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Recycling and characterisation of spent lubricating oil

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Abstract: Ten samples of spent lubricating oil (SLO) were obtained from different road side mechanics in Minna, Niger State, Nigeria; five samples were collected from both premium motor spirit (PMS) and automotive gas oil (AGO) engines respectively. Samples were digested to reduce organic, hydrocarbon and organo-metallic compounds. Samples were analysed using atomic absorption spectrophotometer (AAS). Metals average concentration (ppm) in PMS and AGO samples were as follows: 1.1, 1.43; Iron 4.78, 10.10; copper 0.39, 0.19; zinc 10.5, 17.43; chromium 2.4, 3.83; magnesium 0.633, 0.60 and calcium 135.39, 128.06. Results showed that copper and magnesium were the least concentrated while calcium has the highest concentration. SLO was recycled using locally sourced soap (surfactant) and tetraoxo sulphate (VI) acid, H₂SO₄, producing cleaner SLO suitable for combustion in industrial burner. Recycled SLO was found to have a specific gravity of 0.8972, water content of 10%wt, carbon residue of 3.89% and a flash point of 127°C which complied with standard properties given in the literature. It can be inferred from various analyses conducted that the treatment method employed is efficient and suitable for SLO treatment.

Keywords: recycle; digested; surfactant; spent lubricating oil; SLO; standard properties.

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1 Introduction

Lubricant undergoes chemico-physical transformations during use making it unsuitable to perform its original function; thus, require replacement. Spent lubricating oil (SLO) constitutes one of the major pollutants today, especially from automotive industries and automobiles where they are constantly generated and subsequently disposed. They present the largest amount of liquid, non-aqueous hazardous waste on earth. One of the serious pollution problems worldwide is related to SLO, when dealing with SLO there are two main issues to be solved: lack of sound SLO management and need for efficient technologies to regenerate and/or remediate SLO (Ekanem et al., 1997; Dalla et al., 2003; Kanokkantapong et al., 2009).

Environmental highly toxic polycyclic aromatic hydrocarbons (PAHs), polychlorinated biphenyls (PCBs) and heavy metals are the major contaminants in SLO. When indiscriminately disposed, and released to the environment, for example, water courses, it hindered O₂ from atmosphere and sunlight penetration into water which eventually endangered aquatic lives. Heavy metals and PAHs presence in SLO which is widely belief to be the cause of chronic (long term) hazard including carcinogenicity are introduced to the environment thereby endangering lives of plants, animals, humans and environment. Heteroatoms, such as, bromine, chlorine, nitrogen and sulphur presence in SLO also poses serious problems for its secondary use as fuel or any other applications. Hence, suitable methods and catalyst/sorbents for removing them have been developed (Lukanin, 1990; Roy, 1997; Bhaskar et al., 2004; Oando Training Manual, 2004; Cao et al., 2009; Kanokkantapong et al., 2009).

SLO is recycled to preserve natural resources, protect environment from hazard and pollution. Thus, it is recycling and treatment has become very popular and generated serious concern in modern society. Recycling keeps SLO away from rivers, stream, ground water, beaches, livestock, wildlife and etcetera. For example, if one gallon of it is reprocessed and burned as fuel it will generate enough electricity to power everything in a home for a day. Recovering SLO can be defended on the energy saving and prevention of pollutants to the environment, extensive studies are necessary to justify the promotion of recycling and recovery of SLO. It represents an important energy resource that if properly managed and reused, it could reduce dependency on other fuels and importation of lubricating oil (Lukanin, 1990; Bhaskar et al., 2004; Cao et al., 2009; Kanokkantapong et al., 2009; Josiah and Ikiensikimama, 2010; Njoku et al., 2012).

