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# Modification of Glass Waste and Use for Fractionation of Crude Oil

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**Abstract**

The study aimed to tackle the critical problem of glass waste accumulation in landfills by modifying glass waste into a valuable resource. The process involved meticulous cleaning and purification of the glass waste, which was transformed into a suitable stationary phase for separating crude oil. The modified glass waste was used to fractionate the crude oil into distinct components using the SARA (Saturate, Aromatic, Resin, and Asphaltene) analysis method. The chemical composition accuracy of the separation process was validated through spectral analysis, which highlighted the effectiveness of the modified glass waste as a stationary phase. This research demonstrates the potential for repurposing glass waste and offers an environmentally friendly and economically viable solution for crude oil separation. It underscores the importance of recycling materials for multiple purposes and addressing environmental challenges through innovative approaches.

**Keywords:** Glass waste, Crude oil fractionation, SARA diagram, Chromatography

## Introduction

Petroleum, a mixture of hydrocarbons and their derivatives, is widely used as a global energy source, along with water, sand, and minerals. In petroleum refining, separating hydrocarbons from the mixture poses a significant challenge [1, 2]. Crude oil contains various types of hydrocarbons with diverse physical and chemical properties. Small amounts of sulfur (6.0%), nitrogen (2.0%), and oxygen (1.5%) are also present. The majority of the elemental composition is made up of carbon (83-87%) and hydrogen (10-14%) [3, 4]. Crude oil is the main source of fossil fuels around the world. Determining the physicochemical properties of crude oil is crucial for the oil industry. This involves measuring, the American Petroleum Institute (API) gravity, kinematic viscosity, SARA analysis, pour point, and carbon residue [5,6]. Light oils have a density of 0.8 g/cm3, while heavy oils have a density of approximately 1.0 g/cm$^3$ [7]. Many attempts have been made to classify crude oil compounds into distinct groups or classes. One effective method for this purpose is the SARA separation, which classifies crude oil into four primary chemical classes based on differences in solubility and polarity. These four SARA fractions are saturated (S), aromatics (A), resins (R), and asphaltenes (A). To achieve this separation, the SARA method first removes asphaltenes through precipitation using a paraffinic solvent like n-pentane or n-heptane. Afterward, the crude oil that is left is

separated through chromatography using treated glass waste. These four general fractions saturates, aromatics, resins, and asphaltenes, help to categorize and separate crude oil components efficiently [8-10]. Maltenes dissolve in regular hexane, is a mixture of saturated hydrocarbons and aromatic components. It may also contain heterogeneous molecules [11]. Petroleum asphaltenes are the heaviest and most aromatic component of crude oil, forming a brittle, infusible solid colloidal dispersed in crude oil with a mass fraction of 0-10% or more. Asphaltenes are a complex molecular combination [12]. Compounds consisting of benzene and its derivatives are referred to as aromatics. All petroleum contains aromatics; having alkyl chains, cycloalkane rings, and other aromatic rings. Depending on how many aromatic rings are present in the molecule, aromatics are frequently categorized as mono-, di-, and tri-rings [13,14]. This portion comprises polar molecules, frequently with heteroatoms like nitrogen, oxygen, or sulfur. One practical definition of the resin fraction is the fraction that is soluble in light alkanes like pentane and heptane but insoluble in liquid propane [15, 16] throughout the majority of human history, glass has been primarily based on silica. Modern technology extensively utilizes silicate glass, which serves numerous specialized purposes in communications, electronics, and composites in addition to its traditional uses for containers, windows, lamps, and optical components [17, 18]. Glass is a much-diversified industry, and can be broken down into many subsectors, such as container glass, flat glass, special glass, and the glass fibers [19]. In daily life, glass is used in various industries to manufacture multiple goods, such as plate glass, bottle glass, LCD glass, and fluorescent lamp glass [20,21]. This is due to its benefits, such as good optical properties, chemical stability, and low cost. Glass recycling is essential for waste management, resource conservation, and energy efficiency. Sorting used glass by type and usage is a crucial step in the recycling process [22,23]. Yet, most recycled scrap glass has only been utilized to make bottles and construction materials [24, 25]. Waste glass is prone to fracturing during recycling and requires high-temperature processing, which consumes significant energy [26]. The current study aims to investigate and develop cost-effective, readily available, and environmentally friendly stationary phases for use in chromatography columns. These phases were employed to separate heavy oil components into their basic constituents (asphaltene, maltene, paraffin, aromatic, and resin) and independently studied their chemical elements.

