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ASSESSMENT OF METALS ACCUMULATION IN THE ENVIRONMENT FROM AUTOMOBILE SPENT OILS IN NIGERIA

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Abstract **:** This work assessed the level of metals accumulation in the Nigerian environment from automobile spent oils. Four fresh commercial automobile lubricating oils were introduced into four automobiles with similar specification and the spent oils were drained from the automobiles after two months of being used daily. Quantitative analyses for metal contents with the selected metals Mg, Sn, Zn, Fe, V and Al were carried out on the fresh and used lubricating oils. It was considered that each automobile would have its lubricating oil drained and replaced bimonthly. An extrapolation with the 11,700,000 automobiles registered in Nigeria in 2020, showed that the amounts of Mg, Zn, Fe and Al present in the spent oils within the year were excessively higher than the maximum permissible amounts in the environment; with the excess amount in percentage evaluated as 581000, 879000, 23000 and 965,983% respectively. Sn and V metals were not found in significant amounts in the spent oils. Some performance parameters of lubricating oils such as density, particulate counts, total acid number (TAN) and total base number (TBN) were also quantitatively assessed with the metal contents of the oils. The health risks caused by the poor management of the generated spent oil in Nigeria were also assessed in the study.

Index Terms**:** spent oils, metals, accumulation, toxicity

I. Introduction

The increasing number of automobiles in the world has also contributed to geometric increase in the amount of lubricating oils demand globally and consequently increasing the amount of metals in the environment; if best practices on management of automobile spent oils are not followed, this may post severe risks to the environment. According to the statistics of number of registered vehicles in Nigeria, the trend in four years of five intervals showed total number of vehicles as 1747000, 3210000, 3750000, and 11700000 in 2005, 2010, 2015 and 2020 respectively (Nigeria Registered Motor Vehicle, 2020).

According to NORIA (2023), the longevity of an automobile engine is assured by good maintenance culture; periodic change of the engine lubricating oil. The functions of engine lubricating oils in automobiles include minimizing friction and maximizing fuel economy. This informs the need for automobile users to promptly change the used lubricating oil. The factor used for change of the spent oil are time and millage based, though resent investigations have shown that check for the performance properties (viscosity, API gravity, total acid value, total base value, particulate count, heavy metal) of the spent oil as well as the driving conditions of the automobile are more important factors to consider. On the average, change of spent oil is within three (3) months of usage or driving through a maximum of 3,000 miles. Most newer vehicles have made the monitoring of due time for spent lubricating oil change to be easier as oil-life monitoring devices are inputted in them and period due for change is notified with an alert displayed on the automobile instrument panel. The lubricants are found in the engine in-between metal parts. The lubricant functions include keeping moving parts apart to reduce friction; transferring heat out of combustion units; remove contaminants and particles from the lubricating unit as well as inhibit corrosion in the engine parts (Ahmed and Nassar, 2013).

Metals in the lubricating oils may be introduced in form of additives and these are usually trace metals while heavy metals may barely be introduced as additives but frequently as the depleted parts of the engine. Some metals like Al, Mg, Cu, Ti and Mo are used as additives in the engine oils and are also component of the engines (Myshkin *et al.*, 2003; Shihab *et al.*, 2019 and Sánchez-Alvarracín *et al.*, 2021).

According to the work of Wenten *et. al.*, (2018), the metal contents in automobile spent oils either as additives (antioxidants, anti-wear, and de-foaming agents) or wear metals (component of degraded engine parts) contribute to the hazardous nature of the oils and influence their toxicity when released into the environment.

Studies have shown that ash contents of used automobile spent oils increase with increase in the distance covered by the automobile after the fresh engine oil is changed. An investigation of ash contents in the used oil of an automobile over 621.37, 932.06 and 1242.74 mile distance covered after refilling the oil in the engine block, gave ash contents of 1.230, 1.256 and 1.282 wt %

respectively with the value for the ash content in the fresh oil before refilling recorded as 0.825 wt %; this showed an increase in ash content of about 260 ppm in every 300 mile. This same variation is also expected for metal (noncombustible) components in the automobile lube oils (Shihab *et al.*, 2019).

Sánchez-Alvarracín (2021), did a work to characterize and regenerate twenty-three used oils in a Latin American medium size city. Most of the oil samples were collected from official storage sites and few collected directly from the vehicles; the heavy metals reported in this work were Al, Mg, Fe, V, Sn, and Zn with their amounts contain in the spent oils as 8.0 , 37.80, 40.6, 3.3, 9.2 and 781 mg/kg respectively.

It is very likely that when the used oils are not well managed, the contaminated components are exposed to lands, leached into water bodies and contribute to fumes in the air. Relevant bodies that monitor our environments to determine the based line values of contaminants have published the maximum allowable amounts of metals in our environments. Some of the permissible values for the metals considered in this work are as shown below on Table 1.1.

