

Article

Effect of the Polymerized Titanium Ferric Sulfate (PTFS) Coagulant on Sedimentation of Coal Slime Water

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Abstract: High efficiency slime water settlement is very important for ensuring washing water recycling in coal preparation plants. In order to improve the sedimentation of coal slime water, an iron ion-based coagulant was prepared by titanium ion complexation action using titanium sulfate as a main raw material, Fe^{3+} and NaH_2PO_3 as a stabilizer and NaHCO_3 as an alkalizing agent. The particle size distribution of coal slime was measured, and the sedimentation test for coal slime water was carried out with PAM, polyaluminum chloride and polymerized titanium ferric sulfate (PTFS), respectively. Then, coal slime water sedimentation was investigated at different PAM dosages and polyaluminum chloride or coal slime water of PTFS with various molar ratios of PAM and ferrotitanium. The results showed that PAM and polyaluminum chloride could not make coal slime settle down, and PTFS showed a poor settlement effect. When polyaluminum chloride and PTFS were used together with PAM, it produced a molecular weight of 3 million respectively. Polyaluminum chloride needs 6.66×10^{-10} mol of PAM, PTFS needs 0.66×10^{-10} mol of PAM and the effect of sedimentation is improved. When the molar ratio of PTFS was 1:7, polymerization performance was effective. The sedimentation effect of PTFS was better than that of polymeric aluminum chloride coagulant usually used in coal preparation plants, especially when combined with flocculant, and high efficiency was reached. The surface potential and surface free energy of coal slime particles before and after adding coagulant were measured and analyzed by XDLVO theory to explain the action mechanism of PTFS coagulant. Experimental results demonstrated that PTFS can significantly improve the sedimentation of coal slime water, save the dosage of PAM and increase economic benefit.

Keywords: coal slime water; sedimentation; coagulation; PTFS; XDLVO theory



Citation: Shi, C.; Guo, D.; Li, Y.; Zhang, Z.; Tang, L. Effect of the Polymerized Titanium Ferric Sulfate (PTFS) Coagulant on Sedimentation of Coal Slime Water. *Minerals* **2022**, *12*, 26. <https://doi.org/10.3390/min12010026>

Academic Editors: Chiharu Tokoro, Shigeshi Fuchida and Yutaro Takaya

Received: 28 November 2021

Accepted: 21 December 2021

Published: 24 December 2021

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1. Introduction

Coal slime water treatment is a very important link in the production of coal preparation plants [1–3]. The main function is to recover low calorific value slime and clear water in order to achieve the closed cycle of washing water [4–6]. Coal slime water treatment includes coal slime water sedimentation and pressure filtration recovery and other operation units, among which include coal slime water sedimentation as the key to coal slime water treatment [7,8]. Coal slime in coal slime water has fine particle size, high ash content, large specific surface area and negative charge on the particle surface; therefore, it is very difficult to settle down the coal slime by adding a coagulant alone. In order to enhance the rapid sedimentation of coal slime water, coagulants and flocculants are generally used together in coal preparation plants [9–11].

The coagulants commonly used in coal preparation plants are aluminum salts and iron salts, such as polyaluminum chloride and sulfate, polyferric chloride, ferric sulfate,

magnesium chloride, etc., and polyaluminum chloride is commonly used [12,13]. The main function of coagulant is to neutralize the surface charge of slime and condense the fine slime particles in coal slime water system. Anionic polyacrylamide (PAM) is the most widely used flocculant [14], and its main role is to bridge and sweep particles for the slime settlement. Since aluminum salt and iron salt coagulants are not able to compress the double electric layer on the surface of slime particles, the effect of using them with PAM which is small molecular weight is not ideal. Therefore, most coal preparation plants can only use PAM with high molecular weight, which increases production cost. Different coagulants have different properties and sedimentation effects. The study of high efficiency coagulant has become an important parameter of coal slime water treatment. In recent years, titanium coagulants have been developed rapidly and have been applied in many water treatment fields. Tang [15] used the acid dissolving one-step method to prepare poly titanium sulfate and determined the ratio of reactants, reaction temperature and time. Xue [16] studied the application of PFTS in drinking water purification. The preparation of titanium coagulant is simple, the flocs are large, the separation effect is good and the removal rate of organic matter is high. It is an environmentally friendly coagulant. Liu [17] prepared Polysilicon Titanium Aluminum Sulfate (PTAS) and Polysilicon Titanium Ferro Sulfate (PTFS). It was found that the coagulation mechanism of PTAS and PTFS was mainly adsorption neutralization and hydroxyl adsorption network scavenging on the surface of the complex, and the bridging network scavenging played an auxiliary role. The results showed that they could coagulate simulated river water, emulsified oil wastewater and phosphating wastewater as well. Chen [18] prepared a new type of non-integral covalent compound PFTS. By using single factor and response surface design methods, the optimal parameters of PFTS were obtained. The main morphology and phase structure of PFTS were characterized. The application effect of PFTS in algae removal and control of algae source membrane pollution was studied.

