Asphaltene Precipitation

by Asif Zaman Srizon

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Asphaltene Precipitation Through Partial Dissolution Using C-Si-SARA Method

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Project Report Approval

The thesis titled “Asphaltene Precipitation Through Partial Dissolution Using C-Si-SARA Method” submitted by Asif Zaman Srizon (180051142) has been found as satisfactory and accepted as partial fulfillment of the requirement for the Degree Bachelor of Science in Civil Engineering.

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Declaration of Candidate

We hereby declare that the undergraduate research work reported in this thesis has been performed by us under the supervision of Professor Dr. Nazmus Sakib and this work has not been submitted elsewhere for any purpose (except for publication).

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Asif Zaman Srizon
Student No: 180051142
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Dedication

I dedicate this thesis to my loving family and supportive friends who have been unwavering pillars of strength throughout my academic journey. Your unconditional love, constant encouragement, and belief in my abilities have been instrumental in my success. Your unwavering support and understanding during late nights, countless hours of research, and moments of doubt have fueled my determination to push forward. You have celebrated my achievements, listened to my challenges, and provided a comforting presence during both triumphs and setbacks. Your belief in me has been a constant reminder of the importance of perseverance and the power of genuine support. This thesis stands as a testament to our collective dedication and serves as a token of my deepest gratitude for your unwavering presence in my life. Thank you for being my rock, my cheerleader, and my inspiration.

Table of Contents

List of Tables

List of Figures

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Abstract

Keywords: N-heptane, Toluene, Ratio, Yield, Bitumen, Asphaltene, SARA

Asphaltene is one of the key chemical species of bitumen and generally dictates its stiffness building behavior. Separation of asphaltene from bitumen and its gravimetric as well as chemometric analysis paves the way for a better understanding of bitumen behavior. Asphaltene is generally precipitated from a solution of asphalt in a non-polar solvent (such as n-heptane) where more polar asphaltene precipitates from solution. This study investigates the effectiveness of different solvent ratios of n-heptane/toluene solution, in separating asphaltene from bitumen due to changes in Hansen/Hildebrand Solubility Parameters. The experiment involved gradually varying ratios of n-heptane and toluene solvents such as 90:10, 80:20, and so on. A 50:50 solvent ratio was also examined to compare its effectiveness against using 100% of either solvent. Hansen Solubility Parameters were utilized to see the changes in different parameters due to the mixing of different solvents. The findings revealed that the solvent ratios influenced the asphaltene's gravimetric yield percentage. Remarkably, the 50:50 solvent ratio demonstrated comparable results to those achieved using 100% of a single solvent of n-heptane. This outcome highlights the potential for cost optimization as well as redundancy by reducing the amount of solvent required while maintaining the asphaltene separation efficiency. The gradual changes of asphaltene yield also indicates that asphaltene is not a single chemical element, but a distribution of molecules of varying polarity.

1 CHAPTER ONE: INTRODUCTION

1.1 Literature Review

In order to learn about the unidentified chemical features of bitumen, several writers and researchers conducted prior study. Additionally, researchers have created a variety of techniques and procedures related to the "SARA" approach. Some of these elements have been covered in this literature study

1.1.1 Significance of Bitumen

Bitumen, also known as asphalt or tar, plays a crucial role in transpiration infrastructure worldwide. Its significance stems from its versatile properties and numerous applications in the construction and maintenance of roads, highways, airports and other transportation systems.

One of the key reasons for the significance of bitumen in transportation is its exceptional binding properties. Bitumen acts as a strong adhesive that binds vigorous aggregates together to form asphalt concrete commonly known as asphalt pavement. About 100 million metric tons of bitumen is produced annually from which the pavement industry utilizes around 95% (Lesueur and España 2009, Jones *et al*. 2011, Hajj and Bhasin 2017)

Furthermore, bitumen's significance in transportation extends to its use in the manufacturing of various pavement- related products. It serves as a binder in the production of asphalt shingles, waterproofing membranes, and sealants, contributing the overall quality and performance of these materials.

1.1.2 Physiochemical Properties of Bitumen

Based According to the notion of colloidal structure, bitumen is a viscous substance made up of non-metallic byproducts and a mixture of molecular hydrocarbons. (Lesueur and España 2009). The characteristics and effectiveness of asphalt concrete and paving are determined by the bitumen binder's condition. To ensure that the resulting asphalt mixture and pavement meet realistic criteria of efficiency and service life, the inherent ability of the asphalt binder to bear disturbance, such as rutting and cracking (Jones *et al*. 2011).