## Materials and Methods

Crude oil obtained from Al- Rashidiya field in Iraq, Ethanol (British drug,98%), n-hexane (Fluke 99%), heptane (British drug,98%), Methanol (British drug 99%), toluene (pancreatic 99%), benzene (GGR 98%) were of high purity. Waste glass broken (glass containers from the trash) was used for the study, and Whatman Quantitative Filter Papers, Ashless, Grade 42 was used.

### *Instrumentation*

A separation column of German origin, with dimensions of height of 50 cm and an inner diameter of 2.2 cm was utilized, stone furnace and Nuclear Magnetic Resonance (NMR) measuring device was from Bruker (400 MHZ), Germany.

### *Pre-treatment of Glass*

The setup for the stationary phase separation was as follows: Glass waste was taken, specifically glass juice containers, from the garbage. Properly wash glass waste with water to remove dirt, labeled paper, and dried.

The crushed and mashed glass containers prepared in the initial step into medium-sized pieces to proceed with the work steps. The treated glass was placed in a ceramic crucible and heated to 700 °C in a blazing oven for 4 h and fused the fragments into a single mass.

Removed the treated glass samples from the burning unit after cooling and broke the glass block into small pieces [27]. The glass processed and prepared in the previous work steps varies in size, with some parts forming large blocks, others being medium-sized, and some being small. Volumetric corrections were necessary for these modified glass pieces to address this variability by employing ceramic mortar was initially employed. Subsequently, particle size correction was conducted using sieves with various granular sizes. The sieving process involved three stages, employing sieves with granular diameters of 50 µm, 75 µm, and 150 µm, respectively. The remaining samples were collected within these sieves [28]. Due to the negative effect of asphaltene on the processes of separating the chemical components (aromatic, naphthenic, and paraffinic), including the interferences caused by the impact of the polarity factor through the presence of nitrogen, sulfur, and oxygen atoms in its structural formula. It was necessary to remove the asphaltene by using hydrocarbon solvents to ease the fractionation of the asphalt components. The n-hexane was used in the precipitation of asphalts, where (100 mL) of n-hexane was added to (5 g) of oil and accurately weighed in a ratio of 100:5 (vol: weight). The mixture was shaken for an hour at room temperature. The solution was filtered with ashless filter paper No. 42. The asphaltene was washed with sufficient n-hexane until the drops received from the filter paper into the collecting container became colorless. It was dried at (100 °C) in the oven for 24 h and then weighed accurately and found the percentage of asphaltene in the pure crude oil; after that, it was dried at room temperature; the soluble fraction was obtained in a collecting container containing the maltene, which was then weighed accurately and its percentage was found in the crude oil used [29,30]. The separation columns used in this research have a height of 50 cm and a diameter of 2.2 cm. They were equipped with a glass spout at the bottom to control the speed of solvent flow. A piece of glass wool was positioned at the base of the column, and the tap opening was filled with a predetermined weight of 50 g of the previously prepared stationary phase material, pre-treated glass. The separation process is shown in the Fig. 1.

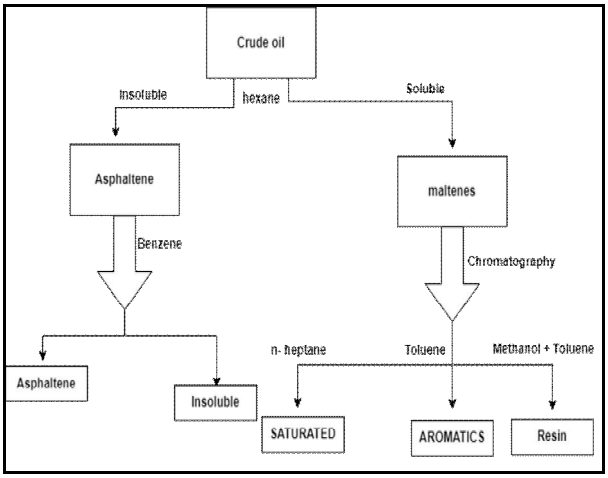


***Figure 1.*** **SARA diagram**

## *Separation of high paraffin content by modified glass*

50 g of pre-treated glass (150 µm) was treated with heptane to facilitate the process of transferring it to the separation column, with tapping on the column to prevent the formation of gaps inside the column, added 5 g of maltene and treated with heptane for the separation of compounds with high paraffin content. In the end, it was noticed that the drops of heptane coming down from the bottom of the column became colorless. After that, toluene was added to the column to

separate the compounds with high aromatic content, and as in the previous time, until colorless drops were obtained. After that, a mixture of coloring was added: methanol and toluene at a ratio of 50:50, and the separation of the last part, which is represented by compounds with high polar content (resin), separation products were collected, and distilled to separate the solvent. The process occurred under laboratory temperature and normal atmospheric pressure, and the velocity of the droplet descending from the column was at a 5-7 drops per minute.