Due to the advantages of titanium salt coagulant, a PTFS coagulant was prepared in this study. The feasibility of its application in the field of coal slime water treatment was studied by conducting sedimentation experiments, and sedimentation performance was evaluated for certain coal slime water.

2. Materials and Methods

2.1. Materials

The chemicals used in this study were titanium sulfate (98% pure) (Alighting Chemical Products Co., Ltd., Shanghai, China), ferric sulfate (22% pure) (Tianjin Fuchen Chemical Reagent Factory, Tianjin, China), anionic PAM with molecular weight of 3 million and 7 million, polyaluminum chloride (Tianjin Yuanli Chemical Co., Ltd., Tianjin, China), sodium dihydrogen phosphate (99% pure) (Tianjin Guangfu Science and Technology Development Co., Ltd., Tianjin, China) and sodium bicarbonate (99% pure) (Tianjin Yongda Chemical Reagent Co., Ltd., Tianjin, China). The water sample used was deionized water with a conductivity of 18 $\mu\text{S}/\text{cm}$ and a resistivity of 480 $\mu\text{S}/\text{cm}$.

Main instruments included a 500 mL settling cylinder, 85-2A digital constant temperature speed measuring magnetic stirrer (Jintan Ronghua Instrument Manufacturing Co., Ltd., Changzhou, China), UV752N ultraviolet visible spectrophotometer (Shanghai Aoxi Scientific Instrument Co., Ltd., Shanghai, China) and electrophoresis instrument (Shanghai Zhongchen Digital Equipment Co., Ltd., Shanghai, China).

The main mineral element composition of coal slime water used in the test is shown in Table 1. The coal slime water used in the test has a high concentration of calcium and magnesium ions. The total hardness is 8.72, which belongs to medium hard water. The pH of water is 8.06, and the water quality is slightly alkaline.

Table 1. Composition of main mineral elements.

Elements	SiO ₂	Al ₂ O ₃	Fe ₂ O ₃	SO ₃	CaO	MgO	Others
Content/%	46.63	25.43	7.03	8.57	6.77	0.72	4.85

2.2. Preparation Method of PTFS

PTFS is mainly made of titanium sulfate and ferric sulfate obtained through polymerization reactions. Firstly, the mass of various reagents was calculated according to a certain molar ratio, then the weighed ferric sulfate was placed into the beaker. Then, water was added and stirred for dissolution. After stirring for 30 min, the weighed titanium sulfate was added, 10 mg NaH₂PO₄ and 10 mg NaHCO₃ at an interval of 30 min. The coagulant was prepared by stirring continuously for 1 h and standing at room temperature for 24 h.

2.3. Experimental Procedure

- (1) The slime used to prepare coal slime water was sieved to determine the size composition of slime;
- (2) Coal slime water was prepared with a volume concentration of 20 g/L;
- (3) Flocculant and coagulant solutions were prepared. An anionic PAM (molecular weight: 3 million and 7 million) solution was prepared with the concentration of 0.1% by weight; polyaluminum chloride solution with the concentration of 5% by weight;
- (4) After repeated determination, the wavelength was determined to be 540 nm based on deionized (DI) water;
- (5) The transmittance of clear water after coal slime water sedimentation was taken as the evaluation index. The higher the transmittance, the better the settlement effect;
- (6) Comparison test of coal slime water treatment. The light transmittances of the test with no reagent, one reagent and different reagents were measured, and the test was observed;
- (7) The optimum ranges of dosage and the optimum molar ratio (Ti:Fe) of polymeric ferric titanium sulfate coagulant were studied;
- (8) The contact angle, surface energy and zeta potential of coal slime water without reagent, with polyaluminum chloride and with PTFS, were measured.