However, Bitumen is a compound made up of a very wide range of mostly sophisticated chemical compounds. As a result, it is customary to evaluate the bitumen's chemical properties using factors such the relative polarities of molecules, size distribution, ionic character, and functional group concentrations. In addition,

It's crucial to note that bitumen is known to be microscopically diverse on a rather small scale. According to Loeber et al. (1996) and Claudy et al. (1992), 'bee' structures are frequently seen on bitumen surfaces. Additionally, Ramm et al. (2016) shown that bitumen possesses bulk microstructures, and these features may influence how bitumen behaves under different temperatures.

Bitumen may be extracted using a number of chemical procedures. It can be produced by oil sand cracking, fractional distillation of crude oil (McNally 2011), refined petroleum (jones et al. 2011), or both. Different products with diverse chemical compositions might result from different extraction methods used to extract crude oil. The chemical composition of the bottom leftovers may alter throughout the processing step itself. For commercial and specification reasons, the extracted bitumen may also undergo further procedures such oxidation, cracking and blending with softer or harder grade bitumen.

Besides that, the necessity of adapting to a more environmentally acceptable approach in the pavement industry influenced the industry to use waste products or bio-degradable products as extenders resulting in further unknown changes in the chemical composition of bitumen (Sakib *et al*. 2020)

1.1.3 Bitumen Fractionation Methods

Fractionation of the bituminous components is often dine in order to undertake a through examination of bitumen (Cuadri 2011). According to Yean et al. (2019), researchers have employed a verity of techniques to separate bitumen, although adsorption-chromatography is the most often utilized. Other techniques include solvent extraction, chemical precipitation, distillation, and adsorption. According to Ashoor et al. (2016), experiments on chromatographic methods were originally carried out for the grouping of hydrocarbons and SARA fractionation as well. Later research concentrated on creating alternative approaches to this strategy leading to the creation of the ASTM D 2007 technique. Other traditional techniques were created for convince because this approach needed a lot od bitumen, adsorbent, and solvents (khrrat et. Al. 2007)

1.1.4 SARA Fractionation

The SARA fraction of bitumen refers to the separation of bitumen into four distinct components: saturates, aromatics, resins, and asphaltenes. These subdivisions are briefly addressed as SARA (Sakib and Bhasin 2018). This separation process is based on the different solubility characteristics of these fractions. Each fraction plays a specific role in the overall properties and performance of bitumen SARA (saturates, aromatics, resins, and asphaltenes) fractionation is a typical & one of the most popular as well as effective procedures for bitumen composition analysis, according to Kharrat et al. (2007). According to Silva et al.'s (2011) investigations, as crude oil's API gravity drops, it gets heavier and contains more asphaltene and resins.

1.1.5 Asphaltene Extraction

Asphaltene extraction differs by various methods using pentane, hexane, or heptane. Asphaltenes and maltenes are separated by dissolving bitumen into a non-polar solvent followed by filtration of the precipitated asphaltene. There are mainly four standard practices of mixing solvents to create specimens. They are summarized below:

1. 10 w/v solution in n-pentane swirled in a warm water bath dissolved; filtered using rapid filter paper (ASTM D2007 2016)

2. 30 w/v solution in n-heptane prepared under reflux for 60 minutes; filtered with Whatman Grade 42 (2.5 m pore) filter paper (ASTM D6560)

3. 100 w/v solution in n-heptane prepared under reflux for 20 minutes; filtered using a glass microfiber filter pad with 1.5 µm pore size (ASTM D3279 1997)

4. 100 w/v solution in iso-octane (previously n-heptane) prepared under reflux for 3-4 hours; filtered using a medium porosity (10-15 m pore size) Buchner funnel fritted glass filter (ASTM D4124, 2009)

1.1.6 Maltene Fractionation

Maltene fractions can be further subdivided into Saturates, Aromatics, and Resins (SAR), in order of increasing polarity. Despite the fact that maltenes are typically divided into three subfractions, it is also possible to fractionate maltene into more than three fractions by appropriately choosing a solvent.

1.2 Objective of the Study

- • To replace the vacuum manifold and drying oven, the two most expensive pieces of equipment utilized by Sakib and Bhasin in 2019 and to replace them with a metal box or pot
- To reduce the cost of the setup
- To create a more sustainable system in which no hazardous chemicals are emitted into the environment.

