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APPLICATION OF FUSION PROPERTY CALCULATIONS FOR KAZAKHSTANI OIL SAMPLES

J.A. ISMAILOVA1 , PhD, associate professor j.ismailova@satbayev. university

I.A. SPIVAKOVA2 , Petroleum engineering bachelor student at KBTU, *i_spivakova@kbtu.kz*

I.M. BAYANOV2 , Petroleum engineering bachelor student at KBTU, *i_bayanov@kbtu.kz*

D.N. DELIKESHEVA1 , Master, Senior lecturer, *d.delikesheva@satbayev. university*

A.R. KHUSSAINOVA3 , Master, *S289850@studenti.polito.it*

A.M. KAIDAR1 , Master, Lead Engineer, *a.kaidar@satbayev.university*

1 SATBAYEV UNIVERSITY, 22/5, Satbayev str., Almaty, 050000,Republic of Kazakhstan

2 KAZAKH-BRITISH TECHNICAL UNIVERSITY Republic of Kazakhstan, 050000, Almaty, Tole Bi, 59

3 POLITECNICO DI TORINO, Corso Duca degli Abruzzi, 24, Turin, 10129, Italy

This study thoroughly investigates the melting properties and solid-state transition temperatures of crude oil from Kazakhstan's oil field, which is critical for predicting wax precipitation. Utilizing advanced laboratory equipment such as gas chromatographs, differential scanning calorimeters (DSC), and pour point testers, we obtained comprehensive data on these parameters. Based on the results, we developed new correlations for the melting point and solid-state transition temperature of crude oil, which demonstrate closer alignment with the reference values for various hydrocarbons' melting points compared to standard prediction models. These findings provide more accurate models for wax deposition, essential for efficient crude oil transportation and processing. The results indicated that implementing these new correlations could lead to significant cost savings by reducing pipeline blockages and maintenance requirements, thereby improving overall *operational efficiency.*

KEY WORDS: wax determination, wax concentration, wax appearing temperature, Won Correlation, Nichita correlation, gas chromatograph, DSC, pour point tester, ASTM-2887 Standard.

ҚАЗАҚСТАНДЫҚ МҰНАЙ ҮЛГІЛЕРІ ҮШІН БАЛҚУ ҚАСИЕТТЕРІНІҢ ЕСЕПТЕУЛЕРІН ҚОЛДАНУ

Д.A. ИСМАИЛОВА1 , PhD, қауымдастырылған профессор, *j.ismailova@satbayev.university* **И.А. СПИВАКОВА2 ,** ҚБТУ мұнай инженері бакалавры, *i_spivakova@kbtu.kz* **И.М. БАЯНОВ2 ,** ҚБТУ мұнай инженері бакалавры, *i_bayanov@kbtu.kz* **Д.Н. ДЕЛИКЕШЕВА1 ,** магистр, аға оқытушы, *d.delikesheva@satbayev.university* **А.Р. ХУСАИНОВА3 ,** магистр, *S289850@studenti.polito.it* **А.М. ҚАЙДАР1 ,** магистр, ғылыми қызметкер, *a.kaidar@satbayev.university*

> 1 SATBAYEV UNIVERSITY, Қазақстан Республикасы, 050013, Алматы қ., Сәтбаев қ., 22

> 2 ҚАЗАҚСТАН-БРИТАН ТЕХНИКАЛЫҚ УНИВЕРСИТЕТІ, Қазақстан Республикасы, 050000, Алматы қ., Төле би қ., 59

3 POLITECNICO DI TORINO, Италия, 10129, Турин қ., Corso Duca degli Abruzzi қ., 24

Бұл зерттеу балауыздың жауын-шашынын болжау үшін өте маңызды болып табылатын Қазақстандық мұнай кен орнынан шикі мұнайдың балқу қасиеттері мен қатты күйдегі ауысу температурасын мұқият зерттейді. Газ хроматографиясы, дифференциалды сканерлеу калориметрлері (DSC) және құю нүктелерін сынаушылар сияқты озық зертханалық жабдықты пайдалана отырып, біз осы параметрлер бойынша жан-жақты деректерді алдық. Нәтижелерге сүйене отырып, біз шикі мұнайдың балқу температурасы мен қатты күйдегі ауысу температурасының жаңа корреляцияларын әзірледік, олар стандарты болжау үлгілерімен салыстырғанда әртүрлі көмірсутектердің балқу температураларының эталондық мәндеріне жақынырақ сәйкестікті көрсетеді. Бұл нәтижелер

шикі мұнайды тиімді тасымалдау және өңдеу үшін қажет балауызды тұндырудың дәлірек үлгілерін қамтамасыз етеді. Нәтижелер осы жаңа корреляцияларды енгізу құбырлардың бітелуін және техникалық қызмет көрсету талаптарын азайту, осылайша жалпы пайдалану тиімділігін арттыру арқылы шығындарды айтарлықтай үнемдеуге әкелетінін көрсетті.

