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Oxidative Extractive Desulphurisation of Crude Oil Using Ghanaian Alcoholic Beverage (Akpeteshie) as an Extractant for Petroleum Coke Production

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Abstract:
Background*: Sulphur in petroleum coke makes it cheaper or less desirable because of regulations set by nations and environmental problems caused by the sulphur. Manufactures of petroleum coke mostly remove the sulphur from the product after manufacturing using high heat that makes the cost of producing the product having less sulphur more expensive. To reduce the cost of sulphur removal from the petroleum coke, this research employed oxidative extractive desuphurisation method.*
Materials and Methods*: In this research, desulphurization of both spilled and unspilled crude oil were conducted to verify the effectiveness of the method employed. Some crude oil was poured into seawater, cleaned, and then tested for sulphur content using sulphur in crude analyser. About 3530 ppm of sulphur was detected in the crude oil. Then, the crude oil was desulphurised using acetic acid (CH_3COOH) and Hydrogen peroxide (H_2O_2) as oxidising agents, and Ghanaian locally manufactured alcoholic beverage (Akpeteshie) as an extractant before conducting the sulphur test again. After that, the desulphurised crude oil was distilled.*
Results*: The sulphur left in the desulphurised crude oil after the test gave 1680 ppm, which translated to about 52.41 % reduction. Doubling the reagents and further desulphurising the crude oil reduced the sulphur content to 282.4 ppm. This result translated to about 83.19 % overall reduction of sulphur. The test of the raw crude oil (unspilled) before and after desulphurisation initially gave 3000 ppm and 1820 ppm respectively. Doubling the oxidising and extracting reagents using the same quantity of the unspilled crude oil reduced the sulphur content to 335 ppm. This was 81.59 % reduction. After distilling the desulphurised spilled crude oil, the tar (a raw material for petroleum coke production) was analysed using Fourier Transform Infrared spectroscopy and Gas Chromatography-Mass Spectrometry analysis.*
Conclusion: *The results showed that Apketeshie could be used as an extractant in the desulphurisation of crude oil to provide a bituminous tar that could be used to manufacture petroleum coke. It was observed that with the same quantity of crude oil, doubling the oxidising and extracting reagents produced better desulphurisation results. The cost of Akpeteshie is cheaper than ethanol used by most refineries as extractant. Hence, it is recommended that oxidative extractive desulphurisation using CH_3COOH and H_2O_2 as oxidants and Akpeteshie as an extractant should be employed to desulphurise crude oil that provides the raw material used in the production of petroleum coke.*
Key Word*: Petroleum coke, Aluminum, Unspilled crude oil, Steel, Oxidative extractive desulphurisation, Akpeteshie, Spilled crude oil*

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I. Introduction

Petroleum coke (Petcoke) is an essential commodity used in industries such as aluminum, iron, steel, cement, etc. Sulphur content above a certain threshold found in the Petroleum coke makes it undesirable because of regulatory policies and environmental problems posed by the sulphur. Because the sulphur in the fuels is a major problem confronting the entire world, nations have enacted laws and regulations regarding the maximum amount of sulphur content allowable in the fuels utilised in their nations. If sulphur is not removed from the crude oil but it is allowed to enter the atmosphere in the form of SO_2, acid rain may result and cause harm to the environment and humans' lives. Nevertheless, humans' lives must not be left to chances (Asante *et al*., 2017). A high sulphur content in petcoke reduces its market value, and may preclude its use as fuel due to restrictions on sulphur oxides emissions to the environment. Most petcoke manufacturers desulphurise it after it has been produced.Xiao et al., (2016) studied desulphurisation of petcoke using ammonia as a reducing agent. They demonstrated the effectiveness of the NH_3-reducing desulphurisation using a high-sulfur coke calcining desulphurisation experiment at 1000 °C, and the sulphur chemical reactions of the three aromaticities of thiophene. The removal of sulphur using 1000 °C is an enormous amount of energy to expend. After producing petcoke, the