2.4. Method for Measuring Zeta Potential

A one milligram sample of coal slime, ground to 0.20 mm or less, was placed into 100 mL deionized water and dispersed for 2 min with an ultrasonic disperser as the liquid to be tested. One milligram of the solution to be tested was taken into the electrophoresis cup, inserted into the cross label and soaked twice. An amount of 0.5 mL of the solution to be measured was injected into the electrophoresis cup, and the cross markers were inserted to adjust the focal length. Then, 1 mL of the solution to be tested was injected into the electrophoresis cup, and the electrode was inserted and bathed twice, then 0.5 mL of the solution was injected into the electrophoresis cup. The electrode was then placed on a three-dimensional platform to measure the zeta potential. After 10 times of repeated measurement, the suspicious value was removed, and the average value was calculated as the final measurement result.

2.5. Properties of Slime

The slime used in the experiment was provided by Yitai Coal Mine in Ordos, China when passing through the coal seam section. The small screening test was carried out by wet screening methods, and the results are shown in Table 2.

Table 2. Particle size distribution of slime sample used in this study.

Particle Size (mm)	Weight (%)	Ash Content (%)
0.25–0.5	1.66	12.01
0.125–0.25	5.60	10.95
0.075–0.125	7.34	13.40
0.045–0.075	7.56	17.17
–0.045	77.84	39.16
total	100.00	33.58

The color of coal slime is gray. It can be seen from Table 1 that the amount of –0.045 mm in the slime was up to 77.84%, ash content was 39.16%, coarse particle content was less and average particle size was 0.046 mm. It is preliminarily judged that the coal slime water prepared from the slime had difficulty settling down.

2.6. Performance Index of PTFS

The performance indexes of PTFS used in the test are shown in Table 3.

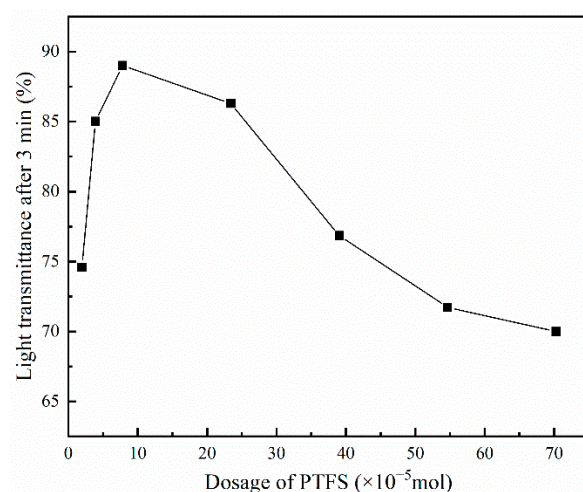
Table 3. Performance index of PTFS.

Performance	Density (g/cm ³)	Basicity (%)	Viscosity (Pa·s)	Iron Content (%)	Insolubles (%)	pH
Index	2.57	13	1.36	>17.85	<0.35	4–11

3. Experimental Results and Analysis

3.1. Single Agent Tests

PAM (7 million molecular weight), polyaluminum chloride, and PTFS (Ti:Fe = 1:7) were added into coal slime water separately, and the sedimentation experiment was carried out according to the method (GB/T26919-2011) of coal slime water settling. The experimental results were as follows: When PAM and polyaluminium chloride were added separately, no slime flocculation occurred and coal slime water could not settle even if the dosage of PAM and polyaluminium chloride was high. The test results of only adding PTFS coagulant are shown in Figure 1.

