Experimental Study on Precipitation Regularity of Asphaltene in Underground Water–Sealed Caverns under Varying Temperature and Pressure Conditions

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ABSTRACT

Through experimental research, this paper reveals the influence of different storage temperatures and pressures on the amount of asphaltene deposition in Russian ESPO crude oil in underground water-sealed caverns. Based on Stokes' settling theory and the experimental results of asphaltene settling at different temperatures and pressures, the Stokes particle settling calculation formula was optimized for temperature and pressure terms. Finally, a particle settling calculation formula that takes into account different sedimentation systems, different crude oil systems, and different temperature and pressure conditions was achieved. The research results show that the asphaltene precipitation of the crude oil system is more affected by temperature than pressure at 10 C-30 C and 0.12MPa-0.28MPa. The precipitation amount decreases with increasing temperature, and the precipitation amount does not show an obvious correlation trend with pressure changes. The research results can provide important theoretical and practical basis for inhibiting the sedimentation loss of long-term storage of crude oil in underground water-sealed caverns.

1. INTRODUCTION

Currently, research on the precipitation patterns of asphaltene in crude oil primarily focuses on the exploration stage of oil fields, particularly within underground reservoirs. Changes in pressure, temperature, and displacement media within porous media result in the deposition of asphaltene, leading to changes in the permeability of the reservoir [1]. However, there is a lack of research on the sedimentation of asphaltene in underground water-sealed storage caverns and above-ground storage tanks under undisturbed conditions. The main objective of this study is to investigate the influence of pressure and temperature variations during static storage on the sedimentation of asphaltene in underground water-sealed caverns [2].

There are currently three recognized mechanisms for asphaltene precipitation in crude oil: (1) Asphaltene exists in crude oil as a true solution, and the precipitation process is considered irreversible. However, some experiments have demonstrated that asphaltene precipitation is partially reversible [3]. (2) Under normal conditions, colloidal-asphaltene and resin-asphaltene form a colloidal solution in crude oil, reaching a dynamic equilibrium state.

Changes in temperature, pressure, and crude oil composition disrupt this equilibrium, resulting in asphaltene precipitation and aggregation into flocculent solids. From a thermodynamic perspective, the aggregation of flocculent sediments is irreversible [4]. (3) There are intermolecular forces, such as van der Waals forces and electrostatic interactions, between asphaltene molecules. When these forces exceed the solvent force, asphaltene molecules aggregate and eventually precipitate [5].

The research methods related to the precipitation behavior of asphaltene in crude oil mainly include laboratory experiments, multi-component equation of state theoretical calculations, and molecular dynamics numerical simulations. The experimental methods encompass: (1) visually observing the precipitation of asphaltene by adding precipitants. (2) Using techniques such as laser, infrared, and ultraviolet transmission spectroscopy to determine and quantify the amount of asphaltene precipitation [6]. (3) Utilizing high-energy X-rays and CT scans with higher transmittance for high-resolution and multi-density imaging of the crude oil solution to analyze the quantity, shape, and distribution of asphaltene at a microscopic scale [7]. Multi-component equation of state theoretical calculations are based on the Peng-Robinson (PR) and Soave-Redlich-Kwong (SRK) equations of state, which calculate the equilibrium phase distribution of all pure components and their contents in the crude oil mixture at specified pressure and temperature conditions. The concept of equilibrium phase distribution means that the fugacity of gas, liquid, and solid phases are equal, achieving thermodynamic equilibrium where the components dispersed or dissolved in each phase reach a consistent state. Iterative calculations continuously adjust the temperature and pressure to determine the solubility of asphaltene in crude oil, and based on the solubility and supersaturation, the amount of asphaltene precipitation in crude oil can be identified. The molecular dynamics numerical simulations, on the other hand, focus on the microscopic scale, establishing models for crude oil and asphaltene molecules [8]. By simulating the motion and interactions of molecules, the micro-mechanisms of asphaltene precipitation can be revealed. During the simulation, the impact of parameters such as temperature, pressure, and time on asphaltene precipitation behavior can be explored by altering physical boundary conditions. This allows for the determination of asphaltene precipitation rate, morphologies, and deposition locations [9].