Table 1.1: Maximum permissible limits of some metals in air, water, soil and vegetables

Metal	Permissible limit in soil (ppm)	Permissible limit in Water (ppm)	Permissible limit in Air (ppm)	Permissible limit in Vegetable (ppm)
Fe	58664.44	0.30	3.40	425.50
Mg	100.00	10.00	0.015 *	86.00
V	82.00	0.015	-	-
Al	16.33	< 0.20	0.0025	-
Zn	494.89	5.00	0.005 *	99.40
Sn	-	< 0.10	0.002 *	50.00

After; New Jersey Department of Health (2007);WHO, 2008; Mensah, 2009; Haase, 2010; Mahmud, 2016;FAO/WHO, 2019 ;Liu *et al.*, 2020; Ogunlana *et al.*, 2020; Morakinyo *et al.*, 2021; Zondo, 2021; Qutob *et al.*, 2022; Wnuk, 2023. *OSHA published legal air-born permissible limit for respirable dust averaged over an eight hour work-shift.

II. Materials and Methods

2.1 Materials

This work involved the use of both used and unused automobile lubricating oils as well as necessary reagents and instrumentation for the analysis of the amount of metals, density, total base number, total acid number and particle count in oil samples.

2.2 Methods

2.2.1 Sampling of Automobile Lubricating Oils

Four fresh commercial lubricants (L_1, L_2, L_3 and L_4) were obtained in Nigeria and were filled into four different Toyota Camry vehicles with similar specification (salon car 2001 model). The vehicles were allowed to drive for two months before the spent lubricants were drained completely from the engine into four different sample containers labeled U_1, U_2, U_3 and U_4 according to the numerical values assigned to each vehicle. The termed Unused Oils (UnO) was used for the fresh oils and labeled UnO_1,UnO_2,UnO_3 and UnO_4, Used Oils was used for the oil drained from the vehicles and marked UO_1, UO_2, UO_3 and UO_4 according to the serial numbers assigned to the vehicles. Both unused and used or spent oils were kept in cold and dry places until required for laboratory investigations.

2.2.2 Measurement of the Amount of Metals in Lubricating Oils

The lubricant to be analyzed was ashed using a crucible, then dissolved in 50 ml aqua regia solution (1:3 volume HNO_3 and HCl) using a 250 ml volumetric flask. The mixture was placed in a hot plate and heated until the volume reduced to half and distilled water was added to make up the displaced volume; the heating process continued with addition of distilled water for three times. The concentration of six heavy metals (Mg, Al, Sn, Fe, Zn and V) were detected from the extracted sample solutions using a BUCK Scientific Model 210VGP Atomic Absorption Spectrometer (AAS) (Ekpo and Obi, 2017; Shihab *et al.*, 2019). The method used here is in conformance with ASTM D6598.

2.2.3 Density Measurement Using ASTM D-1298

With ASTM D-1298 the used and unused oil samples were measured for API gravity at ambient condition using API thermohydrometer and correction table was used to correct the value of API gravity from the ambient condition to 60°F. Then carried out evaluations to obtain specific gravity and density as shown in Equ. 2.1 and 2.2.

$$\text{API gravity at } 60°\text{F} = \frac{141.3}{\text{Specific gravity at } 60°\text{F}} - 131.1 \qquad \textbf{(2.1)}$$

$$\text{Specific gravity at } 60°\text{F} = \frac{\text{Density of oil at } 60°\text{F}}{\text{Density of equal volume of water at } 60°\text{F}} \qquad \textbf{(2.2)}$$

2.2.4 Analysis of Total Base Number (TBN)

2.5 g of sample was weighed into the titration beaker and 60 mL of titration solvent (one volume of glacial acetic acid to two volumes of chlorobenzene) added and stirred. The titrant was prepared by adding 0.1 eq/L Sodium acetate in acetic acid (5.3 g of anhydrous Na_2CO_3 in 300 mL of glacial acetic acid, then diluted to 1 L). A Back titration was carried out by taking 4 mL of 0.1 eq/L $HClO_4$ excess in a pipette to be titrated with the titrant. At the end of the titration. TBN was calculated in mg of KOH per g of sample as shown in Equ. 2.3 and 2.4 (ASTM D2896-11, 2015).

Calculations:

$$TBN(mgKOH/g) = \frac{C_{back\ titrant}\ (eq/L) \times V_{back\ titrant} - C_{titrant}\ (eq/L) \times V_{titrant}\ (mL) \times M_{KOH}\ (g/mol)}{m_{sample}\ (g)} \quad \textbf{(2.3)}$$

$$= \frac{0.1\ (eq/L) \times 4\ (mL) - 0.1\ (eq/L) \times V_{titrant}\ (mL)}{2.5\ g} \times 56.11\ (g/mol) \quad \textbf{(2.4)}$$

2.2.5 Analysis of Total Acid Number (TAN)

The procedure in ASTM D664 was used as it is designed to determine the sum of total acid compounds that are present in petrochemical samples, using the principle of acid-base titration and the result expressed in mg of KOH per mass (g) of sample. 0.1 eq/L KOH was used as titrant; the solvent was a mixture of 5 ml water, 495 ml of isopropyl alcohol and 500 ml chloroform. .

