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Enhancing Sulphur Removal from Iraqi Qayyarah Crude Oil Through Glass-Waste-Based Adsorption at Various Temperatures and Contact Durations

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Abstract: Glass waste has become widespread nowadays as a result of using glass for various popular applications and products including smartphones. Samples of cracked Gorilla Glass were collected from mobile phone repair centers and modified for use with samples of crude oil. This research was conducted in light of the high sulfur content in local oil products and how it contributes to corrosion and reduced engine efficiency. The research involved modifying and thermally activating Gorilla Glass waste, which improves its efficiency in sulfur adsorption. This research investigated the influence of contact duration and temperature on the efficacy of adsorption desulfurization (AD) and used XRD technology to analyze the results. In XRD tables, note that the sulfur content decreases after treatment with Gorilla Glass, from 6.72% to 3.83% and decreases further after furnace treatment, which the sulfur content decreased from (3.83% to 2.24%). The extended duration of treatment, the lower the sulfur content, as there is a continuous decrease in the sulfur content with time from 6.16% at 15 min to 4.52% at 45 min. We also note that temperature plays a major role in reducing the sulfur content, which decreases from 5.26% at 30°C to 4.11% at 60°C. Thus, the higher the temperature, the greater the sulfur loss. This research aims to remove sulfur from Iraqi Qayyarah crude oil and to study the relationship between temperature and contact time on one hand and sulfur adsorption rate on the other.

Keywords: Glass waste, Crude oil, Gorilla glass waste, Adsorption, Desulfurization

Introduction

Desulfurization of crude oil is essential prior to its refinement into final products. The requirements governing transportation fuels have become stricter over time in regard to their sulphur content. Moreover, numerous petrochemical products are engineered to be virtually devoid of sulfur. Further, the sulfur level of crude oil adversely impacts its pricing and processing costs; hence, the removal of sulfur from oil has emerged as a primary conversion necessity in most refineries. (Javadli et al., 2012; Leprince, 2001).

Hydrodesulfurization (HDS) and carbon rejection methods, such as coking and fluid catalytic cracking (FCC), are the main commercial procedures used to desulfurize heavy oil. Even though these technologies are extremely capable of desulfurizing heavy oil, they have significant drawbacks. One of the environmental drawbacks are the significant carbon footprints. These processes, including creating the hydrogen required for HDS, involve high temperatures. Additionally, when heavier and more sulfur-rich crude oils are processed, the cost of refining increases. Thus, there is a rising interest in alternative desulfurization routes (Rana et al., 2007).

The third most common ingredient in petroleum is sulphur, followed by carbon and hydrogen. Crude oil has an average sulphur concentration that ranges from (0.03 to 7.89) mass% (Soleimani et al., 2007). There are two types of sulfur compounds: both organic and inorganic. Pyrite, H2S, and elemental sulfur are examples of inorganic sulfur that can be dissolved or suspended (Agarwal et al., 2010). The primary source of sulfur in crude oil is organic sulfur compounds, such as thiols, sulphides, and thiophene compounds. Some of the important classes of organic sulfur compounds are shown in Fig. 1 (Javadli et al., 2012). More complex sulfur compounds are usually found in higher amounts in crude oils with higher densities and viscosities. Using hydrodesulfurization or thermal methods, the aliphatic acyclic sulphides (thioethers) and cyclic sulphides (thiolanes) can be easily removed. On the other hand, sulfur-containing aromatic rings, such thiophene and its benzo-analogs, like benzothiophene, dibenzothiophene, and benzonaphthothiophene, are more resistant to sulfur removal by thermal conversion and hydrodesulfurization (Gray et al., 1995). Transparent and non-crystalline, glass is an inorganic material used in practically every facet of daily life. glass can be produced in a wide variety of shapes including sheets, wires, fibres, and many others. Modern commercial glass can have weight fractions of more than eight distinct oxides and substituents. Each of these components has a considerable impact on glass's structural, rheological, thermal, and mechanical characteristics in terms of composition and property engineering. These qualities are essential metrics for applications involving displays. Glass composition must be developed according to the applications' desired glass qualities (Künzi, 2002).

