

Physical and Chemical Properties of Virgin and Used Engine Oils

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المخلص

نظراً لأهمية المواصفات القياسية لزيت المحركات و اضرار عدم مطابقتها، يتركز إهتمام هذه الدراسة على التحقيق في تدهور خصائص زيت المحرك لعلامات تجارية مختلفة منتشرة في السوق الليبية. من خلال إجراء اختبارات معملية لمقارنة نتائج لعدد محدود من العلامات التجارية بالمعايير القياسية الليبية و ذلك من حيث قابليتها للاستخدام في ظل ظروف المحرك الفعلية. تم قياس كلا من الخصائص الفيزيائية والتركيبات الكيميائية لزيت محرك البنزين للزيوت الجديدة والمستعملة لعينات من علامات الزيوت المشار إليها ، كما تم تحديد تركيزات العناصر المضافة الرئيسية (Na و Mg و Ca و Zn) و أيضاً المعادن الناتجة من الاحتكاك داخل المحرك (Fe و Cu)، وكذلك الخصائص الفيزيائية مثل اللزوجة عند 40 و 100 درجة مئوية ومؤشر اللزوجة و الكثافة ودرجة حرارة سكب و تبخر الزيت لعينات الزيت الجديدة و المستعملة بمسافة 5000 كيلومتر. كشفت النتائج عن انخفاض عام و متفاوت في جودة عينات الاختبار للعلامات المختلفة من حيث الخصائص الفيزيائية وانخفاض تركيز المواد المضافة للزيت مصحوبة بزيادة تركيز عناصر Fe و Cu وهو ما يمثل علامة على زيادة معدل التآكل . إلى جانب ذلك، كشفت النتائج عدم وجود تغير ملحوظ في نتائج الخصائص الفيزيائية للزيوت بعد استخدامها بما يتطلب دعماً بنتائج أخرى مثل تحديد تركيزات العناصر المضافة بما يمكن من تحديد جودة زيوت المحركات.

الكلمات المفتاحية: زيت المحرك المستعمل، الزيت المقلد ، مراقبة الزيت ، المضافات الزيتية.

Abstract

The importance of the standard specifications of engine oils and the damages of their non-conformity is investigated in this study. Comparison is made between genuine and counterfeit oils in terms of their response to usage under actual engine conditions. Physical properties and chemical compositions of gasoline engine oil are measured for new and used oils of different oil brands spread in the Libyan market. The concentrations of the main additive elements (Na, Mg, Ca and Zn) and wear metals (Fe, Cu) as well as the physical properties such as: viscosity at 40 and 100°C, viscosity index, flash point, pour point and density have been determined for fresh oil samples and after mileage of 5000 km. Results exposed a general trend of decline in oil quality in terms of deteriorated physical properties and decreased concentration of oil additives accompanied by increased concentration of Fe and Cu elements which is a sign of increased rate of wear. It also revealed that the quality of engine oils cannot be determined by the results of the physical properties only; they need promote it with chemical additives results, because the results of physical properties showed that there is no significant change of the oils after used.

Key Words: used engine oil, counterfeit oil, oil monitoring, oil additives.

1. INTRODUCTION

Automotive industry has been growing since the development of gasoline engine in the 1860s. In 2019, 67.1 million passenger cars were produced worldwide [1]. Development of automobile engines is going toward increasing performance, lower emissions, and higher fuel economy which could have not been possible without advanced lubricating oil formulation[2].

Engine lubricating oil serves number of functions to prolong the service life of the engine parts. These include reduction of friction and wear by forming a thin layer between rubbing parts, dissipation of heat, corrosion control, prevention of excessive deposit formation, dispersion of use-generated contaminants, and water separation or demulsibility [3]. Lubricating oil consists of base fluid and viscosity improver (72- 96%), and chemical additive (4-28%)

[4]. The main function of base fluid is to perform lubrication, i.e. minimize friction, hence heat and wear. Furthermore, base fluid serves as a carrier of chemical additives which are chemical compounds blended in the base fluid in relatively small quantities to improve its performance. They do so by enhancing desirable properties, introducing new and useful ones; and suppressing unwanted properties.