In summary, the current methods of experimentation, theory, and numerical simulations provide multi-scale and accurate data and theoretical foundations for studying the precipitation patterns of asphaltene in crude oil. However, some limitations exist. For experimental methods, there is a lack of convenient and cost-effective ways to consider the influence of temperature and pressure on asphaltene sedimentation. Optical techniques require expensive optical receivers and signal conversion equipment, making them costly for long-term testing in water-sealed caverns spanning several months or years. Imaging techniques using high-energy X-rays and CT scanning are unable to guarantee sample stability without physical disturbances and are associated with high costs in terms of time and investment. Theoretical calculations and molecular dynamics simulations, while capable of calculating asphaltene sedimentation under different temperature and pressure conditions, require further validation to ensure accuracy. Additionally, these methods often make stringent assumptions, such as neglecting water content in crude oil for equations of state calculations or using generalized molecular models for asphaltene with typical carbon chain lengths. Therefore, theoretical and numerical simulation methods are suitable for studying the regularities of asphaltene sedimentation in crude oil from a mechanistic perspective [10].

In this study, we have improved upon experimental research by combining the method of visual observation with the addition of precipitating agents to simulate different temperature and pressure conditions in a pressure vessel and constant-temperature box. By measuring the difference in asphaltene content before and after static storage through titration with precipitating agents, we can calculate the sedimentation amount of asphaltene in crude oil under different temperature and pressure conditions in a low-cost and highly accurate manner [11]. The experimental results, combined with Stokes settling theory calculations, refine the calculation formula of Stokes settling theory by incorporating the dimensional influence factors of temperature and pressure. Ultimately, an engineering applicable formula considering thermodynamic factors for sedimentation calculations is established [12]. By improving experimental techniques, we can gain a more comprehensive understanding of the mechanisms by which temperature and pressure affect asphaltene sedimentation, thereby enhancing our understanding and predictive capabilities regarding its behavior. This research contributes to the further understanding of sedimentation loss patterns of crude oil during long-term static storage in water-sealed caverns and provides more reliable guidance for practical engineering applications.

2. EXPERIMENTAL STUDY OF CRUDE OIL ASPHALTENE SEDIMENTATION LOSS CONSIDERING TEMPERATURE AND PRESSURE VARIATIONS UNDER STATIC STORAGE CONDITIONS

Experimental Simulation of a Cave-Type Water-Sealed Cavern Located in Northern China, with a Storage Temperature Range of 10°C to 30°C and Storage Pressure Range of 0.12 MPa to 0.28 MPa. The Stored Crude Oil is Low-Sulfur Dewaxed Crude Oil Sourced from the East Siberia-Pacific Ocean (ESPO) Pipeline. The Crude Oil Samples were Collected from the Lin Yuan Oil Pumping Station in Daqing. The Fractional Components of the ESPO Crude Oil were Determined Using Silica-Alumina Column Chromatography [13], and the Carbon Composition of the Crude Oil was Determined Using Gas Chromatography-Mass Spectrometry [14]. Density and Viscosity Measurements of the ESPO Crude Oil Sample were Performed at a Standard Atmospheric Pressure of 20°C [15]. The Physical Properties and Composition of the ESPO Crude Oil Sample are Presented in Table 1 and Figure 1.

Table 1 Data on fractional component analysis of ESPO crude oil

Saturated Components saturate/%	Aromatic Component saromatic/%	Resins resin/%	Asphaltenes asphaltene/%
56.30	26.47	10.72	6.51
Density kg/m³	**Dynamic Viscosity Pa·s (20°C)**	**Kinematic Viscosity mm²/s (20°C)**	**Sulfur Content mass %**
841.5	0.01147	13.63	0.535

