CHARACTERISATION OF A NAPHTHENIC BASED UNINHIBITED VIRGIN TRANSFORMER OIL AND THE USE OF SYNTHETIC ANTIOXIDANTS.

V. Dukhi[1], A. Bissessur[1*], C. J. Ngila[2] and N. Ijumba[3]
[1]School of Chemistry, [3]HVDC, University of KwaZulu-Natal, Private Bag X54001, Durban, 4000, South Africa
[2]University of Johannesburg, P.O. Box 17011, Doornfontein, 2028, South Africa
*bissessura@ukzn.ac.za

Abstract: The bulk of insulation material used in a transformer is comprised of mineral oil. Exposure of these mineral oils to oxidative, thermal and electrical stresses [1] induce ageing. Degeneration of the oil imparts poor quality thus causing it to lose its ability to function optimally. This work focuses on the characterisation of a naphthenic based virgin uninhibited transformer oil (VO) commonly used in South African transformers. While antioxidants (AO's), such as 2,6-di-tert-butyl-4-methylphenol (BHT) (fig. 1) and 2,6-di-tert-butylphenol (DBP) (fig. 2) have been blended into oils to increase its stability, more work needs to be done to highlight the effects on the oil and efficiency of these AO's. In addition this paper also characterises antioxidant-virgin oil blends as well as the effect of antioxidants on the oil. The common diagnostic chemical characteristics of acid and saponification values showed no detection as expected for virgin transformer oil. The proton and carbon nuclear magnetic resonances (^{1}H NMR and ^{13}C NMR respectively) as well as fourier transform infra-red (FTIR) data showed no presence of acids, alcohols or esters, which are common by-products of transformer oil ageing. The ^{1}H-NMR spectra, however indicated the presence of aromatic groups, which later was identified as tetralin and decalin by gas chromatography-mass spectrometry (GC-MS). The blending of antioxidants into virgin transformer oil proved to be successful based on evidence sourced from ^{1}H NMR, ^{13}C NMR and IR data. The 2,2-diphenyl-1-picrylhydrazyl (DPPH) assay, served to confirm that blending of BHT and DBP into transformer oils increased the free radical scavenging ability of the oil. Thermal gravimetric analysis (TGA) data revealed that blending of AO's into transformer oil improved thermal stabilities of the oil. An attempt to identify degradation products of the AO's by subjecting the oil blends to UV irradiation over a period of 24 hours was successfully carried out using GC-MS. Evidently blending of antioxidants has potential to enhance the performance and increase the life-span of transformer oil.

1 INTRODUCTION

South Africa is currently facing a major energy crisis with respect to a shortage in power supply. As a result of urban growth and development, increased pressure has been placed on power service providers for more efficient production and delivery. In addition, the increasing demand for electricity coupled with a limited number of power stations has placed huge emphasis on power conservation and generation. Transformers form an integral part of power-stations and play key role in the transference of power. For efficient transference from power-stations to end-users, the performance of a transformer has to be considered.

Transformer oil functions both as an insulator and coolant [2]. However the oil suffers a drawback in which it is susceptible to ageing, whereby the main mechanism entails oxidation. During ageing, oxidation reactions lead to the formation of carboxylic acids as well as oil sludge resulting in poor functioning of the transformer oil and the transformer unit as a whole [3]. There are two classes of transformer oils: a) uninhibited oils (containing no antioxidants) and b) inhibited oils (containing antioxidants at specified concentrations) [4]. This study has focused on naphthenic-based virgin transformer oil with synthetic antioxidants added. Note that these oils are a key component of transformer insulation and for maintaining optimum functioning of the transformer unit.

In the methodology, naphthenic based virgin transformer oil was chemically characterised in order to relate its chemical properties to its functioning. The oils are characterised using both conventional and international standard routine oil analysis techniques as well as other analytical techniques such as gas chromatography with mass spectrometry. The procedure used involved the blending of antioxidants (free radical scavengers) with virgin uninhibited oil to enhance oil stability.