### *Separation of aromatic by column*

50 g of pre-treated glass with a particle size of 150 µm was used, and it underwent the typical heptane treatment to facilitate its transfer to the separation column. Careful packing was ensured by tapping the column to prevent gaps. Subsequently, 3 g of previously separated aromatic content was added to the column. The treated glass was further processed with n-hexane and introduced into the separation column. Following this, the column underwent treatment with a mixture of regular hexane and benzene in a ratio of 5:95 to separate the monoaromatic content. The product drops were collected at a rate of 5-7 drops per minute.

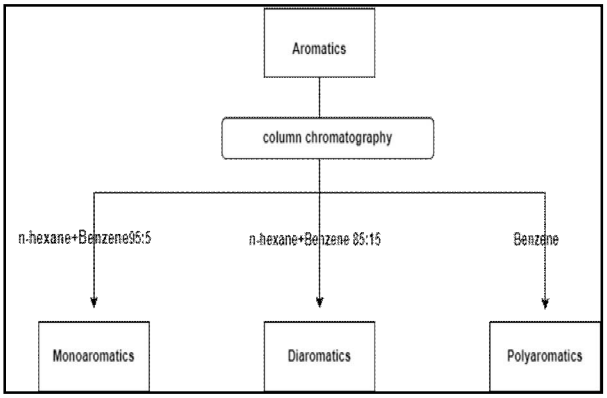


***Figure 2.*** **Aromatic compound separation**

The solvent changed until colorless drops were obtained, using a mixture of n-hexane and benzene at a ratio of 15:85 to separate the diaromatic content. The product drops were collected at 5-7 drops per minute. Further solvent change was carried out until colorless drops were achieved, using benzene to separate polyaromatic content. The separation process is illustrated in the Fig. 2. [31].

## Results and Discussion

### *Microstructural Analysis Using SEM-EDS Techniques*

Scanning electron microscopy (SEM) coupled with energy dispersive spectroscopy (EDS) is the preferred method for identifying the presence of elements in the sample and determining particle size [33]. The results are presented in Fig. 3 and Fig. 4.

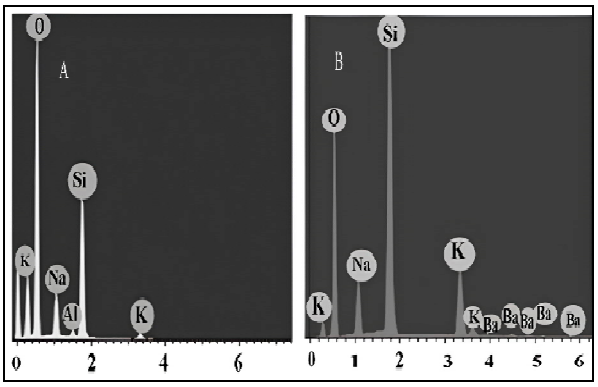


***Figure 3.*** **(a) EDS pre-treated glass (150 µm) (b) EDS for glass waste**

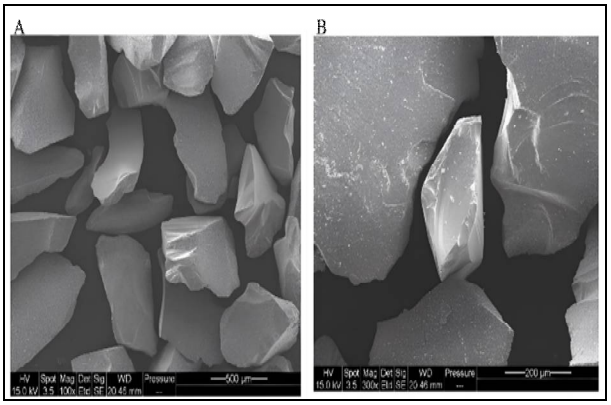


***Figure 4.*** **(a) SEM of pre-treated glass (150 µm) particles at ×100 magnification (b) SEM pre-treated glass (150 µm) particles at ×300 magnification**

The results of the EDS analysis revealed that the presence of silica and alumina oxides in the treated glass played a significant role in the separation processes. It is well-established that alumina and silica are commonly utilized as stationary phases in separation columns [34]. A SEM is an instrument employed to examine and record the surface topography of a specimen at a resolution considerably higher than that achievable with light microscopy [35]. This versatile instrument provides valuable structural insights into the surfaces of various specimens. Before undergoing pretreatment, the majority of glass particles typically exhibit irregular shapes with sharp edges. This inherent characteristic of untreated glass particles underscores the importance of proper pretreatment methods to enhance their suitability for various applications. Through appropriate pretreatment processes, such as grinding or milling, the irregular shapes and sharp edges of glass particles can be modified, leading to improved properties and performance in subsequent applications. As observed in the SEM, pretreated glass exhibited improved mechanical properties compared to untreated glass, revealing a noticeable similarity in particle sizes and crystal structures.

