

Article

Rheological Characterization of Structural Stability for Black Soils from Northeast China

Jian Sun ¹, Lin Zhou ^{2,3}, Yuyang Yan ^{1,3}, Chenyang Xu ¹, Zhe Liu ⁴, Zhenghong Yu ⁵ , Jiangwen Li ^{2,3,*} and Feinan Hu ^{1,2,3,*}

¹ College of Natural Resources and Environment, Northwest A&F University, Yangling 712100, China

² Institute of Soil and Water Conservation, Chinese Academy of Sciences and Ministry of Water Resources, Yangling 712100, China

³ State Key Laboratory of Soil and Water Conservation and Desertification Control, College of Soil and Water Conservation Science and Engineering, Northwest A&F University, Yangling 712100, China

⁴ Shaanxi Provincial Land Engineering Construction Group Co., Ltd., Xi'an 710075, China

⁵ College of Agricultural Science and Engineering, Hohai University, Nanjing 211100, China

* Correspondence: ljw0077@nwafu.com (J.L.); hufn@nwafu.edu.cn (F.H.)

Abstract: Soil structural stability is fundamentally linked to soil functionality and sustainable productivity. Rheological properties describe the deformation and flow behavior of soil under external stress, playing a crucial role in understanding soil structure stability. Despite their importance, the studies about rheological properties of black soils in Northeast China remain limited. This study aims to assess the rheological properties of two kinds of black soil with different degrees of degradation in Northeast China. The rheological parameters of these soils under various water contents and shearing were quantified by conducting Amplitude Sweep Tests (ASTs) and Rotational Sweep Tests (RSTs). Both AST and RST results showed that as soil water content and shear rate increased, shear strength, viscosity, and hysteresis area all decreased in Keshan and Binxian black soils. The increase in soil water content reduces the friction between soil particles, leading to a decrease in soil structure stability. Additionally, the viscosity and hysteresis area of the two soils decreased with the increase in water content, making it more flowable and exhibiting shear-thinning behavior. Keshan black soil exhibited stronger recovery and shear strength compared to Binxian black soil; this is mainly due to the higher organic matter content in Keshan soil, which could increase structural stability by bonding the soil particles at the micro-level. These findings enhance our understanding about the structure stability of the black soils based on the rheological parameters via rheometer.

Keywords: structural stability; slurry; rheology; amplitude sweep test; rotational shear test



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1. Introduction

The black soil zone in Northeast China is one of the four major black soil regions globally. Black soil is a kind of soil with good properties and high fertility, making it very suitable for the growth of plants. It is a cornerstone of global food security and environmental health [1] and plays a crucial role in ensuring food security of China [2]. However, due to the combined influence of natural factors (e.g., soil erosion) and human activities (e.g., intensive farming), this region is subjected to varying degrees of soil structural degradation [3]. The black soil layer is increasingly characterized as “thinner”, “harder”, and “poorer”, which has severely affected soil structural stability [4–6], leading to a decline in soil fertility and erosion resistance, and ultimately threatening China’s food security and

agricultural sustainability. Therefore, research on black soil's structural stability is crucial for soil health and agricultural sustainability.

Soil structural stability refers to the soil's ability to resist deformation and maintain its integrity under external stress. Soil aggregates, the fundamental building blocks of soil structure, are critical indicators for evaluating this stability [7–9]. As an important component of the soil, organic matter significantly affects soil aggregate stability through its cementing effect on soil particles. With increasing organic matter content, the stability of soil aggregates improves, which is reflected in enhanced soil erosion resistance characteristics [10,11]. Furthermore, because soil structure is a three-dimensional system composed of both aggregates and pores, studying either aggregate stability or pore characteristics in isolation may not provide an accurate assessment of overall soil structural stability. Some studies have focused on the soil mass as a whole, using classical mechanical methods such as triaxial shear tests to investigate soil structure. It has been observed that the shear strength of black soil increases with higher organic matter content [12]. Despite these advancements, significant gaps remain in understanding the flow and deformation behavior of black soil under dynamic external disturbances. This limitation hinders the development of predictive models for soil degradation processes.