ТҮЙІН СӨЗДЕР: парафин құрамын, парафин концентрациясын, парафиннің пайда болу температурасын анықтау, Вон корреляциясы, Никита корреляциясы, газ хроматографы, DSC, қатаю температурасын сынаушы, ASTM-2887 стандарты.

ПРИМЕНЕНИЕ РАСЧЕТОВ СВОЙСТВ ПЛАВЛЕНИЯ ДЛЯ ОБРАЗЦОВ КАЗАХСТАНСКОЙ НЕФТИ

Д.A. ИСМАИЛОВА1 , PhD, ассоц. профессор, *j.ismailova@satbayev.university* **И.А. СПИВАКОВА2 ,** студент по специальности «Нефтегазовое дело» КБТУ, *i_spivakova@kbtu.kz*

И.М. БАЯНОВ2 , студент по специальности «Нефтегазовое дело» КБТУ, *i_bayanov@kbtu.kz*

Д.Н. ДЕЛИКЕШЕВА1 , магистр, старший преподаватель, *d.delikesheva@satbayev.university* **А.Р. ХУСАИНОВА3 ,** магистр, *S289850@studenti.polito.it* **А.М. ҚАЙДАР2 ,** магистр, ведущий инженер, *a.kaidar@satbayev.university*

> 1 SATBAYEV UNIVERSITY, Республика Казахстан, 050013, г. Алматы, ул. Сатпаева, 22

2 КАЗАХСТАНСКО-БРИТАНСКИЙ ТЕХНИЧЕСКИЙ УНИВЕРСИТЕТ, Республика Казахстан, 050000, г. Алматы, ул. Толеби, 59

3 POLITECNICO DI TORINO, Италия, 10129, г. Турин, ул. Corso Duca degli Abruzzi, 24

Рассматриваются свойства плавления и температуры перехода в твердое состояние сырой нефти с казахстанского нефтяного месторождения, что имеет решающее значение для прогнозирования осаждения парафина. Используя современное лабораторное оборудование, такое как газовая хроматография, дифференциальные сканирующие калориметры (ДСК) и датчики температуры застывания, мы получили исчерпывающие данные по этим параметрам. Основываясь на полученных результатах, мы разработали новые корреляции для температуры плавления и перехода сырой нефти в твердое состояние, которые демонстрируют более близкое соответствие с эталонными значениями для различных температур плавления углеводородов по сравнению со стандартными моделями прогнозирования

Данные результаты позволят получить более точные модели осаждения парафина, необходимые для эффективной транспортировки и переработки сырой нефти. Результаты показали, что внедрение этих новых соотношений может привести к значительной экономии средств за счет уменьшения засорения трубопроводов и требований к техническому обслуживанию, тем самым повышая общую эффективность эксплуатации.

КЛЮЧЕВЫЕ СЛОВА: определение содержания парафина, концентрации парафина, температуры появления парафина, корреляция Вона, корреляция Ничиты, газовый хроматограф, DSC, тестер температуры застывания, стандарт ASTM-2887.

ntroduction. Crude oil is a complex blend of various hydrocarbon and non-
hydrocarbon components. Typically, the hydrocarbon components include
asphaltenes, resins, aromatics, naphthenes, and paraffins. These components hydrocarbon components. Typically, the hydrocarbon components include asphaltenes, resins, aromatics, naphthenes, and paraffins. These components usually remain stable within the crude oil system until there is a disturbance in the equilibrium. Factors such as changes in pressure, temperature, and oil composition are crucial in causing disequilibrium, leading to instability in the crude oil system.

