applied sciences

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Study on Sulfide Distribution in the Operating Oil of Power Transformers and Its Effect on the Oil Quality

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Received: 13 August 2018; Accepted: 4 September 2018; Published: 7 September 2018

Abstract: Corrosive sulfides in transformer oil could react with copper wire to produce cuprous sulfide, causing insulation failure. At present, both the quantitative measurement method and distribution of sulfur components in operating oil are not clear yet. In this paper, the existing types and contents of sulfides in oil samples with different alkyl groups and different voltage levels were investigated. With quantitative testing methods, the distribution of sulfur composition in the operating oil was analyzed. Results showed that the thiophene sulfide in transformer oil existed mainly in the form of benzothiophene with an unsaturation of 6 and dibenzothiophene with an unsaturation of 9. The content of monosulfide sulfide with unsaturation of 3 or 6 was the highest. The disulfide existed basically in the form of Dibenzyl disulfide (DBDS). The influence of sulfides on the oil quality were analyzed on this basis. Results showed that the existence of sulfides would increase the moisture content in oil. The absorbed moisture could cause the decrease of the breakdown voltage and rise of the dielectric loss. The above study could provide some engineering practice for understanding the sulphide distribution in transformer oils and further prevent the sulfur corrosion faults.

Keywords: transformer oil; sulphide distribution; oil quality

1. Introduction

Currently, transformer oil is mostly mineral oil refined from petroleum. In addition to carbon and hydrogen, sulfur is the third most abundant element, ranging from a few parts per million to as much as five percent. The active sulfides are easy to react with metal parts of equipment to produce cuprous sulfide [1,2], causing partial discharges in electrical equipment and eventually serious insulation accidents. According to current studies by scholars, the source of corrosive sulfurs in insulating oil exist mainly four forms: (1) Corrosive sulfur contained in crude oil, (2) sulfur contained in electrical equipment, (3) corrosive sulfur compounds generated by crude oil pyrolysis reaction, and (4) corrosive sulfur compounds produced by hydrogenation reactions [3,4]. In the past few decades, the sulfide deposition phenomenon was discovered successively in transformer fault inspections of 220 kV and above voltage classes in areas within China Southern Power Grid, East China, and North China [5,6]. Studies and reports on corrosive sulfur faults were also conducted in other countries such as Japan, Europe and other countries [7,8]. The insulation fault caused by sulfur corrosion has become a major threat to the safe operation of oil-immersed power transformers.

As the sulphide damage in transformer oil was not exposed until recently, there are relatively few studies on this problem. Previously, the corrosion of sulfides in crude oil on petroleum refining equipment and the corresponding detection methods have been studied extensively. With the outbreak of corrosive sulfur problems, the detection of total sulfur content in transformer oil has been taken as an indicator by the power grid company [9–12]. However, the types and contents of sulfides in different transformer oils are different, so there is no direct relationship between the total sulfur content and corrosiveness. For the detection of corrosive sulfur in transformer oil, the methods prescribed in different standards are different [13–15]. In the quantitative analysis of corrosive sulfur in insulating oil, most scholars believe that Dibenzyl disulfide (DBDS) is the main corrosive sulfur in transformer insulating oil [16,17]. Therefore, various methods for detecting the DBDS content in oils have been proposed by scholars [18,19]. Solid phase micro extraction (SPE) and gas chromatography-mass analysis (GC-MS) have been combined with these methods, increasing the measurement accuracy to 0.1 ppm [20]. The corrosiveness of a variety sulfides that may exist in transformer oil were examined by Kamishima Shota [21], and the sulfur compounds such as mercaptans, bisulphid, and thioether were all found to have a certain degree of corrosion. Moreover, one sulfide may convert to another under certain conditions, so the study on sulfur corrosion requires further analysis of other sulfide existing in the insulating oil.

In this paper, the sulfur distribution in the transformer oil and its effect on the oil quality were studied. By investigating the types, contents, and sources of sulfides in the transformer oil samples with different alkyl and voltage levels, the quantitative test methods for various sulfide in transformer oil were established. The distribution of sulfur in oil was detected by these methods, and the influence of sulfide on oil quality was analyzed.

2. Quantitative Test Methods for Different Sulphides in Transformer Oil

The insulation oil used in oil-immersed power transformers is extracted from natural oil and by distillation, refining, blending, and other procedures. Therefore, the components of insulation oil include not only hydrocarbons such as alkane, cycloalkane, and small amounts of aromatic hydrocarbons, but also trace elements such as sulfur, nitrogen, and oxygen in crude oil. Their contents are closely related to the origin of crude oil. According to the incomplete statistics of corrosive oil samples given by the Fujian Electric Power Research Institute (as shown in Table 1), the naphthenic oil samples account for most of the composition. The oil samples were detected according to the test method for detection of potentially corrosive sulphur in used and unused insulating oil (IEC 62535-2008). Results showed that the corrosion of Nynas oil samples is more serious, while the corrosion degree of oil samples has no direct relationship with the composition and the voltage class of the insulation oil.

Table 1. Investigation statistics of corrosive oil samples in Fujian power grid.