Samples were non-aqueous and were diluted in a mixture of chloroform and isopropyl alcohol , when required.

The indicator was prepared with 200 ml of toluene and 2 ml of water as solvent, then 0.5g of phenolphthalein was dissolved in 10 ml of the solution of toluene.

The titration was carried out with 5.0 ± 0.5 g of the engine oil and 3 drops of indicator was equally added and titrate with the prepared KOH standard solution. At the end point, the color changes from colorless to pink. The titer value ($V_{titrant}$) was noted and the TAN calculated as shown in Equ. 2.5 and 2.6 (ASTM D664, 2023).

Calculations:

$$TAN\ (mgKOH/g) = \frac{C_{titrant}\ (eq/L) \times 0.1N \times V_{titrant}\ (ml)}{n_{e\text{-}titrant}\ x\ m_{sample}\ (g)}\ x\ MKOH\ (g/mol) \quad \textbf{(2.5)}$$

$$= \frac{0.1\ (eq/L) \times 0.1N \times V_{titrant}\ (ml)}{1\ x\ 5\ g}\ x\ 56.11\ g/mol \quad \textbf{(2.6)}$$

2.2.6 Analysis of Particulate Count

This was carried out using ASTM D7647. The sample was properly agitated and 20 ml taken in a conical flasks. 50 ml of reagent grade kerosene was added to the sample and mixed properly. The mixtures was filtered using vacuum filtration apparatus and dried filter paper of 10 microns . The analysis was carried out in triplicate and the particle count calculated as shown in Equ. 2.7 (ASTM D7647,2010; Quesnel & Geach, 2023)

$$\text{Particulate count (particles/ml)} = \frac{\text{particles}}{\text{volume of samples (ml)}} \quad \textbf{(2.7)}$$

III. Results and Discussion

The fresh lubricating oils used for this study are classified by the Society of Automobile Engineers (SAE) as SAE 20W-50; 20 determines the flow behavior of the oil at cold condition, such that at low temperature conditions the oil still performs its function of lubrication; this shows that the oil can remain in liquid state at cold condition more than another oil with a higher number at this position. The number after W "Winter" shows the Kinematic viscosity rating of the oil at 100 ^{0}C; here the higher the number, the more resistance it is for the oils to withstand loads (ADINOL, 2023). The standard specification of SAE 20W-50 lubricating oils for density at 15 ^{0}C is 889 kg/m^3 and the four unused oils for this work have their density in the range of 883.8-899.0 kg/m^3 (Fig.3.1); this could deduced that the fresh oils attended the standard specification for density of this category of engine oils (Ettefaghi *et al.*, 2013; REPSOL, 2018). Fig. 3.1 also shows that the used oil samples were denser than the unused oil samples. This shows that within the period of running the engine oils in the automobile, the oils performed functions of cleaning the engine block preventing it from malfunction, alongside its primary function of providing lubrication to ease friction in the engine block.

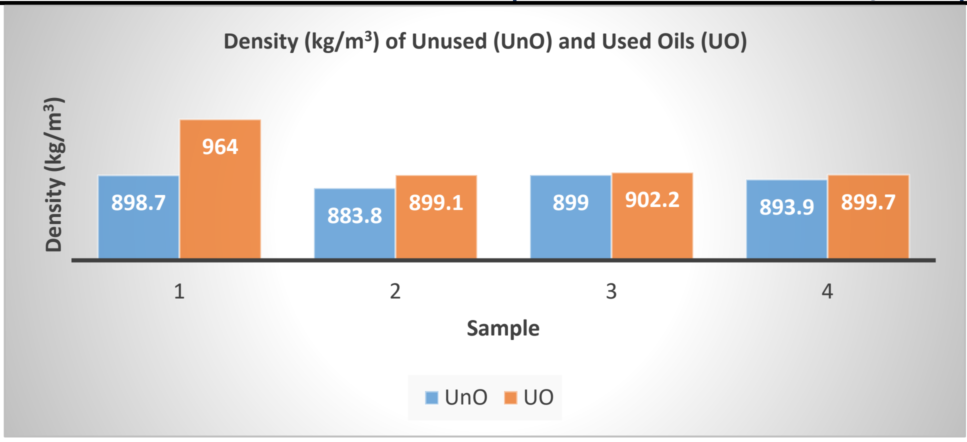

Figure 3.1: Density (kg/m^3) of Unused Oils (UnO) and Used Oils (UO)

The percentage increase in particulate count from unused oil to used oil samples is 99.59 - 12,300 % as shown on Fig. 3.2, while the percentage increase in density from the unused oil to used oil samples is 0.36 - 7.27 % (Fig. 1). This could show that the contaminants in the used oils were not only solid suspended particles from wearing parts of the engine blocks, fluids from byproducts of combustion and other sources were likely to be more dominants as contaminants than the suspended particles.