Most crude oil in Kazakhstan contains heavy hydrocarbons that precipitate as paraffin (wax) solids at low temperatures. Paraffin, a heavy component of crude oil, solidifies below the pour point. The deposition of paraffin on pipeline walls is a significant flow assurance issue, causing reduced or complete blockage of oil flow by decreasing the pipeline's crosssectional flow area. Additionally, onshore facilities face higher energy consumption and equipment failures due to paraffin plugs. Wax deposition also increases the viscosity of the oil mixture, necessitating higher energy for crude oil transportation. Effective management of wax-related issues in crude oil requires a comprehensive understanding of its composition and behavior under different temperature conditions. Various mitigation strategies are employed, including chemical inhibitors, thermal insulation, and mechanical cleaning of pipelines to prevent or remove wax deposits.

Materials and methods. A gas chromatograph is an analytical instrument used for the chromatographic separation of a substance mixture, allowing the analysis of its components and the properties of complex mixtures. This method is highly effective for accurate laboratory studies to refine equations, as it helps assess the uniformity of a substance and enables the separation and identification of substances with similar properties. A gas chromatograph boasts high accuracy, rapid analysis, sensitivity, and the ability to integrate with other research methods.

Gas chromatography is a form of chromatography where the mobile phase is an inert gas (carrier gas) that flows through a stationary phase with a large surface area. Common carrier gases include helium, nitrogen, argon, hydrogen, carbon dioxide, or air. In our work, helium was used as the mobile phase.

The carrier gas must be inert towards the substances being separated and the sorbent, explosion-proof, and sufficiently clean. The selection of a carrier gas should ensure its physical properties are suitable for achieving high column efficiency and adequate detector sensitivity. Gas chromatography separates volatile compounds and can analyze gaseous, liquid, and solid substances with a molecular weight of less than 400, provided they meet requirements such as volatility, thermal stability, inertness, and ease of production. Quantitative analysis is feasible only if the substance is heat-resistant, meaning it can evaporate consistently in the dispenser and elute from the column without decomposing.

When a substance decomposes, false peaks related to decomposition products can appear on the chromatogram. The substance should not form stable solvates in the stationary liquid phase or react with the materials from which the chromatograph parts are made. Organic substances typically meet these requirements, which is why GC is often used to analyze organic compounds, though almost all elements of the periodic table can be determined as volatile compounds using this method. Depending on the state of the stationary phase, gas chromatography is classified into gas adsorption (with a solid adsorbent as the stationary phase) and gas-liquid (with a liquid deposited on a solid

carrier as the stationary phase). Gas chromatography primarily uses an eluent method for carrying out the chromatography process.

Results and discussion. The schematic diagram of the chromatograph Gas analytical chromatograph is a set of interacting systems designed to perform analysis in the optimal mode of chromatographic separation of the mixture under study in order to determine its composition. A gas chromatograph consists of the following main parts: a carrier gas treatment system, a dispenser, a chromatographic column, a detector, a temperature control system, and a recording device. A schematic (functional) diagram of an analytical laboratory gas chromatograph is shown in *Fig. 1*. The carrier gas from the high–pressure cylinder 1 through the flow regulator 2, seizing a sample of the analyzed mixture from the metering tap or evaporator, is sent to the chromatographic column 5. After the column, the carrier gas, together with the mixture component, enters the detector 6 and then into the atmosphere. The detector converts a change in the physical or physic–chemical properties of binary mixtures (carrier gas component compared to pure carrier gas) into an electrical signal, which is recorded by a recorder 7. The temperature of the column and detector is maintained constant by thermostats 4.

Figure 1 – **Schematic diagram of a gas chromatograph** *1 – cylinder with carrier gas; 2 – flow regulator; 3 – sample entry point (metering valve, evaporator); 4 – thermostats; 5 – column; 6 – detector; 7 – recorder. (Source: Gas chromatography. Guidelines for laboratory work)*

1. Carrier Gas Treatment System. The carrier gas, when it emerges from the cylinder, may contain impurities such as oxygen, water, and organic compounds. These contaminants can affect both the efficiency of the detection system and the accuracy of substance separation within the chromatographic column. Thus, it is crucial to remove these impurities before using the gas. Oxygen is effectively removed using specialized catalysts operating at room temperature, water vapor is eliminated by molecular sieves, and organic compounds are absorbed by activated carbon. The velocity at which the gas moves through the chromatographic system is critical for precise component separation. Fluctuations in gas flow rate during analysis can lead to compromised results. Therefore, chromatographs are equipped with advanced devices to stabilize gas flow and accurately measure its velocity, ensuring consistency and reliability in data acquisition. These measures enhance the efficiency of the chromatographic process and contribute to the overall reliability and reproducibility of analytical outcomes.