resulting green coke produced from heavy crude oil processing still has a higher sulphur content, which requires a significant increase in the calcination temperature and residence time before the sulphur content can be reduced (Gemmer, 2017). Because of the high temperature needed to desulphurise the petcoke after it has been produced, it is economically beneficial to desulphurise the crude oil before it is used to manufacture the petcoke. The most abundant sulphur compounds found in gasoline are thiophenes (2-methylthiophene, 3-methlythiophene, 2,4-dimethylthiophene, benzothiophene and 2-methylbenzothiophene) (Mohamed *et al*., 2018). Methods have thus been proposed to reduce or eliminate the sulphur content of petcoke. Most of them involve the desorption of the inorganic sulphur present in the pores or surface of the coke, and the partition and removal of the organic sulphur attached to the aromatic carbon skeleton (Javadli and Klerk, 2012;Tanzadeh, *et al*., 2020; Rodríguez-Cabo, *et al*., 2013; Bhutto, *et al*., 2016). This research developed a method to desulphurise spilled crude oil for the production of petcoke using Ghanaian locally manufactured alcoholic beverage as an extractant. The method desulphurises the spilled crude oil before it is poured into the coke unit to produce the petcoke. The Ghanaian locally manufactured alcoholic beverage is cheap and can save manufacturers of petcoke a great amount of money if it is used to desulphurise the crude oil. In desulphurising the spilled crude oil, the amount of sulphur content in a spilled crude oil was determined. Then the oxidative extractive desulphurisation process was performed. After that, the amount of sulphur left in the desulphurised spilled crude oil was determined. Before providing the entire process followed to desulphurise the crude oil, it is deemed appropriate to talk about the types of petcoke and the processes of manufacturing such a commodity. The four basic types of petcoke are needle coke, sponge coke, shot coke, and honeycomb coke. The needle coke has a lower sulphur and impurities levels. It has anisotropic structure or highly layered structure. Its thermal coefficient is less than 2.0×10^{-6} K^{-1}. The green petcoke refers to the raw coke that has been produced from the Delayed Coking unit that has not undergone any further treatment. It contains relatively high level of sulphur and metals. It is a fuel grade used in generating electricity and fuel. According to the article presented by the National Association of Petcoke Manufacturers of the United States of America entitled " Petroleum Coke: Essential to Manufacturing", about 80 percent of worldwide petcoke production is "fuel grade" petcoke that is used to generate electricity and the production of cement kilns (Anon, 2022). Its high heat and low ash content makes it a decent fuel for power generation in coal-fired boilers. Among the problems associated with petroleum coke having high sulphur content and low in volatile content is the environmental (and technical) problems posed with combustion. Edwards (2015) gives account of the green petcoke production process in a Delayed Coker. According to him, delayed coking is a "carbon rejection" process used to upgrade the heavy residuum or residue from the bottom of vacuum distillation towers and other refinery process units. He added that delayed coking significantly increases the recovery of valuable gas and liquid products such as gas, gasoline, gas oils, and so on, because of the higher temperatures (approximately 500°C) and more severe thermal treatment. As a result, some of the larger, higher molecular weight hydrocarbons are cracked into smaller molecules,which are then recovered in a fractionator. Polymerisation and cross-linking reactions lay down a solid residue of carbon referred to as GPC.

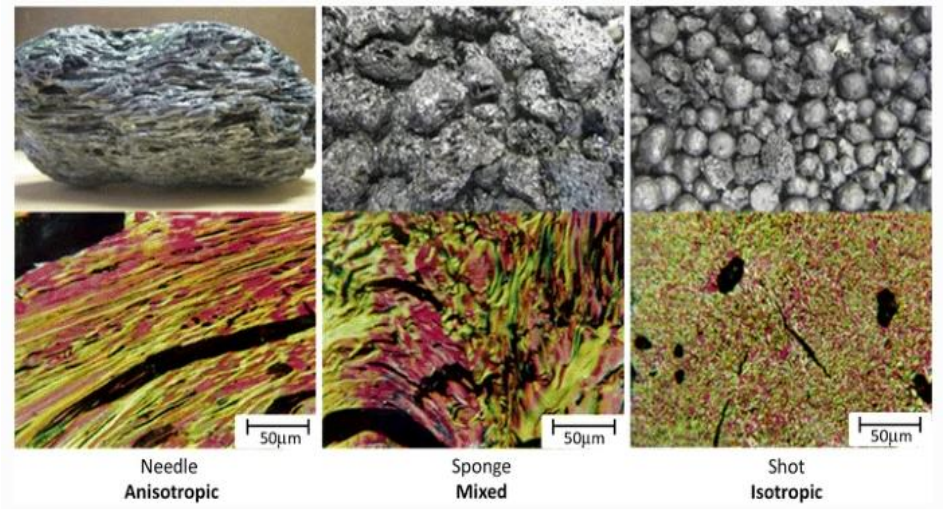


Fig. 1 Delayed Coke type and their optical texture
Source: (Edwards, 2015)

