Bitumen Fractionation Using a Rugged and Economic Variant of 'Si-SARA' Method

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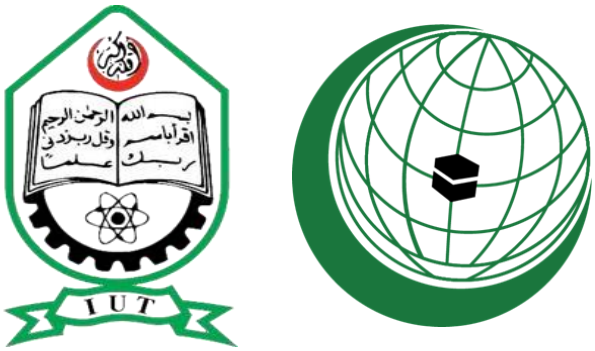

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Project Report Approval

The thesis titled “Bitumen Fractionation Using a Rugged and Economic Variant of ‘Si-SARA’ Method” submitted by Rakibuzzaman Shafi Siam (160051041), Sabrina Islam Tasnim (160051078), and Kazi Tamim Ahmed (160051086) have 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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Dedication

We dedicate our thesis work to our family. A special feeling of gratitude to our loving parents.

We also dedicate this thesis to our many friends who have supported us throughout the process. We will always appreciate all they have done.

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Abstract

Keywords: SARA, Si-SARA, SPE, Syringe filter, Bitumen, Asphaltene.

The chemical composition of bitumen is a reinvigorated aspect of looking into the origin of bitumen behavior. Diversification of crude sources, emphasizing high-value products of refining, and addition of rejuvenators, extenders, modifiers lead to unusual behavior of bitumen compared to unmodified bitumen. There are many cases where the integrity of bitumen as a chemical was understood to be compromised but not captured by usual lab tests. Therefore, the use of bitumen chemistry as a predictor of performance and quality control is being pursued in multiple research fronts. However, analysis of bitumen chemistry is challenging due to the need for specialized equipment and a protective environment.

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 key elements of that procedure were (i) a syringe-filter-based separation to isolate asphaltene and (ii) pre-packaged silica cartridges (Solid Phase Extraction– SPE cartridges) for fractionation of maltene into saturates, aromatics, and resins using chromatography in a procedure similar to ASTM D4124 and IP469. The 'Si-SARA' procedure is an order of magnitude speedier, cheaper, and more compact than that of other existing processes. This procedure leads up to the present work which offers a further refinement of the 'Si-SARA' procedure. Proposed modifications in the present paper make the procedure suitable for field labs and labs without a dedicated chemical handling facility, thereby offering even faster results and point-of-use quality control.

The compact new process calls for an enclosed metallic chamber, a manual or motorized vacuum pump, an air pump, a magnetic stirring heating mantle, and a condenser setup. While the method uses syringe filters and SPE cartridges like the original method, this innovation removes two major and relatively expensive equipment; namely glass-made vacuum manifold and drying oven. The new process actually combines the vacuum manifold and drying oven chamber into a single metal 'lunch box'-commercially available as a steel-made airtight food container- alternately used either as vacuum chamber or as drying chamber mode. When operated in vacuum manifold mode, the system is relatively basic with no flow control regulators though an off-on valve facilitates the elution process' initiation and termination. A vacuum pump helps the liquid flow as shown in Sakib and Bhasin (2019). However, when operated under drying mode, a heating plate is placed under the box, which heats up and evaporates solvents. Solvent-rich gas inside the box then passes through a cold condenser coil with the help of an air pump. After condensation, the accumulated liquid is collected in a bottle, and gas/air is returned to the chamber to complete the iteration. This process offers a closed system and thus removes the necessity of a chemical hood. During drying, nitrogen or other inert gas blanket requires only a small amount of gas or is completely made redundant as the same volume of gas is iterated and thus the amount of oxygen is very limited. Additionally, the use of metal cans in place of glass vials to collect elute in the new process offers a quick-drying and rugged system regarding heating and transportation.

To summarize, the newly proposed system uses the 'Si-SARA' method but utilizes more accessible and economic tools, which enables the chemical analysis of bitumen in limited resource labs, field labs, or labs previously not suitable for chemical testing.

1 CHAPTER ONE: INTRODUCTION

1.1 Literature Review

Many authors and researchers previously researched about fractionation of bitumen to know the unknown chemical properties. Also, many methods and processes have been developed by the researchers about the 'SARA' method. In this literature review, some of these aspects have been discussed.

1.1.1 Significance of Bitumen

The demand for bitumen is skyrocketing due to the mounting construction & maintenance of pavement around the globe. The total world road network length is approximately 16.3 million kilometers or 10.3 million miles of which the majority portion of the road is flexible or asphalt pavement. Asphalt binder, in other terms known as Bitumen, possesses good coating and binding properties that allow it to be an appropriate binder agent in asphalt concrete mixtures. 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). Bitumen has various advantages like comfortable driving, convenient maintenance, and low noise. As the quality of bitumen dictates the pavement quality to a great extent, the cost of bitumen leads to substandard bitumen usage for road construction which results in quick deterioration. Analyzing and categorizing bitumen according to its chemical composition may help us surmount this problem from the user end, increasing the efficiency and longevity of the pavement.