Currently 2,6-di-tertiarybutyl-4-methylphenol (BHT) (fig. 1) and 2,6-di-tert-butylphenol (DBP) (fig. 2) are used as antioxidants (AO's) however owing to their carcinogenic and toxic behaviour the International Electro-technical Commission (IEC) governs that these AO's be used at concentrations not exceeding 0.3 % (w/w) [4].

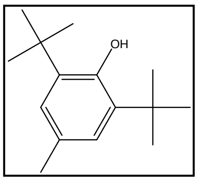

Figure 1: Structure of AO, 2,6-di-tertiarybutyl-4-methylphenol (BHT)

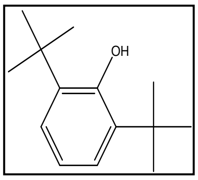

Figure 2: Structure of AO, 2,6-di-tert-butylphenol (DBP)

Despite the protective role which AO's display, they themselves are susceptible to oxidation reactions over time. Hence the analysis of degradation products is crucial to understanding the intensity of AO protection.

In this study, an accelerated ageing study on inhibited oil by UV-digestion was carried out to monitor the impact of AO blending and thus relate this to transformer oil stability.

2 METHODS AND MATERIALS

All analytical grade reagents (AR) were used unless otherwise stated.

2.1 Saponification value

A 10 g sample mass of the naphthenic oil was added to 25 ml of a 0.1 M alcoholic KOH solution in a 250 ml conical flask. The solution was refluxed for 30 minutes and titrated while hot with a standard 0.5 M HCl solution to a phenolphthalein endpoint. A blank was also carried out.

2.2 Iodine number

A 2 g sample mass of naphthenic oil was dissolved in 20 ml of chloroform to which a 10 ml aliquot of 0.1 M Wijs reagent (ICl) was added. The solution was left to stand in the dark for 1 hour after which 20 ml of a 10 % (w/v) KI solution and 100 ml of deionised water was added. The liberated iodine was titrated with a standard 0.0166 M sodium thiosulphate solution to a starch endpoint.

A blank was performed under the same conditions. Replicate titrations were carried out until three iodine values to within 0.1 g I_2/ g oil was obtained.

2.3 Total acid number (TAN)

A 20 g sample mass of transformer oil was dissolved in 100 ml of the titration solvent (toluene : isopropyl alcohol : deionised water; ratio: 100:99:1) and titrated at room temperature to a phenolphthalein endpoint with a standard 0.1067 M alcoholic KOH solution.

2.4 Metal/ non-metal content

Sample preparation: In triplicate, 2 g of the naphthenic oil sample was digested in 20 ml of aqua regia [5] using open beaker digestion. The sample solution was pre-concentrated to approx. 10 ml and the resulting solution was filtered.

A 5 ml aliquot of the pre-concentrate was added to a 100 ml volumetric and diluted to the mark with deionised water.

Preparation of standards:

Multi-element standards of Al, B, Ba, Ca, Cd, Cr, Cu, Fe, Mg, Mn, P, Pb, S, Si, Sn, and Zn in the range of 1, 2, 5, 10, 20, 50 ppm were prepared by serial dilution of 1000 ppm standard stock solutions in 5 % aqua regia. A blank containing 5 % aqua regia was prepared using deionised water.

Instrument: Analysis by inductively coupled plasma-optical emission (ICP-OES) was carried out using the Perkin Elmer Optima 5300 DV ICP-OES Spectrophotometer.

2.5 Thermal Gravimetric Analysis

All virgin transformer oils and blends thereof were analysed using the Universal Analysis 2000 by TA Instruments. A temperature ramp rate of 5 ºC min^{-1} from room temperature to 300 ºC was applied in all cases.

2.6 Water content

The moisture content was determined by the Karl Fischer (KF) method using the Metrohm 870 KF Titrino Plus Analyser.

2.7 Viscosity

The viscosity of transformer oils was determined using Ostwalds capillary viscometer (bore size 2 mm) at a constant temperature of 25 °C using a water bath. Calibration of the viscometer was carried out using a N10 oil standard purchased from Fluka Analytical. Replicate efflux times were recorded to within 0.1 s.