Table I below provides the summary of the types of Delayed Petroleum coke, their coefficient of expansion, their characteristics and their applications. According to the table, it can be said that the honeycomb petcoke has the smallest coefficient of thermal expansion and the shot coke has the largest coefficient of expansion. Other parameters regarding the petcoke can be referred to Table 1.The green petcoke is further treated

to produce a calcined petcoke.The anode grade coke serves as the raw material for the production of calcined coke, which is used to manufacture anodes to produce aluminum or titanium dioxide. On the other hand, steel grade coke is used as a reducing agent in iron and steel metallurgy. It serves the pelletizing and carbide production segments. In addition, it is used to obtain lime and for other industrial applications.The calcined petcoke, on the other hand, contains lower lever of sulphur and metals. It can be used in the manufacturing processes such as the production of aluminum, steel, paint and coloring, fertilizer, Paper, brick, glass, etc. In the production of steel and aluminum, the calcined petcoke is used as an anode coke. Furthermore, it is used as recarburizer in the industries that manufacture steel and iron. The raw materials used in the production of the petcoke include light sweet crude oil, heavy unsweet crude oil, bitumen found in tar sands, etc. According to the article published by Petro Online in 2015, petcoke is a byproduct created when bitumen found in tar sands, like those in Alberta, Canada, is refined into crude oil. In the production of petcoke from heavy crude oil, the product comes out havinga higher sulphur content, which requires a significant increase in the calcination temperature and residence time. Nevertheless, it is challenging to utilize such a high-temperature in the calcination process. It can be emphasised that the production of petcoke from heavy crude oil yields a product fraught with a higher amount of sulfur content. However, in order to attain lower level of sulphur in petcoke using heavy oils, an Advanced Combined Heat and Power (CHP) System was developed to enable the usage of high unsweet crude as a raw material to manufacture lower sulphur level petcoke (Gemmer, 2017).Nonetheless, the method developed using the CHP System has some hurdles to overcome in the process. Unlike the above-mentioned project, this project desulphurises spilled crude oil by innovatively using Akpeteshie as an extractant for the production of low-level sulphur crude for the manufacturing of petcoke. Figures 2A and 2b show the pellets of green petcoke and calcined pekcoke

Table 1 Types of Delayed Petcoke, Properties and Uses

Type of Delayed Petcoke	Coefficient of thermal expansion	Characteristics	Applications
Needle coke	$< 2.0 \times 10^{-6}$/K	Highly layered or anisotropic structure	Production of the graphite electrodes used in steel-producing electric arc furnaces
Sponge coke	$3.5–4.8 \times 10^{-6}$/K	Mixed optical structure and wide range of domain sizes	Anode production
Shot coke	(>5.5)/K	Spherical particle shape and a dense, highly isotropic texture	
Honeycomb	Less than needle coke	Intermediate coke with ellipsoidal pores uniformly distributed	Used in the cement industry as a source of energy

Figure 2a Green Petroleum Coke
Source: (Andrade *et al.*, 2022)

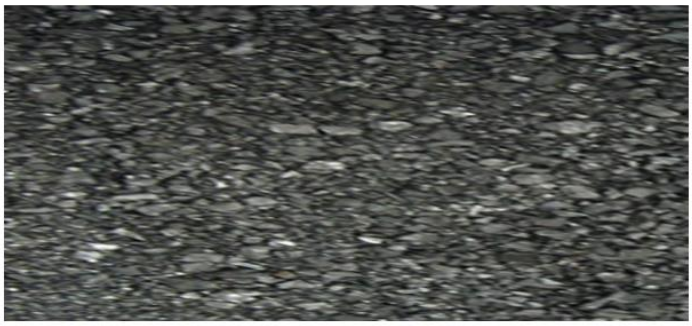


Figure 2b Calcined Petroleum Coke
Source: (Ortega, 2022)