1.1.2 Physiochemical Properties of Bitumen

Based on colloidal structure theory, bitumen is a viscous material comprising of composite molecular hydrocarbons and non-metallic byproducts (Lesueur and España 2009). The conditions of the bitumen binder establish the properties and efficiency of the asphalt concrete and the paving. The intrinsic capacity of the asphalt binder to tolerate disturbance for instance rutting and cracking is essential (nevertheless adequate) to guarantee that the consequential asphalt mixture and pavement have reasonable standards of efficiency and service life (Jones *et al.* 2011). It is obligatory to understand the physicochemical properties of bitumen that regulate its performance in asphalt mixture.

However, Bitumen sources & production method also impacts bituminous properties (Gasper *et al.* 2012, Yang *et al.* 2016). The determination of detailed composition is very difficult as it contains innumerable and complex molecular structures. Any compositional or chemical property change greatly impacts the physiochemical properties of bitumen (Sultana and Bhasin 2014, Fischer and Cernescu 2015). The physicochemical characteristics of the bitumen differ significantly based upon its source, even though the material characteristics are assessed at a given loading rate, temperature, and similar aging conditions (Sakib *et al.* 2020). Following the regenerative component regulation theory, bitumen's performance is determined by its composition & components (Valtorta *et al.* 2007).

Naturally found bitumen is produced from oil through slow natural processes (Spielmann 1937). Conventional oil and alternate deposits, including heavy oil, super heavy oil, oil/tar sand, mineral

oil, shale oil are all-natural petroleum mixes (Rudyk 2017). All of these resources are found in nature and their chemical configuration fluctuates with their terrestrial location of the deposit and for certain instances, the same source too. This heterogeneity is even further boosted by the variations in the residual constituents in the bottom residues after gasoline and diesel or other high-value goods are distilled from the refined source oil (Sultana and Bhasin 2014). Since Bitumen is an organic matter, it is more susceptible to oxidization owing to its complex hydro-carbonous chemical configuration (Lesueur and España 2009). Several researchers have agreed upon the fact that the long-term durability of asphalt concrete mixture is dependent upon the chemical composition of bitumen (Daly 2017). But only mechanical properties are taken into account for design specification if we come from an engineering aspect, which is either quantitative measurements (e.g., penetration test, ring, and ball softening point) or quality measurements (e.g., performance grading system or PG). Many studies have concluded that bitumen with similar performance grades obtained from various sources can perform differently and even meet premature failure.

Bitumen can also be extracted through various chemical processes. It can be obtained from the fractional distillation of crude oil (McNally 2011) or refined petroleum (Jones et al. 2011) or from oil sand cracking. Different extraction processes used to extract crude oil can lead to different products with various chemical compositions. The processing phase itself may change the chemical structure of the bottom residues. In addition, the extracted bitumen sometimes goes through additional processes such as oxidation, cracking, and blending with softer or harder grade bitumen for commercial and specification needs. Apart from these, rejuvenators, polymers,

extenders are added to bitumen for improving bitumen properties or providing commercial incentives.

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 Lack of Correlation between the Existing Methods

Assembling a collection of research findings by other authors that established associations between widely used bitumen output test results and ongoing field deformation, it was observed that the correlation coefficients calculated in various experiments were very distinct, within a range of 0.27 to 0.99. The related variance was also recorded when various forms of bitumen were used such as Pmb also known as polymer-modified bitumen (Batista et al. 2017). Such studies testify that the existing methods to predict the field performance of modified bitumen are inadequate (Delgadillo *et al.* 2006, D'Angelo 2009, Hajj and Bhasin 2017).

1.1.4 Bitumen Fractionation Methods

To conduct a detailed analysis of bitumen, fractionation of the bituminous materials is normally performed (Cuadri 2011). Yang et al. (2019) mention some popular methods used by researchers for bitumen separation are distillation, adsorption, solvent extraction, chemical precipitation,

chromatographic methods, etc. but adsorption-chromatography is the most popular. Ashoori et al. (2016) stated in their work that studies regarding Chromatographic techniques were first done for grouping of hydrocarbons & SARA fractionation as well. Later studies were focused on developing different methods to this approach, which resulted in ASTM D 2007 method development. Since this method required a large amount of bitumen, absorbent & solvents other conventional methods were developed for convenience (Kharrat *et al.* 2007).

1.1.5 SARA Fractionation

SARA fractionation is the segmentation of bitumen into asphaltenes and maltenes based on the polarity of constituents. Maltene constitutes saturates, aromatics and resins. These subdivisions are briefly addressed as SARA (Sakib and Bhasin 2018). Both fixed & mobile phases (where we separate mixed chemicals by heating from Maltene) are applied in this method. Bitumen is easily fractionated based on the polarity & adsorption of detached materials (Chen 2014). Kharrat *et al.* (2007) mentioned in their study that SARA (saturates, aromatics, resins, and asphaltenes) fractionation is a common & one of the most popular as well as efficient practices for bitumen composition analysis. From the studies of Silva *et al.* (2011), we know, as crude oil becomes heavier and has a higher asphaltene & resin content when its API gravity decreases. When the H/C ratio decreases, aromaticity increases.