Rheology, a branch of mechanics that studies the flow and deformation behavior of materials under external stress, has gained increasing application in soil science in recent years. It has proven to be an effective method for evaluating the structural stability of soil samples [13]. For example, studies on bentonite slurry have shown that the addition of cellulose induces pronounced shear-thinning behavior [14]. Carotenuto et al. have found that organic matter content significantly affects the rheological properties of soil. The viscosity and yield stress of soil decrease as organic matter content decreases [15]. Different rheological properties indicate variations in the strength of the microstructural framework in different directions within the same soil [16]. Additionally, substantial differences in rheological properties are observed across different soil types. When the water content of soil samples is relatively low ($w < 50\%$), the stress and strain in the linear viscoelastic region are greater for purple and brown argillaceous sandstone slurries compared to red and white argillaceous sandstone slurries [17]. The rheological method is a feasible approach to explore soil structural stability under external stress. Although numerous studies have been carried out using the rheological method at present, there are few studies that have used the rheological method to assess the structural stability of the black soil in Northeast China.

Therefore, the primary objective of this study is to reveal the rheological characteristics of black soils under rotational shear and amplitude sweep tests. Additionally, this study investigates the fundamental effects of organic matter content and water content on the rheological properties of two typical black soils. The findings of this research will deepen the understanding of soil structure stability based on rheological property and provide more information about the degree of soil degradation and soil quality.

2. Materials and Methods

2.1. Investigated Sites of Soil Samples

Two representative black soils were collected from Heilongjiang Province, China. The first sampling location was at Bin County, Harbin City ($45^{\circ}45'22''$ N, $127^{\circ}25'36''$ E). This area is typified by a thin-layered black soil, with topographical gradients running southeast to northwest, descending from 600 m to 300 m in elevation, with a mean altitude of 450 m. The site experiences a cold temperate continental monsoon climate, with a mean annual temperature of 4.4°C and an average annual precipitation of 570 mm. The secondary location at Keshan County, Qiqihar City ($48^{\circ}08'27''$ N, $126^{\circ}04'59''$ E), straddles the border of mountainous regions and alluvial plains. Here, plateau-like topography is

prevalent in the northeastern section, with elevations changing by over 200 m within the sample area. Although under the same climatic classification as the primary site, Keshan County endures harsher thermal conditions, with an annual mean temperature of 2.4 °C and a prolonged frost duration (ranging from 220 to 274 days, averaging 242 days). The precipitation exhibits a monsoonal distribution, with 65% of the yearly 500 mm total falling between June and August.

2.2. Soil Properties

We adopted the five-point sampling method to collect soil samples from the surface layer (0–20 cm) of the sampling site and mixed them evenly. Then, we removed impurities such as stones and plant residues, and we put the samples into clean plastic bags and marked them clearly. For the samples that need to be analyzed, physical and chemical changes in the samples should be minimized. Basic soil properties were analyzed using conventional methods. Organic matter content was determined via external heating with potassium dichromate. Soil particle size distribution was analyzed using an MS2000 laser particle sizer (Malvern Panalytical, Malvern, UK), classifying particles into sand (2–0.02 mm), silt (0.02–0.002 mm), and clay (<0.002 mm) according to international standards. Soil pH was measured using the electrode method with a soil-to-water ratio of 1:2.5. The cation exchange capacity (CEC) and specific surface area (SSA) were determined using the combined surface property method [18]. The basic physical and chemical properties of the soils are summarized in Table 1.

Table 1. Basic physical and chemical properties of Keshan and Binxian black soils.

Soils	SOM (g·kg ^{−1})	CEC (cmol·kg ^{−1})	SSA (m ² ·g ^{−1})	pH	Clay (%)	Silt (%)	Sand (%)
					<0.002 mm	0.002–0.02 mm	0.02–2 mm
Keshan black soil	49.9	23.4	66.5	5.88	35.5	25.4	39.1
Binxian black soil	28.9	21.3	58.6	6.32	32.9	24.7	42.4

Note: SOM, soil organic matter; CEC, cation exchange capacity; SSA, specific surface area.

2.3. Rheological Tests

Rotational shear tests (RSTs) and amplitude sweep tests (ASTs) were conducted to obtain specific rheological parameters, guided by the previously established soil rheology literature [19–23].

Rheological tests were conducted using the MCR302 rheometer (Figure 1a), manufactured by Anton Paar (Austria, Purchased from Anton Paar Trading Co., Ltd., Shanghai, China), which is capable of various measurement modes for either strain or stress control. In this research, both rotational shear tests (RSTs) and amplitude sweep tests (ASTs) were performed using a cross-plate rotor (model ST22-4V-40, Figure 1b) (Austria, Purchased from Anton Paar Trading Co., Ltd., Shanghai, China), with a length of 40 mm, a diameter of 22 mm, and an outer cylinder diameter of 28.92 mm (Figure 1c). This rotor was rotated within the sample-holding measuring cylinder of the rheometer to exert shear stress on the soil samples.