The methods used in desulphurisation include hydrodesulphurisation, extractive desulphurisation, alkylation desulphurisation, desulphurisation using critical water, biodesulphurisation, oxidative desulphurisation, etc. After producing green petcoke containing relatively high amount of sulphur, metals, etc.,there is the need to reduce the sulphur content to enable the product to be used for the production ofnodes and other products. The methods used to reduce the sulphur content in the petcoke is calcining (Vauramo, 2021;Gemmer, 2017; Carlson, 2012; Edwards, 2015).The calcining process in Figure 3 includes preheating the reactants. Proper calcining assists the petcoke to obtain the deserved quality. In the calcining process, temperature and time are important variables needed to take into consideration. Calcining is done in an environment having almost no oxygen. The steps used in the petroleum coke calcining process are drying, devolatisation and densification. The document provided in 2022 by Metso Outotec, one of the leading manufacturers of calcining equipment, presents that obtaining the calcined coke properties required by the carbon and graphite industries, the coke must be subjected to temperatures of 1150-1350 ºC or higher to achieve density and conductivity. The document added that the final quality of the calcined coke is directly related to the specific characteristics and quality of the green coke fed to the calciner; also, the important control variables are heating rate, air addition rate and final calcination temperature. While calcination cannot improve upon certain quality limits inherent in the green coke, potential quality can be lost by improper calcining. The simulation process for calcination is shown in Fig. 4. Zang and Wang (2019) conducted simulation of combustion and thermal flow of the calcination in order to gain insight of how the energy in the calcinator can be utilised efficiently for sustainability and profit making

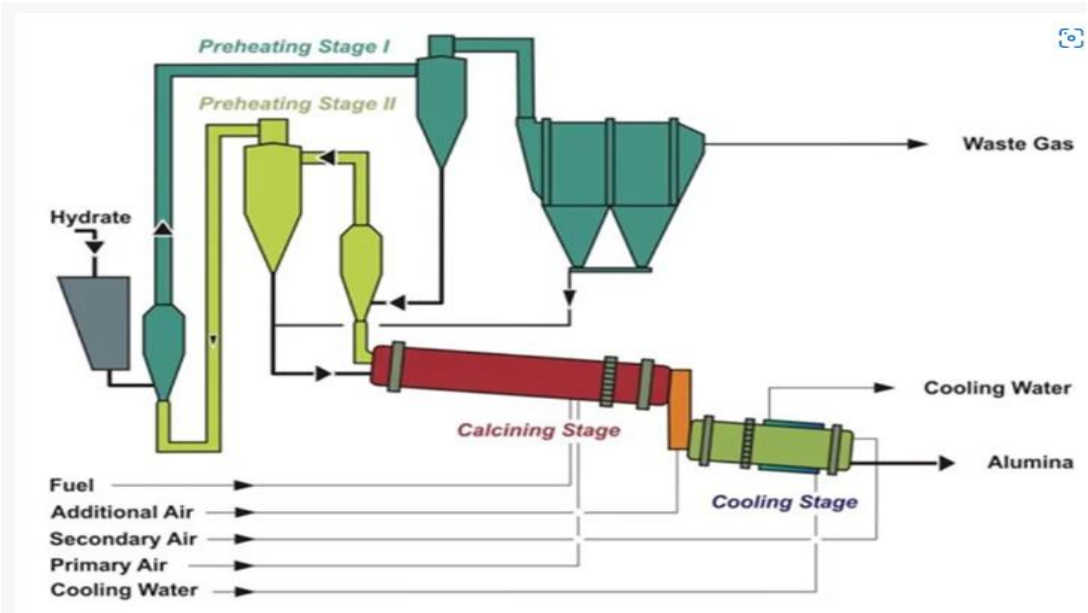


Fig. 3 Schematic Diagram of a Rotary kiln Calciner with Preheating Stage
Source: (Anon, 2022)