1.1.6 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)

1.1.7 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 sub-

fractions, it is also possible to fractionate maltene into more than three fractions by appropriately choosing a solvent.

Procedure	Solvent	δ_D	δ_P	δ_H	δ_{Tot}
Saturates					
TLC, SPE, IP469	n-heptane	15.3	0.0	0.0	15.3
ASTM 4124	n-heptane	15.3	0.0	0.0	15.3
	Toluene*	18.0	1.4	2.0	18.2
Aromatics					
TLC, SPE, IP469	80:20 Toluene:n-heptane	17.5	1.1	1.6	17.6
ASTM 4124	Toluene	18.0	1.4	2.0	18.2
	50:50 Toluene:Methanol*	16.4	6.9	12.2	21.5
Resins					
TLC, SPE	90:10 DCM:Methanol	17.8	6.9	7.7	20.6
IP469	95:5 DCM:Methanol	18.0	6.6	6.9	20.4
ASTM D4124	Trichloroethylene	18.0	3.1	5.3	19.0

Figure 1: Solvent Options for Elution

1.2 Observation from Literature Review

Analyzing bitumen, following the existing methods can be difficult due to the need for specialization in equipment and the security of the environment. To overcome these difficulties Sakib & Bhasin (2019) came up with an innovative process known as 'Si-SARA' where they used a syringe-filter based separation to isolate asphaltene and pre-packaged silica cartridges (Solid Phase Extraction– SPE cartridges) for fractionation of maltene into saturates, aromatics, and resins using chromatography in a procedure similar to ASTM D4124 and IP469. Using this technique bitumen can be chemically disintegrated up to milligrams & offers a further refinement of the 'Si-SARA' procedure. But the method requires time and necessary equipment which is expensive. So in this thesis, we tried to carry on the experimental method but by finding an time-efficient and economic variant.

1.3 Objective of the Study

- To replace the two most expensive equipment used by Sakib and Bhasin (2019) which are vacuum manifold and drying oven.
- To combine vacuum manifold and drying oven into a single chamber metal box
- To replace a $5000 project into a $200 project
- To make a more sustainable system where no toxic chemical will be released into environment
- To make it suitable for field use without a dedicated chemical handling facility.

2 CHAPTER TWO: “Si-SARA” EXPERIMENTAL SETUP

2.1 General

As the initial proposal was to simplify the method of chemical analysis and making it available for fieldwork, a lot of modification has to be done and replaced by cheap and flexible materials. The first concern about the prototype is using of plastic material. Because chemicals like n-heptane, toluene, methanol will be used to separate asphaltene, maltene, and other substances which effectively breaks down the surface of the rubber. So the total system will be disabled then. Due to this, it was decided that the use of any kind of plastic material will be avoided. We can see the sketch of the prototype in the picture below.