The purpose of the asphaltene sedimentation experiments under different temperature and pressure conditions is to determine the influence of temperature and pressure on the sedimentation loss of asphaltenes in crude oil [16]. The experiments were conducted using a dual-phase stainless steel pressure vessel capable of withstanding up to 3 MPa as the pressure
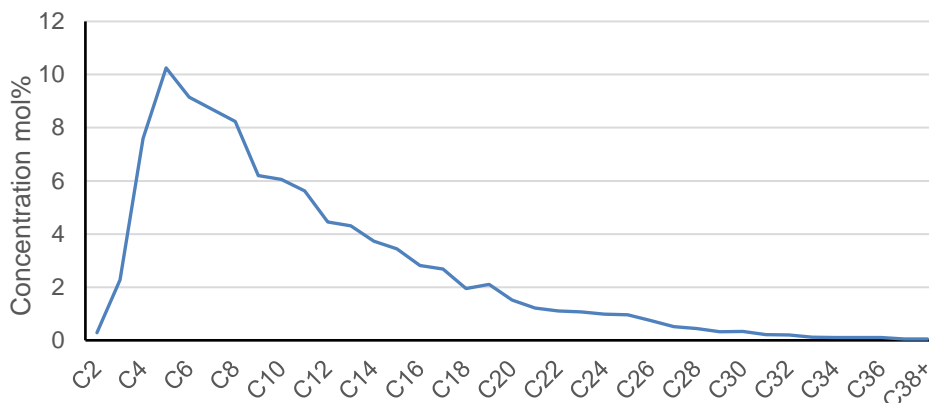
Figure 1 Carbon composition analysis of ESPO crude oil by gas chromatography-mass spectrometry of total hydrocarbons.

environment simulation device, and a thermostatic chamber as the temperature environment simulation device. The simulated storage pressure and temperature conditions were set to match the real conditions of the underground water-sealed cavern: pressures of 0.12 MPa, 0.2 MPa, and 0.28 MPa (By injecting nitrogen gas at varying pressures, different storage pressures can be achieved.); temperatures of 10°C, 20°C, and 30°C (Different storage temperatures are achieved by injecting water into the underground cavern's water curtain for sealing.). In the experimental process, the crude oil sample was first placed in an open cone-shaped flask, which was then placed in the intermediate container. Nitrogen gas was used to pressurize the intermediate container until the desired pressure was reached, and then the intermediate container with the flask was placed in the thermostatic chamber to adjust the environmental temperature [17]. The experimental procedure is shown in Figure 2.

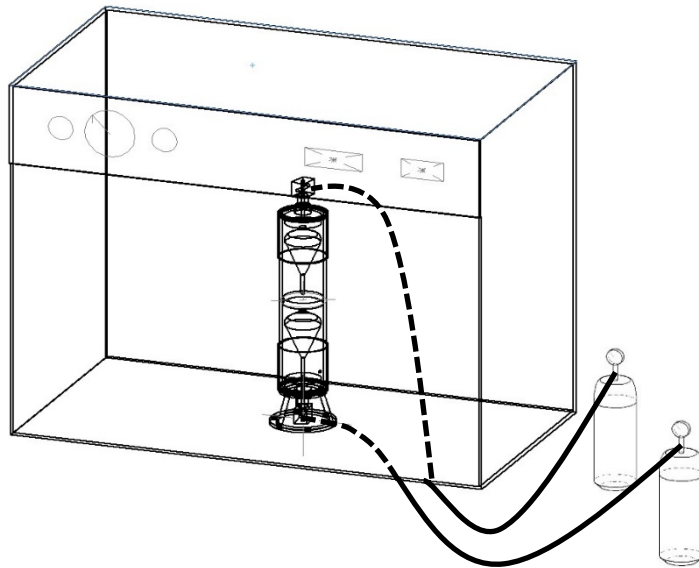(a) The oil sample was filled into the conical flask.

(b) The conical flask was placed inside the intermediate container.

(c) Pressurizing the intermediate container using nitrogen gas.

(d) Setting the environmental temperature using a thermostatic chamber.

(e) Overall assembly diagram of experimental equipment

Figure 2. Experimental process diagram

In the physical experiments, the quantification of asphaltene sediment in the crude oil was carried out by measuring the difference in asphaltene concentration before and after the experiment. The measurement of asphaltene concentration involved thorough mixing of the crude oil with n-heptane (n-C_7H_{16}) to precipitate the insoluble asphaltenes, followed by filtration to separate the asphaltenes from the crude oil [18]. By comparing the volume proportion of asphaltenes in the crude oil before and after the sedimentation experiment, the amount of asphaltene sediment in the crude oil can be calculated.

3. RESULTS OF ASPHALTENE SEDIMENTATION LOSS EXPERIMENTS IN CRUDE OIL UNDER - DIFFERENT TEMPERATURE AND PRESSURE CONDITIONS.