**Figure 1.** Sedimentation effect of using PTFS (Ti:Fe = 1:7) alone.

It can be observed from Figure 1 that the effect of adding PTFS alone is positive. With the increase in the dosage of PTFS, the sedimentation of large flocs can be observed obviously, but the supernatant is turbid, and the transmittance of coal slime water increases

first and then decreases. When the addition dosage of PTFS is 7.81×10^{-5} mol, the transmittance was 89%, indicating that the coal slime water had difficulty settling. Therefore, the mixed test scheme of coagulant and flocculant was considered.

3.2. Effect of the Combination of Polyaluminum Chloride, PTFS and PAM

In order to investigate the effect of polyaluminium chloride, PTFS and PAM on the sedimentation of coal slime water, respectively, polyaluminium chloride and PTFS used with different dosage of PAM explored the effect of polyaluminium chloride dosage, PTFS dosage, different ferrotitanium mole ratio and PAM dosage of coal slime water fall. When the PAM dosage was 0.66×10^{-10} mol, the results of the test of polyaluminium chloride combined with PAM of 3 million molecular weight are shown in Figure 2.

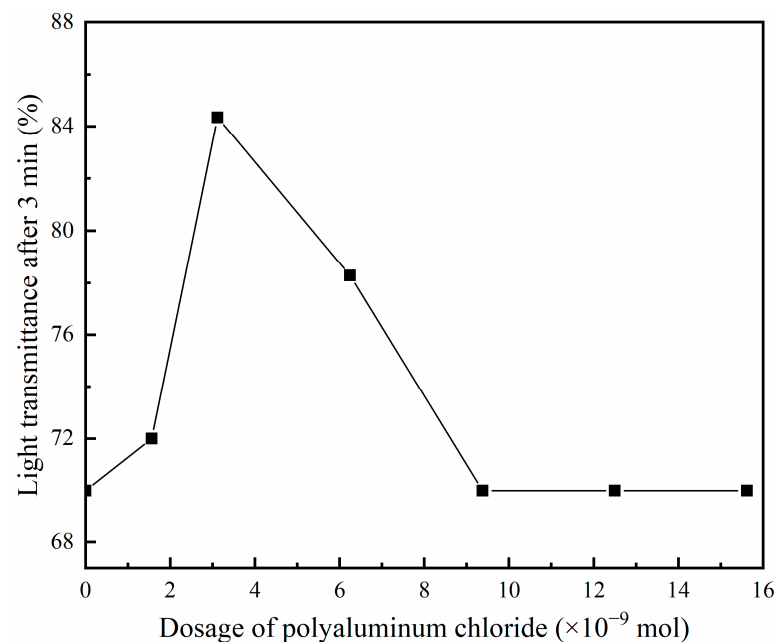


Figure 2. Sedimentation effect of combined use of PAM and polyaluminum chloride (0.66×10^{-10} mol).

It can be observed from Figure 2 that when the dosage of PAM with molecular weight of 3 million was 0.66×10^{-10} mol, light transmittance increases initially and then decreases with the increase in polyaluminum chloride dosage. When the dosage of polyaluminium chloride increased to 9.38×10^{-9} mol, the sedimentation effect was not obvious, the stratification of coal slime water was slow, the supernatant was turbid and the light transmittance was low. When the dosage of polyaluminum chloride was about 3.12×10^{-9} mol, the sedimentation effect was the best, reaching 84.34%. After 3 h, the supernatant would be clear, and the transmittance was 98.71%. However, the settling time was too long to meet production requirements.

In order to investigate the influence of PAM dosage on coal slime water settlement, PAM dosage increased to 6.66×10^{-10} mol, and the test results are shown in Figure 3.

As observed from Figure 3, when the PAM dosage increased to 6.66×10^{-10} mol, the the settling effect significantly improved, the settling speed was high and the light transmittance of the upper clarifying solution was high. The transmittance increased first and then decreased with the increase in dosage of polyaluminum chloride. When the dosage of polyaluminum chloride was 3.12×10^{-9} mol, the sedimentation effect of coal slime water was optimum, and the transmittance was 99.41%. However, when the dosage of polyaluminum chloride increased, the sedimentation effect of coal slime water decreased obviously. When PAM dosage was 6.66×10^{-10} mol, the dosage of polyaluminum chloride should not exceed 3.12×10^{-9} mol in order to ensure the sedimentation effect of coal slime water.