Additionally, the fusion draw rates in production are influenced by Glass's rheological and thermomechanical characteristics, which ultimately determine the throughput and yield of glass production. (Kaur et al., 2019) (Qiao et al., 2019). Modern touchscreen smartphones have protective screens, which are especially important for possible triage applications because they can be removed without causing damage to the phones. Gorilla® Glass (GG), a chemically reinforced glass used by Corning Inc., is typically utilized to make the screens (McKeever et al., 2019). Gorilla Glass is a sturdy and durable kind of glass. Gorilla glass is made of alkali-aluminosilicate sheet glass, which is extremely difficult to break and resistant to scratches. Gorilla Glass comes in several iterations, such as Gorilla Glass 1, Gorilla Glass 2, Gorilla Glass 3, and Gorilla Glass 4. Compared to Gorilla Glass 2, Gorilla Glass 3's strength and scratch resistance have increased by 40%. Gorilla Glass 4 is up to two times stronger than its competitors and can be used on smartphones, laptops, tablets, portable media players, computer displays, and some televisions (Karasu et al., 2017; Seong et al., 2012). The ingenuity behind the discovery of x-rays in 1895 goes back to the German scientist Rutgen, who conducted an electron beam experiment on a gas charge tube ionization tube. Its photons have more energy than visible rays, and X-rays have 10-8 cm wavelengths. Therefore, the expression of the wavelength in the range of X-rays is in units of A•, and we can benefit from X-rays in analysis in several ways that these rays can absorb and thus give qualitative and quantitative information about This absorbing substance. X-ray diffraction allows the analysis of crystalline materials; the rays passing through the crystals will pass, and by measuring the amount of diffraction, the composition of the crystal can be known (Moram et al., 2009; Mosher, 1992).

Method

Crude oil from Al- Qayyarah field in Iraq, sodium hydroxide (GGR 98%), Waste glass. Samples of glass waste from mobile phone repair centers were collected from several local repair centers, and heavy crude oil from the Governorate of Nineveh's Qayyarah field in Iraq. Upon gathering the gorilla glass waste, it was rinsed with distilled water and placed in a beaker, to which water was added and left for some time. After employing stagnation followed by tilting decantation, water was poured while retaining the gorilla glass. This procedure was performed multiple times until it was observed that the water at the top of the glass was devoid of contaminants. Subsequent to the elimination of contaminants from the glass, the models are pulverized to facilitate the ensuing process. This is done by crushing with a ceramic mortar and adjusting the granular size utilizing 150, 75, and 50 mm sieves (Choi et al., 2023; Farhan et al., 2017).

The first step in sample preparation entails acquiring 100 grams of 150mm Gorilla Glass. At a concentration of 5% NaOH, the solution and the treated glass are mixed using an electronic stirrer for one hour, after which the mixture is transferred to a boiling flask for two hours. The reflux process is then performed. The product is then filtered using a separating funnel, keeping the precipitate. In the second part, 100 grams of pre-treated 150 mm glass is moved to a crucible, which is Inserted in the burning unit at 600 °C for four hours. After that, it is extracted from the burning unit and placed in the Desiccator device.

Desulfurization of the Qayyarah Crude Oil

A glass column of 50 cm in height and 2.2 cm in inner diameter was utilized, and it had a faucet at the bottom to regulate the emptying pace. Both the bottom of the column and the bottom of the faucet have a piece of glass wool. As the stationary phase, 50 grams of Gorilla Glass are added to the column, treated, activated and prepared for work previously. Using heat-treated Gorilla Glass once and the Gorilla Glass treated by reflux with sodium hydroxide 5% once again, each separately. After that, 50 grams of Qayyara crude is transferred to the column containing the aforementioned stationary phases. After that, drops of crude oil are received from the tap after different times have passed. The time taken was about three hours. After that, samples were collected to conduct a sulfur measurement.