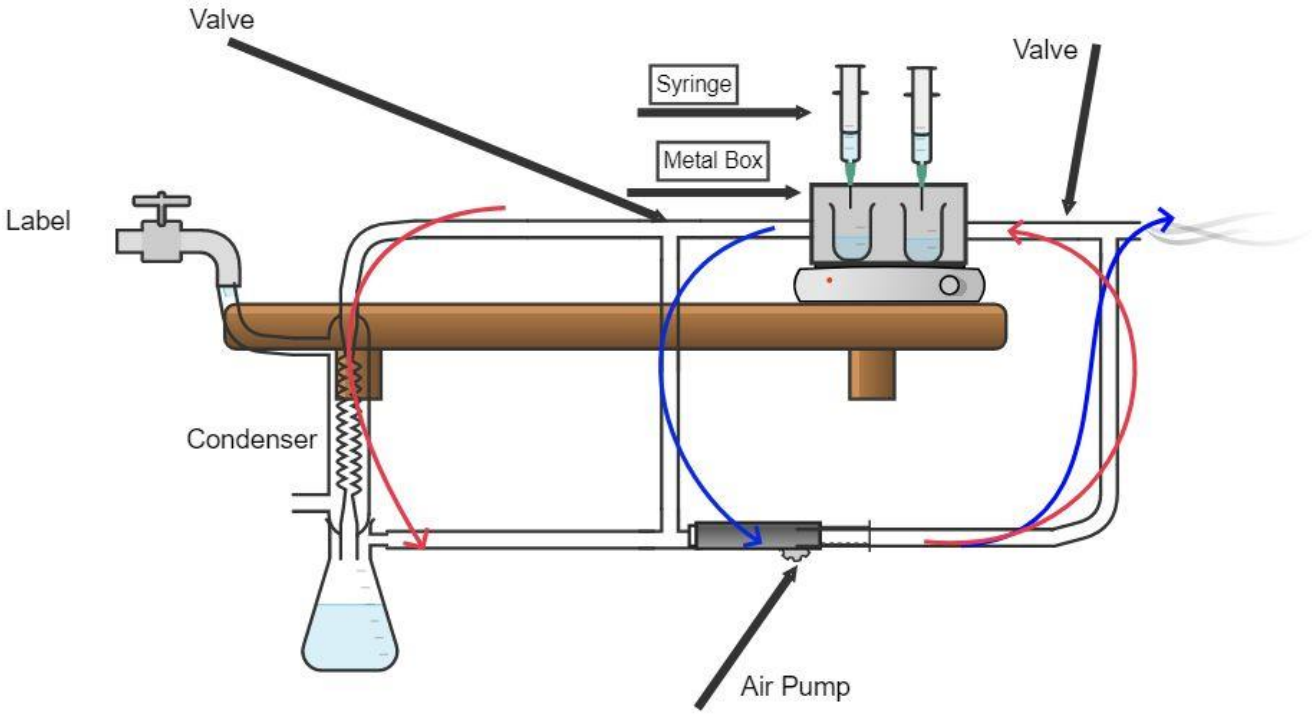

Figure 2 Sketch of the Prototype

We will further discuss details about the prototype. But at this point we can see two indicators, one is blue and one is red in the sketch. The blue one represents the 'filtration cycle' and the red one represents the 'drying cycle'. While analyzing the sketch more, it has been realized that at some point, the plastic joint can be used because it will make no contact with the chemicals. So some plastic joints have been introduced in the design. Mainly the design is based on brass and copper joints. But as it is now under trial, the further modification will be made depending on the results come from the experiment.

2.2 Setup Description

The key innovation or modification is the 'Metal Box' shown in the sketch. It is situated on the base which is a 'Hot Plate' by which heat is given to this system. Previously, drying and filtration have been done in a separate containers. For filtration, a vacuum manifold is used and for drying, an oven is used. To reduce the significant cost, a metal box has been used. Initially, an aluminum box is used. But depending on the experiment result it might be switched to iron. Because iron boxes are easier to weld which provides better finishing. This metal box contains holes multiplier of 2 (2/4/8/12) based on the surface and size of the box. In this experimental case, there are two holes (Figure 3).

Figure 3: Top of the metal box

A metal needle then gone through the holes. We have used No. 10 needles is this purpose. There are separate small boxes under every needle that will collect the filtrated chemicals (Figure 4). The gap between the hole and needle is welded using chemical epoxy to make it airtight. Epoxy is used because it is not possible to weld aluminum, copper, and stainless steel together as all are different. Welding could be done if we used the same type of material for every case.

Figure 4: Inside the metal box

The top surface of the metal box accommodates four things. 1. Needles, 2. Syringe Filter, 3. SPE Cartridge and 4. A Pressure Gauge (Figure 3). These needles have two types of off/on mechanisms. A small valve is placed on them (Figure 5). The purpose of them is to make the metal box airtight during the 'Drying Cycle'. At first 'Filtration Cycle' will be done to separate Asphaltene and Maltene. But after that to remove the solvent, the drying cycle will be done. So then, filter syringes will be removed and valves will be placed on them to make the whole system airtight. Polytetrafluoroethylene (PTFE) filters (Figure 6) and 50 ml. syringes are used in the experiment.

Figure 5: Valve covering the needles

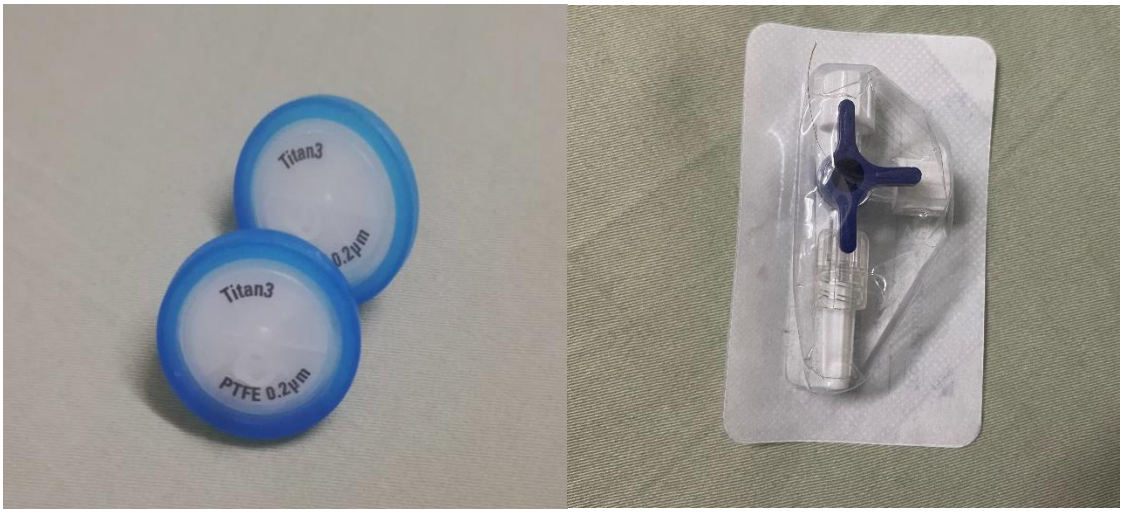

Figure 6: PTFE Filters and Valve for needles

There are two holes in the two opposite sides of the box. Two nuts have been used to joint a two-way valve with the metal box (Figure 7). These valves have an off/on mechanism which helps to take out the box from the system and joining again. These valves are made of brass. There are two types of valves used in this prototype. 1. Male and 2. Female (Figure 8). Both types are used to reduce the overall number of joints, valves. The intention is to reduce the cost.