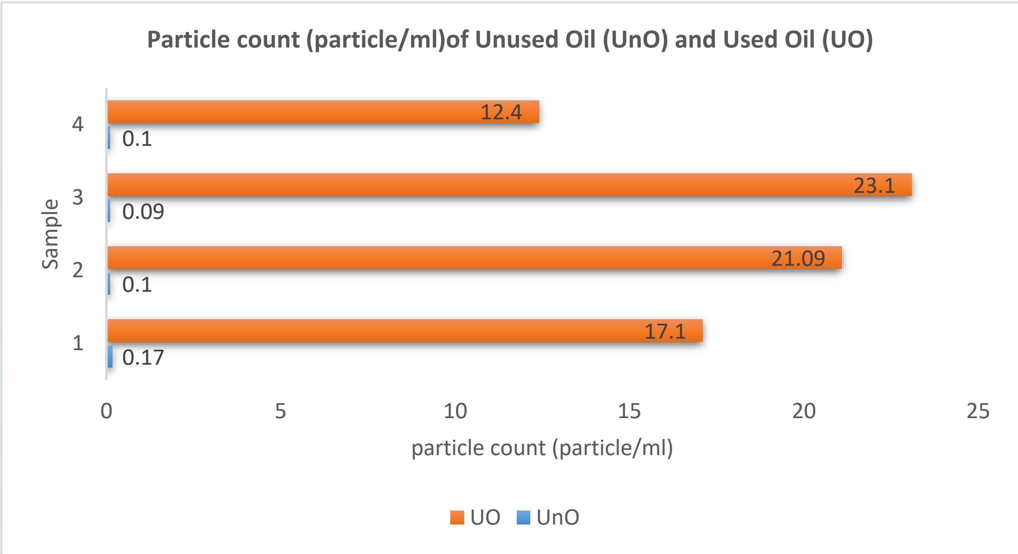

Figure 3.2: Particle count (particle/ml) of Unused (UnO) and Used Oils (UO)

The combustion process in the engine block leads to the formation of acid anhydrides which react with water to form acids. This could be the reason for increasing amounts of total acid number (TAN) from the unused oil samples to the used oil samples as shown on Fig. 3.3. The total acid number From the unused oil samples increased in the used oil samples by 243.70 - 586.80 % (Fig. 3.3). This may cause the engine block to be prone to corrosion via chemical attack. To adjust the effects of acids generated in the engine block, the lube oils are formulated to contain acid neutralizing substances as additives. The technical specification of TBN in automobile lube oils that is to make for acid produced is 10.8 mgKOH/g . The range of TBN in the used oil samples was 4.94-6.8 mg KOH/g as shown on Fig. 3.4; this is an indication that the engine oils were due to be drained from the engine block and replaced as the amounts were below the technical specification for TBN in automobile engines (Papadopoulos, 2016; REPSOL, 2018).

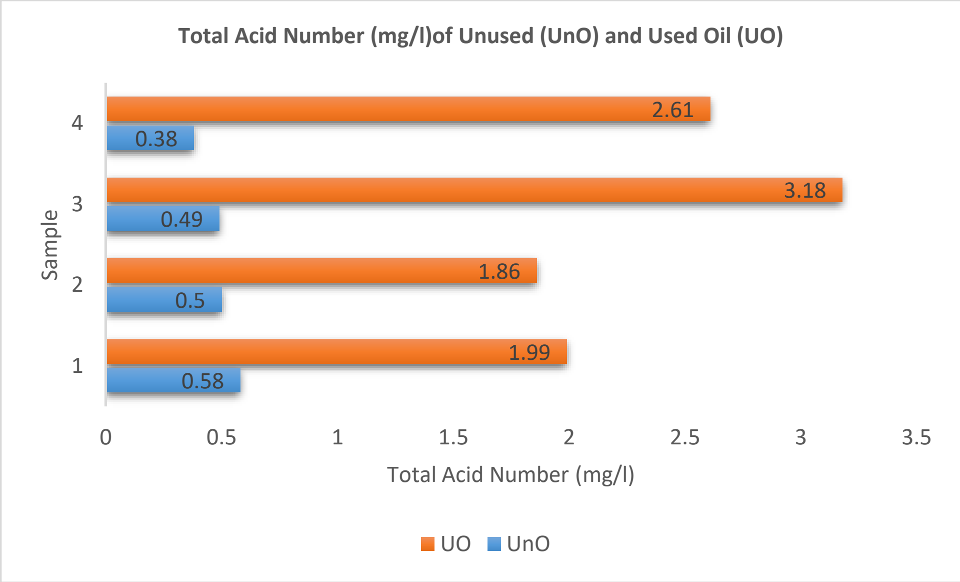

Figure 3.3: Total Acid Number (mg/l) of Unused (UnO) and Used Oils (UO)