Company Name	Transformer Name	Corrosive Degree *	Voltage Level	Delivery Time	Oil Brand	Oil Manufacturer	Oil Group
Quan Zhou	Tuzhai No.1	3b~4a	220 kV	2 December 2004	Nynas 10 GBX	Nynas	Cycloalkyl
	Luotang No.2	4a~4b	220 kV	3 December 2003	Nynas 10 GBX	Nynas	Cycloalkyl
Nan An	Humei No.3	2e	110 kV	14 December 2008	DB-25#	Karamay	Cycloalkyl
Pu Tian	Hualin No.2	2b	110 kV	18 December 2006	DB-25#	Karamay	Cycloalkyl
Xia Men	Wuli No.2	3b~4a	220 kV	18 September 2001	DB-25#	Karamay	Cycloalkyl
Fu An	Bingongchang No.1	1a~1b	Non-operation after detection as corrosive and the oil was returned oil				Paraffin base
	Bingongchang No.2	1a~1b					Paraffin base
Overhaul	Quanzhou No.1	2b	500 kV	29 March 1998	DB-25#	JOMO	Paraffin base
	Dayuan No.2	2b	500 kV	4 January 2009	DB-25#	Karamay	Cycloalkyl
	Quanzhou Gaokang	3a~3b	500 kV	29 March 1998	DB-25#	Karamay	Intermediate base
	Xiamen No.1	2b	500 kV	22 March 1999	DB-25#	Kansai	Cycloalkyl
	Haicang No.1	3a	500 kV	7 September 2007	10#	Nynas	Cycloalkyl

* The corrosive degree are categorized by the standard ASTM D130 (Standard test method for corrosiveness to copper from petroleum products by copper strip test).

The Shanghai Electric Power Research Institute once studied the relationship between the total sulfur content and the active corrosive sulfur in transformer oil. Three typical transformer oils were selected for experiments at four temperature levels. The results showed that the main form of active sulfur in transformer oil is bisulphide and mercaptan sulfur, and the content of elemental sulfur is very low. The content of mercaptan sulfur is lower than the minimum testing standard at low temperatures, while it can be detected when the temperature is higher. It indicates that one sulphide could be converted to another at high temperatures, and some non-corrosive sulfide may be converted to active corrosive sulfide. The sulfur element in the transformer oil mainly exists in the form of mercaptan, bisulphide, thioether, and thiophene, and its content is closely related to the producing area of crude oil. In order to further understand the distribution and content of different sulfur components in oil samples, a quantitative method for the determination of different sulfur compounds in transformer oil was proposed. The test content is total sulfur content, elemental sulfur content, hydrogen sulfide content, mercaptan sulfur content, DBDS content, thiophene sulfur content, and thioether sulfur content. The above operations referring to the following standards and equipment is shown in Table 2.

Table 2. Reference standards and equipment for the quantitative determination of different sulfur compounds in transformer oil.

Test Items	Reference Standard	Equipments
Determination of total sulfur	ASTM D5453-12 Standard Test Method for Determination of Total Sulfur in Light Hy (detection limit is 5 mg/L)	Jena-MultiEA3100 Sulfur nitrogen analyzer
Determination of elemental sulfur	Laboratory method Content of elemental sulfur in the sample should be determined by Oscillographic polarography (detection limit is 0.1 mg/L)	JP-2C Oscillographic polarograph
Determination of hydrogen sulfide	GB/T 26983-2011 determination of sulfureted hydrogen, methyl mercaptan and ethyl mercaptan in crude oil (detection limit is 2 mg/L)	Agilent-6890N gas chromatograph Gas chromatography PFPD detector
Determination of mercaptan sulfur	GB 1792-1988 Determination of mercaptan sulphur (Potentio metric titration method)(detection limit is 3 mg/L)	Kyoto Electronics AT-710M Automatic potentiometric itrant
Determination of DBDS	IEC 62697-1-2012 Test methods for quantitative determination of corrosive sulfur compounds in unused and used insulating liquids-Part 1: Test method for quantitative determination of dibenzyl disulphide (DBDS)	Gas chromatography-mass spectrometry(GC-MS)
Determination of thiophene sulfur and thioether sulfur	Laboratory method The distribution characteristics of thiophene sulfur and thioether sulfur should be analyzed in depth	FT-ICR MS (Bruker Apex Ultra 9.4T AS) Agilent 355SCD Sulfur Chemiluminescence Detector Agilent7890A-5975C Gas chromatography-mass spectrometry

In the above operations, as the determination of thiophene and thioether sulfur is the most difficult, the whole can be divided into the following three steps.

(a) Methylation

The thiophene and thiophene compounds in crude oil are methylated to form the corresponding sulfur salts.

(b) Reduction of thiophene

The thiophene compounds in the methyl thiosulfate/methylated sulfur salt are reduced by 7-azindole, and the amount of thiophene compounds is obtained after separation.

(c) Reduction of thioethers

The 4-dimethylaminopyridine is added to the sample which has been separated from thiophene. The thioether compounds are reduced and the amount can be obtained after separation.

The specific steps are shown in Figure 1.