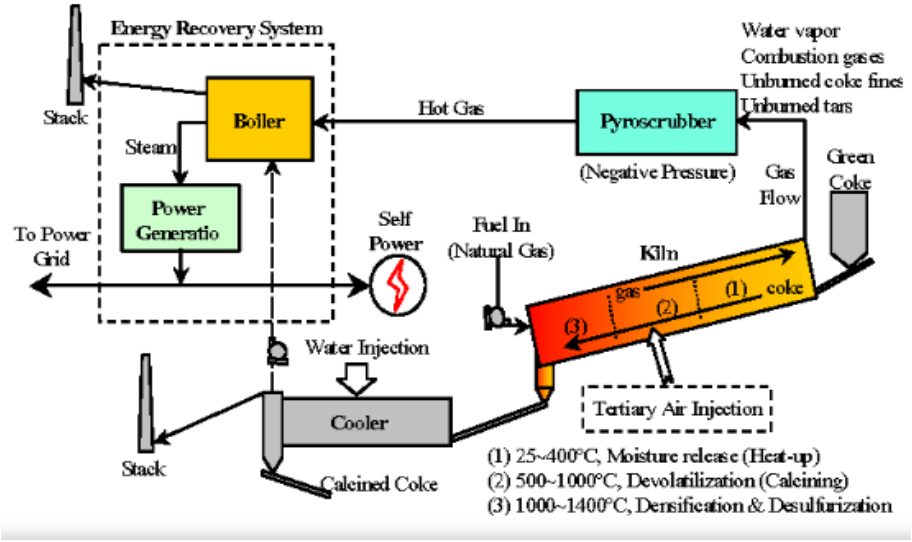


Fig. 4 Simulation Process for Calcination of Green Petcoke
Source: (Zang and Wang, 2019)

Figure 5 shows sulphur-containing compounds found in the crude oil that need to be removed.

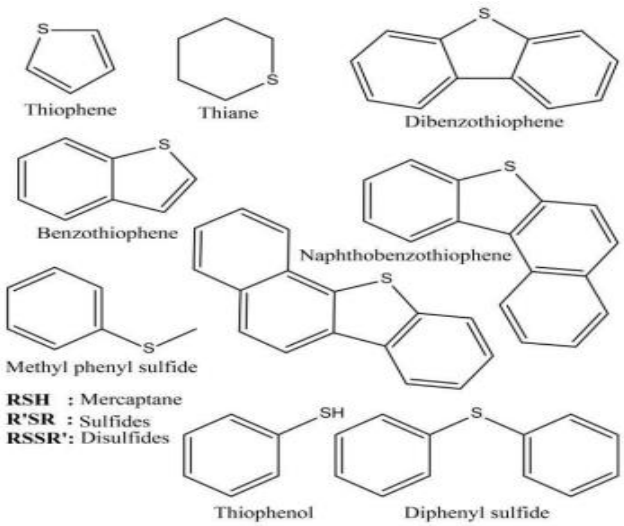


Fig. 5 Chemical Structures of Sulphur Containing Compounds

II. Material And Methods

The oxidative extractive desulphurisation method was used in this research. The crude oil used in this research was obtained from the Tema Oil Refinery (TOR). The crude oil was medium dark sour compound having a density of 900.8 kg/m^3. The American Petroleum Institute (API) index of the crude oil was 25.47 ^{o}.

API = 141.4/ (Specific gravity) -131.5 (1)

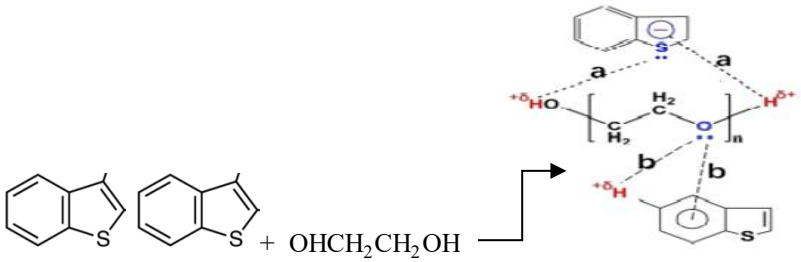
The mix ratio of the initial mixture was 1000 mL (crude oil): 100 mL (CH_3COOH):100 mL (H_2O_2): 100 mL (Akpeteshie).
The mix ratio of the second mixture was 1000 mL (crude oil): 200 mL (CH_3COOH):200 mL (H_2O_2): 200 mL (Akpeteshie).