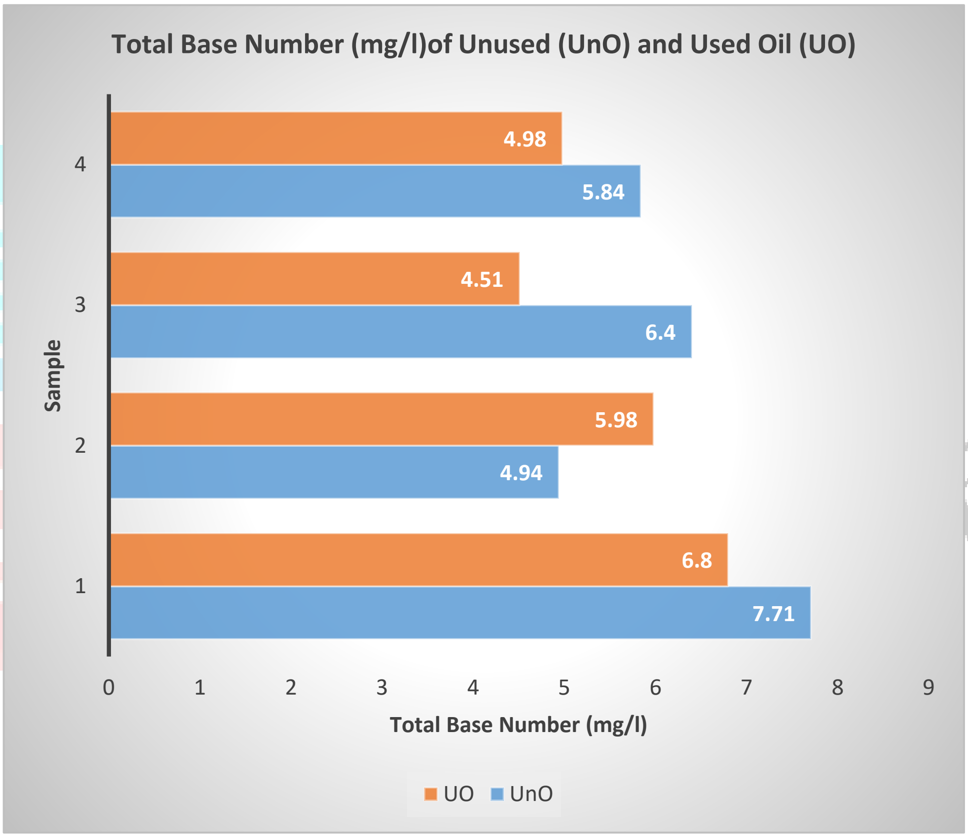

Figure 3.4: Total Base Number (mg/l) of Unused (UnO) and Used Oils (UO)

Table 3.1: Metal Content (ppm) in Unused (UnO)

Sample	Mg	Al	Sn	Fe	Zn	V
UnO_1	0.18±0.0015	<0.01	1.64±0.03	<0.01	<0.01	0.14±0.0011
UnO_2	0.090±0.0002	0.02±0.0006	1.20±0.025	<0.01	<0.01	<0.01
UnO_3	0.11±0.001	<0.01	0.86±0.005	<0.01	<0.01	<0.01
UnO_4	0.18±0.00012	<0.01	0.84±0.002	<0.01	<0.01	<0.01

Data were taken with analysis carried out in triplicate, standard deviation was calculated with P-value = 0.05

Among the six metals investigated, only Mg and Sn were found in significant amounts in the fresh lubricating oils (Table 3.1). This may be due to the used of Mg as detergent and dispersant additive so as to maintain the suspension of particulate matter in the oil. Zn and Sn with densities of 7.133 and 7.310 g/cm^3 respectively are used as anti-wear, anti-oxidant, and corrosion inhibitory additives and the four fresh oils used for this work chose Sn over Zn. The other analyzed metals Al and V might not have had useful applications in the fresh oils (Negash, 2016).