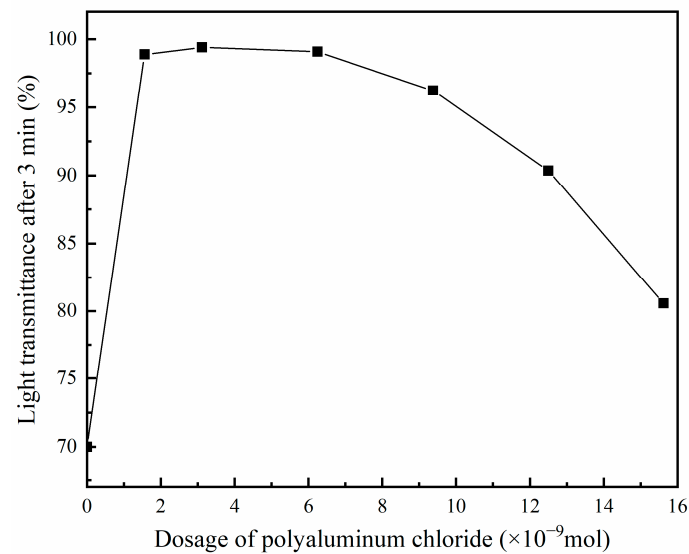


Figure 3. Effect of combined use of PAM and polyaluminum chloride (6.66×10^{-10} mol) on sedimentation.

In order to compare the effects of PTFS and polyaluminum chloride on coal slime water settlement, the dosage of PAM was 0.66×10^{-10} mol, and the influences of different dosage of PTFS and different molar ratio of ferrotitanium on coal slime water settlement were investigated. The test results are shown in Figure 4.

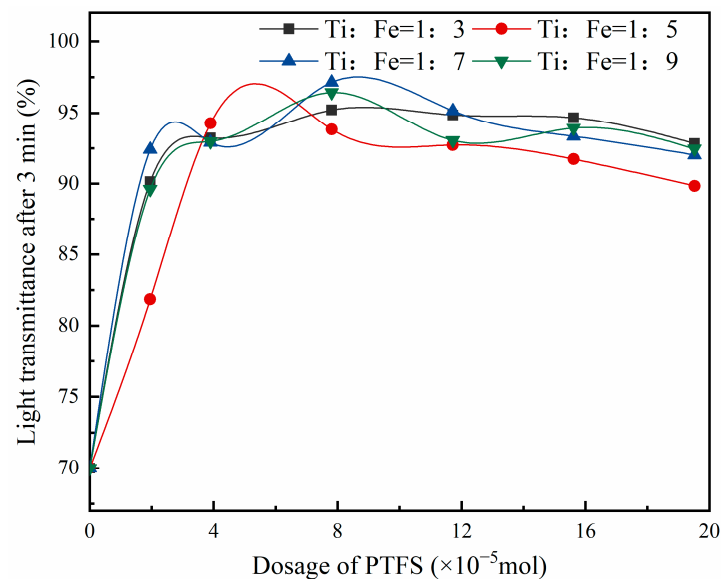


Figure 4. Comparison of the sedimentation effect of PAM and PTFS with different ratios of ferro titanium.

It can be observed from Figure 4 that PTFS combined with PAM can achieve a good settlement effect, and the light transmittance of coal slime water was above 90% on the whole. With the increase in PTFS content, light transmittance of coal slime water first increased and then decreased, and there is an obvious stratification phenomenon of rapid settlement of large particle floc in coal slime water settlement process. Different titanium-iron molar ratios of PTFS have a certain effect on the sedimentation effect of coal slime water. When titanium:iron = 1:7, the peak transmittance is relatively high, and the effect is slightly higher than other molar ratios. In order to obtain good coal slime water settlement effect, when PAM dosage is 0.66×10^{-10} mol, it should be used together with polymerized

titanium iron sulfate with the molar ratio of 1:7, and the dosage of PTFS should not exceed 7.81×10^{-5} mol.