Figure 7: Joint of metal box and valve

Figure 8: Female and Male Valves

Three other important materials in this setup are Male Compression Joint, Female T Joint, and Female I Joint (Figure 9). Male compression joints are used to connect the copper wire from one segment to another. The vapor which will be created in the drying cycle will move through the system by these copper wires. All the joints, valves, and copper wire are 1/4 Inch in diameter. Female T joints are used where the path is divided into three sections as showed in Figure 2. We will do the filtration cycle and drying cycle in the same system. This is why it is very important to use the valves and joints properly. Some of the valves will be closed in the drying cycle and some of the valves will be closed during the filtration cycle.

Figure 9: (From Left) Female T Joint, Female I Joint, Male Compression Joint

One of the copper wire connections will connect with the condenser as shown in Figure 2. In this case, we are using a graham condenser. One opening of the graham condenser is connected with the help of a cork. Copper wire has been drilled through the cork (Figure 10). Another opening of the graham condenser will be attached to a flask. We are using two conical flasks which have two openings. The joint of the conical flask and condenser is air-tightened by another cork (Figure 10). Graham condenser is connected with a water source by which continuous water supply will be provided during the drying cycle. It is to be mentioned that these works have been done with bare hands so the finishing is not very smooth. These are done for experimental purposes.

Figure 10: (From Left) Copper Wire Drilled Into Cork, Joint of Condenser and Flask and Cork Covering the Joint of Condenser and Flask

It has been mentioned already that at first, the idea was to use copper and brass joints in all sections. But it is found that some of the sections will not get polluted by toxic chemicals. So we can use some plastic joints to reduce the cost. There are some plastic compression T joints and valve that has been used after the section where condensing is already done (Figure 11). It means the vapor containing chemicals will already be condensed in the condensing chamber. The remaining fresh air will be passed through the next section. So plastic or rubber material can be used in that section. Mainly we have used the plastic valves and joints after the Air Pump shown in the sketch (Figure 2).

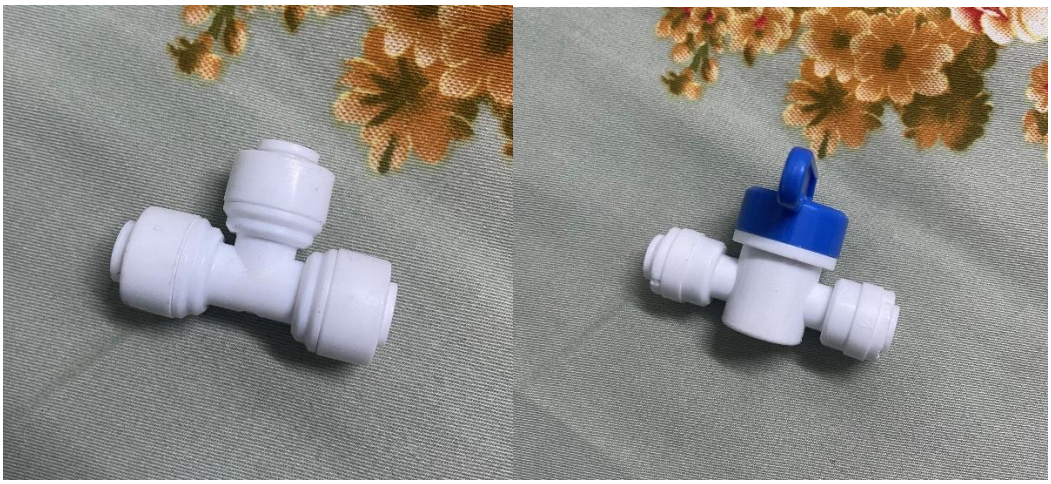

Figure 11: Plastic compression T joint and valve

There is some additional electrical instrument used in this prototype. A submersible pump has been used. The use of the pump is to maintain continuous water flow through the graham condenser. A temperature reader is also put on the top of the metal box. It has been placed to know about the temperature reading from time to time. A small vacuum pump is used. The purpose of this pump is to help to suck the air from the metal box. It will create a vacuum inside the box which will help to fasten the filtration process as well as maintain a constant pressure. Figure 12 is showing these instruments.

Figure 12: Vacuum pump, temperature reader, submersible pump

A total estimation of the materials used in this whole experiment is given in the chart below showing their name and quantity. The number of valves and joints will vary depending on the male valve and female valve used. After the table, a full picture of the setup is shown in Figure 12 after joining the all segments.