### *Separation by Glass Waste*

After collecting samples from the results of the separation of crude oil by treated glass waste, NMR measurements were made using the same solvents and activated glass waste with a granular diameter of 150 um. Three main portions of maltinates were separated according to molecular weight as indicated in Table 1. It shows how crude oil from the Al-Rashdia field was fractionated into its constituent parts. It was discovered that there is a significant proportion of saturated material, which contributes to the oil paraffinic formula. The ratio of the components was found to be aromatic, asphaltene, and resin as the last class. Because of this directive, crude oil has a high viscosity and density. This is consistent with the literature or with the specifications of Al Rashidiya crude oil, as it is characterized by high viscosity, heavy components, and high paraffinity [32].

***Table 1.*** **Percentages and weights of separation products by glass waste.**

| separated components | solvents | % | Wt/gm |
|---|---|---|---|
| Asphaltene | n-hexane | 0.62 | 12 |
| Paraffinic | Heptane | 0.85 | 44 |
| Aromatic | Toluene | 1.55 | 30 |
| Resin | Methanol+ Toluene | 2.10 | 4 |
| Residue | ____ | 0.1460 | 3 |

### *$^{1}$H NMR Spectra Analysis*

The samples of isolated components from Al-Rashdia crude oil were analyzed using NMR spectroscopy (Fig. 5 – 7). The tests involved the area under the curve to identify signals from various protons in the separated regions obtained from treated glass waste [36]. Demonstrate aromatic hydrogen (Ha), hydrogen at the alpha position of an aromatic ring (H), naphthenic hydrogen (Hn), and paraffinic methylene hydrogen signals (Hmy). By looking at the $^{1}$H NMR results for oil and its fractions, paraffinic methyl hydrogen (Hme) Presence was determined. The Ratio of saturated sections was more significant than the other one. Although the values were distributed because the aromatic protons are restricted to the area between (8.5-6.5 ppm), the alpha site protons are found within the range of (3.4-1.7 ppm), and the naphthenic systems protons are located within the scope of (2.2-1.4 ppm).