2. Flow Regulator. The gas flow within the chromatograph system is carefully regulated using throttles to ensure precise control over the carrier gas movement. Stability, which is crucial for accurate chromatographic analysis, is maintained using either a pressure regulator or a flow regulator. These regulators work together to maintain a consistent carrier gas velocity, ensuring accuracy within a narrow margin of about $\pm 1\%$ across the entire temperature range relevant to the column's operation. To counter the increasing resistance encountered within the column as the temperature rises, it is essential to apply sufficient pressure to the carrier gas at its inlet to the chromatograph. For columns with nozzles, a pressure of 550 kPa (80 psi) is generally adequate. However, capillary columns have different requirements, with a recommended pressure range of 10 to 70 kPa (1.5 to 10 psi) to ensure optimal performance and efficiency.

3. Dosing devices. The effectiveness of chromatographic separation depends on the method and accuracy of sample introduction into the apparatus. It's essential that the sample's composition accurately reflects that of the analyzed mixture. Any sample loss during introduction, such as through evaporation, or the presence of residual volumes in the dispenser ("dead" volumes), can compromise the integrity of both the sample and the mixture, impacting the analysis accuracy. The sample size is determined by the sensor's sensitivity and the column's capacity. Maintaining an accuracy range of 1-3% in terms of the sample's volume or mass is crucial. Efforts are made to introduce the sample into the column quickly to prevent peak smearing on the chromatogram and to preserve the device's operational efficiency. For the measurement and injection of gaseous mixtures, specialized metering valves with calibrated loops are used, allowing for sample injections ranging from 0.1 to 10 ml. Liquid samples are introduced into the column through micro-nozzles via a specialized evaporator seal capable of withstanding high temperatures. Depending on the sensor type, the volume of the injected sample can range from 0.1 to 50 microliters.

4. Temperature control system. The thermostat in a gas chromatograph is responsible for generating the additional temperature required for nozzle columns. It comprises a specialized chamber with dual insulating layers. Inside this chamber, a flat heater provides warmth, while a sensor ensures precise temperature control. The temperature of the thermostat is adjusted using the chromatograph switch's tap. Maintaining column temperature stability is crucial in chromatography, typically requiring an accuracy of 0.2 $^{\circ}$ C. To achieve these precise temperature conditions for the column, detector, and metering devices, they are housed within dedicated thermostats regulated by the main thermostat. During the analysis, if there is a need to increase the column temperature, a temperature programmer is used. Additionally, chromatographs often feature air thermostats equipped with fans to assist in temperature control. This setup ensures the thermostat functions optimally, creating the necessary temperature conditions for accurate chromatographic analysis.

1. When the chromatograph is plugged into the outlet, the thermostat begins to adjust to the desired temperature. One end of the heater is always connected to one of the poles of the outlet. The other end of the heater, which is permanently connected to one of the terminals of the resistor, is connected to the other pole of the outlet via an electronic key controlled by the controller. When an electronic key opens, it simultaneously closes another one, and the current passing through this path is relatively small, so it does not affect the main current passing through the "heater - electronic key" circuit.

2. In this state, electronic keys and half-waves of electric current, both positive and negative, with the amplitude of the network, are supplied to the heater. The flaps are closed at the same time, and there is no exchange of air with the external environment inside the thermostat.

3. Each half-wave of the supply voltage transmits to the thermostat chamber an amount of heat proportional to the amplitude of this voltage and the conductivity of the heater. As a result, the temperature in the thermostat becomes pulsating with twice the frequency of the network, but the amplitude of this oscillation is greatly reduced due to the operation of the fan.

4. The thermostat continues to adjust the temperature until the sensor informs the controller that the desired temperature has been reached. From this moment on, the thermostat switches to temperature maintenance mode.

5. In this mode, the thermostat maintains a thermal balance inside the chamber. Now the amount of heat needed to maintain the temperature is reduced, and the controller outputs a certain amount of half-wave voltage required to maintain thermal balance.