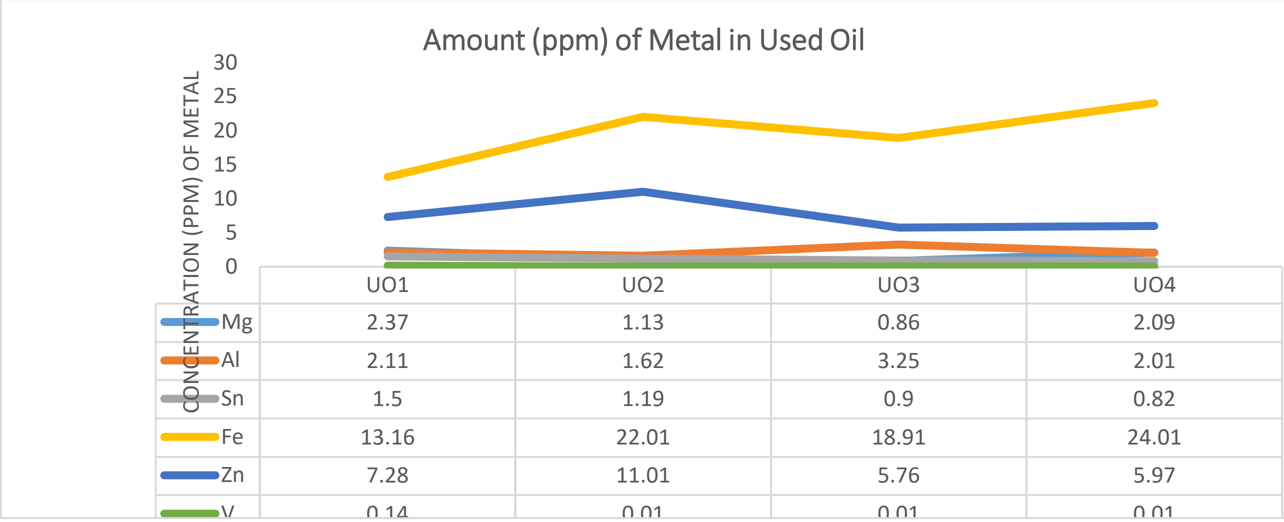

Figure 3.5: Amount (ppm) of Metals in Used Oil

Sánchez-Alvarracín *et al.*, 2021 showed the expected limits of Al and Sn in used automobile oils to be < 20 ppm and that of Fe as < 100 ppm, no limits were shown for Mg, Zn and V. The values on Fig. 3.5 showed that the metal contents in the spent oil samples were far below the maximum limits.