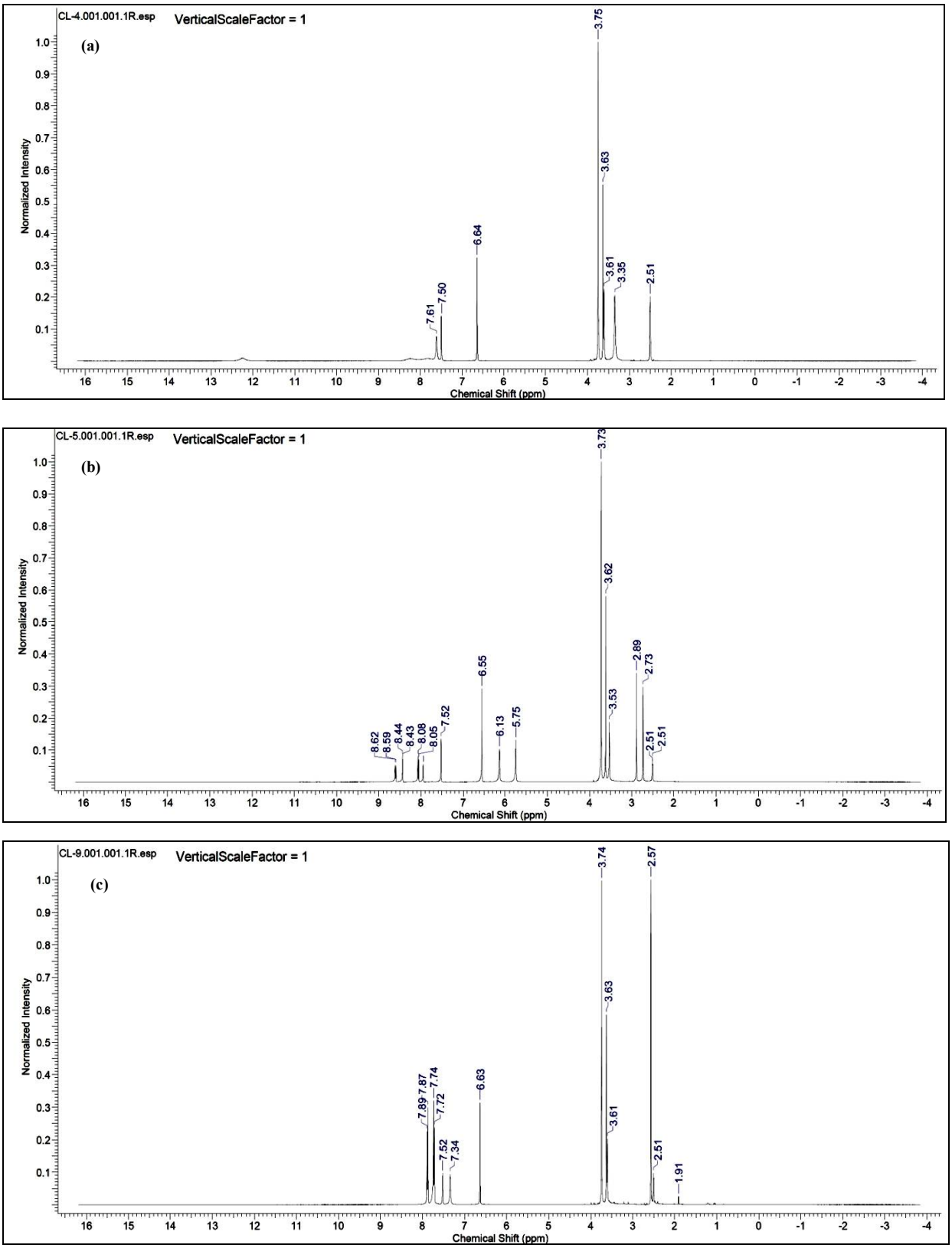


*Figure 5.* **$^{1}$H NMR spectrum of (a) asphaltene (b) maltene and (c) aromatic**