Soil samples were air-dried naturally, and impurities such as stones and plant roots were removed. The samples were then ground and passed through a 0.5 mm sieve before being bagged for storage and subsequent analysis. Deionized water was added to the two types of black soil samples to prepare soil slurries with moisture contents of 40%, 45%, 50%, 55%, and 60% (based on solid-to-liquid ratios).

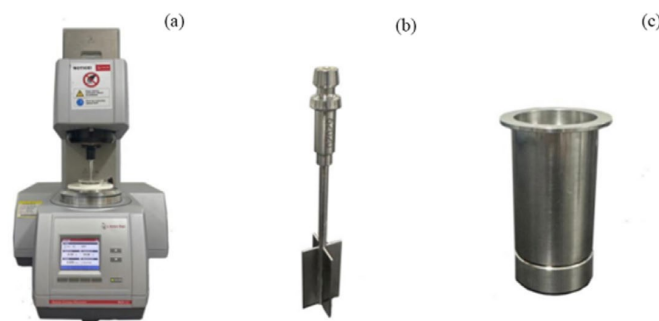


Figure 1. Test equipment: (a) rheometer physical picture, (b) vane rotor, and (c) cup.

In the rotational shear tests, we put the soil slurry into the cylinder and used a vane rotor to conduct the shearing. The shear rate was uniformly increased from 0 to 100 s^{-1} . The flow curves (shear stress vs. shear rate) and viscosity curves (viscosity vs. shear rate) for the two types of black soils were obtained to analyze their rheological behavior. After the shear rate was uniformly increased to 100 s^{-1} , it was then uniformly decreased back to 0 s^{-1} ; the upward curve and the downward curve take the same amount of time. This process was performed to obtain the hysteresis curves of the two types of black soils, reflecting the thixotropic behavior of the soil slurries. The experiments were conducted at a controlled temperature of 20°C , with 50 data points collected during the shear rate variation process. Each test was repeated 4 to 8 times to ensure accuracy and reproducibility. The primary objective was to investigate the rheological response of the soil slurry under varying shear rates, focusing on properties such as shear thinning, viscosity evolution, and thixotropic behavior. Additionally, the study aimed to systematically analyze the effects of soil type and moisture content on the structural stability of the slurry.

We put the prepared soil slurry into the cylinder and used a vane rotor to shear the sample at a frequency of 0.5 Hz, with a shear strain ranging from 0.001% to 1000% [23]. The test temperature was maintained at 20°C , and 30 data points were measured, with 4 to 8 repetitions conducted [24]. This test aimed to explore the effects of different moisture contents and soil types on the viscoelasticity of the black soil slurry, specifically examining shear stress and shear strain in the linear viscoelastic zone, as well as shear stress and shear strain at the yield point.

2.4. Technical Roadmap

The technical roadmap of this paper is shown in Figure 2. The black soil within 0.5 mm is screened and configured into slurries with different water contents. The amplitude sweep test and the rotational sweep test in the rheological method are used to characterize the structural stability of the black soil.

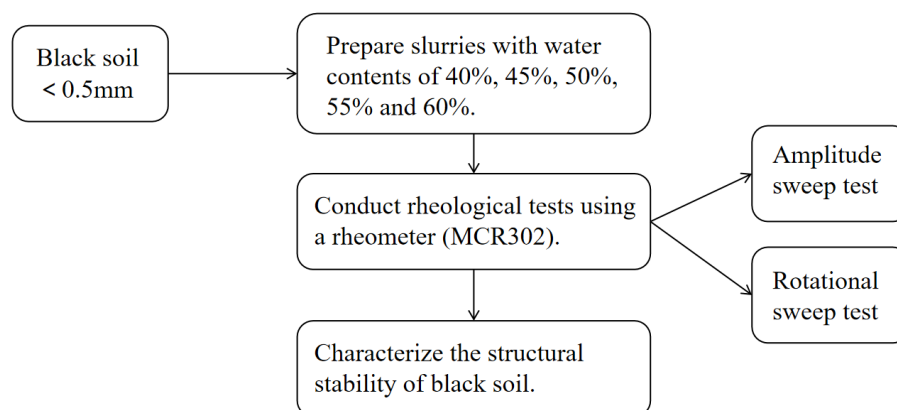


Figure 2. Flowchart of the experimental process.