2 CHAPTER TWO: "Si-SARA" SETUP & SOLVENT CHOOSING

2.1 General

Since the original suggestion was to make chemical analysis more accessible for fieldwork and simpler, many of the original materials had to be modified and replaced with more affordable and adaptable ones. The use of plastic in the prototype is the first area of concern. Because compounds like asphaltene, maltene, and other substances will be separated using chemicals like n-heptane, toluene, and methanol, the rubber's surface will be efficiently destroyed. The entire system will thereafter be deactivated. As a result, it was determined to refrain from using any plastic materials. The image below shows a drawing of the prototype.

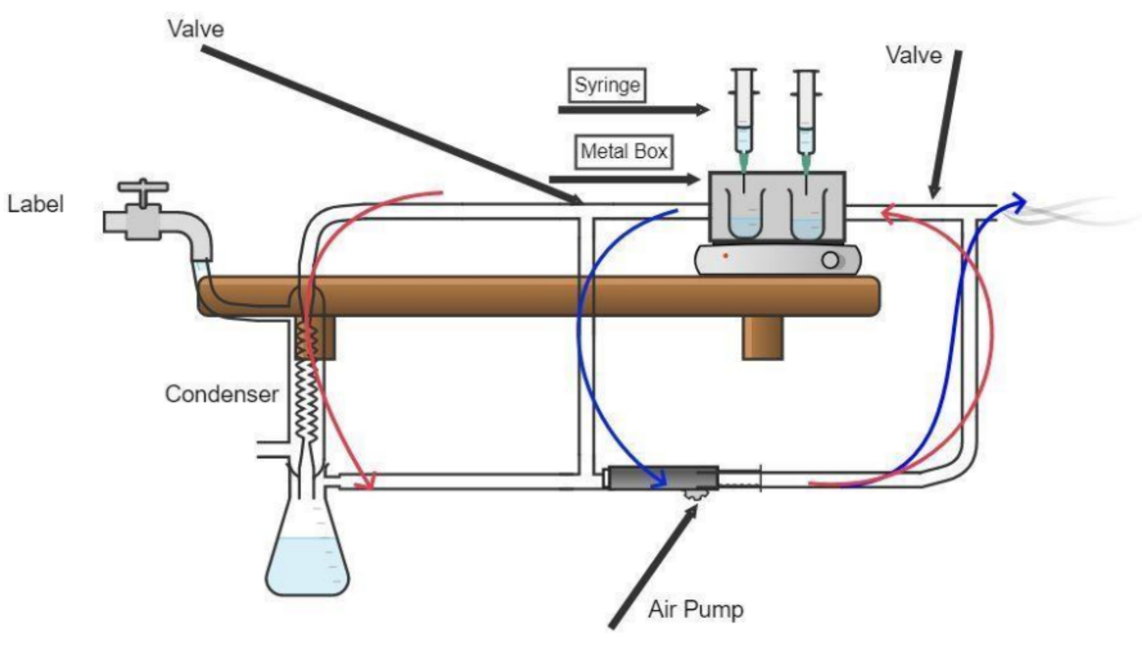

Figure 1 Sketch of the Prototype

We will go over the prototype's specifics in more depth. However, two signs are now visible; one is blue and the other is red in the sketch. The red one stands in for the "drying cycle," while the blue one symbolizes the "filtration cycle." The plastic joint can be utilized at some time because it won't come into touch with the chemicals, it has been discovered after further analysis of the sketch. So, the design now includes some plastic joints. The design is mostly centered on joints made of brass and copper. However, as it is currently being tested, more modifications will be made in accordance with the findings of the study.

In a recent study, Sakib and Bhasin (2019) used a novel method called ‘Si-SARA’ for extracting bitumen chemical fractions on the scale of milligrams. The Hansen Solubility Parameters, specifically δ_D, δ_P, δ_H are commonly used as polarity measurements and they are influenced by the composition of n-heptane and toluene (Redelius 2004). Generally, the proportion of toluene increases the value of δ_D, δ_P, δ_H. Investigating the Hansen Solubility Parameters of n-heptane and toluene mixtures at different ratios provide valuable insights into the solubility behavior. Understanding the changes in these parameters are crucial for optimizing solvent blends in various applications. We examined by using different ratios of n-heptane and toluene to investigate variations in δ_D, δ_P, and δ_H ,and they are tabulated

below:

N-heptane : Toluene	δ_D	δ_P	δ_H	δ_{total} ($MPa^{0.5}$)
100% : 0%	15.3	0	0	15.3
90% : 10%	15.57	0.14	0.2	15.6
80% : 20%	15.84	0.28	0.4	15.9
70% : 30%	16.11	0.42	0.6	16.12
50% : 50%	16.65	0.7	1	16.7
30% : 70%	17.19	0.98	1.4	17.3
20% : 80%	17.46	1.12	1.6	17.6
10% : 90%	17.73	1.26	1.8	17.9

Table 1: Hansen solvent solubility parameters for various non-polar solvent combinations

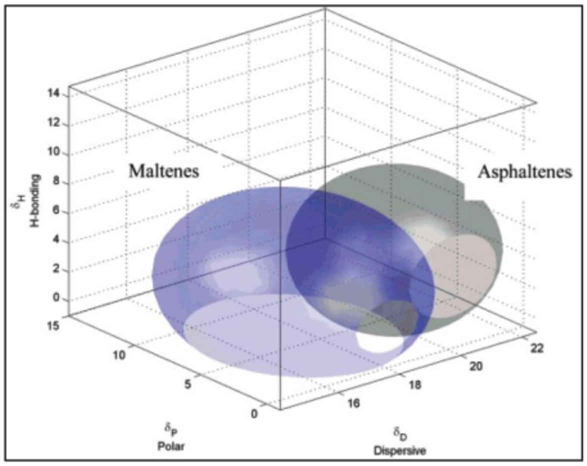

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Figure 2: Hansen solubility parameter of maltenes and asphaltenes bitumen (from Redelius 2004)

3 CHAPTER THREE: METHODOLOGY

The whole fractionation procedure can be divided into three phases.

1. Sample Preparation
2. Sample Filtration
3. Sample Drying

3.1 Sample Preparation

First, we take a beaker and PTFE-coated magnetic stirrer bar and all of these are sterilized using acetone. Then the beaker was placed in the oven for 5 minutes so that there is no reside of acetone. A small amount of bitumen was weighed to a precision of 0.04 mg and placed in a beaker along with a PTFE-coated magnetic stirrer bar. The solvent (n-heptane & toluene) was poured into the jar in different ratios w/v ratio of added bitumen. We have made 7 samples which include different ratios of n-heptane and toluene as 90:10 (36 ml n-heptane and 4 ml toluene), 80:20 (32 ml n-heptane and 8 ml toluene), 70:30 (28 ml n-heptane and 12 ml toluene), 50:50 (20 ml n-heptane and 20 ml toluene), 30:70 (12 ml n-heptane and28 ml toluene), 20:80 (8 ml n-heptane and 32 ml toluene) and at last 10:90 (4 ml n-heptane and 36 ml toluene). All of these samples were placed in a magnetic stirrer and it was stirred for 24 hours at medium speed and at room temperature. A few ml of CO_2 was given in the samples so that the samples don't get oxidized during this time.

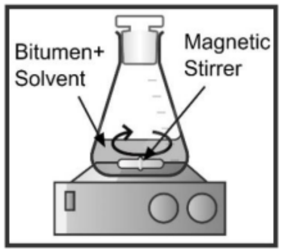

Fig. 3: Bitumen dissolution in non-polar solvent

3.2 Sample Filtration

At first, we took four moisture cans and sterilized them with acetone, and the dried them in the oven so that there won't be any residue of acetone. When the sample has been stirred well and all the solvents have been dissolved completely it is time to filter the samples to remove the precepted asphaltene. For the filtration, we used a disposable syringe to mount a vacuumed chamber. The pan, we can see in Figure 1. Is used as a vacuum chamber. We place a 10 ml solution to each of the syringes that is placed on the vacuum chamber and with the help of the vacuum machine we made the chamber airless and the using the vacuum to drive the solution through the filter which was placed at the tip of the syringes. And a CO_2 jar was attached to the chamber during the process to prevent the solution get oxidized. Two moisture cans were placed in the chamber under the syringes so that when the asphaltene is captured in the filter the maltene can be stored in them. The filter is cleaned using toluene as asphaltene is soluble in it and the solutions were taken in two different moisture cans.