2.8 Hydroperoxide content

The hydroperoxide content was analysed according to a method used by Wikland *et al.* [6]. Solution A was prepared by dissolving 2 g of triphenyl phosphine (TPP) and 0.15 g flourene (internal standard (IS)) in toluene made up to 25 ml. Solution B was prepared by dissolving 0.45 g elemental S dissolved in toluene made up to 50 ml. A sample mass of 0.5 g was weighed into a vial and reacted with 250 µL of solution A for 15 minutes. This was followed by the addition of 250 µL of solution B and reacted for 15 minutes. The amount of triphenyl phosphine oxide (TPPO), produced as a result of the oxidation of TPP, served as an indicator of the hydroperoxide level in the oil.

TPPO was analysed by GC-FID (Agilent Technologies 6820 GC system) using a DB-1 column.

Injection Temp: 280 °C
Detector Temp: 280 °C
Column temp: 70 °C for 1 minute

Column ramp temp:
Ramp 1: 20 °C min^{-1}: to 100 °C
Holding time: 1min
Ramp 2: 15 °C min^{-1}: to 150 °C
Holding time: 1min
Ramp 3: 15 °C min^{-1}: to 300 °C
Holding time: 4 min

The relative response factor (RRF) of TPPO to fluorene was used to quantify TPPO.

2.9 ^{13}C and ^{1}H NMR, Infrared Spectroscopy and Elemental analysis

All ^{1}H and ^{13}C NMR spectra of virgin transformer oils dissolved in deuterated chloroform (Merck) were recorded on a Bruker AVANCE III 400 MHz instrument at room temperature. Chemical shifts were expressed in ppm relative to TMS.

FTIR spectra were recorded using the - Perkin Elmer 100 Fourier transform infrared (FT-IR) spectrophotometer.

The virgin oil was analysed on the Leco 932 CHNS elemental analyser.

2.10 GC-MS analysis (virgin oil)

This analysis was performed using the Agilent 6890 GC system, column type: DB5-MS. Injector temp: 250 °C. Column temp: 60 °C for 2 minute. Column ramp temp: 10 °C min^{-1} from 60 to 300 °C.

2.11 Dissipation factor and Dielectric breakdown voltage (DBV)

The above tests were carried out by Transformer Chemical Services (Durban, 2010) according to the guidelines stipulated in IEC 156 and IEC 247 for dielectric breakdown voltage and dissipation factor respectively.

2.12 DPPH assay [11]

Oil blends comprising of 0.3 % (w/w) of (i) BHT + VO and (ii) DBP + VO were prepared. A 0.1 g sample mass of the oil blend was added to 3 ml of 1.8 x 10^{-3} M DPPH in dichloromethane (DCM). Samples were left to react in the dark for 30 minutes. The absorbance (at 517 nm) was recorded on a Shimadzu UV 1800 Spectrophotometer. All runs were performed in triplicate and a sample blank was used to eliminate background interferences.

2.13 AO monitoring

A VO blend containing AO's was exposed to UV light using a UV digester (Metrohm 705 UV digester) over a period of time. A sample mass of 0.5 g of UV irradiated oil was dissolved in 500 µL of 3.66x10^{-2} M fluorene (IS) in toluene and analysed by GC using flame ionisation detection (FID). The RRF of fluorene to DBP and BHT was used to quantify the antioxidant. In addition to monitoring antioxidant depletion, degradation products of the antioxidants were also investigated. This was achieved through dissolution of UV irradiated samples in DCM and analysis by GC-MS using Agilent Technologies 6890 Series GC coupled with Agilent 5973 Mass selective detector.

Column type: DB5-MS.
Injector temp: 250 ° C.
Column temp: 80 °C for 1 minute
Column ramp temp: 10 °C min^{-1} to 260 °C
Holding time: 15 minutes.