2.5. Statistical Analysis

The data of the rheology tests in this paper were directly exported after being tested and completed by the software RheoCompass 1.24. The experimental data were processed in Excel 2019, the one-way analysis of variance for significance was carried out by using SPSS 26, and the data were plotted by using OriginPro 2021.

3. Results

3.1. Flow and Viscosity Curves of the Soils

The soil flow curve, the relationship between shear stress and shear rate (Figure 3), is crucial for characterizing the rheological behavior of soil. The typical flow curves of soil suspensions showed an increasing then decreasing trend with the increasing shear rate. The curves' maxima represented the highest shear stress tolerable by the soil particle network. The maxima of shear stress decreased with the increase in soil water content. Furthermore, the flow curves of Keshan black soil were consistently higher than those of Binxian black soil at the same soil water content.

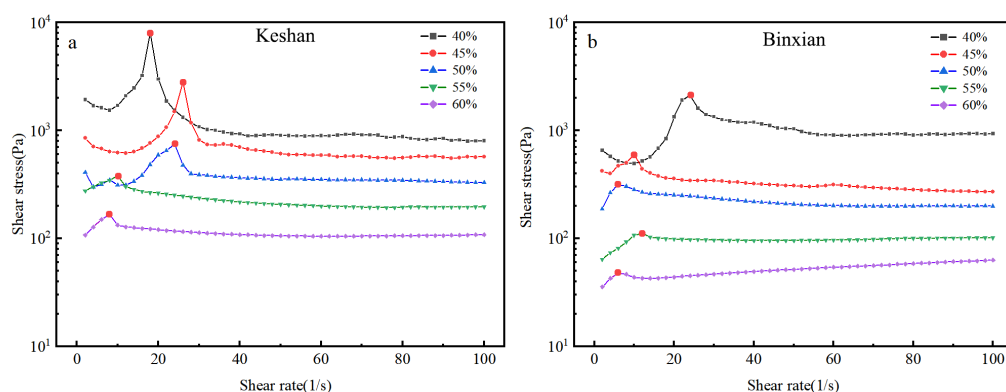


Figure 3. Flow curves of slurry in Keshan (a) and Binxian (b) black soils. The values 40%, 45%, 50%, 55%, and 60% represent soil water content, the same as below.

The relationship between yield stress (the curves maxima) and water content of the two black soils is shown in Figure 4. As the water content increased, the yield stress of both soils decreased. When the water content raised from 50% to 60%, the yield stress began to stabilize. Notably, Keshan black soil consistently exhibited higher yield stress than that of Binxian black soil at the same soil water content level.

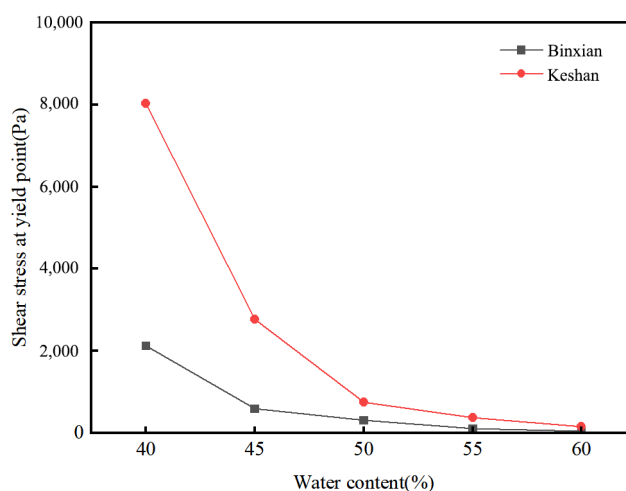


Figure 4. Shear stress at the yield point of two black soils.

The viscosity curve reflects the characteristics of the flow resistance of the material under different shear conditions. The relationship between viscosity and shear rate for the two black soils at various water contents is shown in Figure 5. Both soils exhibited a general decrease in viscosity as the shear rate increased. During the initial shear stage (0 to 25 s^{-1}), viscosity decreased sharply, and then it increased and decreased gradually with the increase in the shear rate. The viscosity curves of the two soils exhibit distinct patterns. For Keshan black soil, at water contents of 40%, 45%, and 50%, the viscosity first decreased, then increased, and subsequently decreased again. In contrast, for Binxian black soil, only at 40% water content, a similar trend was observed wherein the viscosity initially decreased, then increased, and subsequently decreased again. However, this phenomenon for the two soils eventually disappeared as the water content increased.