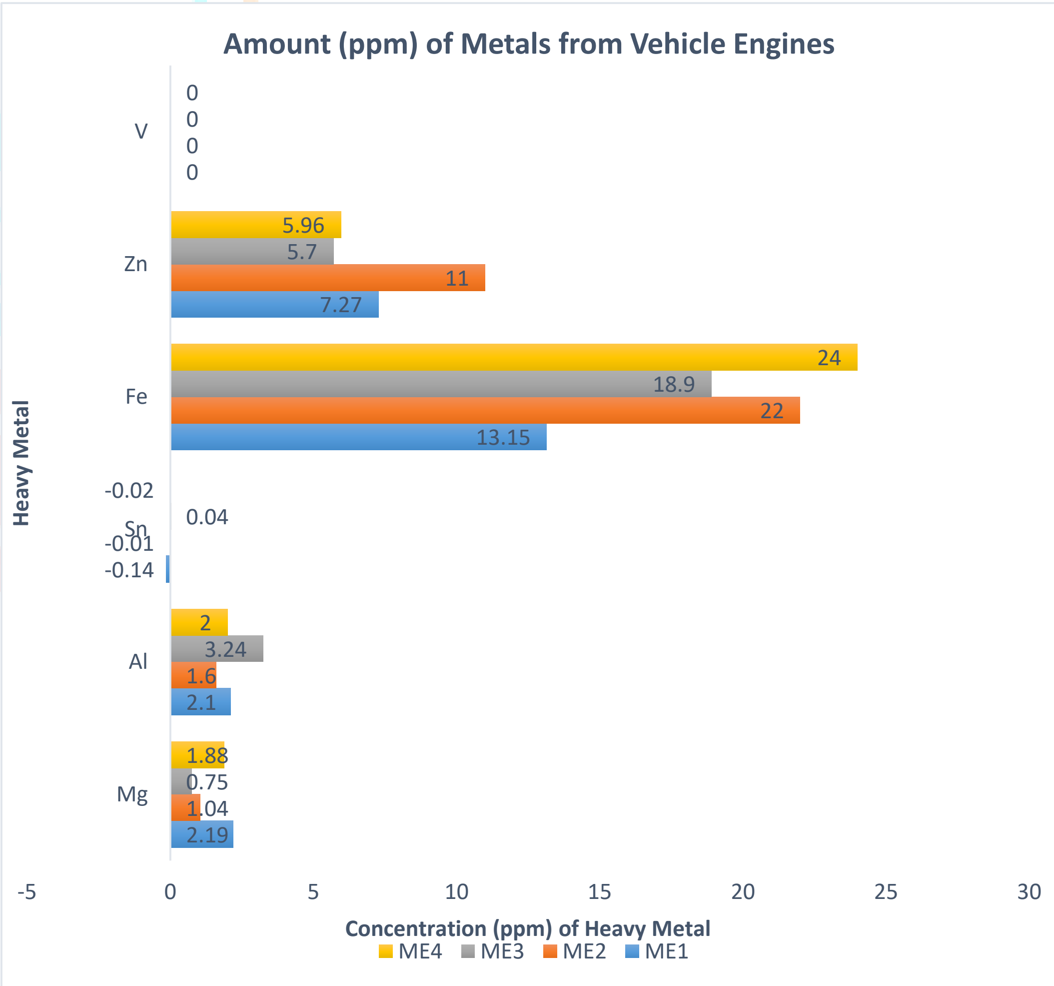

Figure 3.6: Amount (ppm) of Metals Extracted from Vehicle Engines to the Used Oils.

Comparing the distribution of metal contents in the fresh oil (Table 3.1) to the distribution of metals extracted from the vehicle engines to the used oils (Figure 6), it is observed that Sn which was dominant in the fresh oil followed by Mg appeared in least amounts in the used oil, except for vanadium. The boiling points of the considered metals arranged in increasing order is Zn < Mg < Al <Sn < Fe < V. It is expected that Zinc metal with a boiling point of 907 ^{0}C would have been vaporized first from the oils rather than metals with higher boiling points such as V, Sn and Mg with boiling points of 3380, 2602 and 1091 respectively. So, the reduction in the amount of Sn and Mg in the spent oil samples may be due to the fact that these metals were used up for their functions as additives and in the process denatured. The estimated temperature in the automobile engine is 350 - 400 ^{0}C; this is not sufficient to emit any of the considered metals into the gaseous phase as well as subsequent emission via the automobile exhaust.

On Fig. 3.7, Iron is the most dominantly extracted metal into the engine oil from the engine block; the sources of iron in the engine are cylinder liners, camshaft lobes, crankshaft and oil pumps (Negash, 2016). The crank cases, cylinder blocks, heads and pistons are made from Aluminum metals as well as other metals. The amounts of metals extracted into the spent oil arranged in decreasing order is Fe > Zn > Al > Mg > Sn > V.