Table 1 – **Technical characteristics of the thermostat**

Figure 2 – **Thermostat**

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5. Chromatographic column. The chromatographic column is arguably one of the most crucial components of a chromatograph. It operates by being packed with stationary phase material, enabling the precise separation of substances into their individual components. There are two primary types of columns:

Nozzle Columns: These columns have larger diameter tubes, typically around 2 mm, and are pre-filled with adsorbent material. They can be commercially purchased or selfconstructed, often referred to as "self-packed" columns.

Capillary Columns: These are hollow or open columns with small diameter capillaries, usually measuring 0.53 mm, 0.32 mm, 0.25 mm, or 0.1 mm. The small diameter significantly reduces peak blurring due to diffusion, thereby increasing efficiency. Capillary columns help shorten analysis time and improve the differentiation of substances into their components. In our study, we used a RESTEK 800-356-1688 MXT-2887 5MS 10m x 0.53 mmID, 2.65µm capillary column.

Figure 3 – **Capillary column RESTEK 800-356-1688**

6. Detectors. A chromatographic detector is a device positioned at the end of a column, designed to detect and measure components as they exit in the flow of carrier gas from the analyzed mixture. In gas chromatography, the most commonly used detectors are differential detectors, which measure the instantaneous concentration of components in the carrier gas stream, unlike integral detectors, which do this with a delay. Today, there are various types of detectors used in modern chromatographs, leveraging different physical properties of gases such as thermal conductivity, density, heat of combustion, and the ability of gas molecules to ionize, among others. In all cases, detectors rely on the difference in physical properties between the carrier gas and the components of the analyzed mixture. When only carrier gas passes through the detector, it remains inert and its signal is zero. However, as components of the analyzed mixture begin to flow into the carrier gas stream, the detector registers a signal that is proportional to their concentration. It's important to note that the detector is installed after the chromatographic column, where it interacts not with the complex multicomponent mixture, but with a pure carrier gas or its mixture with one of the components from the sample being analyzed. This setup ensures that the

detector's response is specific to individual components as they elute from the column. The separation achieved by the chromatographic column isolates each component, enabling the detector to provide precise and accurate measurements of each substance in the sample. The detector plays a critical role in gas chromatography (GC) by converting the physical or chemical properties of the eluted compounds into measurable signals.

Figure 4 – **Chromatograph's functionality modules**

7. Temperature setting. Setting the column temperature is a strategic approach used in the analysis of complex mixtures that span a wide range of boiling points. The primary goal is to streamline the analysis process, resulting in significantly reduced overall analysis time compared to traditional isothermal methods. This technique is particularly beneficial when analyzing samples containing compounds with varying volatility profiles. Two commonly used modes of temperature programming include the linear ramp, where the temperature increases at a constant rate, and the linear-step mode, which alternates between temperature increase phases and isothermal intervals. By dynamically adjusting the column temperature during the analysis, temperature programming enhances separation efficiency and peak resolution. Modern chromatographic systems offer advanced temperature programming capabilities, allowing precise control over heating rates. These systems typically offer a wide range of heating speeds, ranging from as low as 0.1 \degree C/min to as high as 140 \degree C/ min. This flexibility enables researchers to customize the temperature profile according to the specific needs of the sample, thereby optimizing chromatographic performance and analytical results.

8. Registration of the analysis results. In modern chromatographs, the data processing system is organized around a personal computer on which special software is installed. This software allows the operator to interact with the chromatograph through a dialog interface. All visual information is displayed on the monitor and, if necessary, on the printer. Thus, operators like us can monitor and control the operation of the chromatograph, as well as analyze the results in real time.

Figure 5 – **Gas chromatogram result**

After the gas chromatograph experiment has been carried out, it is possible to work to determine its composition using a standard.

Figure 6 – **ASTM D2887 standard**

Differential Scanning Calorimetry (DSC) and pour point testers are two analytical techniques used to determine temperature-related properties of materials, such as the wax appearance temperature (WAT) and pour point of crude oil.

Differential Scanning Calorimeter (DSC): DSC is an analytical technique used to measure the heat flow associated with phase transitions of materials as a function of temperature. When used on crude oil, DSC can detect the onset of wax crystallization by measuring the heat flow changes as the sample is cooled. The temperature at which a noticeable exothermic peak occurs is often associated with the wax appearance temperature (WAT).

Procedure:

A small sample of crude oil is placed in the DSC.

The sample is cooled at a controlled rate.