4. Analysis of PTFS Condensation Mechanism Based on XDLVO Theory

4.1. XDLVO Theory

The composition of coal slime water is very complex. It was found that the classical DLVO theory cannot reasonably explain the coalescence of coal slime water. Recent studies [19] have shown that there is a special interaction energy between hydrophilic or hydrophobic colloidal particles, in addition to Van der Waals energy and electrostatic interaction energy, that plays a decisive role in the stability of colloidal dispersion system. This energy is called interfacial polar interaction energy ($A-B$ interaction energy for short). The classical DLVO theory is extended to XDLVO theory. Both van der Waals energy and electrostatic interaction energy can only act on short distances < 5 nm [20], while $A-B$ interaction energy is more than two orders of magnitude larger than that of electrostatic interaction energy and van der Waals interaction energy. It can act on medium and long distance and play a leading role in the stability of colloids. Therefore, XDLVO theory can more reasonably describe the aggregation behavior of fine particles and colloids in solution. The total potential energy between particles is composed of van der Waals potential energy, electrostatic interaction energy and $A-B$ interaction potential energy. The total potential energy between particles is expressed in Equation (1):

$$\Phi_T = \Phi_{LW} + \Phi_{EL} + \Phi_{AB} \quad (1)$$

where Φ_T is the total potential energy between particles in the process of condensation; Φ_{LW} is the energy between particles caused by van der Waals gravity potential energy; Φ_{EL} is the electrostatic potential energy when the electric double layer between particles is formed; and Φ_{AB} is the $A-B$ interaction potential energy between particles.

(1) Van der Waals potential energy

Van der Waals force is an attractive force between particles, which comes from dispersion force, induction force and orientation force. The van der Waals potential energy Φ_{LW} between two fine coal particles is described as follows in Equation (2).

$$\Phi_{LW} = -\frac{A}{6h} \times \frac{R_1 R_2}{R_1 + R_2} \quad (2)$$

In Equation (2), R_1 and R_2 are the radii of two spherical particles, h is the distance between two particles and A is the Hamaker constants.

The Hamaker constants of material 1 and 2 in medium 3 are calculated as $A_{132} = (\sqrt{A_{11}} - \sqrt{A_{33}})(\sqrt{A_{22}} - \sqrt{A_{33}})$, where A_{11} , A_{22} and A_{33} are the Hamaker constants of material 1, material 2 and medium 3 in vacuum, respectively. For coal slime water system, it follows from Equation (3).

$$A = (\sqrt{A_{11}} - \sqrt{A_{33}})^2 \quad (3)$$

According to the data [21], the Hamaker constant of coal is 6.1×10^{-20} J, and the Hamaker constant of water is 3.7×10^{-20} J.

(2) Electrostatic interaction energy

In the system of coal slime water, the slime particles are negatively charged, and a certain number of anti-sign ions are adsorbed on the periphery of the particles, forming an electric double layer structure and generating electrostatic repulsion. When the electric double layers of two particles overlap, the repulsive force becomes greater. The interaction energy of electric double layer between particles is expressed as follows [22] in Equation (4).

$$\Phi_{EL} = \pi \epsilon_0 \epsilon_r \frac{R_1 R_2}{R_1 + R_2} \left\{ 2\psi_1 \psi_2 \ln \frac{1 + \exp(-kh)}{1 - \exp(-kh)} + (\psi_1^2 + \psi_2^2) \ln[1 - \exp(-2kh)] \right\} \quad (4)$$

In Equation (4), ε_0 is the absolute permittivity of vacuum, $\varepsilon_0 = 8.854 \times 10^{-12} \text{ C}^2/(\text{J} \times \text{m})$, ε_r is the relative permittivity of dispersive medium (the relative dielectric constant of water is 78.5), ψ_1 and ψ_2 are the surface potential of two particles and k^{-1} is the Debye length.