3 RESULTS AND DISCUSSION

Table 1: Properties of naphthenic based uninhibited virgin transformer oil. (n.d. #= not detected)

Property evaluated	
Acid Number/ (mg KOH/ g oil)	n.d. $^{\#}$
Saponification number/ (mg KOH/ g oil)	n.d. $^{\#}$
Iodine value/ (g I_2/ 100 g oil)	0.6313 ± 0.01270
Water content/ ppm	129.2 ± 39.21
Kinematic viscosity/ mm^{2} s^{-1}	15.64 ± 0.03480
Dissipation factor	0.0121
Dielectric Breakdown Voltage/ kV	50
Hydroperoxide content/ (g TPPO/ g oil)	0.07734 ± 0.006126
% C	86.00
% H	13.51
% N	0.3815

In the evaluation of total acid number and saponification values, none were detected, which seemingly is an expected result for virgin transformer oil. Ideally no acidic or saponifiable constituents must be present in the unused original oil. Generally, the acid number of new unused transformer oils does not exceed a value of 0.05 mg KOH per gram [7]. Although some forms of transformer oils are classified as naphthenic based, additives present may be aromatic in nature. The iodine value, an indication of saturation, suggests the presence of unsaturated components in the oil. A moisture content of 129.2 ± 39.21 ppm obtained for the virgin oil was significantly higher than the max. moisture content value of 35 ppm prescribed for most oils [7]. According to Dong *et al.* [14] in new and used oils, additives, and contaminants containing OH group make determination of moisture content at low levels (< 2%) difficult. Moisture determination using the KF method for oils with low water content (< 20 ppm) and additives have been very problematic. An alternate method has been proposed by Dong *et al*. [14]. Consequently, a low water content in transformer oil is a desirable property as an increase in moisture content reduces the insulating properties of the oil [8]. In addition an increase in water content may accelerate ageing of other components of the insulating material [7].

The dissipation factor, which essentially is the leakage current through the oil [9], is expected to be less than 0.1 for oils used in Category A transformers (i.e. transformers exceeding 170 kV but less than 400 kV) [9]. A dissipation factor of 0.0121 and DBV of 70 kV was recorded for this naphthenic based oil, these values are deemed to be acceptable.

The two classes of metals that may be present in transformer oils, are classified as wear metals and additive metals [10]. Wear metals are known to accelerate the degradation process and these metals include Fe, Cu, Sn, etc.[10] In the case of wear metals they may accumulate in oils as a direct result of contamination arising out of the transformer itself. Additive metals exhibiting antioxidant, anti-wear and anticorrosion activity may be blended into the oil in order to protect the oil as well as the transformer.

Table 2: Metal/ non metal content of a naphthenic virgin transformer oil.

Metal	Concentration/ (µg/g)
Al	3.877 ± 0.021
B	16.23 ± 5.51
Ca	91.478 ± 9.42
Mg	27.03 ± 2.68
P	0.5244 ± 0.211
Si	226.1 ± 5.87

In this study a host of metals/ non metals that included Al, B, Ba, Ca, Cd, Cu, Cr, Fe, Mg, Mn, P, Pb, S, Si, Sn and Zn were investigated and quantified. The absence of wear metals (table 2) was largely attributed to non-contact of the oil with a transformer. The presence of Si in highest concentration is due to the use of Si derivatised compounds as defoaming agents [10]. The presence of B, Ca, Mg and P or derivative compounds thereof, as additives, may act as potential anti-wear agents or antioxidants [10].

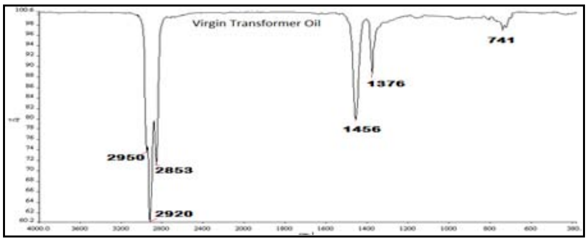

Figure 3: FTIR spectra of naphthenic based virgin transformer oil.