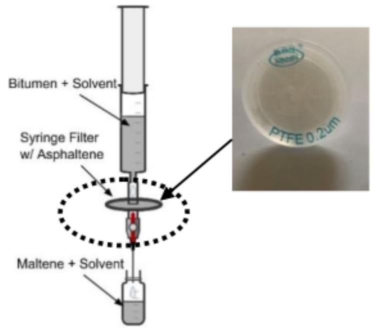

Fig. 4: Asphaltene filtration

3.3 Sample Drying

Now we put the two moisture cans holding the asphaltene solution in the chamber. The chamber was placed over an induction oven. The oven was put up on medium heat. We heat the solution for 30- 45 minutes and within this time the maltene is evaporated but as there is a coil condenser attached to the chamber, it liquefies the evaporated toluene in the beaker that is placed under the condenser.

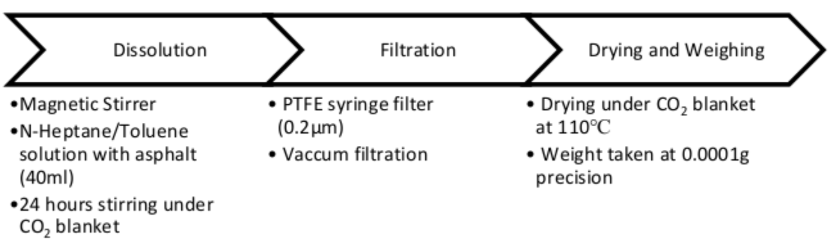

Fig. 5: Asphaltene separation process

4 CHAPTER FOUR: RESULT

We have made around 8 samples using different toluene and n-heptane ratios. And the results are shown in a tabular form below:

n-heptane to toluene ratio	Asphaltene (%)
100% and 0%	30.1
90% and 10%	29.2
80% and 20%	28.9
70% and 30%	28.2
50% and 50%	30
30% and 70%	25
20% and 80%	22
10% and 90%	19
0% and 100%	28

Table 2: List of Asphaltene Percentage for Different Ratios

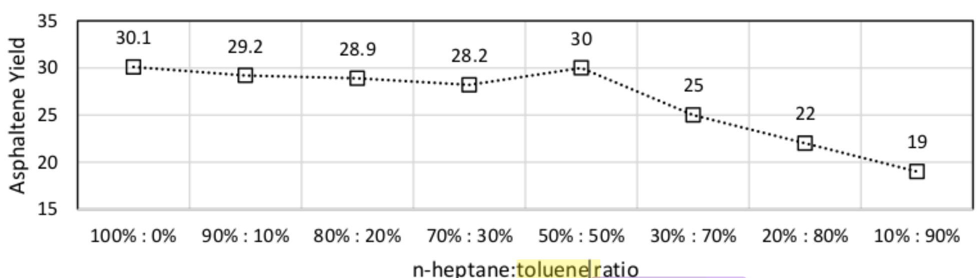

Figure 6: Graphical presentation of changing in asphaltene percentage

From the graph and test result we can see that the more we are increasing the toluene percentage the asphaltene percentage is reducing. And if we look when we are using 50:50 ratio, we get the same result when we were using 100% n-heptane. Knowing the asphaltene percentage of bitumen is crucial for road construction. Asphaltene is the high molecular weight component of bitumen that contributes to its binding and cohesive properties. By assessing the asphaltene percentage engineers and contractors can select bitumen grades with suitable asphaltene content that meet the specific road project.

These changes have meaningful asphaltene chemistry implication. For example, the asphaltene collected from 50:50 solution of n-heptane: toluene will be slightly more polar than the asphaltene collected from 30:70 solution of n-heptane: toluene.