The results of the asphaltene sedimentation loss experiments in Russian ESPO crude oil, after being stored under different pressures and temperatures for 60 days, are presented in Table 2. From the experimental results, it can be observed that temperature has a significant influence on the sedimentation of asphaltenes in the crude oil, with a decrease in sedimentation as temperature increases, the data results curve in Figure 3. The effect of pressure variations on sedimentation is not clearly evident and does not exhibit a consistent trend. However, the data results curve in Figure 4 indicates a very minimal change in sediment content after increasing the pressure.

Table 2: Sedimentation Experiments of Russian ESPO Crude Oil at Different Pressure and Temperature

Temperature °C	Pressure MPa	Sedimentation ml	Sedimentation Volume Percentage
10		0.052	0.052%
20		0.05	0.05%
30	0.2	0.045	0.045%
40		0.043	0.043%
	0.12	0.05	0.05%
20	0.2	0.05	0.05%
	0.28	0.051	0.051%
	0.36	0.05	0.05%

According to the solvency theory of colloidal solutions, solubility in the liquid phase is temperature-dependent but independent of pressure. Based on the experimental conditions, it can be assumed that the increase in pressure leads to a decrease in the fugacity of light hydrocarbons in the crude oil system, resulting in lower concentrations of light hydrocarbons compared to lower pressure conditions [19]. Consequently, the content of n-heptane, the asphaltene precipitant in the crude oil, is higher than that in the samples under lower pressure, leading to a slight increase in asphaltene sedimentation in the crude oil [20]. However, some literature studies suggest that the solubility of asphaltenes in crude oil is influenced by pressure. Generally, with increasing pressure, the solubility of asphaltenes tends to increase [21]. This is because increasing pressure lowers the boiling point of the oil, which in turn increases the solubility of the system, allowing for more dissolution of asphaltenes in the oil.
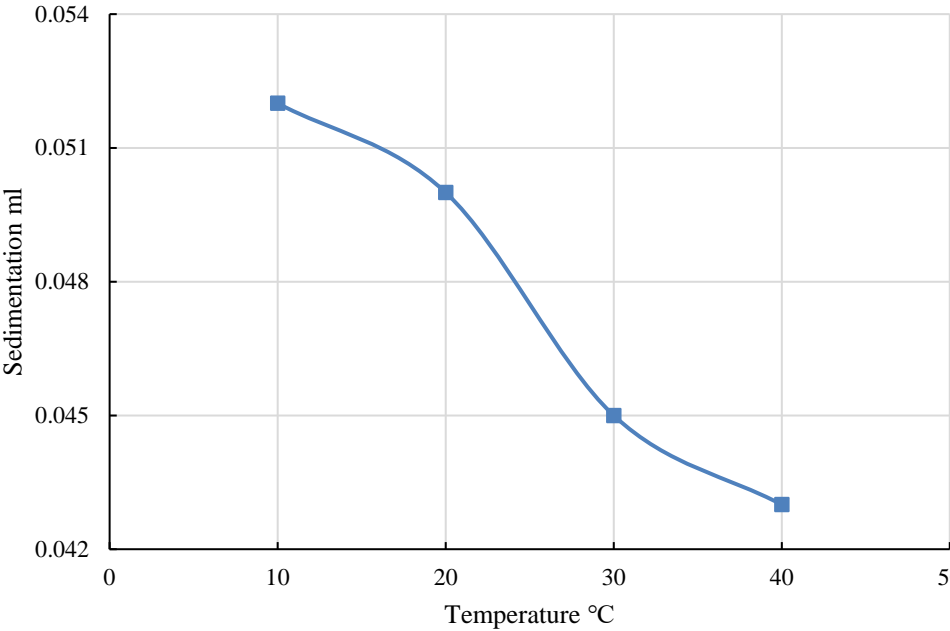
Figure 3. Trends of sedimentation experiments for colloid-asphaltene precipitation under different temperature conditions
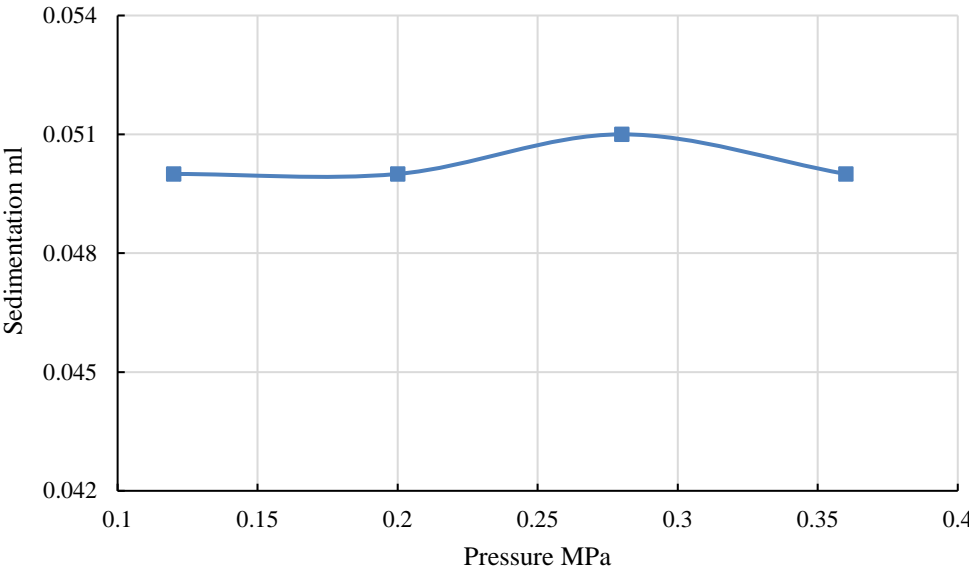