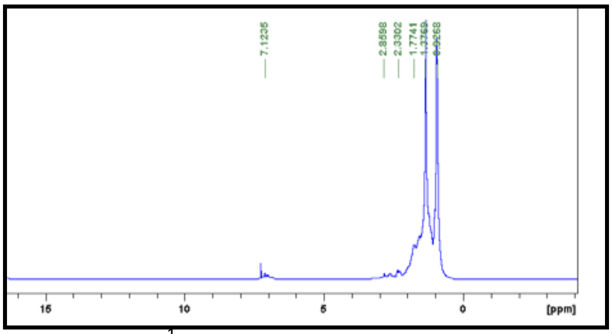

Figure 4: ^{1}H NMR of virgin transformer oil.

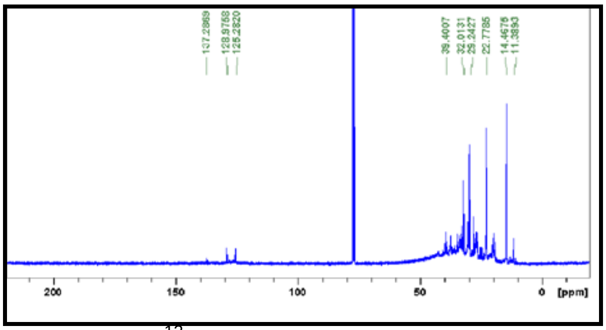

Figure 5: ^{13}C NMR of virgin transformer oil.

Characteristic FTIR data of a naphthenic based virgin transformer oil shown in Figure 3, illustrates the C-H stretching of methyl groups at 2950 and 1376 cm^{-1}, C-H stretching in methylene bonds at 2920 and 2853 cm^{-1}, C=C stretching of aromatic groups at 1456 cm^{-1} and C-H of aromatic groups (Ar-H) at 741 cm^{-1}. Data obtained from FTIR spectra was reinforced by ^{1}H and ^{13}C NMR analysis (fig. 4 and 5 respectively). Peaks obtained in ^{1}H and ^{13}C NMR at 7.1-7.2 ppm and 125-137 ppm regions respectively are characteristic of aromatic groups. Other peaks identified in ^{1}H and ^{13}C NMR in the 0.2-2.9 ppm and 11-39 ppm regions respectively are indicative of -CH$_3$, -CH$_2$ and -CH groups.

The identification of characteristic components in a naphthenic based virgin transformer oil by GC-MS (figure 6, table 3) was achieved through comparison of data from a NIST library. Components shown in table 3 were identified using mass spectral data that was based on a minimum certainty factor of 80 %. A characteristic base line drift (oil hump) is observed in GC spectra for naphthenic based oil (fig. 6).

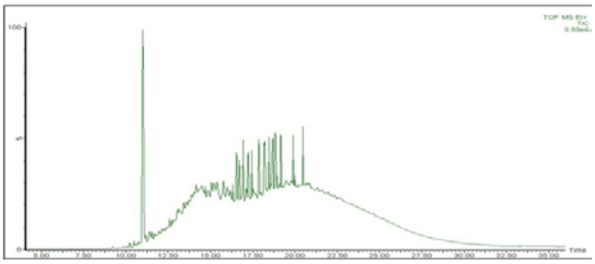

Figure 6: GC spectra of a naphthenic based virgin transformer oil.

Table 3: Components of a naphthenic based uninhibited virgin transformer oil and their retention times identified by GC-MS.