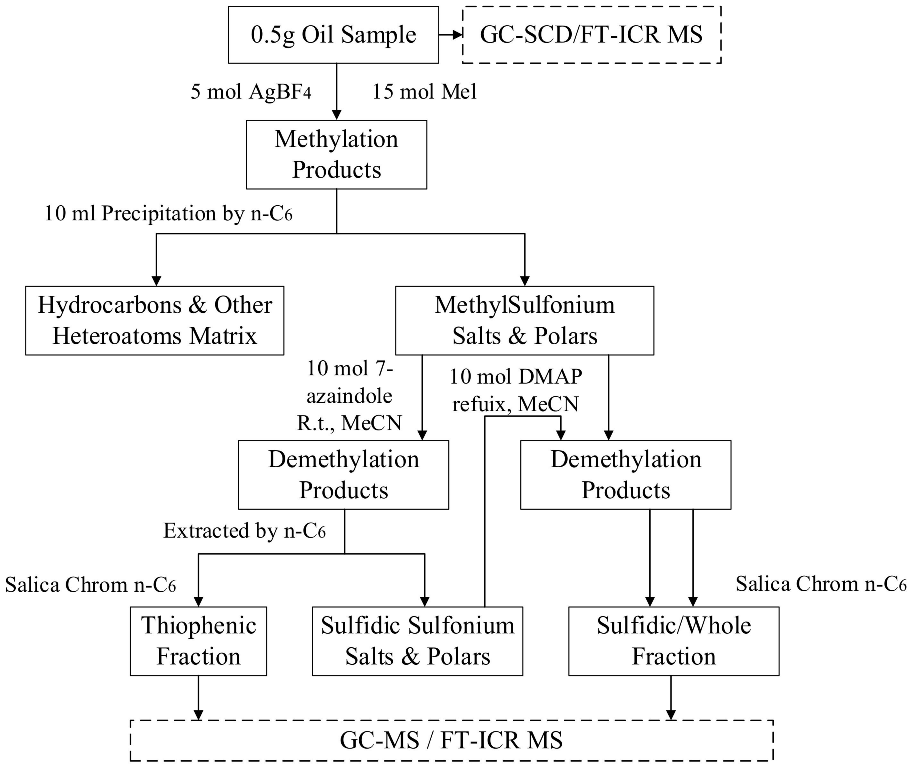


Figure 1. The experimental schematic diagram.

In order to further analyze the distribution characteristics of sulfur in thiophene and thioether, a combination of GC-SCD (gas chromatography-sulfur chemiluminescence detection), GC-MS, and FT-ICR MS (Fourier transform ion cyclotron resonance mass spectrometry) was adopted. The distribution spectra of sulfur in thiophene and thioether, with different carbon number and different unsaturation, can be generated by FT-ICR MS, respectively. The molecular formula of sulfur compounds with higher content in oil samples could then be obtained. Then, the molecular structure could be speculated on. Gas chromatography has an effective separation and resolution capability for organic compounds, and mass spectrometry is an effective means to accurately identify compounds. The combination of the two forms the technology of chromatographic mass spectrometry. The sulfide with low molecular weight could not be detected by the FT-ICR MS and only the sulfide with more than 10 carbon atoms could be identified. Only substances with boiling points below 400 °C could be detected by GC-SCD and GC-MS, but their resolution is far lower than the FT-ICR MS. In addition, isomers with the same molecular weight could not be distinguished in the detection process of FT-ICR MS, while different peaks of isomers could be shown in GC-SCD and GC-MS. Structural formula information could be matched and compared in the database of GC-SCD and GC-MS to obtain the accurate one. Therefore, more information about sulfur in thiophene and thioether could be obtained by the combination of FT-ICR MS, GC-SCD and GC-MS.

3. Test Results of Different Sulfur Components in Transformer Oil

In order to explore the distribution of sulfur in the transformer oil, the above method was used to test five operating oil samples. The test results were shown in Table 3. The five operating oil samples were taken from phase B of Quanzhou No. 1 500 kV Transformer (QZT, Quanzhou, Fujian, China), Wulv No. 2 Main Transformer (WLT, Xiamen, Fujian, China), phase A of Haicang No. 1 500 kV Transformer (HCT, Xiamen, Fujian, China), and Hualin No. 2 Main Transformer (HLT, Putian, Fujian, China).

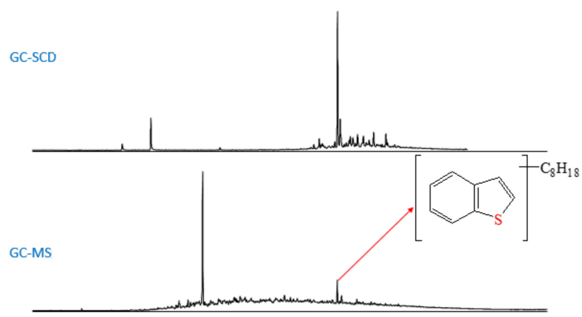
Table 3. Test results of sulfur composition in operating oil.

Sulfur Type (mg/L)	QZT	WLT	HCT	HLT
Total sulfur content	1562	190	203	457
Hydrogen sulphide	0	0	0	0
Elemental sulfur	<0.1	<0.1	<0.1	<0.1
Mercaptan sulfur	<3	<3	<3	<3
Thiophene sulfur	1380	150	42	65
Thioether sulfur	86	30	159	376
Others	52	0	2	16
Sulfur balance	97.18%	100%	99.99%	99.96%

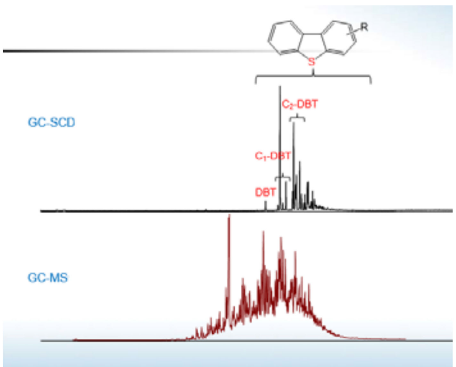
It can be seen that the total sulfur content of the operating oil samples range from 190 mg/L to 1562 mg/L. The hydrogen sulphide gas may escape out during storage, so its content cannot be detected. The content of elemental sulfur in transformer oil is lower than the detection limit. The thiophene sulfur and thioether sulfur are the main components of sulfide in the transformer oil. Their specific content is related to where the sample was produced. The thiophene sulphur accounts for the majority in samples from QZT and WLT, while the thioether sulfur dominates in samples from HCT and HLT.