The unit of k^{-1} is in meters, and it is related to cation valence, absolute temperature and ion concentration. The reciprocal is the thickness of the electric double layer, and the expression is provided in Equation (5).

$$k = \left[\frac{2e^2 N_A \sum (c_m z_m^2)}{\varepsilon_0 \varepsilon_r k T} \right]^{\frac{1}{2}} = 5.58 \times 10^{10} \left(\frac{c_m z_m^2}{T} \right)^{\frac{1}{2}} \quad (5)$$

In Equation (5), e is the elementary charge, $e = 1.6 \times 10^{-19} \text{ C}$; $N_A = 6.022 \times 10^{23} / \text{mol}$, which is the Avogadro constant; k is the Boltzmann number, $k = 1.38 \times 10^{-23} \text{ J/K}$; T is the absolute temperature, K; c_m is cation concentration, mol/L; and z_m represents the cation valence, which is dimensionless. However, it is difficult to accurately determine cation valence and ion concentration in coal slime water; according to the literature, the thickness of the electric double layer is 5 nm.

(3) A–B interaction energy

A–B interaction can determine the coalescence behavior of coal particles to some extent [23].

$$\Phi_{AB} = 2\pi \frac{R_1 R_2}{R_1 + R_2} \lambda_{AB} \Delta G_{y_0}^{AB} \exp(-h/\lambda_{AB}) \quad (6)$$

In Equation (6), attenuation length λ_{AB} is related to the characteristics of the particle itself and has the dimension of unit length. Generally, it is 1–10 nm, and the interaction between hydrophobic particles λ_{AB} takes the maximum [23]; the interaction between hydrophilic particles λ_{AB} takes the minimum value. The value here is 5 nm; $\Delta G_{y_0}^{AB}$ is the A–B free energy per unit area at the interface of two particles in the medium; and the unit is mJ/m^2 . The surface free energy of slime particles is obtained here.

4.2. PTFS Coagulation Mechanism Analysis

Three groups of coal slime water were prepared according to the standard steps of sedimentation test, namely, without any reagent, with 1 mL polyaluminum chloride and 1 mL PTFS (Ti:Fe = 1:7). The slime water was filtered and baked in a 60 °C oven until it is basically dry, and then it was dried at room temperature until it was completely dry. The slime was ground down to a full size of less than 0.075 mm and used to measure its free energy and zeta potential. The results are shown in Table 4.

Table 4. Potential and free energy of slime.

Group	Zeta Potential (mV)	Free Energy (mJ/m^2)
Raw coal group	−71.34	61.07
with polyaluminum chloride	−64.60	57.62
with PTFS	−64.34	58.09

The agglomeration or dispersion between particles is determined by the total acting potential energy Φ_T . If $\Phi_T > 0$, the particles disperse and repel each other; if $\Phi_T < 0$, the particles attract each other. Coagulant can change the flocculation effect by changing the surface electricity or hydrophobicity of particles. The particle size of slime is $R_1 = R_2 = 0.046 \text{ mm}$. The sum of van der Waals energy, electrostatic energy, A–B energy and potential energy is calculated according to Equations (1)–(5). The total potential energy curves of the three are shown in Figure 5.

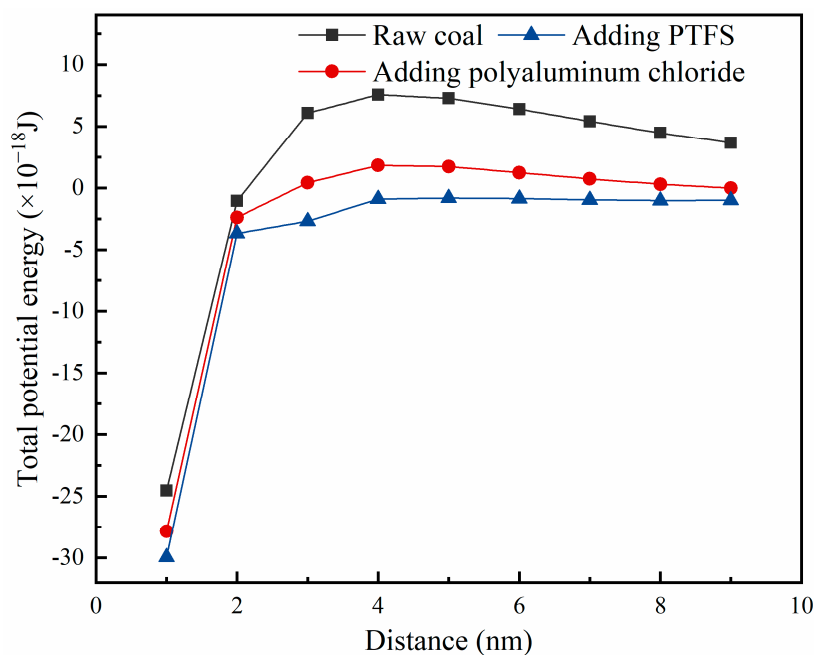


Figure 5. Diagram of distance and total potential energy.