Recycling of SLO has to do with the mechanism and processes involved in removing the impurities in the SLO and bring it to the initial state. Recycling of SLO has been done using membrane filtration employing polyether sulfone (PES), polyvinylidene fluoride (PVDF) and polyacrylonitrile (PAN). Hollow fibre polymeric membranes and efficiency of the membrane separation was characterised using physical and chemical properties of the oil plus membrane rejection. Recycling involves two unit operations: dislodging using H₂SO₄ as reagent and adsorption using activated clay as reagent (Kalnes et al., 1989; Lukanin, 1990; Bhaskar et al., 2004; Cao et al., 2009; Kanokkantapong et al., 2009; Josiah and Ikiensikimama, 2010; Njoku et al., 2012).

Alternatively, SLO has been recycled by combustion process to extract its heat value. Likewise, base-oil regeneration from it has been achieved. SLO is often dumped on garbage, heap, land and sewage systems. Most SLO is incinerated or used as fuel in space

heater, cement kilns or sent for re-refining. It is burning and improper disposal destroys excellent resources. It presents enormous threat to animals, humans, plants and environment. For instance, taste of one million gallons of portable water can be ruin if contaminated by just a gallon of SLO and toxic components present in burnt lubricating oil often escape to the environment with flue gas. They also damage furnace in extreme cases causing serious environmental pollution. Another innovative novel re-refining process is direct contact hydrogen (DCH) which provides a cost effective means of recycling and treating hazardous SLO. Recycling and re-use of contaminated resources are obviously much more preferable to their improper disposal or destruction. The former will preserve natural resources and address problem of environmental pollution. Besides, treating SLO polluted water by conventional methods is difficult. Thus, minimising SLO by recovery and its efficient usage are excellent solutions for its management (Kalnes et al., 1989; Clare, 1997; Morawski, 2000; Bhaskar et al., 2004; Kanokkantung et al., 2009; Josiah and Ikiensikimama, 2010).

A lot of research work has been conducted on recycling and utilisation of SLO by valorisation, co-pyrolysis of SLO and coals. In addition, thermal and catalytic treatment of SLO into chemical feedstock or fuel oil is commonly practise. This preserves valuable petroleum resources and protect environment from toxic and hazardous components of SLO. It can be re-refined to base oil. For example, 1.5 Kg of SLO will produce 1 Kg of high quality-base oil besides other oil products: gas oil, fuel, fluxed bitumen and etcetera. Studies on SLO regeneration have shown significant energy and emission benefits relative to virgin-base oil production (Kalnes et al., 1989; Dalla et al., 2003; Bhaskar et al., 2004; Kalnes et al., 1989; Kanokkantung et al., 2009; Josiah and Ikiensikimama, 2010).

A common and standard method of digestion is by using concentrated acids known as acid digester. This involves the use of concentrated tetraoxo sulphate (VI) acid (H_2SO_4) followed by Trioxo Nitrate (V) acid (HNO_3). Most soaps removes grease and other dirt because some of their components are surface active agents or surfactants which have molecular structure that acts as a link between water and dirt particles, loosening particles from underlying fibres or other surfaces to be cleaned (Josiah and Ikiensikimama, 2010).

This research work intends to recycled SLO into useful products; identify type of metals present, evaluate their concentrations (ppm) and extent of toxicity in SLO.

2 Materials and method

Samples were collected in rubber sampling bottles, which were washed using distilled water and tight-fitting caps were used to prevent foreign matter from entering the bottle. In all, ten different samples were collected in which 5 were from premium motor spirit (PMS) engines and 5 from automotive gas oil (AGO) engines. Data, time and source of samples were recorded. Samples collected were examined within six days of collection (Cao et al., 2009). Every sample bottles were labelled, in format: PMS-1, PMS-2, PMS-3, PMS-4, PMS-5 and AGO-1, AGO-2, AGO-3, AGO-4, AGO-5. Sample bottles were kept close until the time they were required to be filled up. Parameters of the samples investigated were: calcium, magnesium, zinc, iron, copper, lead and chromium.