Procedure methodology

The crude oil was poured into a seawater and stirred for about 2 minutes. The mixture was allowed to stand for about 2 hours. The crude oil was cleaned from the seawater and tested for the sulphur content. Then the crude was desulphurised. The method used in desulphurising the spilled crude oil employed two steps, namely, the oxidation of the sulphur containing compounds, and the extraction of the oxidised sulphur compounds. In the oxidation method, 100 mL of CH_3COOH was added to 1000mL of crude oil in a bottle. The mixture was shaken vigorously for about 4 minutes. Then 100 mL of H_2O_2 was added to the mixture of the crude oil and the acetic acid. Again, the mixture of the crude oil/ acetic acid/ hydrogen peroxide was shaken for about 4 minutes. Then the mixture was allowed to sit for about 30 minutes. This step completed the oxidation step. The oxidation step was followed by the extraction step. The Ghanaian locally manufactured alcoholic beverage (Akpeteshie), was used as an extractant to remove the oxidised sulphur containing compounds from the crude oil. One hundred milliliters of the Akpeteshie was added to the mixture. The mixture was shaken vigorously for about 2 minutes. Then the mixture was allowed to sit for about 2 hours. Sample from the mixture was taken and tested for the sulphur content. Equations 2 and 3 below present the chemical mechanisms of the oxidation of the sulfur containing compounds in the spilled crude oil and the extraction of the oxidised sulphur containing compounds respectively. The desulphurisation process was performed again using augmented quantities of the oxidising and extracting agents. Two hundred mL of acetic acid and 200 mL of hydrogen peroxide were added to 1000 mL of crude oil. Then, the procedure detailed above was followed to oxidise the sulphur containing compounds. In the extracting step, two hundred milliliters of Akpeteshie was used to extract the oxidised sulphur containing compounds. The entire processes of desulphurisation were repeated using raw crude oil (unspilled). The unspilled refers to the crude oil that was not poured into the seawater. Equation 2 shows the mechanism of the oxidation step using hydrogen peroxide and acetic acid In the extraction step, the Akpetshie containing predominantly ethanol is dehydrated in the presence of the acetic acid to form ethylene (Tagba, *et al*, 2017; Efeovbokhan *et al*., 2017, Idoneje *et al*., 2012; Adakporia, 2021). Then the ethylene is oxidised in the presence of the oxidising agent (hydrogen peroxide) to form the ethylene glycol. Using benzothiophene as an example, the partial positive end of one of the hydroxide groups in the ethylene glycol interacts with the negative molecules in one benzothiophene. The negative end of the hydroxide group in the ethylene glycol bonds with the positive ends of another benzothiophenes. The complex then enters into the aqueous phase with velocity in accordance with the Stokes equation. Then the crude oil and the water are separated. The desulphurisation process was repeated. Equation 4 shows the reaction of the benzothiophene and ethylene glycol.

$$RR'S + HO\text{-}OH + CH_3COOH \rightarrow \underset{CH_3COO\text{-}H}{R'RS\ H\text{-}O\text{-}OH} \rightarrow \underset{CH_3COOH}{R'RS\ldots OH} + H_2O \rightarrow RR'S\text{–}O + H_2H + CH_3COOH \quad (2)$$

$$CH_3CH_2OH + CH_3COOH \rightarrow C_2H_4 + H_2O_2 \rightarrow OHCH_2CH_2OH \quad (3)$$



(4)

Figure 6 shows the Sulphur in Crude Analyser used in testing the sulphur contents in the crude oils.

III. Result

After conducting two desulphurization processes, the spilled and the unspilled crude oils were tested for sulphur contents and subsequently distilled. The results are presented in the Tables 4 and 5 below. The FTIR spectrum of the desulphurised bituminous tar is also presented below.

Table 4 Tests Results of Sulphur Contents in the Crude Oils Before Desulphurisation

Type of crude oil	Sulphur content (wt. %)	Sulphur content (ppm)
Spilled	0.353	3530
Unspilled	0.3	3000

Table 5 Tests Results of Sulphur Content in Desulphurised Crude Oils

Type of crude oil	Sulphur content (wt. %)	Sulphur content (ppm)
Spilled	0.168	1680
Unspilled	0.182	1820

Table 6 shows the extent of sulphur removal from the spilled and the unspilled crude oils after the second desulphurisation process.

Table 6 Tests Results of Sulphur Contents in Desulphurised Crude Oils

Type of crude oil	Sulphur content (wt. %)	Sulphur content (ppm)
Spilled	0.02824	282.4
Unspilled	0.0335	335

Table 7 shows the extend of sulphur reduction in both the spilled and the unspilled crude oils. The values have been presented in percentages. Figure 9 presents the FTIR spectrum of the bituminous tar obtained from the distillation.

Table 7 Percent Reduction of Sulphur in Spilled and Unspilled Crude Oils after Desulphurisation

Type of Crude Oil	Initial Sulphur Reduction (%)	Overall Sulphur Reduction (%)
Spilled	52.41	83.19
Unspilled	39.33	81.59