As the soil water content increased, the viscosity of both soils decreased. Specifically, when the water content of Binxian black soil increased from 40% to 60%, the initial viscosity decreased by 37,000 $\text{mPa}\cdot\text{s}$, while Keshan black soil exhibited a decrease of approximately 910,000 $\text{mPa}\cdot\text{s}$. This indicates that Keshan black soil had significantly higher viscosity compared to Binxian black soil at the same water content.

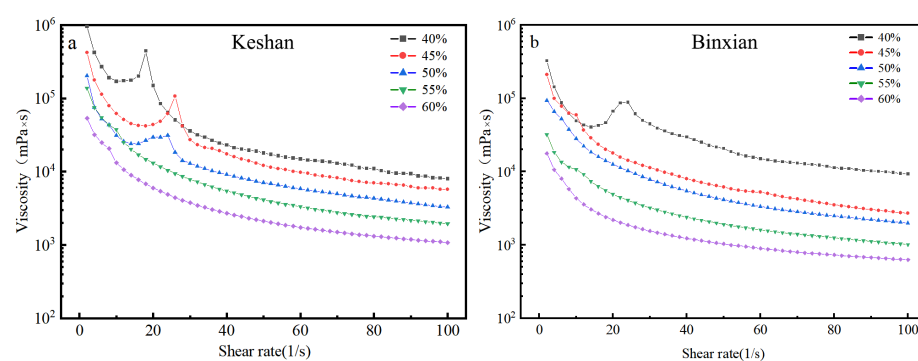


Figure 5. Viscosity of slurries in Keshan (a) and Binxian (b) black soils.

3.2. Hysteresis Curves of the Soils

The hysteresis curve is a curve in rheology used to characterize the energy dissipation and structural recovery capabilities of materials. The hysteresis curves for the two black soils at various water contents are presented in Figure 6. Initially, the difference between the upward and downward curves increased and then decreased as the shear rate raised. At all water contents, the upward curves of both soils consistently lie above the downward curves. Distinct differences were observed in the hysteresis curve characteristics of the two black soils. At 40% water content (Figure 6a), Keshan black soil displayed the largest positive hysteric area. As water content increased, the disparity between the upward and downward curves progressively decreased. Furthermore, the hysteresis curve for Keshan black soil consistently exhibited higher values compared to Binxian black soil.

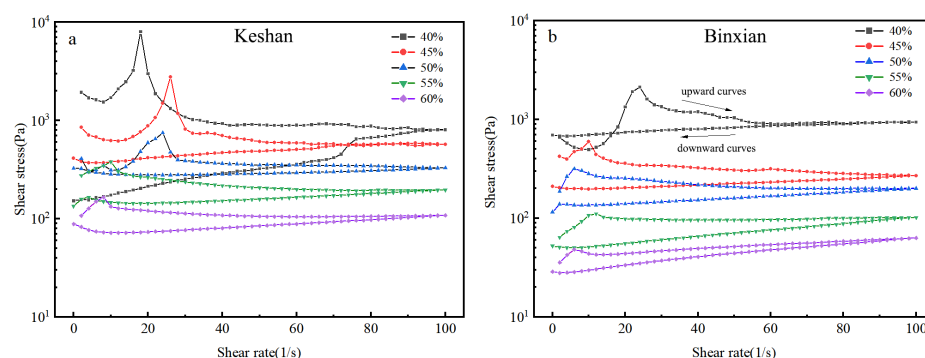


Figure 6. Hysteresis curves for different water contents in Keshan (a) and Binxian (b) black soils.

The hysteresis area is positively correlated with thixotropy, which characterizes the time-dependent structural recovery ability of the material. The hysteretic areas of the two black soils are presented in Figure 7. The hysteretic area of Keshan black soil was consistently higher than that of Binxian black soil, especially at the low soil water content. As the water content increased, the hysteretic area displayed a decreasing trend.