Figure 4. Trends of sedimentation experiments for colloid-asphaltene precipitation under different pressure conditions

During the experimental process, ultraviolet light (imaging) and distilled water were utilized to test the density of the precipitate [22]. Distilled water was injected into the bottom of the pear-shaped bottle containing the oil sample using a syringe and a 20 cm long needle. The density difference between the precipitate and the distilled water was observed, and it was found that within a short period (60 days of experimentation), the density of the precipitate in the crude oil did not exceed 1000 kg/m^3, as shown in the figure 5. The precipitate could not penetrate the bottom water cushion layer of the water-sealed underground caverns and settle to the bottom of the tanks. Additionally, different density precipitates were formed in crude oils with varying densities. For light or medium crude oils, under conditions of sealed, non-volatile light alkanes, and absence of oxygen, it is difficult to generate pure asphaltene precipitates with a density greater than 1000 kg/m^3 [23].
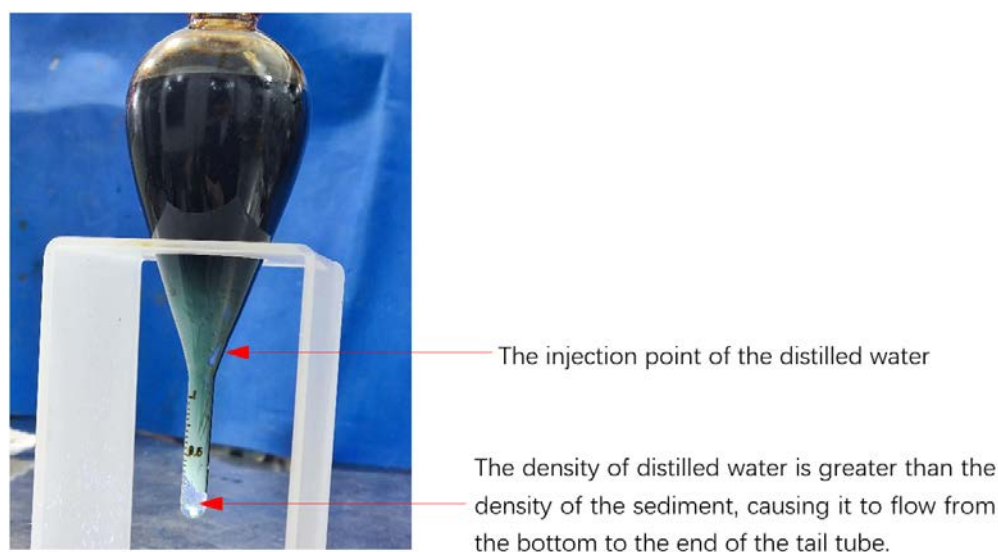

The injection point of the distilled water

The density of distilled water is greater than the density of the sediment, causing it to flow from the bottom to the end of the tail tube.