Engine oil additives typically serve a number of functions such as: anti-foaming (dimethyl silicone); anti-oxidants (zinc dithiophosphates); anti-rust and anti-corrosion (sulfonates materials, calcium and barium sulphonates); anti-wear (compounds of phosphorus, sulphur and chlorine) and detergent and dispersant (sodium hydroxide, calcium hydroxide, barium hydroxide and magnesium oxide). Most of these additives are surface active agents [5]. Typical composition of engine oil is shown in Table 1.

Table 1 Typical Concentration range of main additives in engine oils [6]

Component	Weight %	Component	Weight %
Base oil	71.5 to 96.2	Antioxidant/wear	0.1 to 2.0
Metallic detergent	2.0 to 10.0	Friction modifier	0.1 to 3.0
Ashless dispersant	1.0 to 9.0	Anti-foam agent	2 to 15 ppm
Zinc dithiophosphate film	0.5 to 3.0	Pour point dispersant	0.1 to 1.5

The optimal selection of lubricating oil is an important factor in engine performance and the service life of its parts. However, physical and chemical properties of lubricating oil do change gradually as a result of operation of the engine and depletion of additives. Manufacturers recommend oil change after certain mileage as to ensure engine oil does not degrade to an extent that could harm the engine. For instance, increased friction and wear in automobile engines due to deterioration of lubricating oil performance are accountable for failure of different engine parts such as bearings and gears, and energy losses due to the high friction. It is estimated that about 19% of the total energy generated

by the engine is wasted due to mechanical frictional [7]. This highlights the importance of monitoring the physical and chemical properties of engine oil during usage.

The first known application of used oil analysis dates back to the early 1940s in the United States. Railway companies conducted chemical analysis using simple spectrographic equipment and physical tests to monitor steam engine oil [8], this helped maintenance technicians make maintenance decisions. The benefits of oil analysis prompted The American Navy in 1950s to introduce spectrometric techniques to monitor their jet engine oil [9].

Currently a wide variety of analytical procedures and methods of oil analysis are in use such as potentiometry, polarography [10,11], atomic absorption spectroscopy, X-ray fluorescence spectroscopy, laser-induced break down spectroscopy [12,13,14], differential scanning gravimetry [15], spectrography [16], ferrography [17], mass spectrometry [18], and chromatography [19] These methods help determine the concentration of number of important elements and measure some physical properties of used oil such as viscosity, viscosity index, density, flash point, pour point, total acid and base numbers, and water content [20,21]. The resulting data are then used to diagnose the condition of the oil and its suitability for further use.

Counterfeit lubricating oils could pose great threat to automobile engines due to their inadequate functioning and to the environment which is further polluted by using these low grade oils.

Counterfeit lubricating oils have become serious issue for car owners in Libya over the past decade due to lack of government control over the local market as a result of the difficult political and economic situation after 2011 uprising and the consequent civil war.

In a previous study the current authors investigated the chemical composition of new gasoline engine oil samples representing the common brands in the local market using flame atomic absorption spectroscopy [22]. The study found that 70 % of them lack the recommended additives concentrations and therefore classified as counterfeit according to the Libyan standards [23] for lubricating oil additives [24].

The aim of this paper is to analyse samples of virgin and used gasoline oils from the local market. Chemical composition and

physical properties are to be compared for genuine and counterfeit engine oil to explore the differences in their response to operation conditions. The concentrations of essential additive elements (Na, Mg, Ca and Zn) and wear metals (Fe, Cu) in addition to the physical properties such as: viscosity at 40 and 100°C; viscosity index; flash point; pour point and density are to be measured.



2. MATERIALS AND METHOD

To evaluate the engine oils that spread in the Libyan market physically and chemically, nine samples of mineral engine oils were chosen to be investigated. Three of those nine samples are branded names globally, while the rest are just known locally. The physical properties including Viscosity Index, Flash point, Pour Point, Density and Kinematic Viscosity will be taking into consideration. In addition, the chemical compositions to find the concentration of the chemical additives will be tested before and after been used.

2.1 Investigated Materials

As above mentioned, Table 2 shows the nine engine oils and the coded number of each sample. For research purposes, from this point on, each sample of the chosen ones will be mentioned according to its sample code number instead of the oil name.