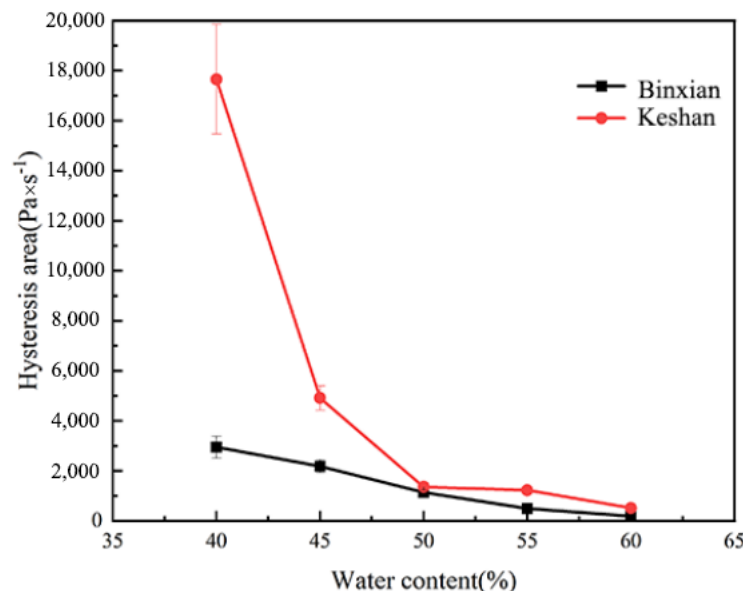


Figure 7. Hysteresis area of two black soils.

3.3. Storage Modulus and Loss Modulus

The relationship between the storage modulus and loss modulus versus shear strain at varying water contents for the two black soils are shown in Figure 8. As the shear strain increases, both the storage modulus and loss modulus kept constant and then displayed a decreasing trend. At the point where the storage modulus was equal to the loss modulus, the soil reached its yield point. With increasing water content, both the energy storage modulus and loss modulus generally decreased for both soils. Furthermore, there were observable differences in the storage modulus and loss modulus between the two soils. For example, the reductions in Binxian black soil were consistently smaller than those recorded for Keshan black soil at the same water content. Specifically, the decrease in the storage modulus of Binxian black soil amounted to only 25% of that observed in Keshan black soil.

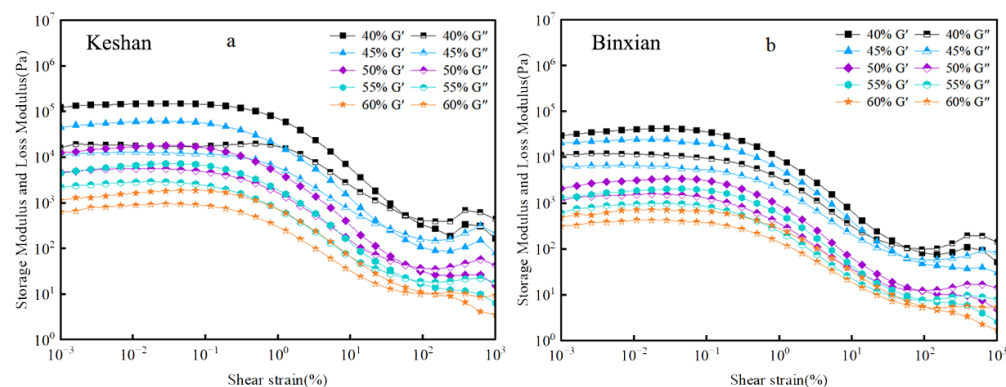


Figure 8. Relationship between rheological parameters and shear strain for various water contents in two black soils. G' , for storage (elastic) modulus; G'' , loss (viscous) modulus; (a): Keshan; (b): Binxian.

3.4. Rheological Parameters at the End of the Linear Viscoelastic Range

The relationship between the storage modulus and shear stress at the linear viscoelastic range of the two black soils, as a function of water content, is presented in Figure 9. As the water content increased, both the storage modulus and the shear stress at the yield point in the linear viscoelastic region exhibited a decreasing trend for both soils. Notable differences were observed between the two soils. Keshan black soil consistently exhibited higher values for both the storage modulus and shear stress compared to Binxian black soil. However, the disparity in both parameters diminished as the water content became >50%.

