa·s for an activated sludge with 13.5% TSs.

Table 2. Parameters of Herschel–Bulkley and Cross models for ADS.

	Herschel–Bulkley Model				Cross Model			
TSs (%)	K (Pa·s^n)	τ_y (Pa)	n	R^2	η_0 (Pa·s)	$\eta\infty$ (Pa·s)	$\dot{\gamma}_c$ (s^{-1})	R^2
5	5.317×10^{-4}	0.319	1.400	0.9984	0.7773	0.0050	10.34	0.8859
10	1.728×10^{-3}	1.941	1.276	0.9841	0.0463	0.0134	104.3	0.6538
15	2.225	22.19	0.595	0.9920	4.927	0.1692	16.70	0.9978
20	10.88	178.3	0.963	0.9770	40,004	0.5102	1.503	0.9999

Note: K—consistency coefficient; τ_y—yield stress; n—flow index; η_0 and η_∞ are the viscosity in Pa·s for $\dot{\gamma} \to 0$ and $\dot{\gamma} \to \infty$ (limit viscosity), respectively, and $\dot{\gamma}_c$ (s^{-1}) is a characteristic shear rate.

Another factor that may influence the rheological behavior of SS is the organic fraction, here represented by the VS content. Wang et al. (2020) [39] indicate that the higher the organic load, the stronger the binding intensity between the flocs, and consequently, the less the fluidity of the sludge. Thus, it is expected that the higher the organic fraction in SS, the higher the energy needed to transport the sample within the dryer. In addition, the hydrolysis of organic matter in the anaerobic digestion process weakens the microstructures of the sample inside the digester, which may interfere with the rheological behavior of the final ADS sample, allowing an easy transition to a liquid-like material [40].

After the addition of GLDs, the consistency index (K) is reduced by about 51% (Table 3) compared to raw ADS. Similarly, the limit viscosity ($\eta\infty$) also decreases, with a positive effect on the material shear resistance and the energy necessary to convey it in the equipment. The decrease in the organic fraction can also justify these results. As aforementioned, GLDs are an inorganic material, and after mixing with SS, they will reduce the organic matter of the mixture from about 51% TSs (ADS) to 40% TSs (ADS_GLDs).

Table 3. Parameters of Herschel–Bulkley and Cross models for ADS with GLDs.

	Herschel–Bulkley Model				**Cross Model**			
	K (Pa·s^n)	τ_y (Pa)	n	R^2	$\eta\infty$ (Pa·s)	η_0 (Pa·s)	$\dot{\gamma}_c$ (s^{-1})	R^2
ADS	2.225	22.19	0.5952	0.992035	0.1692	4.927	16.70	0.9978
ADS_GLDs	1.080	13.33	0.6337	0.993015	0.0712	2.356	4.832	0.9995

Note: K—consistency coefficient; τ_y—yield stress; n—flow index; η_0 and η_∞ are the viscosity in Pa·s for $\dot{\gamma} \to 0$ and $\dot{\gamma} \to \infty$ (limit viscosity), respectively, and $\dot{\gamma}_c$ (s^{-1}) is a characteristic shear rate.

3.3. Practical Applications

There is already extensive knowledge about SS rheology. However, the practical and industrial applications of these measures are less explored. The ultimate purpose of this work is to provide more information about SS characteristics to construct an efficient industrial dryer for SS management. In this scope, the rheology findings will help improve sludge handling from the dewatering unit to the dryer (e.g., pumping, transporting, and extrusion). For example, the rheological behavior can influence the transporting by the pressure losses in the pipes and, consequently, raise the operating costs [41]. This study demonstrates that the addition of GLDs may reduce the yield stress and the consistency coefficient by about 40 and 51%, respectively, compared to the raw ADS sample. These results indicate that the sludge plus GLDs offers less resistance to flow and reduces the pressure losses in the pipes during transportation. There are already some models that use these rheological parameters to design piping systems and evaluate the pressure drop in pipes [22]. Additionally, the rheological properties need to be considered to understand and optimize an extrusion process. The use of an extrusion process is widely studied in the polymers area, and that knowledge indicates that it is difficult to understand and optimize an extrusion operation without knowing the rheological behavior of the material under process.

4. Conclusions

According to the rotational tests, it is possible to observe a predominant shear-thinning behavior for ADS. Nevertheless, the ADS flow curves reveal some complexity at <10% TSs showing both pseudoplastic and dilatant behaviors. As expected, the limit viscosity and yield stress are proportional to the total solids content. The oscillatory measurements revealed that at 5 and 10% TSs, ADS emphasizes a liquid-like regime, while at TSs > 10%, it behaves as a viscoelastic solid. The addition of GLDs to ADS had a positive effect on the shear resistance, leading to a reduction in the limit viscosity. These results can be useful for selecting equipment, such as feeders and extruders, based on viscosity values and their variation as the TSs increase.

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