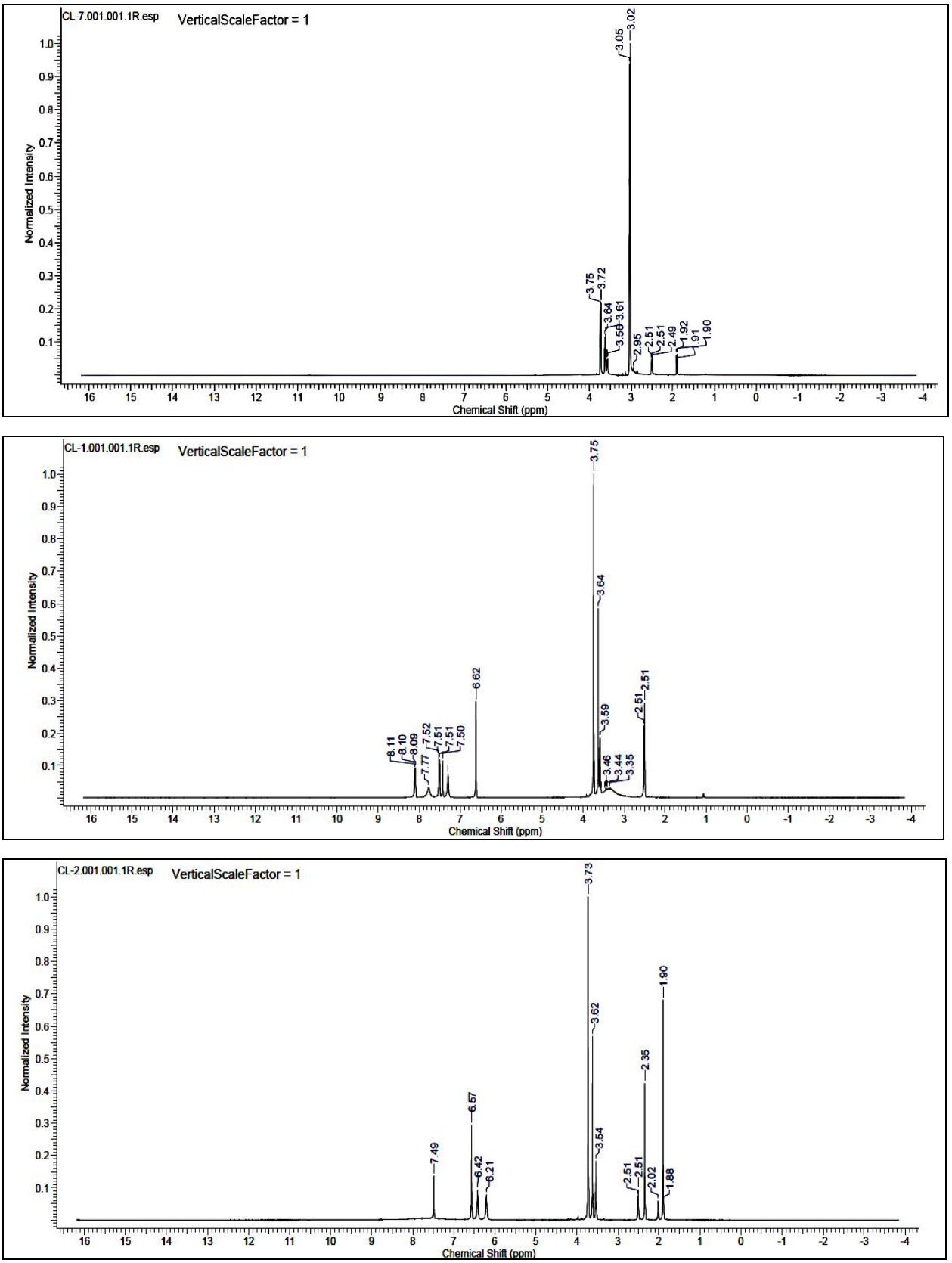


**Figure 6. $^{1}$H NMR spectrum of (a)paraffin (b) resin (c) mono-aromatic compounds**

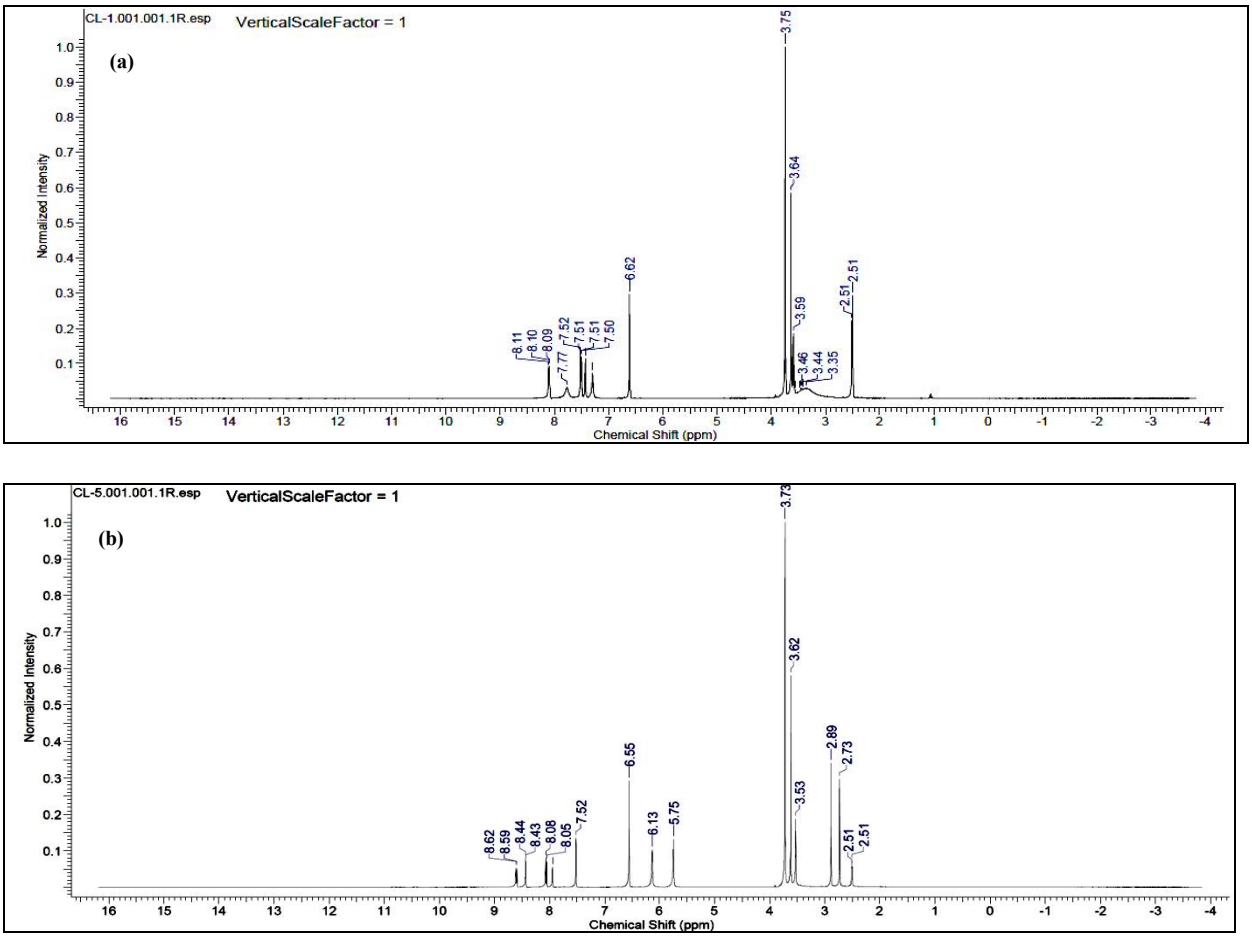


*Figure 7.* **$^{1}$H NMR spectrum of (a) di-aromatic (b) poly-aromatic**

The paraffinic alpha methylene groups range from (1.8 to 0.9 ppm), while the methyl groups fall between (1.4 and 0.5 ppm) [37]. The Table 2 and 3 for $^{1}$H NMR spectrum shows the number of protons and their chemical shift value.

Table 2 The $^{1}$H NMR chemical shift region and number of protons of asphaltene, maltine, aromatic, paraffin, and resin were obtained from the fractionation of crude oil. The results showed the efficiency of the work. After analyzing the results obtained from measuring the $^{1}$H NMR spectrum of malten and its parts, it was discovered that the percentage of paraffin materials is higher than the other parts. and this is consistent with the literature regarding the Al-Rashidiya field, as the values were distributed on the basis that the area confined between (8.5-6.5 ppm) belongs to aromatic protons, and within limits (3.4 -1.7 ppm) the protons of the alpha site are located in the aromatic ring, but within the range (2.2-1.4 ppm). So the protons belonging to the naphthenic systems appear, and the paraffinic alpha methylene groups fall within the range (1.8-0.9 ppm) and the methyl groups within the range (0.5-1.4 ppm). Through information from the values of the $^{1}$H NMR results, the methylene proton of paraffin compound were in higher percentage in asphaltene part of Al-Rashidiya field. From (Fig. 5 - 7), one can observe that asphaltene contains many methylene groups, followed by methyl groups, the ratio of naphthenic protons to the alpha aromatic were close o each other in the relative distribution. The fractionated parts of maltenes (paraffinic, aromatic, resin)