Table 2 Engine oils coding numbers

Sample Code	Oil Name	Commercial Package
A1	MANNOL (SAFARI)	
A2	Q8	

A3	LUKOIL	
A4	GOLD SERIES	
A5	BENDIX	
A6	ELISTER MAX3	
A7	SALALA OIL	
A8	GASOLINE MOTOR OIL	
A9	ZAWIA OIL	

2.2 Physical Properties

The physical properties to be tested in this study are showed as follow:

2.2.1 Flash Point

Basically, the Flash Point test deals with relatively low temperatures of liquid at which the vapour of the tested material mixes with air producing a flammable mixture then “flashing” could

happen when ignition source is sparked. In this study, the Flash Point test is conducted with the Pensky–Martens closed-cup flash-point test. The test apparatus, as shown in Figure 1, includes a closed-cup which holds any vapors generated and basically simulates the condition in which a potential source of ignition is accidentally occurred into a compartment. For this test, the oil sample is put into a cup and then closed by its fitted lid. The cup and the oil sample are heated and stirred; openings are then opened in the lid to permit air into the cup then after the ignition source to be plunged into the vapors to test for a flash. The standard test method is following the regulation of ASTM D93, EN ISO 2719 and IP 34[25].



Fig.1 Flash Point device [26]

2.2.2 Pour Point

The pour point of particular oil is the temperature below which the liquid loses its flow characteristics. It is characterized as the base temperature in which the oil can pour down from a measuring beaker. There are two standards to measure the Pour Point of the sample, namely, ASTM D97 for the manual method and ASTM D5949 for the automatic method. In this study, the manual method is applied by using the Pour Point test device shown in Figure 2. According to ASTM D97, the oil sample should be cooled in a cooling bath to permit the formation of paraffin wax crystals. At approximately 9 °C above the expected Pour Point, and for every consecutive 3 °C, the test jar is removed and tilted to be tested for surface movement. If the sample does not flow when tilted, the jar is held in horizontal mode for 5 seconds. In case it does not flow,

3 °C should be added to the corresponding temperature and the result is the Pour Point temperature [27].



Fig. 2 Pour Point device [28]

2.2.3 Density

The density test is done according to ASTM1298 by using a glass hydrometer which is a device implemented to find the relative density of liquids according to the concept of buoyancy, see Figure 3. The hydrometer is already calibrated and scale-graduated narrow stem with one or more properties such as density, relative density, or American Petroleum Institute (API) gravity of crude petroleum, petroleum products. The liquid to measure should be poured into the [graduated cylinder](#), and then the hydrometer is gently dropped into the liquid till it floats freely. The point at which the surface of the liquid touches the stem of the hydrometer correlates to relative density [29].



Fig. 3 Glass Hydrometer [30]

2.2.4 Kinematic Viscosity and Viscosity Index

The Kinematic Viscosity (KV) is tested for oil samples by using test method described in ASTM D455. The time is recorded for a specified volume of a liquid to flow under gravity crossing through a calibrated viscometer under a repeatable driving head and at a firmly controlled and capillary known temperature inside a viscosity bath. The KV is the value needed to be measure by finding the flow time of the oil and the calibration constant of the viscometer. The test should be done twice and the average value among these two results should be computed and considered as the KV. Figure 4 shows the KV test apparatus.[31] By using the value of the KV, the Viscosity Index (VI) can be calculated. Where the VI is an arbitrary dimensionless number that identify the lubricant oil and to what extend its viscosity can be affected by the change in temperature. If the VI is high enough (above 100) it can be said that the oil is less influenced by the change in temperature.



Fig. 4 Kinematic Viscometer [32]

2.3 Chemical Compositions

The chemical compositions of the collected oil samples are done by using a standard test method ASTM D1318 and the Atomic absorption spectrophotometer Device model (AA6800F), as shown in Figure 5. The spectrophotometer is an apparatus used to measure light at a specific wavelength. The apparatus consists of two main parts, namely, a spectrometer and a photometer. The spectrometer delivers light at a particular wavelength while the photometer measure the intensity of the light by computing the amount of light that a solution is able to absorb and applying Beer's law, the spectrophotometer can determine the concentration of a coloured

solution[19]. The test method is described by which requires the steps of converting the oil samples to inorganic solution by burn a few quantities from each sample to convert it to ash, then adding a Hydrochloric acid to the sample. Finally, to find the results in part per million (ppm), the next equation should be applied:

$$ppm = \frac{device\ reading \times solution\ volume}{solution\ weight} \quad (1)$$