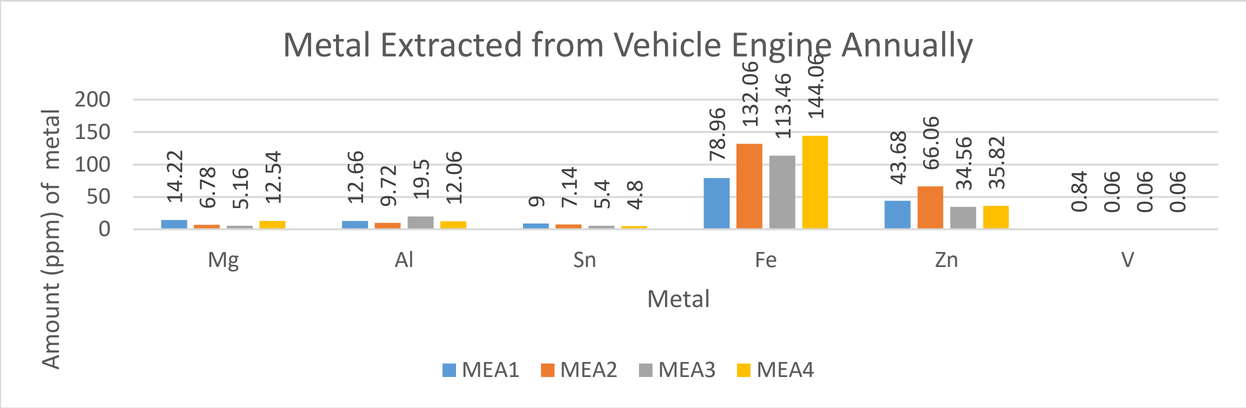

Figure 3.7: Amount of Metals Contained in Used Oils Estimated Annually

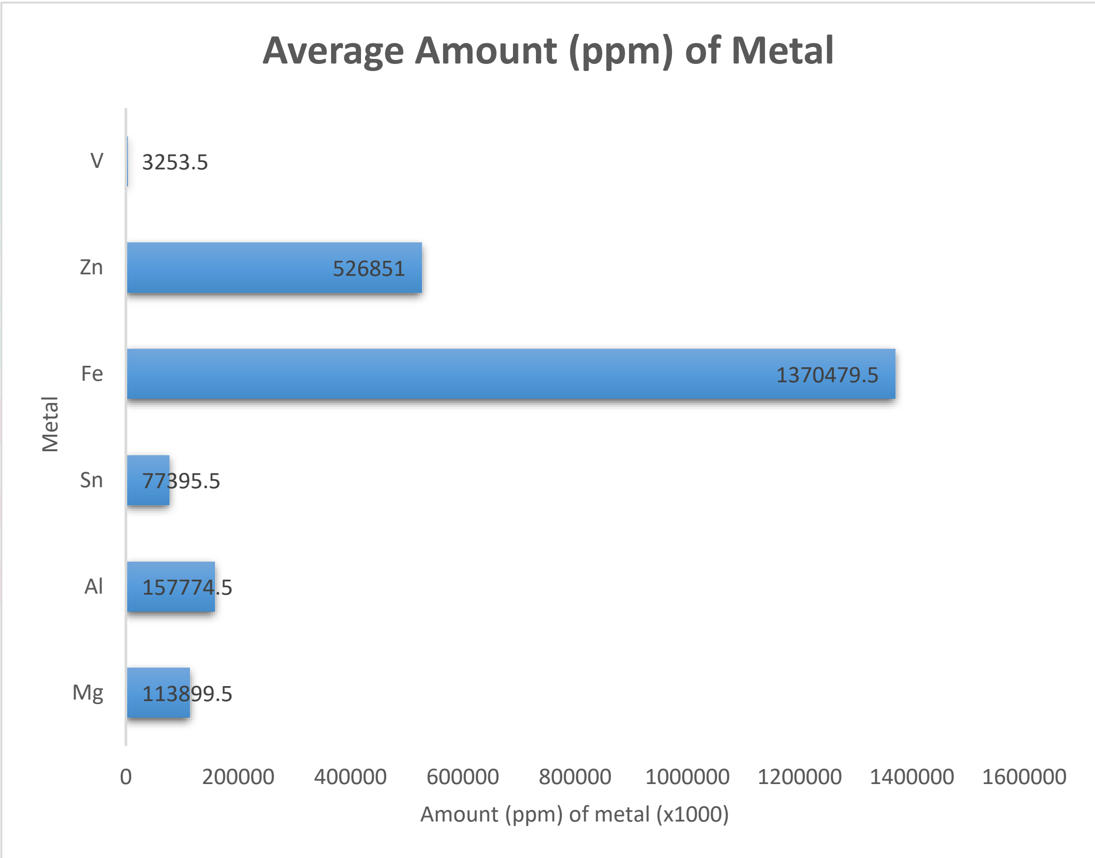

Figure 3.8: Amount of Metals Accumulated in the Environment from Used Oils Estimated for the Year 2020.

Fig. 3.8 was evaluated from the total number of 11,700,000 automobile vehicles registered in Nigeria in 2020, using the annual average of the amount of the four samples for each metal analyzed in the spent oils as shown on Fig. 3.7. Nigeria is a country with a population of about 210 million in 2020 and numbered above 2020 million towards the ending of 2023 and increasing in population yearly like that of any other country. The data on Table 1.1 shows that iron metal detected with the highest amount in the used oil sample has a maximum permissible limits of < 59,000 ppm as total in soil, water and air. The estimated accumulation of Fe contributed from spent automobile oils in this work is 1,370,479,500 ppm annually; this is greater than the maximum permissible limit in the environment by 23,000 times, while Mg, Zn and Al are 581,000, 879,000 and 965,983 respectively times greater than the maximum permissible limits in the environment. Evaluation for V and Sn were not considered because these metals were not present in the spent oil samples; Sn was likely to have been used-up in the process while V was present in trace amounts in the fresh oil samples.

The two major source of exposure of this metals to human is inhalation from aerosols (as the beginning of this study has shown that these metals cannot be found easily in the air through evaporation because of their extremely high boiling points) and ingestion through vegetables. Table 1.1 has shown that the maximum permissible limits for Fe, Zn and Sn in vegetables are 425.50, 99.40 and 50.00 respectively; it could be possible for plants classified as vegetables to absorbed these metals above their permissible limits, especially in regions where adequate management of used engine oils is not in place.

WHO (2008) and Johnson (2023), have outlined the health risks associated with the overload of these metals to human. This include hemochromatosis (genetic disorder), vomiting, diarrhea as well as damage to intestine and other organs for Iron; alzheimer (memory loss) disease associated with Aluminum; anorexia (losing of weight), vomiting and diarrhea, interference with Copper metabolism causing impaired immunity, low blood Copper levels, red blood cell microcytosis as well as neutropenia (low count of a type of white blood cell) for Zinc overload; excess of Tin could cause argyria (this is identified with bluish-gray colour of the skin and nails); Magnesium overload in human could cause digestive issues, lethargy (feeling sleepy or fatigued and sluggish) as well as irregular heartbeat and Vanadium overload have been identified to be carcinogenic, causing cancer of the lungs.

V. Conclusion

Engine oils reduce the hash effects that could be caused in mechanical systems with moving parts by reducing frictional forces; this is indispensable to the use of machines. The outlined risks of accumulation of metals in the environment; contributed from engine oils is an indication that if spent lubricating oils are not properly managed, our environment may be too hash for human and other biota to survive in due to the estimated toxicity of these metal pollutants.

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