3.1. Thiophene Sulfur

The GC-SCD and GC-MS spectra of thiophene sulfur in WLT, HCT, QZT, and HLT are shown in Figure 2. The GC-SCD spectra of thiophene sulfur have better separation effect. Several of the neighboring peaks are classified together. They are isomers of the same molecular formula. Results show that the thiophene compounds in transformer oil exist mainly in the form of benzothiophene, dibenzothiophene, and its homologues.



(a)



(b)

Figure 2. *Cont.*

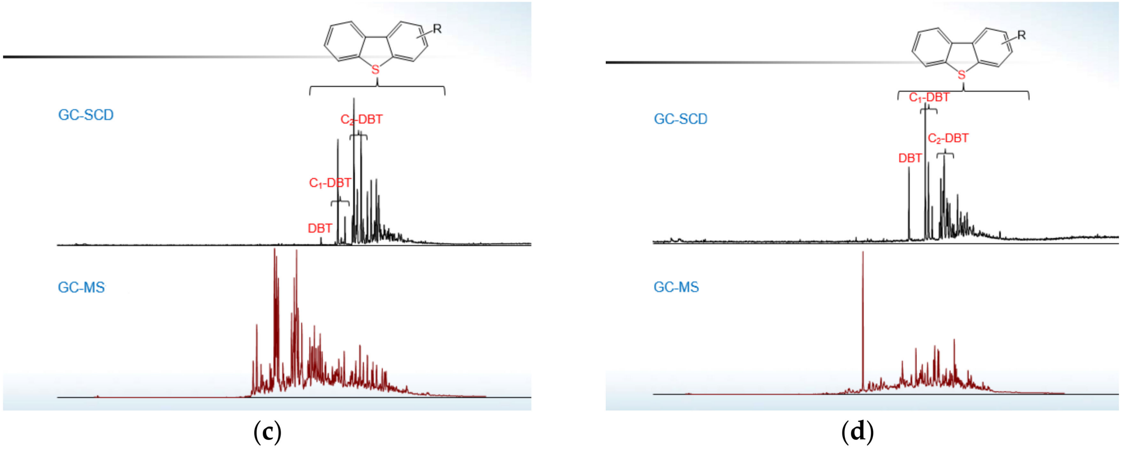


Figure 2. The GC-SCD and GC-MS spectra of thiophene sulfur: (**a**) WLT, (**b**) HCT, (**c**) QZT, and (**d**) HLT.

The FT-ICR MS distribution spectra of thiophene sulfur in WLT, HCT, QZT, and HLT are shown in Figure 3. The horizontal axis represents the number of carbon atoms, and the vertical axis represents the degree of unsaturation. The size of the dot represents the relative abundance of the substance. It can be seen that thiophene sulfur in the transformer oil is mainly in the form of benzothiophene with unsaturation of 6, while dibenzothiophene with unsaturation of 9 as well as their homologues.