Component	Retention time/ min
Cyclohexane, butyl-	8.63
Naphthalene, decahydro-, trans-	9.22
Undecane	9.73
Naphthalene, decahydro-, cis-	10.02
trans-Decalin, 2-methyl-	10.21
trans-Decalin, 2-methyl-	10.49
1H-Indene, 2,3-dihydro-5-methyl-	10.59
Naphthalene, decahydro-2-methyl-	10.62
2,4- Dimethylstyrene	10.74
Tetralin	10.97
Dodecane	11.43
Cyclopentylcyclohexane	11.54
Hexylidencyclohexane	11.55
Dodecane, 6-methyl-	11.65
Naphthalene, 1,2,3,4-tetrahydro-6-methyl-	12.72
Naphthalene, 1,2,3,4-tetrahydro-5-methyl-	12.72
n. id	13.41
1,1'-Bicyclohexyl, 4,4'-dimethyl-	14.88
1,1'-Bicyclohexyl, 4,4'-dimethyl-	14.90
Decahydro-4,4,8,9,10-pentamethylnaphthalene	15.79
2-Dodecen-1-yl(-)succinic anhydride	18.17
Z,E-2,13-Octadecadien-1-ol	18.46
1-Heptatriacontanol	19.89
Z,Z-3,15-Octadecadien-1-ol acetate	20.46
n. id	24.33

n. id – non identifiable component.

Table 4: Functional groups of blended virgin transformer oils from FTIR spectra.

Blends	Wavenumber/ cm^{-1}	Functional Group	Explanation
0.3 % BHT-Virgin Transformer Oil	3649	OH	Hyroxyl group attached to a benzene ring
	1231	C-OH	C on the benzene ring attached to OH group
	859	CH	CH on the benzene ring that is meta di substituted
0.3 % DBP-Virgin Transformer Oil	3650	OH	Hyroxyl group attached to a benzene ring
	1230	C-OH	C on the benzene ring attached to OH group

Table 5: Identification of functional group in blended virgin transformer oils from ^1H and ^{13}C NMR spectra.

Blend	NMR	Chemical Shift/ ppm	Explanation
0.3 % BHT-Virgin Transformer Oil.	^1H	5.08	OH
		2.4	CH_3 (para methyl group)
		1.57	CH_3 (tert. butyl groups)
	^{13}C	34.26	C (quantenary carbon)
		32.1	CH_3 (para methyl group)
		30.39	CH_3 (tert. butyl groups)
0.3 % DBP-Virgin Transformer Oil.	^1H	5.08	OH
		1.57	CH_3 (tert. butyl groups)
	^{13}C	30.39	CH_3 (tert. butyl groups)
		34.4	C (quantenary carbon)

Successful blending of antioxidants with the virgin transformer oil has been verified by the functional groups as identified from FTIR data (table 4), ^1H and ^{13}C NMR (table 5).

The antioxidants, BHT and DBP, which are free radical scavengers are used as additives to terminate, reduce or significantly slow down free radical process [4]. The antioxidant activity was monitored by a change in the concentration of DPPH, a stable free radical [11]. An electron transfer to DPPH leads to a decrease in absorbance observed at 515 - 517 nm [11].

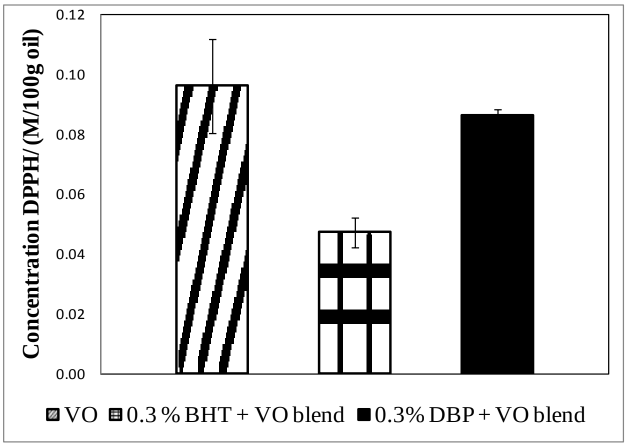

Figure 7: Change in concentration of DPPH (per 100g of virgin transformer oil/ blended transformer oil) monitored over a 30 minute period.

A decrease in the concentration of DPPH when comparing uninhibited oil (VO) to inhibited oil (doped with antioxidants) is clearly evident. Thus an addition of the specified antioxidants has increased free radical scavenging ability of the oil. The BHT-oil blend is shown to have a higher DPPH scavenging ability than the DBP-oil blend.