2.1 *Experimental procedure*

Twenty cm³ of sample to be analysed was transferred into a conical flask and 20 cm³ of concentrated tetraoxo sulphate (vi) acid was added. The mixture in the conical flask was slowly heated in a sand bath at 60°C. This temperature was maintained for three hours with constant mixing. The mixture in the conical flask was then heated to 120°C for the acid to evaporate. It was allowed to cool down, then 6 cm³ of concentrated trioxonitrate (v) acid was added. Temperature of the mixture was gradually raised to 60°C and maintained until the solution became homogeneous. The mixture was heated further to 100°C for the acid to evaporate until 1 cm³ was left inside conical flask. This amount was added to 100 cm³ capacity volumetric flask and distilled water was then added to make it up to 100 cm³. Filter papers were used to remove residues and filtrate was then collected in new sample bottles and labelled accordingly. The samples were then analysed using atomic absorption spectrophotometer (AAS) machine.

2.2 *Recycling procedure*

200 cm³ of the sample was transferred into a beaker and 12 cm³ of concentrated tetraoxo sulphate (vi) acid, H₂SO₄ (5.6 wt % of used oil) was added. Mixture of the used oil and tetraoxo sulphate (vi) acid was stirred continuously under heat at 60°C for 20 minutes. 9 g of dissolved soap (surfactant) was added and stirred continuously. The mixture was then allowed to stand undisturbed for 24 hours, this allowed the mixture to separate into two phases, that is, oil and water (aqueous)-base. Clarified oil at upper layer was decanted and both residue and supernatant were separated. Aqueous phase contains acid, SLO contaminants including metals and some of the additives. Finally, the product was analysed to obtain its physical properties.

2.3 *Analyses of samples*

2.2.1 *Carbon residue test (micro-method IP 398)*

0.5 g of the sample was transferred into the bottom of the vial and the vial loaded into the vial holder. The vial holder was then transferred into the oven and the lid was shut. Sample was heated to 500°C for 15 minutes after which the oven was disconnected from power source and allowed to cool under pure nitrogen at 600 cm³/minute. When oven temperature was exactly 250°C, vial holder was removed and placed in a desiccator for further cooling. The residue was removed, weighed and the carbon residue was calculated.

2.2.2 *Flash point test*

Cleveland open cup tester was filled so that the top of the meniscus was exactly at filling line. Test flame light diameter reading was adjusted to 4.8 mm and heat was applied on the heating plate. As the flash point was observed, temperature reading was taken immediately using thermometer and recorded. This thermometer reading indicated the flash point. The flash point test was carried out inside fume cupboard.

2.2.3 Specific gravity at 15°C

Sample was transferred into a clean hydrometer cylinder; hydrometer was depressed into the sample in the cylinder and then released. Sufficient time was allowed for the hydrometer to come to rest, floating freely away from the walls of the cylinder. Afterwards, hydrometer reading was taken and recorded. Steady sample temperature was also noted and recorded at that time.

3 Results and discussion

Adverse health effect that do occur as a result of contamination of well-water, streams, rivers, beaches and farmland. Through indiscriminate dumping of SLO could be averted if it could be recycled into other useful products. Cost of energy is rising incredibly globally. Most industries in the world, particularly those located in Nigeria are finding it difficult getting return on their investment. This is because of the fact that the cost of firing furnaces, boilers, burners and etcetera is going up almost on a daily basis due to the changing prices of oil in the international market. Use of industrial burner oil produced from recycled SLO will reduce the price of generating energy thereby reducing the cost of doing business and also protect the environment. Furthermore, SLO is an excellent and cheap way of producing industrial burner oil which is a very good energy alternative. This study was carried out because used oils are major source of air, water and soil pollution.

Table 1 Analysis of recycled SLO

<i>Test</i>	<i>Recycled oil for combustion</i>
Appearance	Dark
Specific gravity (at 15°C)	0.8972
Flash point °C	127
Carbon residue % wt	3.89
Water content % wt	10
Ash content % wt	NA

Note: NA – not available.