Study Relationship Between Temperature and Contact Time on Sulfur Content

15 g of 150 mm gorilla glass was placed for work (burnt at a temperature of 600 °C) and placed inside a volumetric vial, and 100 ml of Qayara crude was added to it. In the first stage, the water bath temperature was fixed at 45 °C and the work was repeated with different contact times. The volumetric flask was placed inside the water bath for 15 minutes with continuous stirring using an electronic stirrer. The samples were collected in a sealed test tube to make the necessary measurements of the sulfur content, and the connection between sulfur content and temperature was studied. The same steps were repeated, but with contact time increased to 30 minutes, and the samples were collected and kept for study. This is repeated one more time, but by making the contact time between the crude oil and the treated glass 60 minutes. The results were then collected for the sulphur content using concentration. For the second stage, 15 g of prepared Gorilla Glass 150 mm (reflux with NaOH 5%) was put to work and placed inside a volumetric flask using the electronic stirrer, where the contact time was constant, which is one hour for all models in this stage. The work is repeated for the three models with a temperature change. 30°C for the first model, 45°C for the second model, and 60°C for the third. The samples were collected for the study and examinations (Hussein Hatem Meteab et al., 2024).

Desulfurization by Absorption

Because of the dangers and harms associated with the sulfur compounds accompanying the fumes of engines that operate using various types of fuel, to health and the environment, many regulatory bodies are issuing strict legislations and laws to limit the sulfur content of all petroleum products, which must be to very low limits approaching 15 ppm, and this is not possible through the traditional hydrogenation units utilized in refineries. For this reason, non-traditional methods are being sought. The most important sulfur compounds in oil come in several varieties, namely thiols, sulfides, and disulfides (Figure 1) (Speight, 1999). In this research, the proportion of sulfur in crude oil from Qayyarah was measured and was found to be 6.7%, which is consistent with those found in literature. The full results are depicted in Table 1. The adsorption of sulfur with Gorilla glass burned at 600 °C was utilized in this research as a method of desulfurization.

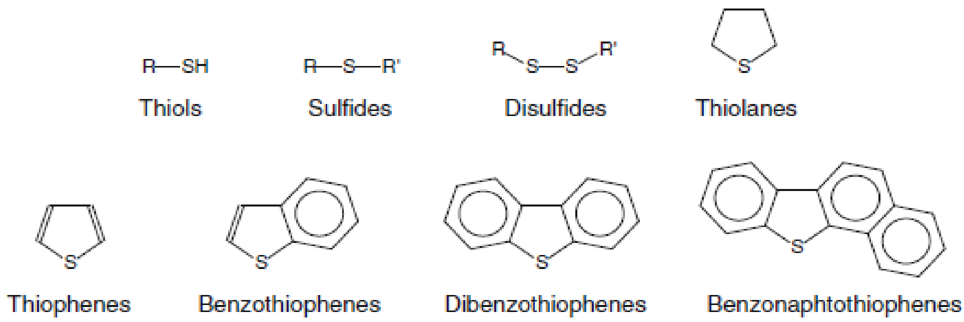

Figure 1. The most important sulfur compounds in the oil

Results and Discussion

Gorilla Glass comprises several oxides, including calcium oxide (CaO), magnesium oxide (MgO), alumina oxide (Al_2O_3), and Silicon dioxide (SiO_2) When the Gorilla Glass is prepared for work (150) mm, a reaction occurs between silica and sodium hydroxide 5%, as in the following equation:

$$SiO_2 + 2NaOH \longrightarrow Na_2SiO_3 + H_2O.$$

Zeolites are frequently utilized as water softeners in detergents and as catalytic materials in the petrochemical sector. They are also referred to as molecular sieves because they can selectively extract a specific chemical from a fluid mixture, such as removing H_2S from biogas. Zeolites are primarily made up of a three-dimensional framework of SiO_4 and AlO_4 tetrahedrals and are microporous crystalline minerals. Their frameworks are normally anionic, and to maintain electrical neutrality, Charge-compensating cations such as sodium and calicium are found in catalysts. These cations are useful for selective H_2S adsorbents because they can be used in ion exchange procedures, like in the well-known Claus procedure (Kerr et al., 1960; Ozekmekci et al., 2015). Qayyarah crude oil's sulfur level was measured, and the results are presented in the (Table 1-a). The sulfur percentages are depicted in (Table 1-b) after the adsorption procedure using Gorilla glass treated to work with sodium hydroxide. The percentage of sulfur in Qayyarah crude oil was 6.7% before adsorption, and it decreased to 3.8% after adsorption with the treated Gorilla glass. Gorilla Glass that had been pre-treated and burnt at 600°C demonstrated excellent sulfur adsorption capabilities, depicted in (Table 1-c) with the sulfur content in the sample decreasing to 2.2% after adsorption. Gorilla Glass, with its aluminosilicate composition, contains silicon dioxide, aluminum, magnesium, and sodium. Metal oxides have demonstrated significant efficiency in the desulfurization process. Consequently, mixed metal oxides have become increasingly popular for desulfurization as a result of their ability to operate effectively within 300–800°C is a broad temperature range (Garces et al., 2010). The overarching chemical interaction between the sulfur compounds found in the oil and a metal oxide can be described as follows, with 'M' denoting the representative metal: (Charisiou et al., 2019; Gangwal et al., 1988; Montes et al., 2013)

$$M_XO_Y + R_2S \longleftrightarrow M_XS_Y + YR_2O$$

Table 1. a: XRD for sulfur percentage in Qayyarah crude before treatment, b: XRD for sulfur percentage after treatment with reflux Gorilla Glass, c: XRD for sulfur percentage after treatment by furnace

SEQ	(a) before treatment	(b) after treatment with reflux Gorilla Glass	(c) after treatment by furnace
1/3	6.72827%	3.83132%	2.24131%
2/3	6.71794%	3.83132%	2.24331%
3/3	6.72405%	3.83332%	2.24131%
Average	6.72342%	3.83198%	2.24197%
K	1.46339	1.50702	1.42020
STD DEU	0.00520%	0.00400%	0.00704%
K	0.00197	0.00152	0.00267

Measuring the Impact of Temperature and Contact Time on Sulfur Content

The challenge of eliminating sulfur compounds becomes more pronounced as the oil fraction's boiling point increases, with thiophenes representing one of the most difficult sulfur compounds to remove (Shi et al., 2021). With that in mind, Qayyarah crude oil was chosen because of its high sulfur content. The outcomes of the experiments performed in this study are displayed in (Tables 2, and 3 a, b and c). This table illustrates the impact of contact duration on sulfur concentration at a fixed temperature, with measurements taken at intervals of 15, 30, and 60 minutes.

Table 2. Results of measuring sulfur levels at different times using modified Gorilla Glass

Time	15 min	30 min	60 min
Sulphur con%	6.1	5.8	4.5

Table 3. XRD for sulfur levels at different times using modified Gorilla Glass a: at 15 min, b:at 30 min, c:at 60 min.

SEQ	(a) at 15 min	(b) at 30 min	(c) at 60 min
1/3	6.16126%	5.83142%	4.52132%
2/3	6.16226%	5.83242%	4.52185%
3/3	6.16226%	5.83342%	4.52182%
Average	6.16226%	5.83242%	4.52166%
K	1.46339	1.42020	1.42020
STD DEU	0.00520%	0.00704%	0.00704%
K	0.00197	0.00267	0.00267

The correlation between the amount of sulfur and the duration of contact at a constant temperature is depicted in Figure 2.