TGA data shown in fig. 8 reflects the thermal stability and weight loss of the virgin oil and oil blends upon accelerated degradation. The onset of degradation for virgin oil, a BHT-oil blend and a DBP-oil blend was found to be 137.72, 139.64 and 144.63 °C respectively. This indicates that the onset of degradation of the oil shifts to a higher temperature upon blending with AO.

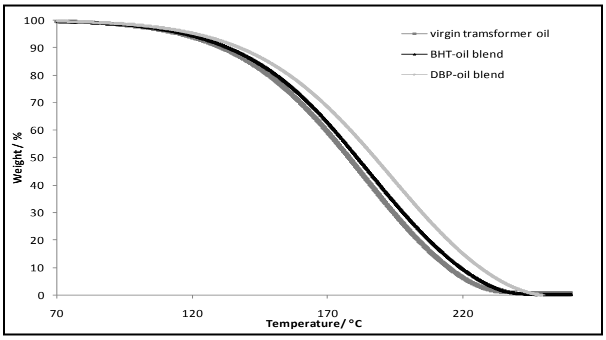

Figure 8: TGA of virgin transformer oil and AO-oil blends.

The accelerated ageing study was carried out in a UV digester that initiated rapid ageing over a limited period of time. In this study the concentration of BHT and DBP was monitored over 24 hours with exposure to UV radiation.

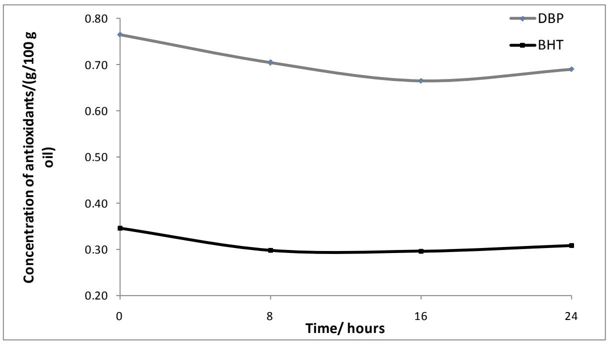

Figure 9: Concentration of BHT and DBP over a 24 hour UV exposure period.

A decrease in concentrations of DBP and BHT was observed over the 24 hour time period relative to their initial concentrations. This may be attributed to the oxidation of antioxidants themselves as predicted by the analysis of oxidation products following UV digestion by GC-MS. Identification of products resulting from BHT oxidation included: 2,4-di-tert-butyl-6-methylphenol [m/z = 220, 205, 191, 55] (a tautermization product), 4,4'-(ethane-1,2-diylidene)bis(2,6-di-tert-butylcyclohexa-2,5-dienone) [m/z = 433, 218 and 207] and 4,4'-(ethene-1,2-diyl)bis(2,6-di-tert-butylphenol) [m/z = 435, 234, 123, 57]. The formation of dimerised products in dry media has been reported by Foley and Kimmerle (1979) [12]; however in media with trace amounts of water present it is possible that degradation of BHT follows a radical and ionic pathway [12]. In the oxidation of DBP, the isomeric product 2,5-di-tert-butylphenol was successfully identified.

4 CONCLUSION

Naphthenic based uninhibited virgin transformer oil has been successfully blended with AO's and their indicative characteristic chemical properties identified. Aromatic additives, such as tetralin, that is used to reduce the gassing tendency of oil [13] is prevalent in virgin oil. The presence of metals and non metals such as Mg, Ca or P can be attributed to the fact that they possess antiwear or antioxidant properties. However virgin oil was found to be void of wear metals that catalyse oxidation reactions. Blending of virgin transformer oils with DBP and BHT was found to be successful as shown by NMR and FT-IR data. DPPH assay showed an increase in the free radical scavenging ability of blended oils compared to that of virgin oil; TGA confirmed an increase in thermal stabilities of AO-oil blends. Small changes in the concentration of BHT and DBP over 24 hour exposure to UV digestion was observed.

5 ACKNOWLEDGEMENTS

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