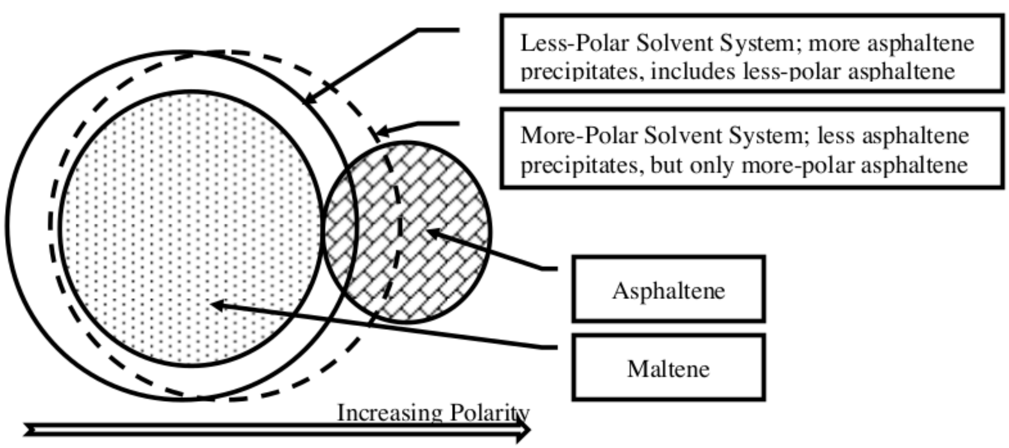

Fig. 7: Graphical representation of Asphaltene precipitation from bitumen solution

The reason is that the 30:70 solution of n-heptane: toluene is comparatively more polar than the 50:50 solution, though both solutions are less polar comparative to asphaltene. Now, polarity of the 30:70 solution will be closer the polarity of some lower-polarity asphaltene molecules, thereby dissolving them and reducing asphaltene yield. However, as lower-polarity asphaltene molecules are with maltene solution now, remaining asphaltene are more polar but less in amount. This discussion can be summarized in the following figure 8.

In Figure 8, the solvent system with less polar solvent leaves a significant portion of asphaltene to be precipitated which also includes asphaltenes which have lower polarity. On the other hand, more polar system will dissolve lower polarity asphaltene species, thereby reducing yield but the precipitated asphaltene species will be more polar.

Asphaltene content affects the viscosity of bitumen. Viscosity determines the flow characteristics of bitumen at different temperatures. This ensures that the bitumen can be properly mixed, spread, and compacted, leading to a smooth and well-compacted road surface. Additionally, asphaltene content

influences the aging and long-term performance of bitumen. As bitumen ages, its asphaltene content can change, leading to changes in its properties over time. By knowing the initial asphaltene percentage, engineers can better predict how the bitumen will age and adapt their road construction and maintenance strategies accordingly. This knowledge helps in designing appropriate pavement structures and implementing effective maintenance practices to extend the service life of the road.

In conclusion, knowing the asphaltene percentage in bitumen provides valuable information for road construction. It helps in selecting suitable bitumen grades, determining appropriate handling and laying temperatures, assessing waterproofing properties, and predicting long-term performance. By considering the asphaltene content, engineers can optimize road design, construction methods, and maintenance strategies to ensure the durability, stability, and longevity of the road infrastructure.

5 CHAPTER FIVE: CONCLUSION

In conclusion, this thesis research demonstrates that using a 50:50 ratio of n-heptane and toluene for extracting asphaltene from bitumen yields similar results to using 100% n-heptane or toluene. This cost-effective method provides a practical alternative for asphaltene extraction, reducing solvent expenses without compromising quality. The developed lab setup enables efficient extraction using affordable solvents, offering potential cost savings in asphalt production and relates industries. Further research is needed to validate the method's scalability and explore its broader implications. Overall, this research contributes valuable insights into cost optimization in asphaltene extraction

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Asphaltene Precipitation

PAGE 1

Proper Noun If this word is a proper noun, you need to capitalize it.

PAGE 2

Article Error You may need to use an article before this word. Consider using the article **the**.

PAGE 3

Article Error You may need to use an article before this word.

PAGE 4

Article Error You may need to use an article before this word. Consider using the article **the**.

P/V You have used the passive voice in this sentence. Depending upon what you wish to emphasize in the sentence, you may want to revise it using the active voice.

PAGE 5

Missing "," You may need to place a comma after this word.

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Article Error You may need to use an article before this word. Consider using the article **the**.

Article Error You may need to use an article before this word.

Frag. This sentence may be a fragment or may have incorrect punctuation. Proofread the sentence to be sure that it has correct punctuation and that it has an independent clause with a complete subject and predicate.

Wrong Article You may have used the wrong article or pronoun. Proofread the sentence to make sure that the article or pronoun agrees with the word it describes.

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Prep. You may be using the wrong preposition.

PAGE 14

Proofread This part of the sentence contains a grammatical error or misspelled word that makes your meaning unclear.

PAGE 15

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PAGE 22

Missing "?" Remember to use a question mark at the end of a question.

S/V This subject and verb may not agree. Proofread the sentence to make sure the subject agrees with the verb.

PAGE 23

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PAGE 30

PAGE 31

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PAGE 33

PAGE 34