Fig.5 Atomic Absorption Spectrophotometer [19]

3. RESULTS AND DISCUSSIONS

The analysis of the samples has been categorized into two parts, part one is physical properties analysis and part two is chemical additives analysis. The physical analyses have been done at the laboratory of Arabian Gulf Oil Company (AGCO) and the Chemical additives analyses have been implemented at Sirte Oil Company, Raslanuf Oil and Gas Company and Alzawia Oil Refining Company.

Physical Properties

For the above mentioned oil samples, it has been decided to make the travelling distance at nearly 5000 km (about 3000 miles) to show the real effect on the engine oil. Then, the samples collected after use and tested to find the values of the required physical properties. In Table 3, the values of the physical properties of the selected oil samples before and after being used are showed. In addition, the last row of the table is the Libyan standard for the same properties for ease the comparison.

Table 3 physical properties of the different oil samples

Code number	Flash Point °C		Pour Point °C		Density kg/m ³		Viscosity Index	
	N	U	N	U	N	U	N	U
A1	194	250	-12	-24	891	891	117	129
A2	200	242	-12	-18	883	897	128	133
A3	204	242	-12	-24	883	889	141	147
A4	200	250	-12	-21	887	892	126	126
A5	190	236	-12	-30	873	880	160	154
A6	190	252	-12	-21	879	883	144	139
A7	175	242	-12	-21	867	878	168	149
A8	184	238	-12	-21	878	884	197	145
A9	197	238	-12	-21	870	881	171	150
Libyan standard	230		-21		891		117	

Where N denotes to New oil and U denotes to Used oil.

To show the effect of engine oil running on the physical properties, the results obtained have been compared with Libyan stranded, that will help us to see if this oils satisfies the Libyan stranded or not.

3.1.1 Flash point

Figure (6) shows the change of flash point for oil samples.

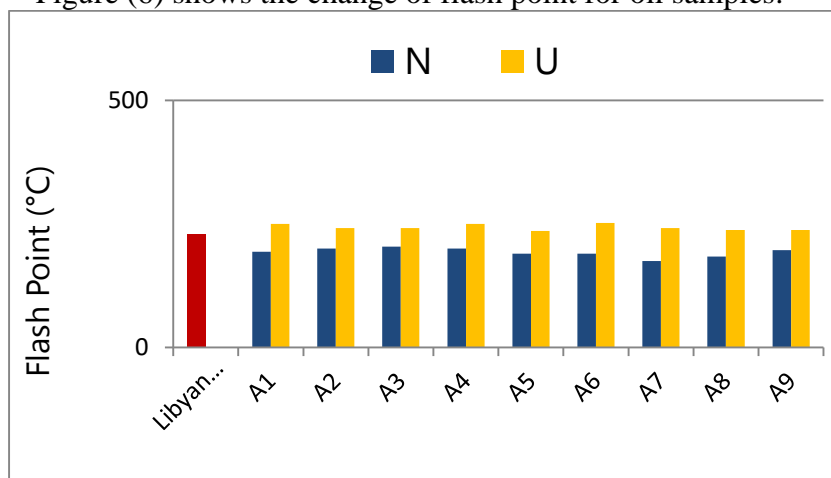


Fig. 6 Comparison the results of the flash point value of N and U samples with Libyan standard

from this figure we can see that the value of flash point for all N samples lowered the value of the Libyan standard, but the value of all the U samples are increased, due to flying light metals and remain heavy metals and these metals are difficult to evaporate so the degree of flash temperature increases.

3.1.2 Pour point

And from Figure (7) for a pour point we note that the value of all N samples and U samples number (A2 and A8) are exceeded the value of Libyan standard, and the value of U samples number (A4,A6,A7 and A9) remains constant, but the value of U samples number (1,3,5) are lowering, and this low because of degradations of additives, which were present in fresh oil as pour point depressants, in general the value of pour point for all samples are close to pour point of Libyan standard.