Table 2 Concentration of metals (ppm) in SLO samples from PMS engines

<i>Samples</i>	<i>Lead</i>	<i>Iron</i>	<i>Copper</i>	<i>Zinc</i>	<i>Chromium</i>	<i>Magnesium</i>	<i>Calcium</i>
1	1.20	1.00	0.00	15.26	2.40	0.77	150.30
2	0.90	8.90	0.00	5.1	0.75	0.60	129.68
3	1.05	3.40	0.00	8.86	3.00	0.46	140.28
4	1.20	1.80	1.003	17.4	1.85	0.775	96.20
5	1.15	8.80	0.96	6.10	4.00	0.56	160.32

Table 1 shows the result of the analysis carried out on recycle SLO. Table 2 shows concentration of metals (ppm) in SLO samples from PMS engines. Table 3 shows concentration of metals (ppm) in SLO samples from AGO engines. Table 4 shows percentage concentration of metals in SLO samples from PMS engines. Table 5 shows

percentage concentration of metals in SLO samples from AGO engines. Table 6 shows average concentration (ppm) of metals in SLO samples from PMS and AGO engines. Table 7 shows average percentage concentration of metals in SLO samples from PMS and AGO engines while Table 8 shows AAS concentration standards for metals.

Table 3 Concentration of metals (ppm) in SLO samples from AGO engines

<i>Samples</i>	<i>Lead</i>	<i>Iron</i>	<i>Copper</i>	<i>Zinc</i>	<i>Chromium</i>	<i>Magnesium</i>	<i>Calcium</i>
1	1.65	12.10	0.52	10.20	3.00	0.475	144.39
2	1.65	11.601	0.00	9.35	4.09	0.99	156.31
3	1.103	11.60	0.30	16.2	5.10	0.396	120.24
4	1.50	8.80	0.00	22.6	3.90	0.64	104.21
5	1.27	6.40	0.00	28.8	3.05	0.495	80.16

Table 4 Percentage concentrations of metals in SLO samples from PMS engines

<i>Samples</i>	<i>Lead</i>	<i>Iron</i>	<i>Copper</i>	<i>Zinc</i>	<i>Chromium</i>	<i>Magnesium</i>	<i>Calcium</i>
1	0.70	0.59	0.00	8.93	1.40	0.75	87.90
2	0.62	6.09	0.00	3.49	0.51	0.41	88.89
3	0.67	2.16	0.00	5.53	1.91	0.29	89.32
4	1.00	1.50	0.83	14.47	1.54	0.64	80.00
5	0.63	4.48	0.53	3.35	2.20	0.31	88.14

Table 5 Percentage concentrations of metals in SLO samples from AGO engines

<i>Samples</i>	<i>Lead</i>	<i>Iron</i>	<i>Copper</i>	<i>Zinc</i>	<i>Chromium</i>	<i>Magnesium</i>	<i>Calcium</i>
1	0.96	7.02	0.30	5.92	1.74	0.28	83.78
2	0.90	6.31	0.00	5.08	2.22	0.54	85.00
3	0.71	7.49	0.19	10.46	3.29	0.26	77.60
4	1.06	6.212	0.00	18.95	2.75	0.45	73.57
5	1.06	23.97	0.00	23.97	2.54	0.41	66.70

Table 6 Average concentrations (ppm) of metals in SLO samples from PMS engines

<i>Samples</i>	<i>Lead</i>	<i>Iron</i>	<i>Copper</i>	<i>Zinc</i>	<i>Chromium</i>	<i>Magnesium</i>	<i>Calcium</i>
PMS	1.1	4.78	0.39	10.50	2.40	0.633	135.39
AGO	1.43	10.10	0.16	17.43	3.83	0.60	121.06

Table 7 Average concentrations (ppm) of metals in SLO samples from AGO engines

<i>Samples</i>	<i>Lead</i>	<i>Iron</i>	<i>Copper</i>	<i>Zinc</i>	<i>Chromium</i>	<i>Magnesium</i>	<i>Calcium</i>
PMS	0.72	2.96	0.27	7.15	1.51	0.42	86.80
AGO	0.94	6.47	0.098	12.88	2.51	0.39	77.33