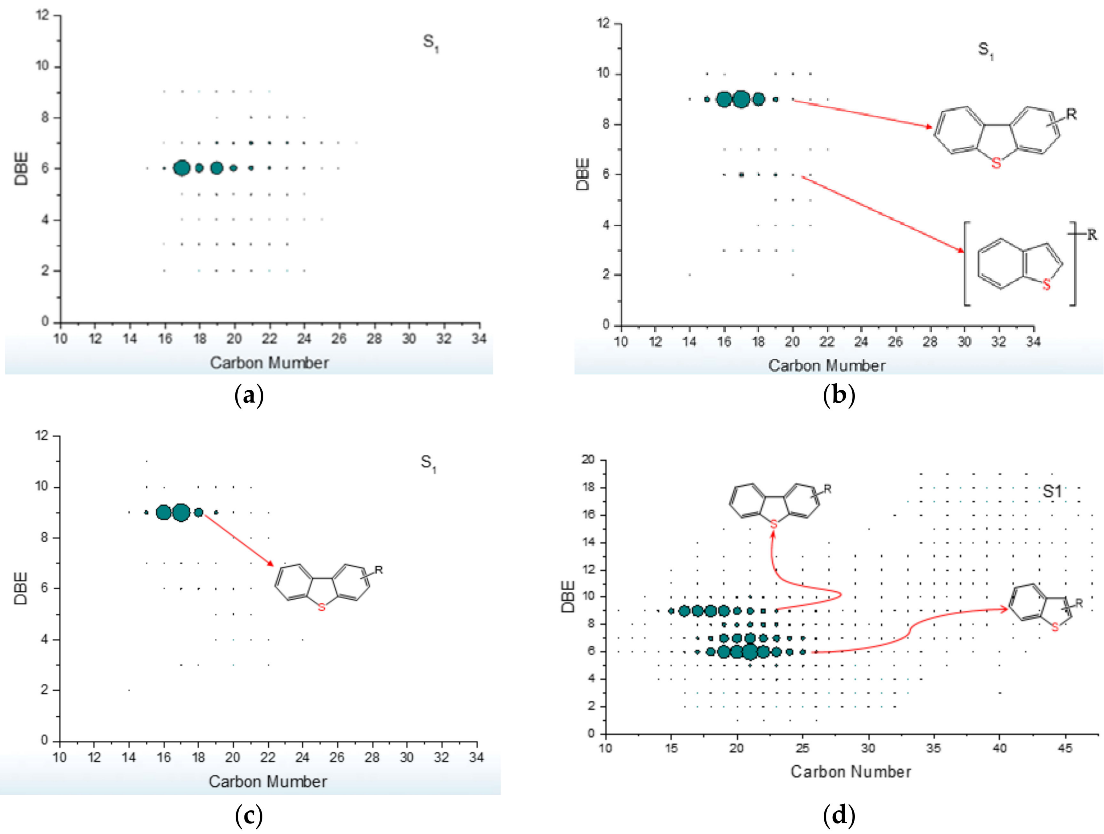
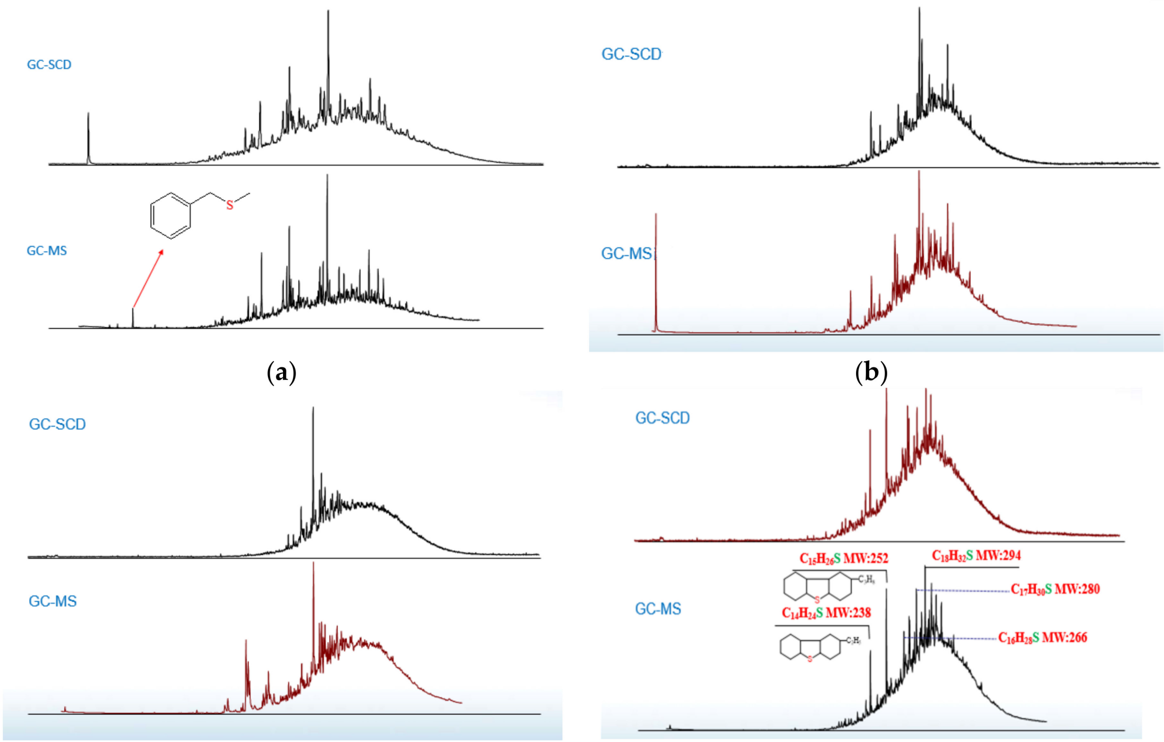


Figure 3. The FT-ICR MS distribution spectra of thiophene sulfur: (**a**) WLT, (**b**) HCT, (**c**) QZT, and (**d**) HLT.

3.2. Monothioether Sulfides

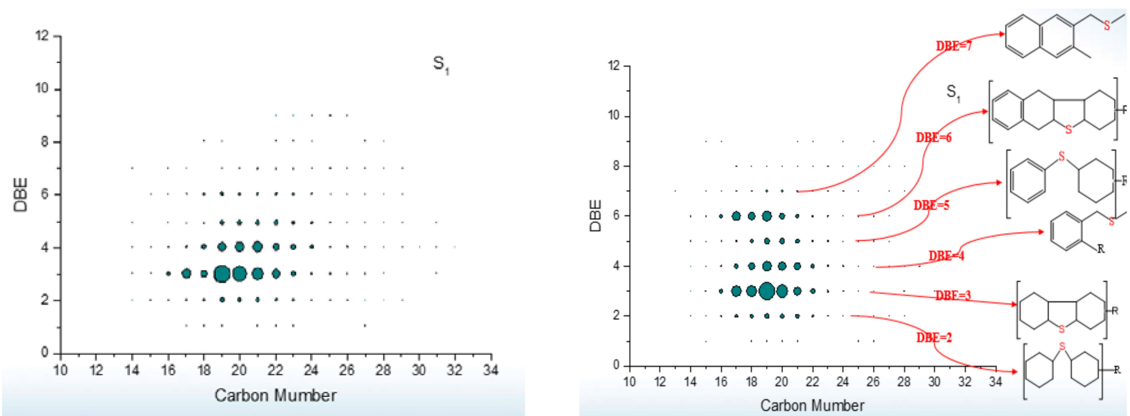
The GC-SCD and GC-MS spectra of thioether sulfur in WLT, HCT, QZT and HLT are shown in Figure 4. Compared with thiophene sulfur, the GC-SCD and GC-MS spectra of thioether sulfur are almost completely correspondent, though their separation effect is not good. A small bump is formed in the spectrum, from which only a small amount of information can be read. The structural formula can be speculated from the molecular formula, but most of the structural formula cannot be determined in the spectral diagram.