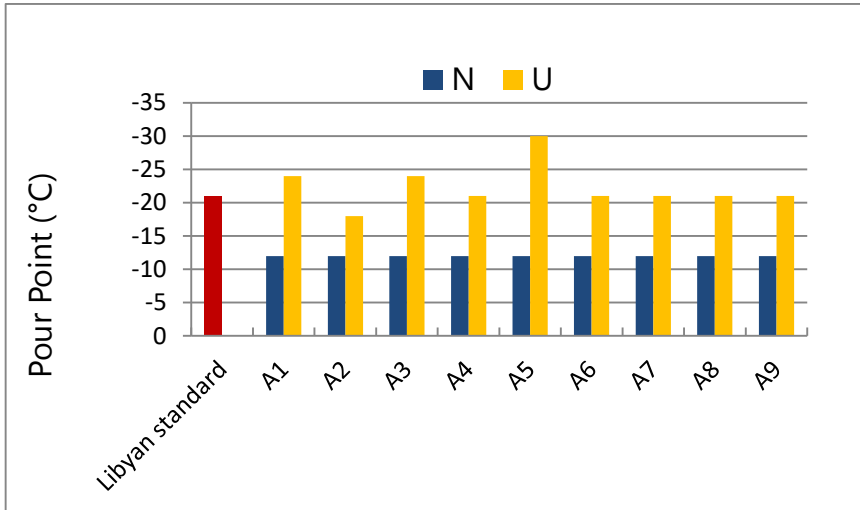


Fig. 7 Comparison the results of the pour point value of N and U samples with Libyan standard

3.1.3 Density

And for a density in Figure (8) we note that the value of N and U samples number (A3,A5,A6,A7,A8 and A9) and N samples number (A2 and A4) are lower than the value of Libyan standard, but the value of U samples number (A1,A2 and A4) are highly. As known the density of a lubricant increases, the fluid becomes thicker. This

leads to an increase in the amount of time it takes for particles to settle out of suspension and it can result in problems such as silt lock in the cavitations and corrosion, and this can cause failure. Knowing that, increase of density in oil after used is due to the presence of sludge and a higher percent of sulfur compounds, in general the value of density for all samples are close to density of Libyan standard.

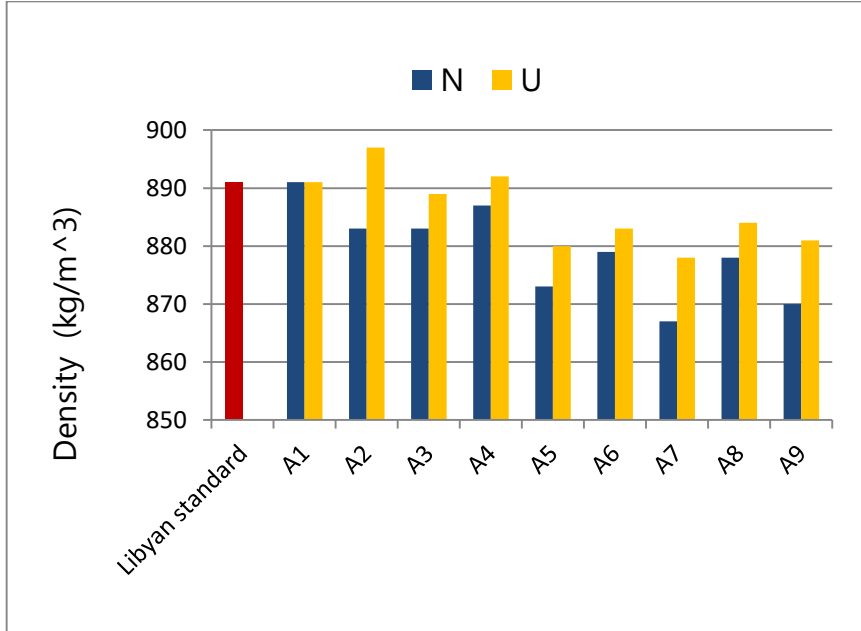


Fig. 8 Comparison the results of the density value of N and U samples with Libyan standard

3.1.4 Viscosity index

Finally, from Figure (9), we note that the value of the viscosity index for all samples is higher than the value of the Libyan standard. And we also note that the viscosity index for U samples number (A1,A2,A3 and A4) are higher than the same N samples and then reversed for the other samples. The reason for increase the value of viscosity index after used is due to exposure the oil to high heat from engine causing the shear of the viscosity index increase, the high temperatures can do shear/crack the oil molecules into smaller molecules, which causes a decrease in viscosity, and this mean not contribute to reduced fuel economy as well as poor oil circulation

throughout the engine, greater oil consumption, higher wear rates and increases emissions, in general the value of viscosity index for all samples are close to viscosity index of Libyan standard.