Table 1: List of Materials

No	Item Name	Quantity
01	Metal Box	1
02	Small Box	4
03	No. 10 Needle	4
04	Needle Blocking Valve	4
05	Digital Thermometer	1
06	Pressure Gauge	1
07	12V Submersible Water Pump	1
08	Vacuum Pump	1
09	Graham Condenser	1
10	Double Opening Conical Flask	1
11	Copper Wire Cutter	1
12	50 ml Syringe	4-8
13	PTEF Filters	10
14	¼ Inch Nuts	2
15	Male Valve	5
16	Female T	3
17	Female I	6
18	Male Compression Joint	6
19	Cork	2
20	Plastic I	1
21	1/4 Inch Copper Pipe	7/8 Ft

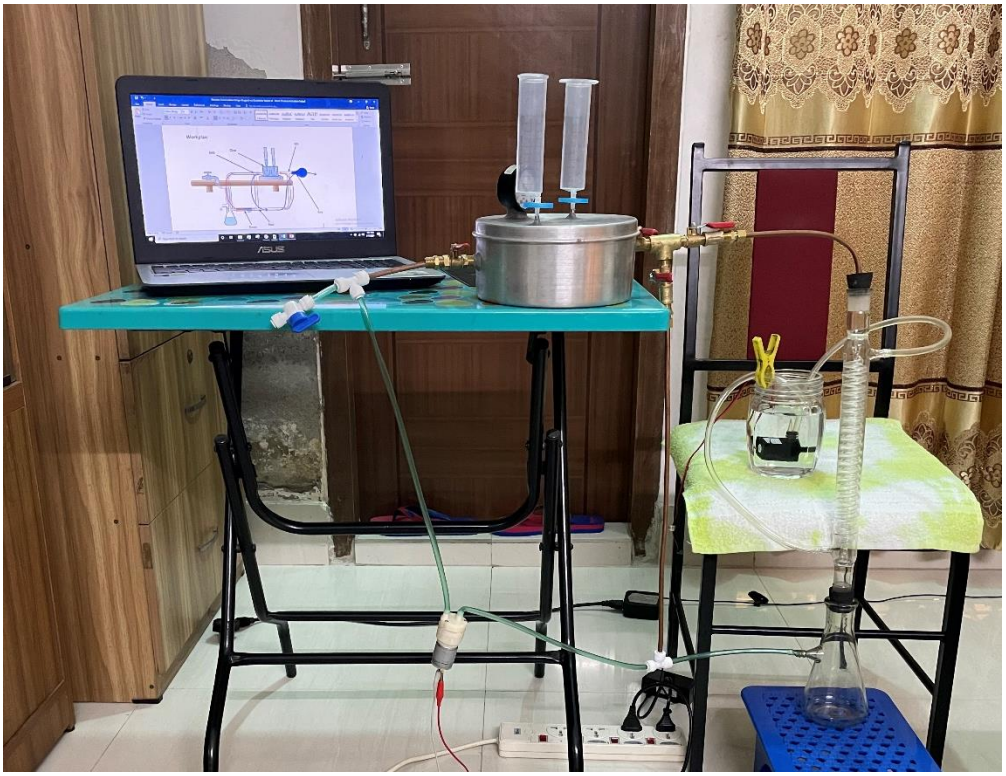

Figure 13: Si-Sara Full Setup

2.3 Blue Cycle and Red Cycle Mechanism

In figure 2, there are two cycles shown by using the arrow. The blue arrows are indicating the filtering cycle and the red arrow is referring to the drying cycle. These arrows are showing the path of how air will go through the whole system while operating. The Blue cycle will be completed only when filtration is done. We can see in figure 1, there is a valve on the left side of the metal box. When the filtration process will be done, that valve will be closed. So no air or any kind of material will go through the copper wire in the direction of the graham condenser. The valve on the right side of the metal box will be open. So air pumped through the vacuum pump will go out in the environment. So it will take out the air from the metal box and release it into the environment.

This pressure will also help to fasten the filtration process automatically. The total path of air flowing in the filtration cycle is shown by the blue arrows.

On the other hand, when we start the heating oven, syringes from the top of the metal box will be removed. Cover will be placed on top of the needles. While the drying cycle, the valve on the left side of the metal box will be open. So the vapor will continue flowing in the red path. It will go through a graham condenser and then the chemical will be consolidated in the conical flask. Only pure air will come out and it will continue traveling through the vacuum pump. The valve on the right side of the metal box will be closed this time. So no air will go out of the system. Instead of going out, the air will enter the box again and it will go through the cycle again. So the best part of this system is, by repeating the cycle, air will be completely purified so no toxic chemicals will be released into the environment. The toxic chemical will be stored on the conical flask after the whole repeating cycle is completed.

3 CHAPTER THREE: METHODOLOGY

There are two phases of the experiment. The first one is the separation of Asphaltene and maltine and the next phase is using pre-packaged silica cartridges (Solid Phase Extraction– SPE cartridges) for fractionation of maltene into saturates, aromatics, and resins using chromatography in a procedure similar to ASTM D4124 and IP469. Here we can see a flowchart of SARA fractionation below.