(**c**) (**d**)

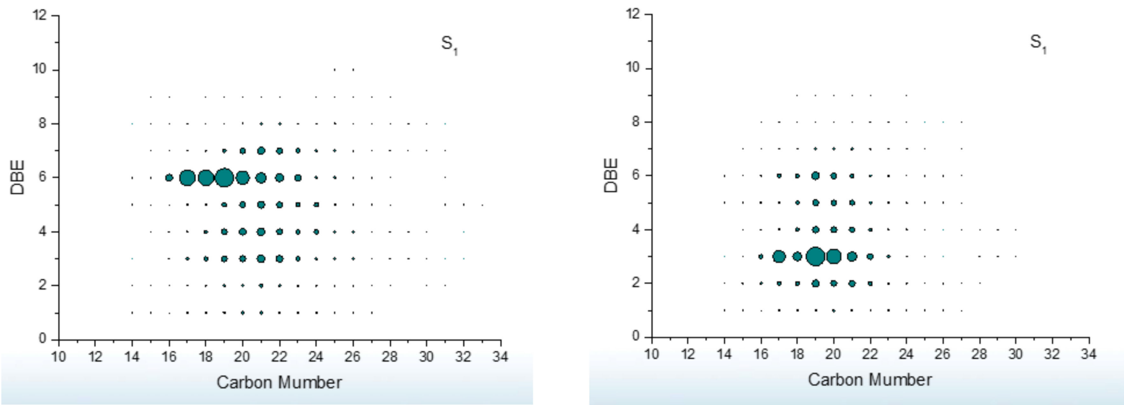
Figure 4. The GC-SCD and GC-MS spectra of thioether sulfur: (**a**) WLT, (**b**) HCT, (**c**) QZT, and (**d**) HLT.

The FT-ICR MS distribution of thioether sulfur in WLT, HCT, QZT, and HLT are shown in Figure 5, from which the distribution characteristics of thioether sulfur in transformer oil can be inferred. The carbon number of thioether sulfur in transformer oil is mainly distributed between 14 and 24, and the unsaturation is mainly distributed between 2 and 8. Compounds with unsaturation of 3 and unsaturation of 6 are the most abundant.



(**a**) (**b**)

Figure 5. *Cont.*

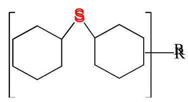
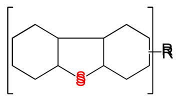
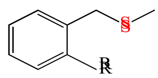
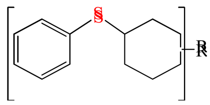
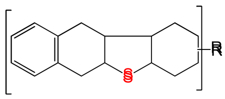
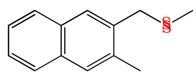
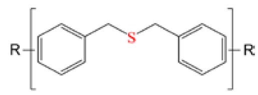


(c) (d)

Figure 5. The FT-ICR MS distribution of thioether sulfur: (**a**) WLT, (**b**) HCT, (**c**) QZT, and (**d**) HLT.

According to the above information, the molecular formula of thioether with different degrees of unsaturation can be calculated and their structural formulae can be estimated. The statistical results are shown in Table 4. However, there are few standard samples of thioether compounds, and the matching results in the database are not good. The following structural formulas are speculative results.

Table 4. Test results of thioether sulfurin transformer oil.

Unsaturation	Molecular Formula	Possible Structural Formula
2	$C_nH_{2n-2}S$	
3	$C_nH_{2n-4}S$	
4	$C_nH_{2n-6}S$	
5	$C_nH_{2n-8}S$	
6	$C_nH_{2n-10}S$	
7	$C_nH_{2n-12}S$	
8	$C_nH_{2n-14}S$	

3.3. Disulfide Sulfides

The FT-ICR MS distribution of disulfide compounds in the transformer oil samples is shown in Figure 6. The dot size in the figure represents the relative abundance of the sulfides. As can be seen from the figure, the disulfide compounds in the transformer oil are mainly the DBDS with an unsaturated degree of 8. Compared with DBDS, the other disulfide compounds are almost negligible. The FT-ICR MS method has high resolution, even if GC-MS cannot detect DBDS content, FT-ICR MS can also detect the presence of DBDS.