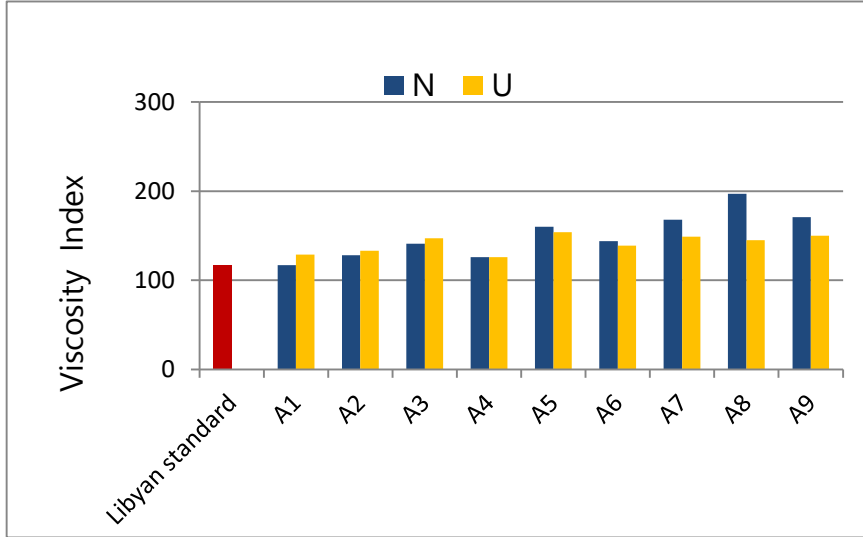


Fig. 9 Comparison the results of the viscosity index value of N and U samples with Libyan standard

From the physical properties analysis, we can note that:

1. After the comparison with Libyan specifications standard for all corresponding samples as presented in the previous figures, almost the samples before and after used have a values close to the Libyan Standers specifications. Therefore, the distinguish between the samples according to physical properties is insufficient.
2. By dealing with the samples before used, we didn't notice any peculiar smell in any sample.
3. For the N samples color's, all samples are transparent by sight as it is listed in the Libyan Standers Specification.

3.2 Chemical Compositions

In Table 4, the values of the chemical additives of the selected oil samples before and after being used are showed. In addition, the last

row of the table is the Libyan standard for the same additives for ease the comparison.

Table 4 Chemical additives of the different oil samples

Code number	Zn (ppm)		Ca (ppm)		Mg (ppm)		Na (ppm)	
	N	U	N	U	N	U	N	U
A1	473.1	491	2227	1514	1.519	17.9	22.63	59.24
A2	1132	648	3083	2417	11.81	68	4.33	66.25
A3	1151	790	3023	2272	88.33	134.9	4.75	46.5
A4	29.73	161	134.1	329	50.18	11.6	3.06	25.8
A5	306.7	435	1270	1435	241.8	72.46	5.71	21.82
A6	268	136	920.3	400	7.34	4.11	2.39	21.12
A7	38.33	55	1.64	108	0.518	2.05	2.96	27.6
A8	0.36	191	1.29	568	0.419	12.7	1.81	23.23
A9	32.47	243	2.035	807	0.523	8.93	3.57	29.4
Libyan standard	1200		1225		590		N/A	

Where N denotes to New oil and U denotes to Used oil.

To show the effect of engine oil running on the chemical additives, the results obtained have been compared with Libyan stranded, that will help us to see if this oils satisfies the Libyan stranded or not.