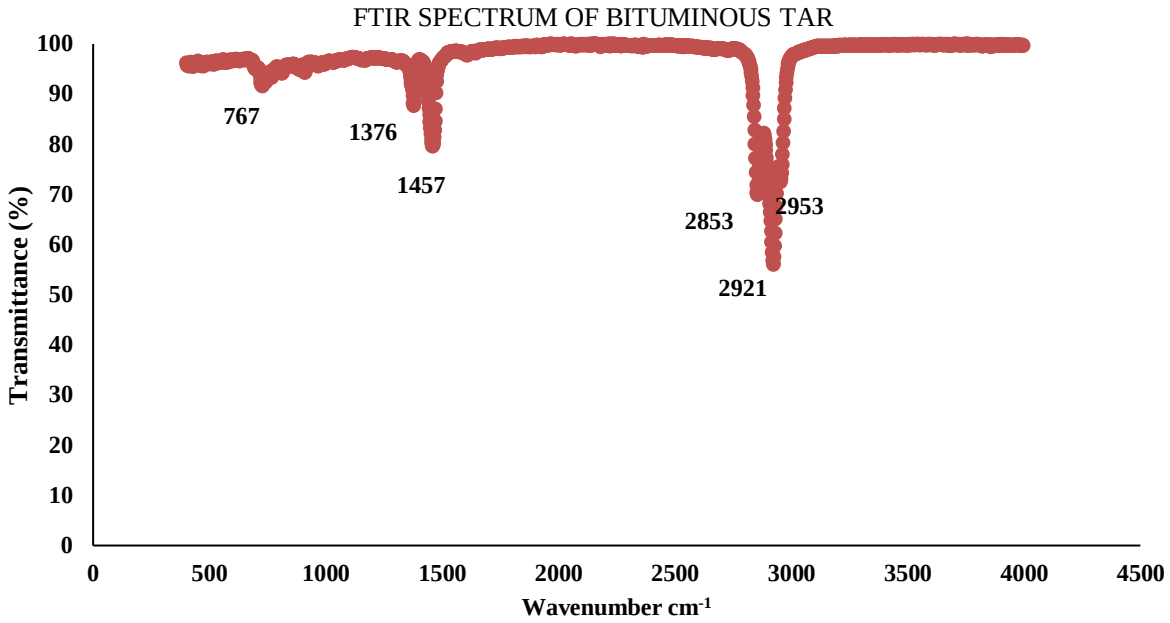


Fig. 7 Wavenumbers and their Corresponding Percentage Transmittance of Bituminous Tar

Table 8 presents the functional groups detected from the FTIR spectrum obtained from the bituminous tar. Table 9 shows the compounds detected in the bituminous tar as a result of the GC-MS analysis**.**

Table 8: Functional Groups Observed from the FTIR Spectrum of the Bituminous Tar

Wavenumber (cm^{-1})	Transmittance (%)	Bond	Functional Group
3996.06	99	C–H stretch	Aromatic
3351.46	99	O–H stretch, H–bonded	Alcohol, phenol
3000.61	97	C–H stretch	Alkanes
2953.20	72	C–H stretch	Alkanes
2921.54	55	C–H stretch	Alkanes
2853.18	69	C–H stretch	Alkanes
2420.30	99	–C≡C– stretch	Alkynes
2277.76	99	–C≡C– stretch	Alkynes
2197.51	99	–C≡C– stretch	Alkynes
2178.12	99	–C≡C– stretch	Alkynes
2131.65	99	C≡C stretch	Terminal Alkynes
2080.70	99	–C≡C– stretch	Alkynes
2072.96	99	–C≡C– stretch	Alkynes
2007.13	99	–C≡C– stretch	Alkynes
1987.43	99	Overtones	Aromatics
1606.95	97	N–H bend	1˚ amines
1457.46	79	C–H bend	Alkane
1376.76	87	-CH(CH3)2 -(CH3)3 bend	Alkanes and alkyls
965.51	95	*trans*-RCH=CHR	Alkenes
909.09	94	=C-H bend	Alkenes
887.64	94	=C-H bend	Alkenes
810.00	94	C-Cl stretch	Alkyl halide
767.14	93	C-Cl stretch	Alkyl halides
741.06	92	C–Cl stretch	Alkyl halide
725.30	91	C–H rock	Alkanes
610.55	96	C≡C –H: C–H bend	Alkynes
593.92	96	C–Br stretch	Alkyl halides
473.28	95	C–H	Alkanes