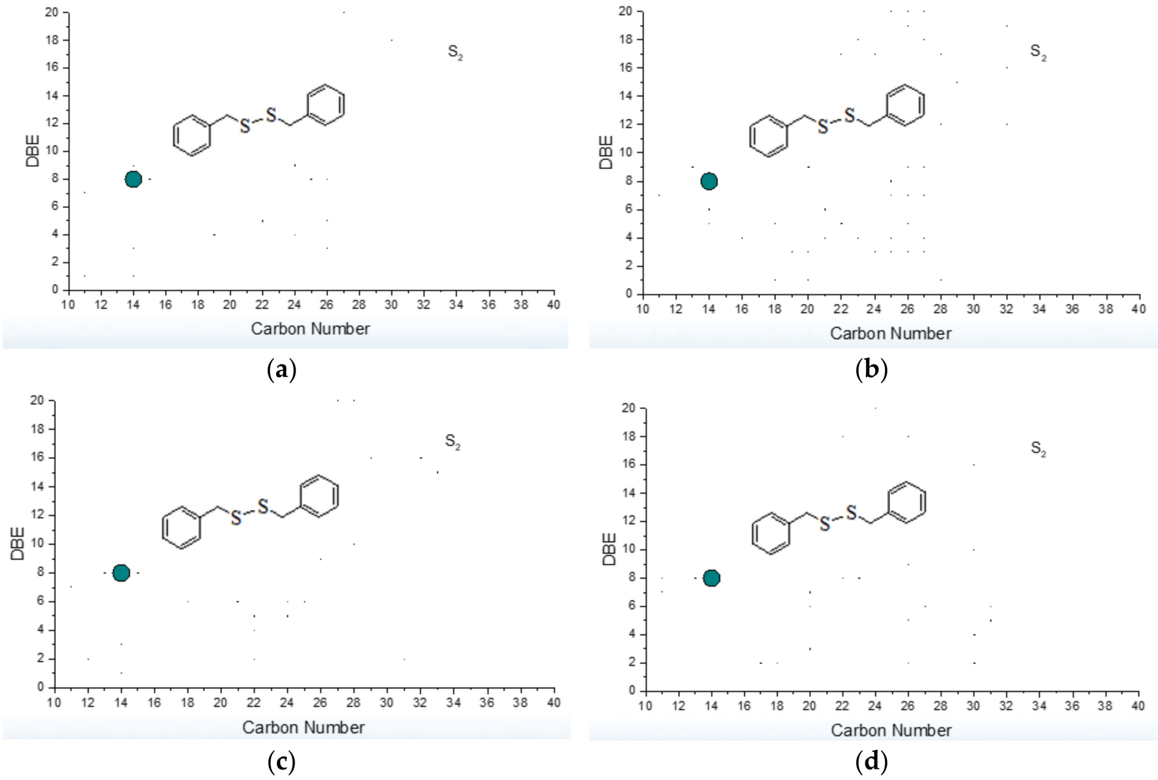


Figure 6. The FT-ICR MS distribution of disulfide compounds: (**a**) WLT, (**b**) HCT, (**c**) QZT, and (**d**) HLT.

4. Effects of Different Sulfur Components on Oil Quality

According to the above investigation, the corrosive sulfur in the transformer oil is mainly mercaptan and disulfide. The mercaptan content is lower than the detection value at environmental temperature. In addition, some non-corrosive sulfide may convert to corrosive mercaptan sulfide when partial overheating occurs in the transformer. According to the test results, the disulfide content in the oil is mainly DBDS. Therefore, the benzyl disulfide and dodecyl mercaptan were selected as typical corrosive sulfurs to add into the new transformer oil in this article. The influence of different sulfur components on oil quality was investigated by testing the breakdown voltage at power frequency, the moisture in oil, dielectric power factor, and volume resistivity of insulation oil.

The test materials used in this paper included the following: new 25# transformer oil, DBDS, and DDM (dodecyl mercaptan). The concentration of sulfides added in oil samples is shown in Table 5. The principle of addition is to ensure that the total sulfur concentrations in the different groups of oil samples are equal.

Table 5. Concentration of sulfides added in oil samples.

Sulfide	New Oil	DBDS	DDM	DBDS + DDM
DBDS (ppm)	-	200	-	100
DDM (ppm)	-	-	400	200

The 15 mL new 25# transformer oils were poured into each of the four prepared 20 mL empty bottles. Then the specified concentration of DBDS, DDM and a combination of the two were added into the bottles. Next, they were heated and stirred at 40 °C to ensure that the sulfide compounds are fully dissolved in the oil. The new 25# transformer oil was used as the blank control group. The above four prepared insulating oils were dehydrated and degassed at 40 °C/50 Pa for 24 h, and then the characteristic parameters were tested. The experimentally tested characteristic parameters of insulating oil include the followings details. (A) The breakdown voltage of insulation oil at power frequency refers to the IEC 60156 standard. The standard ball-plate electrode was used and the voltage was increased at a constant rate of 2 kV/s until breakdown. The breakdown voltage of insulating oil at power frequency was measured by the automatic oil dielectric strength tester. (B) The moisture in oil refers to the ASTMD 1533 standard. The moisture content in oil was tested by the coulomb method moisture meter. (C) The dielectric power factor and volume resistivity of insulation oil refer to the IEC 60247 standard. The dielectric power factor and volume resistivity of insulation oil were measured by the automatic insulation oil dielectric loss and volume resistivity tester.