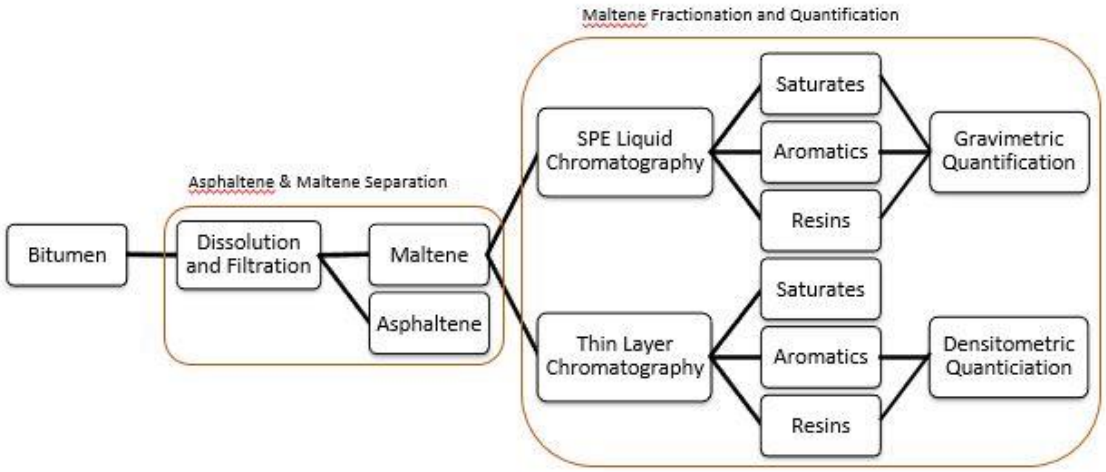

Figure 14: Flowchart of SARA Fractionation

3.1 Phase 1: Seperation of Ashphaltene and Maltene

Asphaltene is the most polar fraction of bitumen which is black/dark brown in color. In general, it makes bitumen stiff, strong, and brittle. Asphaltene is the easiest to remove among four SARA fractions. 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, 2001)

We choose using 1:100 w/v n-heptane stirred at room temperature for 24 hours and PTFE syringe filters

3.1.1 Step One: Specimen Preparation

The binder will have to be weighed to a precision of 0.01 mg and placed in a wide-mouthed jar with septa and a magnetic stirrer bar coated with PTFE. A 1:100 weight-to-volume ratio of solvent (n-heptane) to added bitumen will be poured into the jar. After that, the jar must be placed on the

stirrer and stirred at about 120 rpm. Sakib and Bhasin (2019) have experimented with the use of 0.1 g to 1.0 g binder to make 10–100 ml solution depending on the amount of maltene and the number of replicates desired.

3.1.2 Step Two: Filtration (Blue Cycle)

10 ml of solution (while being stirred) is drawn into a syringe and a pre-weighed syringe filter is placed on the tip of the syringe.The solution was pushed through a PTFE filter which will be and the filtrate is collected in pre-weighed small metal bowls placed inside the metal box. The asphaltene will be contained by the filters.

3.1.3 Step Three: Maltene Separation

Once the filtration process is done, maltene can be recovered from the filtered solution by heating at approx. 120°C for 40-45 min inside the metal box used as a heating oven. It is the drying cycle so the path indicating the red arrows will be open and mixed n-heptene will be consolidated into the conical flask with the help of a graham condenser. The process will continue over and over for 45 minutes so every last portion of n-heptane will be consolidated in the flask and no chemical will be released openly in the laboratory. After that, The residual maltene will be weighed and the amount of asphaltene is resolved gravimetrically.