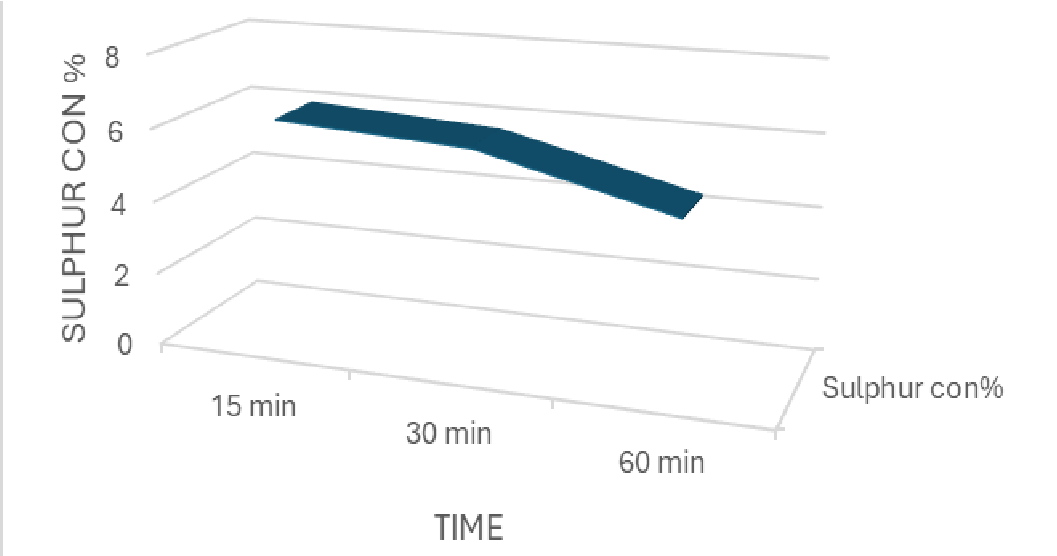

Figure 2. Correlation between the amount of sulfur and the duration of contact at a constant temperature

Sulfur reduction from crude oil is observed to increase significantly when the contact time is extended to 60 minutes, resulting in a 50% improvement in sulfur removal. This outcome represents the highest percentage of sulfur removal achieved at the longest time interval. In contrast, sulfur removal is less efficient at shorter contact times, and decreases from 60 minutes to 30 minutes, and from 30 to 15 minutes. This observation aligns with findings in previous studies and literature, as well as the work of the researcher Fayrouz Al-Bitar, who achieved sulfur content reduction through adsorption on activated carbon using various time intervals and ratios (*Reducing Sulfur and Aromatics percentage in Diesel Oil by the Adsorption on Activated Carbon*, n.d.). The second phase of the experiment aims to examine the effect of temperature on sulfur content while keeping the duration fixed at 60 minutes. Three distinct temperatures were utilized: the initial temperature was set at 30°C, followed by 45°C, and then 60°C. Subsequent to sample collection and measurement, the recorded sulfur percentages are displayed in Tables 4 and 5.

Table 4. Results of measuring sulfur levels at different temperatures using modified Gorilla Glass

Temp	30 °C	45 °C	60 °C
Sulphur con%	5.2	4.4	4.1

Table 5. XRD for sulfur levels at different temperature using modified Gorilla Glass a: at 30 °C, b:at 45 °C, c:at 60 °C.

SEQ	(a) at 30 °C	(b) at 45 °C	(c) at 60 °C
1/3	5.26185%	4.45134%	4.11326%
2/3	5.26181%	4.45135%	4.11326%
3/3	5.26182%	4.45135%	4.11326%
Average	5.26182%	4.45134%	4.11326%
K	1.46339	1.42020	1.42020
STD DEU	0.00520%	0.00704%	0.00704%
K	0.00197	0.00267	0.00267

These observations reveal that the desulfurization process of crude oil becomes more effective with rising temperatures. The optimum sulfur removal rate was achieved at 60°C. However, at lower temperatures, namely 45°C and 30°C, the percentage of adsorbed sulfur was significantly lower, varying in proportion. This is explained by the desulfurization process's slowness at lower temperatures, resulting from catalyzed reactions being strongly influenced by temperature as their efficiency improves at higher temperatures due to the increase in activation energy, and vice versa. (Jalil et al., 2012). The correlation between sulfur concentration and temperature., at a fixed contact time, is depicted in (Figure 3).