Figure 5. Density difference between crude oil sediments and distilled water

4. THE STOKES' LAW DESCRIBES THE SETTLING BEHAVIOR OF PARTICLES IN A FLUID MEDIUM.

In crude oil systems, precipitation can generally be divided into two categories: conventional precipitation of mechanical impurities and flocculation settling of asphaltene[24]. In the case of conventional precipitation related to mechanical impurities, particles primarily settle under the influence of gravity. However, in the context of asphaltene flocculation settling, it involves more intricate physical, chemical, and interfacial electrostatic interactions. This includes the adsorption and coagulation of particles, as well as the disruption of colloidal system dynamic equilibrium due to variations in temperature, pressure, and crude oil composition. It is generally believed that asphaltene and colloids undergo flocculation to form precipitates.

Stokes Particle Sedimentation Theory assumes that the settling material consists of particles of various shapes and suggests that the sedimentation velocity is related to the diameter, viscosity, and density of the particles [25]. When objects move in a viscous fluid, a layer of adhering fluid forms on their surface. This adherent layer moves along with the object's motion, resulting in viscous resistance exerted by the surrounding fluid. Research

indicates that when the relative velocity of the objects is relatively slow, the influence of inertial forces can be neglected. The viscous resistance experienced by an object is directly proportional to the fluid's viscosity, the object's density, and the object's velocity of movement. The proportionality constant is determined solely by the shape of the object. In most cases, the proportionality constant for metallic mechanical impurities in crude oil is 0.7 times that of the spherical proportionality constant, falling somewhere between polygonal and elongated shapes [26]. Stokes theoretically derived that for spherical particles in flow conditions with Re < 1, the proportionality constant is 6π. In other words, when a sphere with a radius of r moves at a velocity v relative to the fluid in a fluid with viscosity coefficient μ, the viscous resistance it experiences can be expressed as:

$$f = 6\pi\mu v r \tag{1}$$

where μ is dynamic viscosity of the solution, Pa·s; v is settling velocity of particles in the colloidal suspension, m/s; r is radius of particles in the colloidal suspension, m.

The forces acting on a small sphere moving in a viscous fluid include gravity, buoyancy, and viscous drag. The resultant force is:

$$F = \frac{4}{3}\pi r^3 \rho_{\text{solid}} g - \frac{4}{3}\pi r^3 \rho_{\text{oil}} g - 6\pi\mu v r \tag{2}$$

where ρ_{solid} is density of particles in the colloidal suspension, kg/m³; ρ_{oil} is density of colloidal suspension, kg/m³; F is the resultant force acting on particles in the colloidal suspension, N.

Under this combined action, the small sphere undergoes accelerated descent, but the viscous drag force increases as the descent velocity increases. When the velocity reaches a certain value, the three forces balance, and the small sphere descends at a constant speed. At this point, we have:

$$\frac{4}{3}\pi r^3 (\rho_{\text{solid}} - \rho_{\text{oil}}) g = 6\pi\mu v r \tag{3}$$

$$v = \chi \frac{1}{18\mu} d^2 (\rho_{\text{solid}} - \rho_{\text{oil}}) g \tag{4}$$

where d is the diameter of particles in the colloid suspension, m; χ is the dimensionless irregularity coefficient of particle shape, representing the degree of irregularity. For mechanically impure substances, χ is taken as 0.7.

The Equation (4) represents Stokes' law of sedimentation, which allows for the prediction of sedimentation patterns based on particle size characteristics. This equation indicates that when spherical objects, such as solid mechanical impurity particles in crude oil, settle in a viscous fluid, the sedimentation velocity is directly proportional to the gravitational acceleration, the density difference between the particles and the fluid, and the square of the radius (diameter) of the spherical particles. However, it is inversely proportional to the dynamic viscosity of the fluid. In this study, during the precipitation experiments, mechanical

filtration similar to that used for crude oil in oil depots was applied. Filtration paper was used to remove existing mechanical impurities.