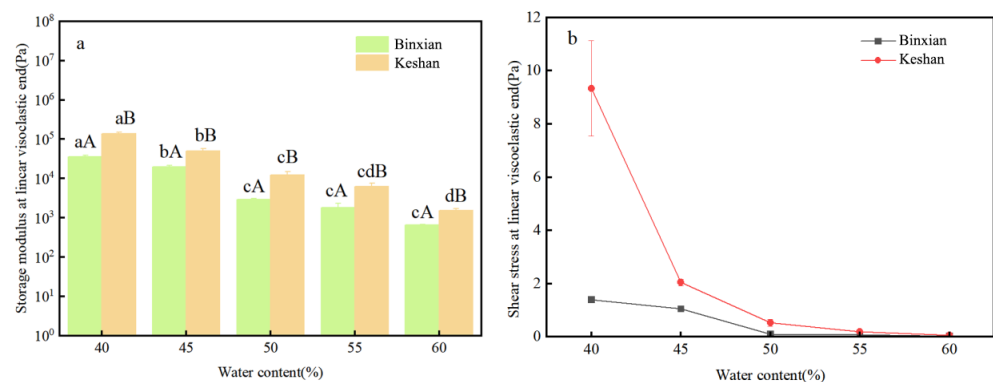


Figure 9. Storage modulus (a) and shear stress (b) at the linear viscoelastic zone of two black soils (Keshan and Binxian). Different lowercase letters represent significant differences in the storage modulus at the linear viscoelastic end between different soil water contents at the same soil ($p < 0.05$). Different capital letters represent significant differences in the storage modulus at the linear viscoelastic end between the two soils at the same soil water content ($p < 0.05$), the same as below.

3.5. Rheological Parameters at the Yield Points

The relationship between the storage modulus, shear stress at the yield point, and water content for the two black soils is illustrated in Figure 10. As the water content increased, both the storage modulus and the shear stress at the yield point exhibited a decreasing trend for both soils. Notable differences were observed between the two soils ($p < 0.05$). Keshan black soil consistently exhibited higher values for both the storage modulus and shear stress compared to Binxian black soil at the yield point. However, the disparity in both parameters diminished as the water content became >50%.

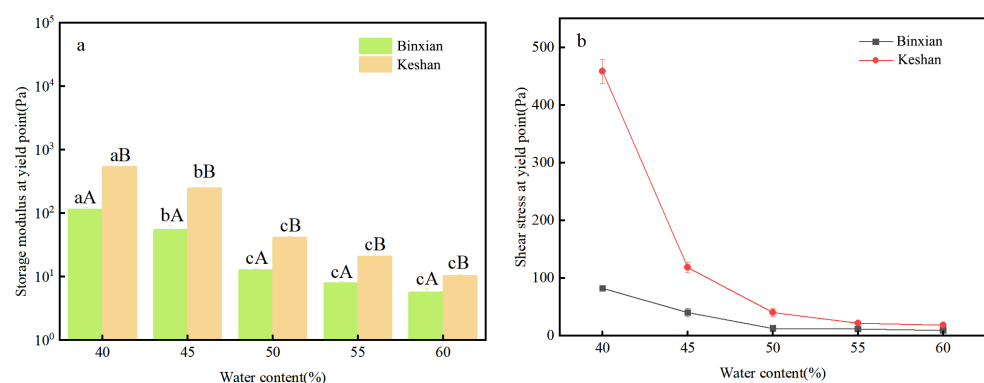


Figure 10. Storage modulus (a) and shear stress (b) at the flow point of two black soils. Different lowercase letters represent significant differences in the storage modulus at yield point between different soil water contents at the same soil ($p < 0.05$). Different capital letters represent significant differences in the storage modulus at yield point between the two soils at the same soil water content ($p < 0.05$).

4. Discussion

This study found that the flow behavior of the two black soils exhibited complex non-Newtonian fluid characteristics. It can be seen from Figure 3 that as the shear rate increased, the shear stress of both black soil slurries showed a decreasing trend, demonstrating shear-thinning behavior; this is consistent with the findings of Markgraf [16]. This behavior can be primarily attributed to the disruption of the internal structure of the fluid and the rearrangement of particles [24]. As external stress increases, the internal structure of the fluid begins to break down. At lower shear rates, the initially disordered state of the soil particles is disrupted, leading to the formation of solid “chains”, in turn reducing viscosity. At higher shear rates, these “chains” are broken, the distance between soil particles decrease, the interaction force between particles can be enhanced, and they could reorganize into a more stable structure, resulting in an increase in viscosity [17].

In this study, at low water content levels (40% and 45%), the viscosity curve shows a trend of initially decreasing, then increasing, and subsequently decreasing again. This behavior reflects a transition process from shear thinning to shear thickening and finally to secondary thinning. In the initial stage, as the shear rate increased, the interactions between soil particles were disrupted, enhancing the fluidity of the system (Figure 3). Consequently, the ability to resist external forces weakened, leading to a decrease in shear stress and viscosity. When the shear rate decreased to a certain range, the soil particles rearranged and reorganized, forming a more compact structure, which increased flow resistance and resulted in a rise in viscosity [25]. However, as the shear rate continued to increase, the particles in the slurry became further dispersed, the gaps between particles widened, the interaction force between the particles decreased, and the structure was disrupted again, enhancing fluidity and reducing viscosity. The viscosity of the soil decreased with the increase in soil water content, which is consistent with the previous study [24]. This is because the increase in water content reduces the friction between soil particles, and the water also has a lubricating effect. As a result, the overall viscosity of the soil decreased as the water content increased (Figure 5).