were separated with column chromatography by using a treated glass trash stationary phase.

***Table 2.*** **The $^1$H NMR chemical shift region and number of protons of asphaltene, maltine, aromatic, paraffin, and resin obtained from fractionation of crude oil.**

| S. No | δ ppm | Number of protons |
|---|---|---|
| | **Asphaltene** | |
| 1 | 2.51 | br. s., 10 H |
| 2 | 3.35 | br. s., 21 H |
| 3 | 3.53 - 3.69 | m, 22 H |
| 4 | 3.75 | s, 26 H |
| 5 | 6.64 | s, 9 H |
| 6 | 7.50 | s, 5 H |
| 7 | 7.61 | br. s., 8 H |
| | **Maltine** | |
| 1 | 2.34 - 2.62 | m, 5 H |
| 2 | 2.73 | s, 7 H |
| 3 | 2.89 | s, 7 H |
| 4 | 3.53 | s, 9 H |
| 5 | 3.73 | s, 22 H |
| 6 | 5.75 | s, 7 H |
| 7 | 6.13 | br. s., 7 H |
| 8 | 6.55 | s, 7 H |
| 9 | 7.52 | s, 4 H |
| 10 | 7.95 | s, 2 H |
| 11 | 8.06 | d, J=8.07 Hz, 4 H |
| 12 | 8.43 | d, J=1.96 Hz, 3 H |
| 13 | 8.60 | dd, J=8.13, 2.02 Hz, 4 H |
| | **Aromatic** | |
| 1 | 2.28 - 2.54 | m, 6 H |
| 2 | 2.54 - 2.60 | m, 14 H |
| 3 | 3.44 - 3.69 | m, 18 H |
| 4 | 3.74 | s, 19 H |
| 5 | 6.63 | s, 7 H |
| 6 | 7.34 | br. s., 6 H |
| 7 | 7.52 | s, 4 H |
| 8 | 7.73 | d, *J*=8.56 Hz, 15 H |
| 9 | 7.88 | d, *J*=8.31 Hz, 11 H |
| | **Paraffin** | |
| 1 | 1.68 - 1.94 | m, 3 H |
| 2 | 2.33 - 2.54 | m, 4 H |
| 3 | 2.95 | br. s., 4 H |
| 4 | 2.99 - 3.06 | m, 32 H |
| 5 | 3.44 - 3.68 | m, 9 H |
| 6 | 3.68 - 3.82 | m, 9 H |
| | **Resin** | |
| 1 | 2.46 - 2.61 | m, 12 H |
| 2 | 3.22 - 3.51 | m, 14 H |
| 3 | 3.52 - 3.68 | m, 18 H |
| 4 | 3.75 | s, 21 H |
| 5 | 6.62 | s, 7 H |
| 6 | 7.30 | br. s., 6 H |
| 7 | 7.43 | s, 3 H |
| 8 | 7.46 - 7.63 | m, 7 H |
| 9 | 7.77 | br. s., 6 H |
| 10 | 8.10 | dd, *J*=5.81, 3.48 Hz, 6 H |