It can be observed from Figure 5 that van der Waals energy is beneficial to the agglomeration of particles. With the increase in the distance between particles, van der Waals energy gradually decreases, and the influence on particle aggregation becomes smaller and smaller. Electrostatic energy is repulsive energy, which makes the particles disperse and is unfavorable to the agglomeration of particles, and the electrostatic potential energy also decreases with the increase in particle distance. The coagulant added in coal slime water can generate positive ions, neutralize the surface charge of particles, compress the electric double layer, reduce the electromotive potential and repulsive force and then cause coagulation. Compared with the data and curves of the above three groups, it can be observed that when polyaluminum chloride and PTFS are added, their electrostatic energy will be significantly reduced compared with raw coal, and the latter electrostatic energy will be reduced more, which also proves theoretically that the coagulation effect of PTFS is better than that of polyaluminum chloride.

For A and B, the interaction energy is the attraction energy, and when the distance is short, the A–B potential energy is larger, which plays a decisive role. It makes the total potential energy negative, which makes the total force behave as the attraction, thus promoting the agglomeration of particles. Compared with the total potential energy of the above three groups, the total potential energy of PTFS group is the lowest, especially in the short distance, which makes the total attraction of PFS group the largest, promotes the agglomeration of particles and makes agglomeration occur faster: The time is then short, and the effect is the best. Moreover, the total potential energy of adding polyferric titanate sulfate is always negative, which proves that only polyferric titanate is added. Ferrotitanium sulfate can make coal slime water settle, and the above experiment also verifies this conclusion.

5. Conclusions

- (1) The coagulant of PTFS has high coagulating speed and good settling effect on coal slime water, and the effect is more obvious when it is used with flocculant PAM. It can clear coal slime water in a short time, obtain clear water with high transmittance and achieve the purpose of solid–liquid separation. The effect is obviously better than that of polyaluminum chloride inorganic coagulant commonly used in coal preparation plants.

- (2) In order to obtain good coal slime water settlement effect, when PAM dosage is 0.66×10^{-10} mol, it should be used together with polymerized titanium iron sulfate with the molar ratio of 1:7, and the dosage of PTFS should not exceed 7.81×10^{-5} mol. When the dosage exceeds it, the transmittance of clear water decreases.
- (3) When titanium-iron = 1:7, the peak transmittance is relatively high, and the effect is slightly higher than other molar ratios.
- (4) Based on XDLVO theory, the coagulation mechanism of PTFS on coal slime water is explained reasonably, which is of great significance to understand the settlement of coal slime water.

Author Contributions: Conceptualization, C.S. and L.T.; methodology, D.G.; software, D.G.; validation, Y.L.; formal analysis, C.S. and L.T.; investigation, D.G. and Z.Z.; resources, C.S.; data curation, Y.L., L.T. and Z.Z.; writing—original draft preparation, C.S. and L.T.; writing—review and editing, Z.Z.; visualization, C.S.; supervision, C.S. and L.T.; project administration, C.S. and L.T.; funding acquisition, C.S. All authors have read and agreed to the published version of the manuscript.

Funding: This work was funded by the Natural Science Foundation of Hebei Province, China (E2018508073), the Science and Technology Project of Hebei Education Department (No. ZC2021025) and the Fundamental Research Funds for the Central Universities of China (Nos. 3142017104, 3142017103).

Institutional Review Board Statement: Not applicable.

Informed Consent Statement: Not applicable.

Acknowledgments: This work is supported by the Natural Science Foundation of Hebei Province, China (E2018508073), the Science and Technology Project of Hebei Education Department (No. ZC2021025) and the Fundamental Research Funds for the Central Universities of China (Nos. 3142017104, 3142017103).

Conflicts of Interest: The authors declare no conflict of interest.

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