The hysteresis area is positively correlated with thixotropy, which characterizes the time-dependent structural recovery ability of the material. Our study showed that with the increase in water content, the hysteresis area of the soil decreased, indicating that the thixotropy of the soil decreased. This is due to the dispersing effect of water on soil particles.

This study found that the shear strength, storage modulus, and viscosity of Keshan black soil were greater than those of Binxian black soil, which may be due to its higher organic matter content [26,27]. Liang et al. found that the storage modulus and loss modulus are related to soil texture, organic matter (SOM), and clay mineralogy [28]. High organic matter content can typically enhance soil cohesion and structural integrity [29]. Organic matter also acts as a binding agent, improving soil stability [30–32]. As a macromolecular organic compound, organic matter has a high specific surface area (SSA), which can enhance the physical–chemical interactions between soil particles, thus significantly improving the shear strength and viscoelastic mechanical properties of the soil. In addition, the surface of organic matter is rich in active functional groups. Compared with inorganic mineral particles, it has a more prominent ability to adsorb cations, so it has a significant regulatory effect on the surface charge characteristics of the soil [33]. Soil with a high cation exchange capacity (CEC) can promote the aggregation of organic and inorganic matter. An increase in the organic matter content can lead to an increase in the Hamaker constant, which can enhance the van der Waals force between soil particles. Therefore, the increase in organic matter can enhance the soil’s resistance to external mechanical disturbances [34].

We found that in the initial stage of shearing, the storage modulus and loss modulus did not change. This is consistent with the previous research [35]. This is because during

the initial shearing stage, the applied stress is insufficient to cause the destruction of the soil structure but can lead to the rearrangement of fine particles [35]. With increasing external shearing, the storage modulus and loss modulus of both soils decrease, exhibiting strain-thinning characteristics. Baumgarten et al. found that strain thinning is related to the reorganization of soil particle network structures [13]. Due to the reorganization of particles, the rheological properties of both soils also change, becoming more prone to flow. Water, as another critical influencing factor, exerts a dispersing and lubricating effect on the soil. It forms a water film on the surface of soil particles, weakening the cohesive forces between soil particles, reducing the stress required to resist deformation, and making flow more likely to occur [36]. The water can increase the interparticle distance and thus reduce the total energy and ionic concentration of the system, which can further alter the structure of the electric double layer and weaken the bonding forces between particles.

5. Conclusions

In the study, the rheology method was used to characterize the structure stability of the two black soils. Soil water content and shear rate could greatly affect soil structural stability. As soil water content and shear rate increased, shear strength, viscosity, and hysteresis area all decreased in Keshan and Binxian black soils. The increase in water content reduces the friction between soil particles, leading to a decrease in soil structure stability. Additionally, the viscosity and hysteresis area of the two soils decreased with the increase in water content, making it more flowable and exhibiting shear-thinning behavior. Keshan black soil exhibited stronger recovery and shear strength compared to Binxian black soil, demonstrating greater structural stability. This is mainly due to the higher OM content in Keshan soil, which could increase structural stability by bonding the soil particles at the micro-level. The findings enhance our understanding about soil structure stability based on the rheological parameters via rheometer.

Author Contributions: Conceptualization, J.S., L.Z., C.X., J.L. and F.H.; methodology, J.S., L.Z. and F.H.; software, J.S., L.Z. and Y.Y.; formal analysis, J.S., L.Z. and F.H.; investigation, J.S.; resources, J.S.; writing—original draft, J.S., J.L. and F.H.; writing—review and editing, J.S., L.Z., C.X., Z.L., J.L. and F.H.; visualization, J.S., J.L. and F.H.; funding acquisition, C.X., Z.Y., J.L. and F.H. All authors have read and agreed to the published version of the manuscript.

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Conflicts of Interest: Author Zhe Liu was employed by the company Shaanxi Provincial Land Engineering Construction Group Co., Ltd. The remaining authors declare that the research was conducted in the absence of any commercial or financial relationships that could be construed as a potential conflict of interest.

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