Table 8 AAS concentration standards for metals

Concentration (ppm)	Zinc	Iron	Copper	Chromium	Lead	Magnesium
1.00	0.040	0.017	0.002	0.455	0.208	0.544
2.00	0.055	0.033	0.004	0.772	0.238	0.743
3.00	0.115	0.050	0.006	0.896	0.361	0.816
4.00	0.150	0.075	0.008	0.941	0.377	0.883
5.00	0.200	0.080	0.010	1.092	0.388	0.915
6.00	0.225	0.102	0.012	1.134	0.404	0.968
7.00	0.265	0.120	0.013	0.000	0.000	0.968
8.00	0.300	0.137	0.017	0.000	0.000	1.000
9.00	0.361	0.143	0.018	0.000	0.000	1.010
10.00	0.375	0.173	0.020	0.000	0.000	1.017

Figure 1 Composite bar chart of metals concentration (ppm) in SLO samples from PMS engines (see online version for colours)

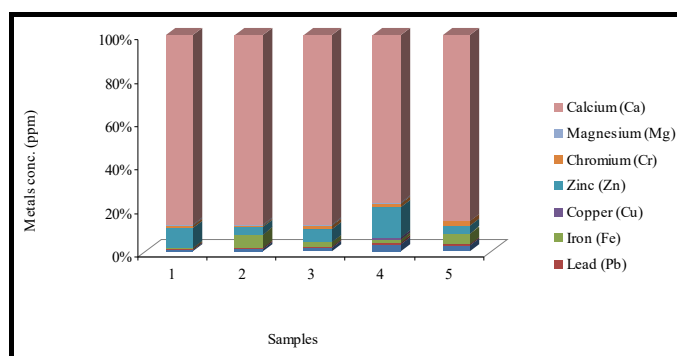


Figure 2 Composite bar chart of metals concentration (ppm) in SLO samples from AGO engines (see online version for colours)

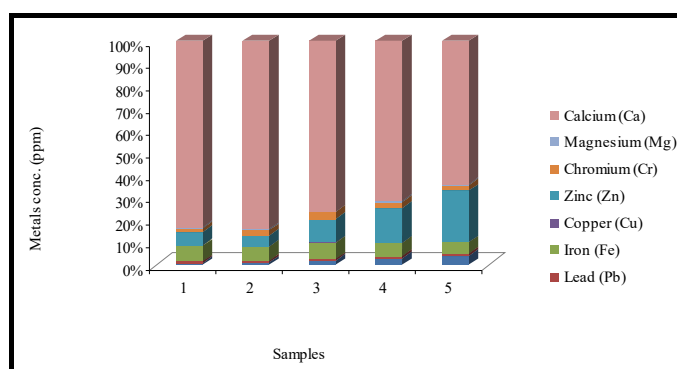


Figure 3 Composite bar chart of average concentration of metals (ppm) in SLO samples (see online version for colours)

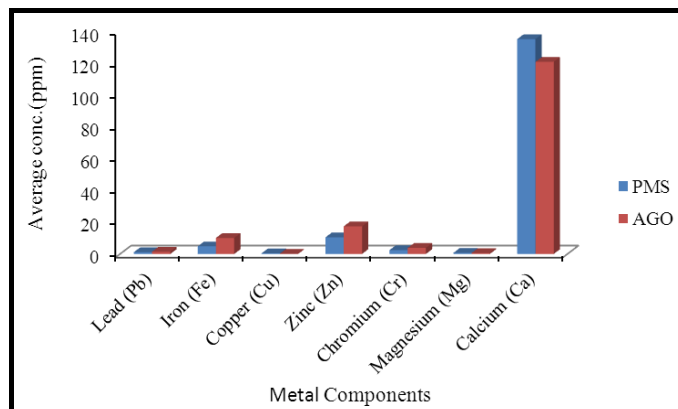


Figure 1 shows composite Bar chart of metals concentration (ppm) in SLO samples from PMS engines. Figure 2 shows composite Bar chart of metals concentration (ppm) in SLO samples from AGO engines. Figure 3 shows composite bar chart of average concentration of metals (ppm) of SLO samples.