Table 9 Compounds Found in the Bituminous Tar Sample

#	Component
1	p-Xylene
2	Nonane
3	1-ethyl-2-methylbenzene
4	1,2,4-trimethylbenzene

5	1-ethyl-3-methylbenzene
6	Decane
7	1,2,3-trimethylbenzene
8	5-methyl-2-(1-methylethyl)-, benzoate, [1R-(1a,2a,5a)]cyclohexanol
9	Undecane
10	1-ethyl-3,5-dimethylbenzene
11	Naphthalene
12	1-Decene
13	Nonylcyclopropane
14	Dodecane
15	2,6-dimethylundecane
16	1,2,3,4-tetradydro-5-methylhnaphthalene
17	7-methyltridecane
18	1-methylnaphthalene
19	Tridecene
20	1H-Indene, 1-ethylidene
21	z-(13,14-Epoxy)tetradec-11-en-1-ol acetate
22	2,6,10-trimethyldodecane
23	Trichloroacetic acid, hexadecyl ester
24	Tetradecane
25	2,7-dimethylnaphthalene
26	1,7-dimethylnaphthalene
27	Octylcyclohexane
28	2,6,10,14-tetramethylheptadecane
29	1-pentadecene
30	Pentadecane
31	3-(2-methyl-propenyl)-1H-indene
32	Trichloroacetic acid, hexadecyl ester
33	Hexadecane
34	2,6,10-trimethylpentadecane
35	Cyclohexadecane
36	Heptadecane
37	2,6,10,14-tetramethylpentadecane
38	Octadecane
39	2,6,10,14-tetramethylhexadecane
40	2-Ethyl-1-dodecanol
41	Octadecane
42	Eicosane
43	Heneicosane
44	Heptacosane
45	2-cyclohexyleicosane
46	Decosane
47	3-ethyl-5-(2-ethylbutyl)octadecane
48	3-ethyl-5-(2-ethylbutyl)Octadecane
49	Tetracosane
50	Tetratetracontane
51	9-(2,2-dimethylpropanoilhydrazono)-3,5-dichloro-2,7-bis-[2-(diethylamino)-ethoxy]fluorine
52	17-Pentatriacontene
53	Hexacosane
54	Heptacosane

IV. Discussion

The oxidative extractive desulphurisation of the spilled crude oil using hydrogen peroxide (H_2O_2) and acetic acid (CH_3COOH) as oxidants, and using Ghanaian locally manufactured alcoholic beverage as an extractant gave promising results. Before commencing the desulphurisation, the samples of the unspilled and the spilled crude oils were tested for their sulphur contents. About 3530 ppm and 3000 ppm of sulphur were found in the spilled crude oil and the raw crude oil respectively. The test of sulphur content after desulphurisation, which involved adding 100 mL of CH_3COOH and 100 mL of H_2O_2 to the spilled and the unspilled crude oils for the oxidation step, and then adding 100 mL of Akpeteshie to the crude oils for the extraction gave 1680 ppm and 1820 ppm respectively. This translated to about 52.41 % and 39.33 % reduction of sulphur contents in the crude oils correspondingly. Other tests were made after adding 200 mL of acetic acid and 200 mL of hydrogen peroxide to the crude oilsand then adding 200 mL of Akpeteshie to extract the oxidised sulphur containing compounds. The tests of the mixtures showed that the spilled crude oil had reduced to 282 ppm, which translated to about 83.19 % overall reduction, and the sulphur in the unspilled crude oil also had reduced to 335 ppm which translated to 81.59 % overall reduction. The tests showed that with the same amount of crude oil, adding twice the oxidising andreducing reagents provided better desulphurisation results. Tables 5 and 6 provide the summary of the results.The residue obtained from distilling the desulphurised crude oils was analysed using FTIR spectroscopy

and GC-MS. The constituents of bitumen are asphaltenes, maltenes, aromatics, saturates, resins (Natalie, 2019; Holy, 2019; Nair, 1978; Whiteoak, 1999; Zhang *et al*., 2019).The information observed from the GC-MS and the FTIR spectroscopic analysis in comparison with the information about bitumen presented by the researchers mentioned above aided in the identification of the sample as bituminous tar.

V. Conclusion

In conclusion, the desulphurisation pathway employed to remove sulphur from the crude oil was successful. The distilled desulphurised crude oil provided bituminous tar that could be used to manufacture petroleum coke. Moreover, it was observed that with the same quantity of spilled or unspilled crude oil, doubling the oxidizing and reducing reagents achieves better desulphurisation results. The future of using acetic acid and hydrogen peroxide as an oxidant, and using Ghanaian locally manufactured alcoholic beverage, as an extractant looks more promising. Hence, it is recommended that refinery companies should look into using Akpeteshie as an extractant in the desulphurisation process because of its great financial economic benefit to petroleum coke manufacturers.

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