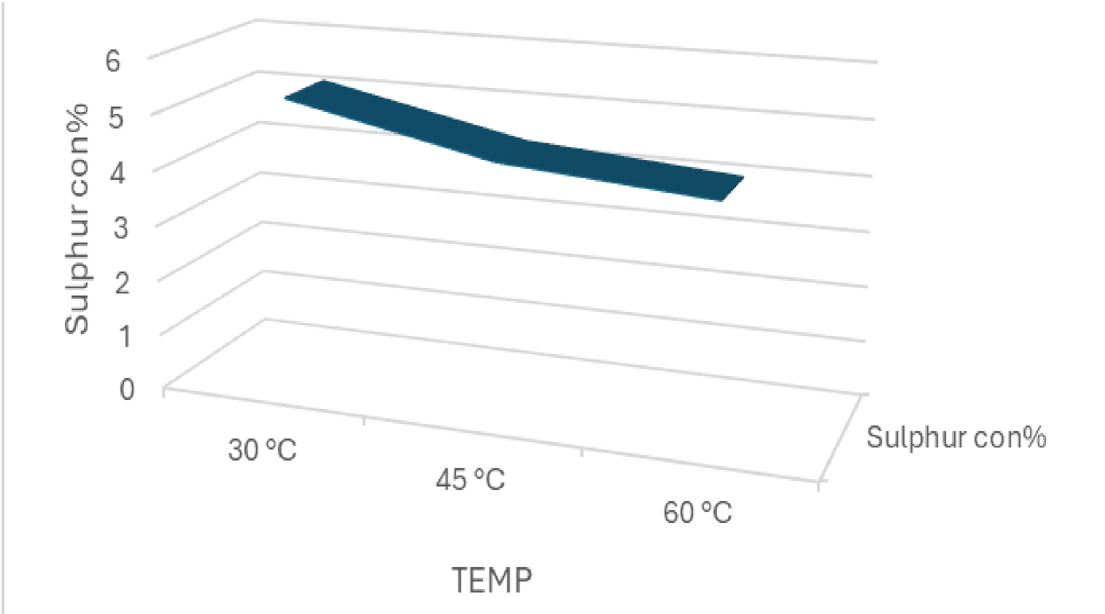

Figure 3. Relationship between sulfur content and temperature with constant contact time

Experiments showed that the interchange of alkali or alkaline earth metal atoms in zeolite with univalent ions was responsible for the adsorption capacity of sulfur compounds. The reduction of sulfur compounds was subsequently made easier by this exchange. It was hypothesized that crude oil's comparatively lower surface potential in comparison to the zeolite framework could be the reason for the reduced adsorption of sulfur compounds. Additionally, it was demonstrated that the dissociation of H_2S, one of the sulfur compounds, into HS^- and H^+ (as illustrated in Figure 4) and physical adsorption played a role in this process (de Oliveira et al., 2019; Karge et al., 1978).