From Table 1 dark appearance of SLO was as a result of the presence of soot or carbon residue which was 3.89% wt. in the spent oil. During recycling a considerable amount of soot was removed but a large quantity still remained, water content was 10%. Ash content could not be determined because of unavailability of equipment to carry out the test. Tables 2 and 3 show the concentration of metals (elements) in parts per million (ppm) in SLO samples from PMS and AGO engines. Tables 4 and 5 shows percentage concentrations of metals in waste oil samples from PMS and AGO engines. Also, Tables 6 and 7 show average concentrations (ppm) of metals in SLO samples from PMS and AGO engines while Table 8 shows AAS standards for metals.

From the results presented in Table 6, percentage concentrations of metals for samples obtained from PMS engines in order of increasing magnitude were: copper 0.27%, magnesium 0.42%, lead 0.72%, chromium 1.51%, iron 2.96%, zinc 7.15% and calcium 86.86%. Similarly, the percentage concentrations of metals for samples obtained from AGO engines in order of increasing magnitude were: copper 0.098%, magnesium 0.3885%, lead 0.938%, chromium 2.51%, iron 6.47%, zinc 12.88% and calcium 77.33% respectively. Average concentrations of some metals were higher in PMS samples, for these metals their differences were shown in parenthesis: calcium (14.33 ppm), magnesium (0.033 ppm), and copper (0.23 ppm), metals that were higher in AGO samples were: lead (0.33 ppm), iron (5.32 ppm), zinc (6.93 ppm), chromium (1.43 ppm).

From the results of average concentrations (ppm) of metals in SLO samples, it was observed that the concentrations of lead, iron, zinc and magnesium were very high. This shows that these metals were present in toxic quantities and can pose an environmental problem when waste oil is not properly disposed.

Average concentration of lead obtained from PMS and AGO engines oil samples were: 1.1 ppm and 1.43 ppm. Presence of lead could be from fuel additives, wear and tear. From literature, threshold limit value (TLV) and allowable concentration of lead in human system is not specified. Therefore, an accumulation of lead in human body can cause symptoms such as, hypertension, fatigue, haemolytic anaemia, abdominal pain,

nausea, constipation, irritability, impotence, depression and etcetera. Average amount of iron in PMS and AGO engines SLO samples were 4.78 ppm and 10.10 ppm. It has been confirmed from literature that full-blown iron intoxication can produce gastrointestinal damage, convulsion and coma. The permissible level of iron in portable water is 0.05 ppm. Also, concentration average of copper in PMS and AGO samples were: 0.39 ppm and 0.16 ppm. Presence of copper may be due to wear and tear. Though, the level was minute, it is worthy to note that an accumulation of copper in the environment is quite dangerous. High copper level may cause neurologic diseases characterised by tremors, dystopia, schizophrenia and etcetera. Copper accumulation mainly affects the liver and brain (Njoku et al., 2012).

Concentration average of magnesium obtained from both PMS and AGO waste oil samples were: 0.633 ppm and 0.60 ppm. Magnesium is part of base metal and also found in some lubricating oil additives. Excess of magnesium intake can cause abdominal pain, disorientation, convulsion and etcetera. Concentration average of calcium in PMS and AGO samples were: 135.39 ppm and 121.06 ppm. Presence of calcium is mostly from lubricating oil additives. Although excess of calcium in the body is not known to cause any terrible disease. But, its presence in water can cause hardness of water and deposition of scales in cooking utensils.

3 Conclusions

From the results obtained carrying out analyses on recycled SLO. It was observed that surfactants play a vital role in the agglomeration of the contaminants in SLO and that by its application. A clean SLO suitable for combustion in industrial burners can be obtained. It was also observed that SLO contains several metals such as, lead, iron, chromium, zinc in toxic concentrations which were introduced to the environment in slightly high proportion. Improper disposal of SLO poses a big environmental challenge. When these metals are found in human system beyond a certain limit, it has the potential to cause life threatening diseases characterised by symptoms such as, depression, abdominal pain, hypertension and haemolytic anaemia.

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