The heat flow is monitored to detect the exothermic transition indicating wax crystallization.

The temperature corresponding to the onset of this transition is recorded as the WAT.

Pour Point Tester: The pour point tester is used to determine the lowest temperature at which crude oil remains pourable or flows. This temperature is critical in assessing the flow characteristics of crude oil at low temperatures and indicates the point at which wax crystals significantly hinder oil flow.

Procedure:

A sample of crude oil is cooled at a specified rate.

The sample is periodically tilted to check if it flows.

The lowest temperature at which the oil can still flow is recorded as the pour point.

Results. There are numerous methods for calculating concentrations, but one of the simplest and least biased is the percentage normalization method. This method relies on the principle that various components will show different peak areas and peak heights on a chromatogram, reflecting their content in the oil. Therefore, the concentration of a compound can be determined using the following equation:

$$
C_i = \frac{R_i}{\sum R} \tag{1}
$$

Where C_i is the concentration of compound i; R_i is the peak response (area or height); ΣR is the sum of the responses of all the peaks in the chromatogram.As an example, consider the results of the chromatogram for field A:

Table 2 – Oil composition for field A (sample 3)

Table 2 – Oil composition for field A (sample 3)

In this case, the sum of all areas is 13115103.7. Knowing the area of each compound, we can find the concentration of each compound. For example:

$$
C_{C_5H_{12}} = \frac{R_{C_5H_{12}}}{\sum R} = \frac{143251,845}{13115103,7} = 0,011 \text{ or } 1,1\%
$$
 (2)

Table3 - Oil composition for field A (sample 3) - finding molecular weight

By applying Kay's rule, the molecular weight for the mixture can be calculated:

$$
\theta = \sum_{i=1}^{N} z_i \theta_i \tag{3}
$$

$$
MW = \sum_{i=1}^{N} z_i MW_i = 0.79 + 5.83 + 7.42 + \dots + 4.11 = 218.43
$$
 (4)

Knowing the molecular weight, it becomes possible to calculate the fusion properties using the equations of Won (1986) and Nichita (2001):

$$
T_i^f = 374.5 + 0.02617 * MW_i - \frac{20172}{MW_i} = 374.5 + 0.02617 * 218.43 - \frac{20172}{218.43} = 287.86 °K = 14.71 °C
$$
\n(5)

$$
T_i^{tr} = 366,39775 + 0,03609M_i - 2,08796 \times \frac{10^4}{M_i} = 366,39775 + 0,03609 * 218,43 - \frac{20879,6}{218,43} = 278,69 \,^{\circ}K = 5,54 \,^{\circ}C
$$
 (6)

After conducting gas chromatography to determine the composition and concentration, as well as utilizing Differential Scanning Calorimetry (DSC) and the Pour Point Tester to identify relevant temperatures, we obtained the following results:

Table 4 – **Temperatures determination by DSC and Pour Point Tester**

Table 5 – **Temperatures determination by correlation of Won and Nichita Correlation**

Conclusion. In this study was investigated the fusion properties, specifically the melting point and solid-state transition temperature for oil sample. The first step involved determining the compositional makeup of the oil sample, which was accomplished using gas chromatography. This technique allowed for the precise identification and quantification of the different components within the oil. The melting and solid-state transition temperatures of the oil was measured using advanced analytical equipment. A Differential Scanning Calorimeter (DSC) was employed to accurately determine the melting point, leveraging its capability to measure heat flow associated with thermal transitions in the sample. Additionally, a Pour Point Tester (PPT) was used to establish the solid-state transition temperature, which is crucial for understanding the low-temperature flow properties of the oil.

The models by Won (1986) and Nichita (2001) are widely recognized and utilized in the field for predicting wax precipitation, a common issue in the oil industry that affects the flow characteristics of crude oil, especially at low temperatures. However, the new formula derived from this study provides a more precise prediction for the specific oil sample tested. This improvement is attributed to the incorporation of more detailed compositional data and the use of advanced regression techniques. This research contributes a novel and more accurate method for predicting the fusion properties of oils, which has significant implications for the oil industry. By enhancing the accuracy of temperature predictions, the new formula aids in better managing and preventing issues related to wax precipitation, thereby optimizing the processing and transportation of crude oils. This advancement underscores the importance of continuous innovation and refinement in predictive modeling to address the evolving challenges in the industry. \bullet

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