The test effect of different sulfur components on the electrical properties of insulating oil is shown in Figure 7. It can be seen from the figure that the addition of sulfide would increase the moisture content in oil. The addition of dodecyl mercaptan nearly doubled, and it could be concluded that the water absorption of the mercaptan is better. The impurity could reduce the breakdown voltage. Therefore, the breakdown voltage of all three groups was decreased, in which the DDM group decreased most. The increase of dielectric loss could be caused by the water inhalation. In addition, the decrease of volume resistivity could cause the increase of conductance loss and dielectric loss.

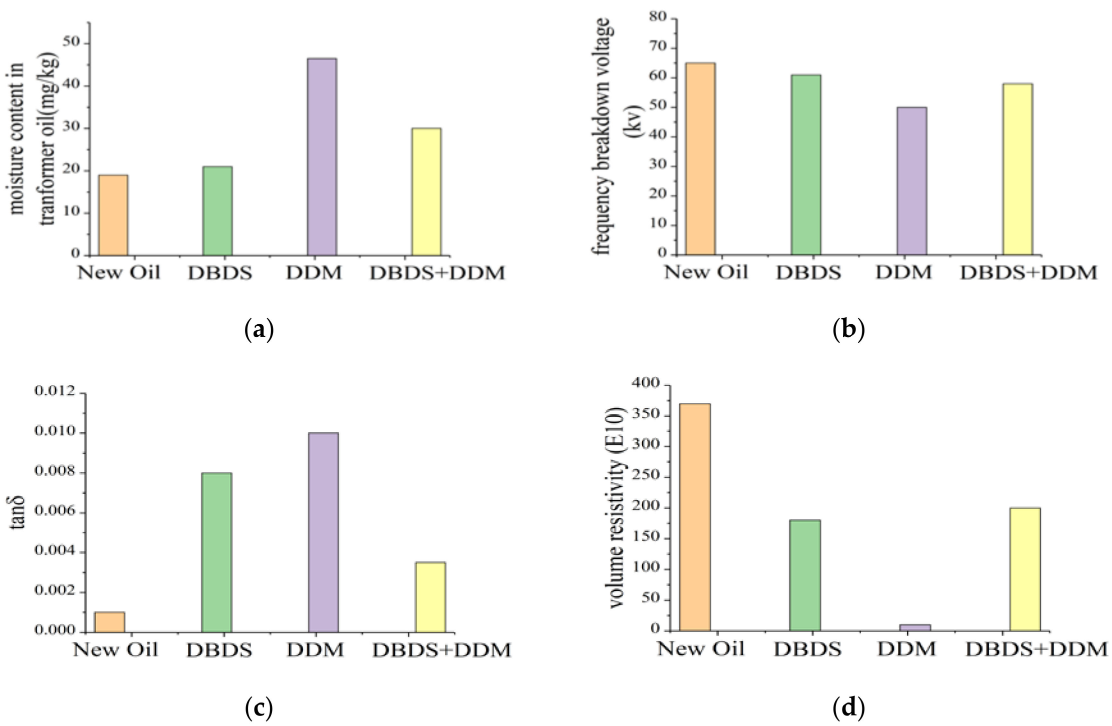


Figure 7. Effect of different sulfur components on the electrical properties of insulating oil: (**a**) Moisture in oil, (**b**) breakdown voltage at power frequency, (**c**) tan δ, and (**d**) volume resistivity.

5. Conclusions

(1) According to the investigation statistics, the naphthenic base oil samples account for a large part of the total corrosive samples in Fujian area. The corrosive oil samples under 500 kV voltage level are more than other voltage levels. Relatively speaking, Nynas oil sample is corroded seriously, while the oil composition and the voltage level are not directly related to the corrosiveness of the oil sample.

(2) The qualitative testing methods of various sulfide compounds in transformer oil was established. On this basis, the sulfur composition distribution in operating oil was analyzed. The thiophene sulfide in the transformer oil mainly exist in the form of benzothiophene with an unsaturation of 6 and dibenzothiophene with an unsaturation of 9. The monosulfide sulfide with an unsaturation of 3 or an unsaturation of 6 has the highest content in transformer oil. The disulfide in transformer oil is basically in the form of DBDS.

(3) The addition of different corrosive sulfurs will lead to the increase of moisture content in oil, indicating that corrosive sulfur has a certain amount of water absorption. The mercaptan has the strongest water absorption capacity. The increase of water content in oil will lead to the decrease of the breakdown voltage and the increase of the dielectric loss, which will seriously affect the quality of oil products.

(4) In view of the sulfide hazard on transformer oil, it is necessary to prevent and control the sulfur corrosion. On one hand, it is essential to prevent the insulating oil with corrosive sulfur from entering the transformers. On the other hand, it is also necessary to conduct corrosive testing on the operating transformer insulating oil to ensure that corrosive sulfur is detected and effectively blocked as early as possible. Recently, the use of vegetable oil for large power transformers is proposed and there is no corrosive sulfur hazard. If the mineral insulating oil is replaced by vegetable oil in the future, it can effectively avoid transformer failure caused by corrosive sulfur.

Author Contributions: H.C. and Q.L. conceived and designed the experiments, M.Z. performed the experiments and analyzed the data, H.C. wrote the main manuscript text. All authors read and approved the final manuscript.

Funding: Finical supported by the National Natural Science Foundation of China (51807061) is here acknowledge.

Conflicts of Interest: The authors declare no conflict of interest.

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