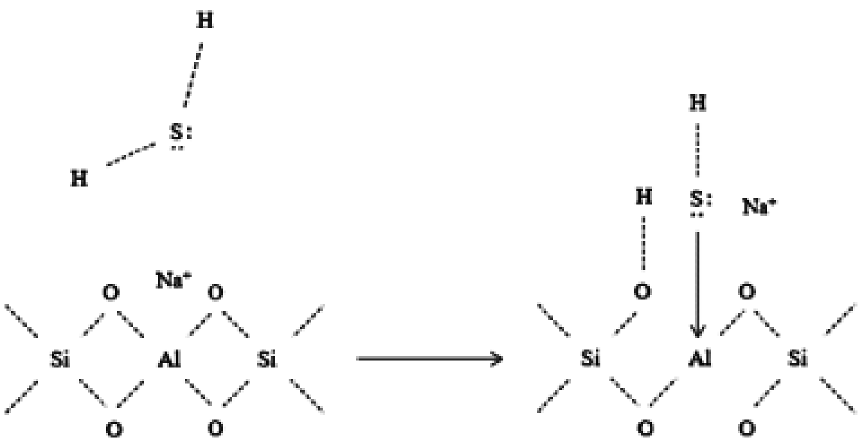

Figure 2. Dissociative H_2S adsorption on an aluminosilicate zeolite

Microstructural Analysis Using SEM-EDS Techniques

Scanning electron microscopy (SEM) in conjunction with energy dispersive spectroscopy (EDS) is the most effective method for determining particle size and identifying the presence of components in the sample (Nasry et al., 2021). Figure 5 displays the findings. The EDS analysis results indicated the presence of silica and alumina oxides in the treated glass. Because they have a big impact on the separation processes, silica and alumina are frequently used as stationary phases in separation columns (Malhotra, 2023). A scanning electron microscope (SEM) is used to examine and record a specimen's surface topography at a much higher resolution than what is possible with light microscopy thus providing valuable structural insight into the surfaces of different specimens. Prior to pretreatment, most glass particles have irregular shapes with sharp edges. According to SEM measurements, pretreated glass has better mechanical qualities than untreated glass, with notable similarities in crystal structures and particle sizes (Venkatarajan et al., 2023).

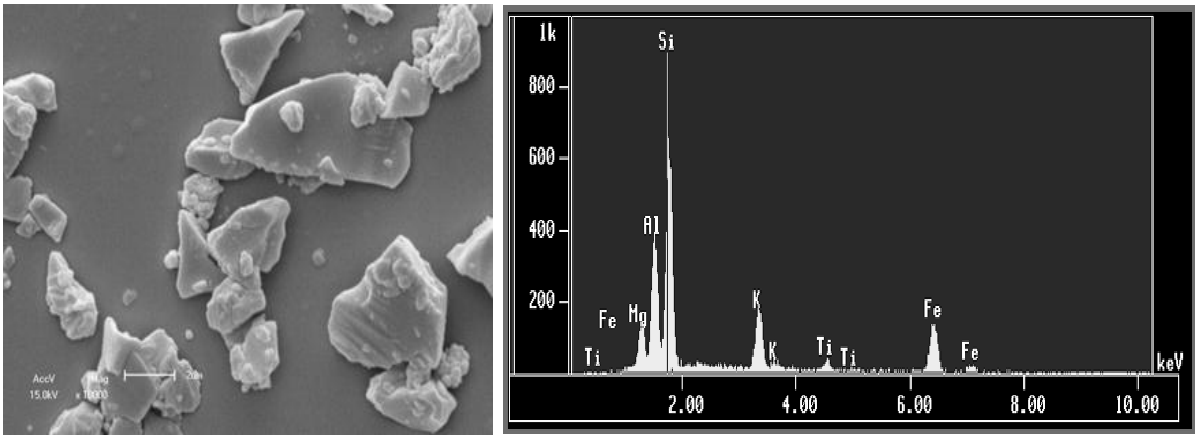

Figure 5. SEM micrographs and EDS spectra of gorilla glass

Conclusion

This study illustrates that recycled Gorilla Glass trash serves as an effective adsorbent for the extraction of sulfur from Iraqi Qayyarah crude oil. The modified glass's sulfur content was considerably decreased by thermal activation, and improvements were seen when the temperature and contact duration were raised. The material's promising use in adsorption desulfurization was highlighted by XRD analysis, which verified the gradual reduction of sulfur from 6.72% to as low as 2.24%. These results suggest that temperature and contact duration are important variables in promoting sulfur adsorption, providing a sustainable and effective way to raise fuel quality and reducing environmental impact.

Recommendations

Researchers recommend for the utilization of eco-friendly, cost-effective, and accessible sources for sulfur removal through innovative approaches.

Scientific Ethics Declaration

* The authors declare that the scientific ethical and legal responsibility of this article published in EPSTEM journal belongs to the authors.

Conflict of Interest

* The authors declare that they have no conflicts of interest

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