***Table 3.*** **The $^1$H NMR chemical shift region and number of protons of mono, di, and poly-aromatic wereobtained from crude oil fractionation.**

| S. No | δ ppm | Number of protons |
|---|---|---|
| | **Mono aromatic** | |
| 1. | 1.76 - 1.95 | m, 11 H |
| 2. | 1.96 - 2.15 | m, 3 H |
| 3. | 2.26 - 2.43 | m, 9 H |
| 4. | 2.43 - 2.61 | m, 5 H |
| 5. | 3.54 | s, 8 H |
| 6. | 3.58 - 3.66 | m, 11 H |
| 7. | 3.67 - 3.87 | m, 23 H |
| 8. | 6.21 | br. s., 7 H |
| 9. | 6.42 | br. s., 7 H |
| 10. | 6.57 | s, 9 H |
| 11. | 7.49 | s, 7 H |
| | **Di aromatic** | |
| 12. | 1.91 | s, 1 H |
| 13. | 2.49 - 2.61 | m, 4 H |
| 14. | 3.45 - 3.67 | m, 9 H |
| 15. | 3.67 - 3.92 | m, 14 H |
| 16. | 6.61 | s, 5 H |
| 17. | 6.95 | d, *J*=8.56 Hz, 21 H |
| 18. | 7.00 - 7.21 | m, 6 H |
| 19. | 7.49 | s, 2 H |
| 20. | 7.54 | br. s., 3 H |
| 21. | 7.68 - 7.80 | m, 20 H |
| 22. | 7.85 | d, *J*=8.56 Hz, 3 H |
| 23. | 9.78 | s, 12 H |
| | **Poly aromatic.** | |
| 24. | 1.91 | s, 4 H |
| 25. | 2.38 | s, 19 H |
| 26. | 2.44 - 2.55 | m, 4 H |
| 27. | 3.56 | s, 4 H |
| 28. | 3.63 | s, 5 H |
| 29. | 3.73 | s, 10 H |
| 30. | 7.50 | s, 2 H |
| 31. | 7.64 - 7.74 | m, 12 H |
| 32. | 7.83 | s, 5 H |

## *Comparison Fractionation of Crude Oil by Glass Waste and Traditional Methods*

Based on the results obtained from crude oil fractionation operations using glass waste and comparing them to traditional methods, and innovative by previous researchers to fractionate crude oil and its components. The compiled findings are shown in Table 4. It was evident thatthe current proposed method is efficient, economical, and environmentally friendly.

***Table 4.*** **Comparison of traditional and Modified Glass Waste method.**

| Aspect | Modified Glass Waste | Traditional Methods | Clays mineral |
|---|---|---|---|
| Efficiency in Fractionating Crude Oil | High | Varies | low |
| Cost-effectiveness | Economical | Costly | Economical |
| Environmental Impact | Low | Varies | Varies |
| Resource Utilization | Glass Waste | Virgin Materials | Consuming more natural resources |
| Recycling of solvent and stationary phase | yes | Varies | yes |
| References | Current study | 28,38 | 39,40 |

## Conclusion

Utilizing modified glass waste as a novel material opens up innovative possibilities in various fields, including the preparation and design of stationary phases for crude oil separation. It becomes feasible to separate crude oil into its primary constituents effectively. Additionally, the utilization of modified glass waste allows for the diagnosis and determination of the weight and percentage of these constituents within the crude oil sample. The results obtained from the $^{1}H$ NMR demonstrate the remarkable separation efficiency achieved, particularly for heavy oil fractions, showcasing the potential of this approach in advancing oil separation and analysis techniques.

## Conflict of Interest

The author declares that there is no conflict of interest.

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