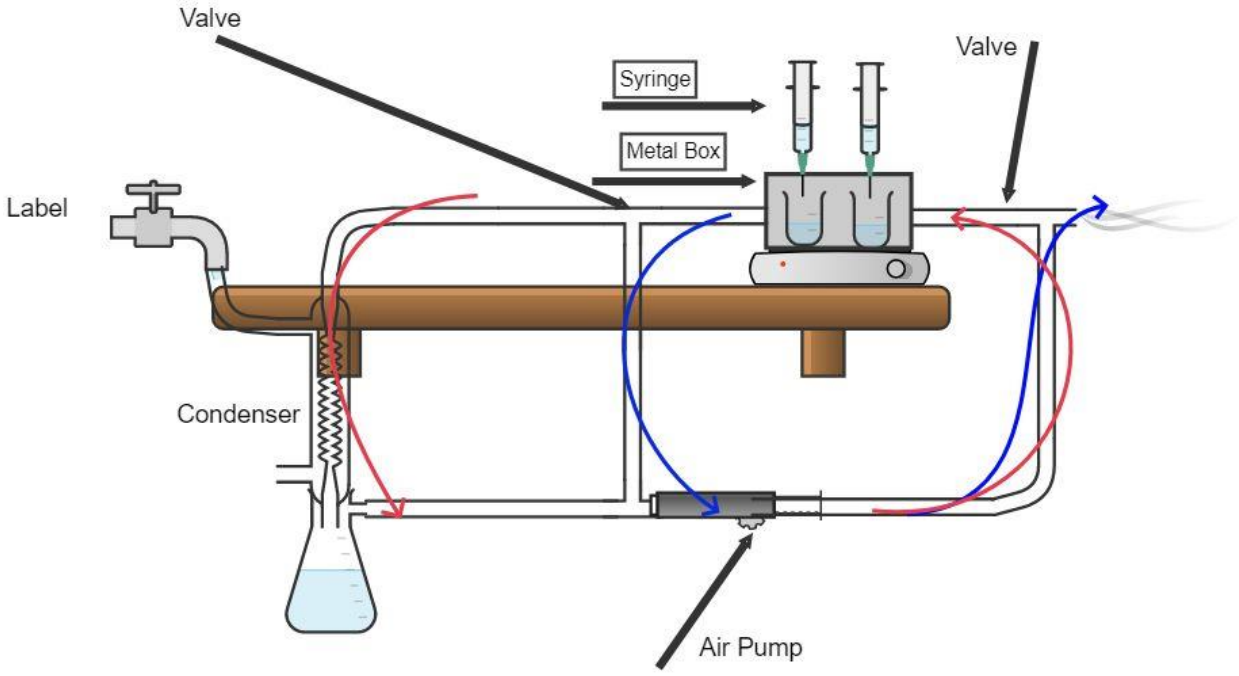

Figure 15: Sketch of the System

3.2 Phase 2: Fractionation of Maltene

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 sub-fractions, it is also possible to fractionate maltene into more than three fractions by appropriately choosing a solvent.

To develop the total process, some choices had to be made. The first is about the chromatographic method. In this case, we have selected a method based on solid-phase extraction (SPE). SPE is an attractive option for SAR separation since SPE cartridges, which can be used as disposable mini-LC columns, are commercially available and pre-packaged. It also saves time and reduces reproducibility issues.

Another two choices that had to be made are the choice of mobile phase and stationary phase. We have selected Silica Gel as a stationary phase. In the case of solvent selection, we have considered some facts. The solvent set should elute three fractions that correspond qualitatively/semi-qualitatively with existing standards. Compatibility with other standards is also a matter of consideration. Last but not the least, toxicity and availability of solvents were also an issue for the selection.

Table 2: Solvent Options for Elution

Procedure	Solvent	δ_D	δ_P	δ_H	δ_{Tot}
Saturates					
TLC, SPE, IP469	n-heptane	15.3	0.0	0.0	15.3
ASTM 4124	n-heptane	15.3	0.0	0.0	15.3
	Toluene*	18.0	1.4	2.0	18.2
Aromatics					
TLC, SPE, IP469	80:20 Toluene:n-heptane	17.5	1.1	1.6	17.6
ASTM 4124	Toluene	18.0	1.4	2.0	18.2
	50:50 Toluene:Methanol*	16.4	6.9	12.2	21.5
Resins					
TLC, SPE	90:10 DCM:Methanol	17.8	6.9	7.7	20.6
IP469	95:5 DCM:Methanol	18.0	6.6	6.9	20.4
ASTM D4124	Trichloroethylene	18.0	3.1	5.3	19.0

3.2.1 Step One: Preparation

Obtaining maltene from the filtrate of bitumen and n-heptane solution. A solution of 3.33 mg/ml maltene/n-heptane (w/v) ratio will have to be prepared for fractionation using SPE. Cartridges mounted on the metal box will be working as a vacuum manifold that allows multiple extractions to be carried out simultaneously.

3.2.2 Step Two: Saturates Collection

20 ml n-heptane will be added to 15 ml maltene solution followed by 10 ml n-heptane flush. The elution products then will be collected in a pre-weighed metal bowl; the collected fraction is deemed as ‘saturates’ dissolved in n-heptane.

3.2.3 Step Three: Aromatics Collection

25 ml of 80:20 (v: v) Toluene: n-heptane solution will be eluted. This portion is regarded as ‘aromatics’.

3.2.4 Step Four: Resin Collection

25 ml of 90:10 (v: v) dichloromethane and methanol solution will be prepared. This solvent eluted is the ‘resin’ fraction.

3.2.5 Step Five: Heating

Maltene fractions (saturates, aromatics, resin) will be recovered by heating inside the metal box used as a heating oven. The residual maltene fractions then will be weighed. The vapor produced during the healing process is run through a graham condenser. The remaining vapor is run through the condenser several times until the vapor is free from any sort of toxic gases.

4 CHAPTER FOUR: RESULT

Initially, the whole process was run with mineral water to make sure the process worked. Due to a lack of adequate lab facilities, we weren't able to run the process with actual bitumen samples. The process is still in work and some modifications have to be made.

5 CHAPTER FIVE: CONCLUSION

The main goal of this study was to develop a fast, inexpensive method to determine the polarity-based distribution. That could be used on a routine basis with high throughput. This would ultimately help create a sizable database of this property alongside typical engineering properties for bitumen from various sources. This compact method will also allow for site quality tests. As the test process is still ongoing, further modifications will be required from time to time.

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