During the precipitation process of asphaltene in crude oil, it can undergo aggregation with colloids or other mechanical impurities, which is known as the flocculation process [27]. After flocculation, chemical bonding and surface electron interactions occur, leading to the formation of larger precipitates with an accelerated settling rate. The efficiency of flocculation settling depends not only on the surface charge and distribution of chemical bonds in the flocculates but also on the settling time (reaction time) [28]. Therefore, different types of compounds, molecular structures, and concentration distributions exhibit significant variations in the flocculation settling behavior. Currently, there is no precise mathematical model to calculate the flocculation settling velocity. Instead, experimental data combined with the Stokes' Law for particle settling theory are used to characterize the flocculation settling velocity [29].

$$V_f^2 = \frac{4g(\rho_f - \rho_w)D_f}{3C_d\rho_w} \tag{5}$$

where V_f is the settling velocity of flocs, m/s; D_f is equivalent diameter of flocs, m; g is the gravitational acceleration, m/s^2; ρ_f is the density of floc sediment, kg/m^3; ρ_w is the density of the crude oil solution, kg/m^3; Re is the Reynolds number, dimensionless; μ is the dynamic viscosity of the solution, Pa·s; C_d is the drag coefficient of floc sedimentation, dimensionless.

$$Re = \frac{\rho_w V_f D_f}{\mu} \tag{6}$$

$$C_d = \frac{24}{Re} + \frac{3}{Re^{0.5}} + 0.34 \tag{7}$$

settling velocity of asphaltene flocs.

$$V_f = \sqrt{\frac{4g(\rho_f - \rho_w)D_f}{(72\mu + 9\sqrt{\rho_w V_f D_f \mu} + 1.02\rho_w V_f D_f)\rho_w}} \tag{8}$$

Based on the indoor experimental results of asphaltene precipitation, it is evident that there is a pronounced relationship between precipitation volume and temperature in the crude oil system. During the experiments, a 100ml pear-shaped flask was used, and the volume of crude oil remained constant, with no changes in other experimental conditions except for variations in temperature and pressure. Consequently, the change in temperature led to differences in the precipitation volume, which is equivalent to alterations in precipitation rate within the same experimental time frame [30]. According to the results of a 60-day crude oil precipitation experiment, as shown in Table 3, it can be inferred that higher temperatures result in less precipitation volume, as shown in the figure 6, and the variation in precipitation rate is inversely correlated with temperature increases.

(a) 0.043ml at 40 ℃

(b) 0.045ml at 30℃

(c) 0.05ml at 20℃

(d) 0.052ml at 10℃

Figure 6. Volume of sedimentation of crude oil at different temperatures within 60 days.

Table 3: Sedimentation Experiments of Russian ESPO Crude Oil at Different
Pressure and Temperature

Temperature difference °C	Temperature range °C	Pressure MPa	Reduction in precipitate amount ml	Change in sedimentation rate %
10	10~20		0.002	3.85
20	10~30	0.2	0.007	13.46
30	10~40		0.009	17.31
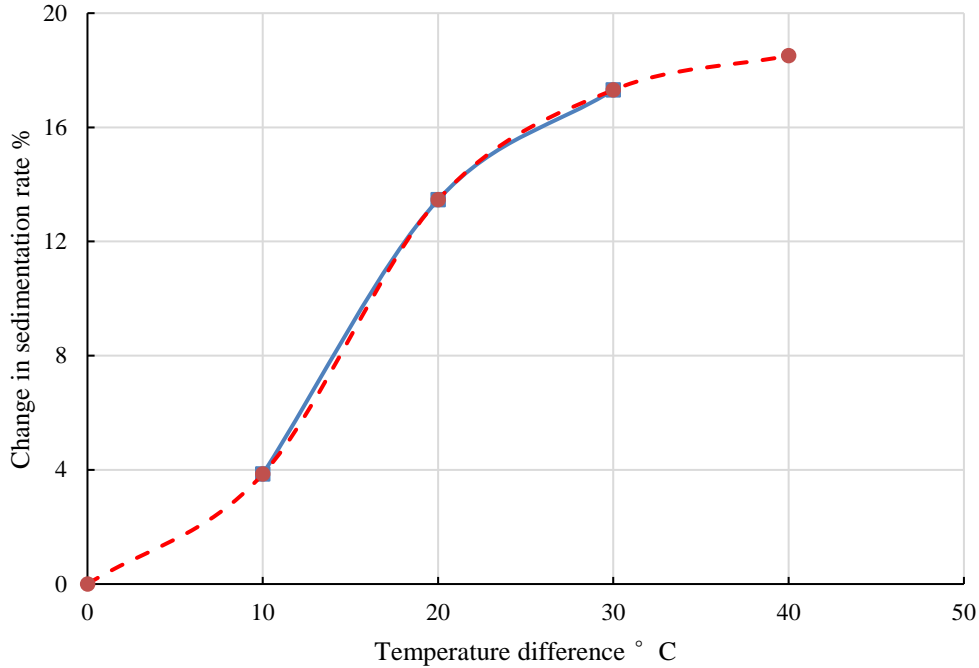
Based on the experimental results, it is evident that the main influencing factor for crude oil sedimentation in underground water-sealed caverns is temperature. Temperature variation significantly affects the movement of particles in the colloidal solution. Therefore, under such practical conditions, the sedimentation rate of the sediment cannot be represented by the equation (8)[31]. Therefore, it is possible to describe the sedimentation velocity at different temperatures by multiplying the sedimentation velocity by the rate of change derived from the temperature-dependent equation fitted to the experimental results. The function curve fitted based on the experimental results is shown in Figure 7, the resulting in a correlation of 0.978, and the expression is as follows:

$$\eta = \frac{19.404}{1+\left(\frac{T}{15.484}\right)^{-3.194}} \tag{9}$$

where T is the temperature difference, ℃; domain is 1℃~30℃; η is the percentage change in sedimentation rate, %.

$$V_f = (1-\eta)\sqrt{\frac{4g(\rho_f-\rho_w)D_f}{(72\mu+9\sqrt{\rho_w V_f D_f \mu}+1.02\rho_w V_f D_f)\rho_w}} \qquad (10)$$

Based on the relationship between temperature difference and sedimentation rate, it can be inferred that as the temperature rises, the sedimentation rate decreases, albeit with varying degrees of reduction. Once the temperature difference exceeds 20°C, the decrease in sedimentation rate becomes less pronounced, indicating an enhancement of Brownian motion in the crude oil system at higher temperatures [32]. Brownian motion refers to the erratic movement of molecules, where suspended particles experience continuous and irregular thermal motion. Brownian motion can persist indefinitely, thereby influencing macroscopic properties such as density, temperature, and the pressure of light hydrocarbons within the system. The intensity of Brownian particle movement depends on their velocity and mass, with velocity increasing with temperature and decreasing with reduced solution viscosity.

The significance of Brownian motion lies not only in its role as evidence of molecular existence but also as a manifestation of the chaotic nature of molecular thermal motion. Regarding the precipitation behavior of asphaltene in quiescent crude oil, more intense Brownian motion weakens the aggregation and sedimentation tendencies of asphaltene. Furthermore, an elevation in the crude oil system's temperature leads to a reduction in system viscosity, resulting in decreased electron density on the outer layer of asphaltene and consequently weakening the coagulation and precipitation effect [33].

5. CONCLUSION

This paper combines experimental research results with theoretical analysis. Taking Russian ESPO pipeline crude oil as an example, the influence of temperature on asphaltene precipitation in the crude oil system is incorporated into the Stokes sedimentation velocity formula in non-dimensional form. This approach enables the development of an engineering-applicable sedimentation velocity calculation formula that considers thermodynamic factors. The following conclusions are drawn:

(1) Precise measurement of asphaltene precipitation in static stored crude oil over a certain period can be achieved using a pressurized pressure vessel, a constant temperature chamber, and n-heptane (n-C_7H16) as a solvent.

(2) The sedimentation of asphaltene in Russian ESPO crude oil under water-sealed tank storage conditions is sensitive to temperature variations. During the operation of underground water-sealed tank storage, it is recommended to maintain pressure above the oil's saturation pressure to reduce the evaporation of light components. Simultaneously, increasing the operating temperature can slow down asphaltene precipitation. Under temperature differences between 10 to 30°C, the sedimentation rate of asphaltene can be reduced by 3.85% to 17.31%.

(3) The density of the sediment from Russian ESPO crude oil over a 60-day period is similar to that of the crude oil, and no solid particles or precipitation with density greater than water are observed.

(4) By incorporating the influence of temperature on sedimentation velocity in non-dimensional form into the Stokes sedimentation velocity formula, the time required for complete asphaltene precipitation in the crude oil system can be calculated for different storage periods.

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