3.2.1 Zinc analysis

From Figure (10) there is a significant difference in zinc amounts between the oil samples, only N samples number (A2 and A3) has an acceptable amount by comparing them with the Libyan Standards, and the other N and U samples have a lower amount of Zinc than the Libyan standard by almost half, and the reason for the low zinc value after used, is the cracking of the protective layer being formed and turning into ash, which means reduce anti-wear and increase erosion, on the other hand, the oils contained zinc mainly in their composition by not more than 300 parts per million to the advent of the seventies and eighties and the emergence of strong engines, which companies raised it to 1,200 to 1400 parts per million after the chemists discovered its benefits, thus the samples that counties high amount of Zinc such as N samples (A3 and A2)

still can be used for a period of time more than that recommended by manufacturer. Moreover;

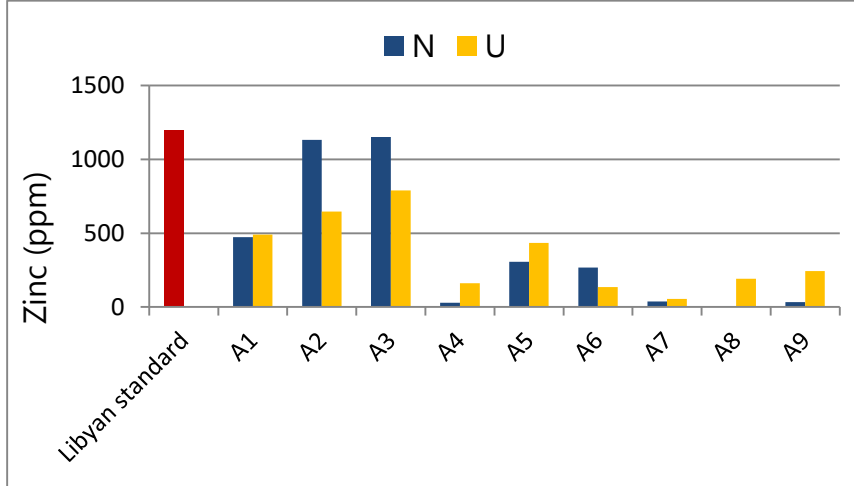


Fig. 10 Comparison the results of the zinc value of N and U samples with Libyan standard

3.2.2 Calcium analysis

The figure (11) presents the amount of the calcium in each N and U samples comparing with the Libyan Standards, as seen in result, the N and U samples number (A1,A2 and A3) have a high values more than Libyan Standards, and N and U samples number (A5) and N sample number (A6) have an acceptable amount of calcium levels that in Libyan Standards range, as for all other N and U samples have a very low amount of calcium element. As known Calcium is working as detergent and dispersant in engine oil to suspend or disperse harmful products like water, fuel, sludge and oxidation products and keeps the engine parts clean, so this is another reason explain the damage that happened in the engine after using those engine oils because of the low values of detergent and dispersant additives and the accumulation of dirt and the sludge inside the engine. And we also note that some of U samples have an amount of calcium more than N samples, this influenced can be explained by the wear of bearings and transition of metal particles to oil during normal car service [32,2].

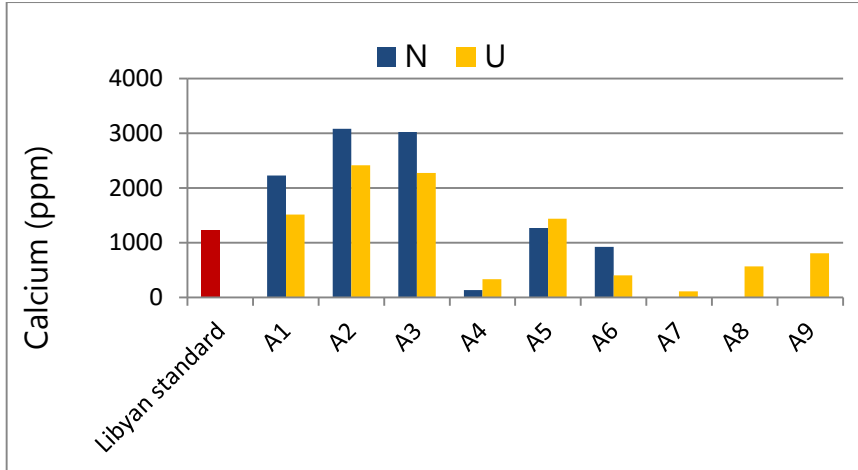


Fig. 11 Comparison the results of the calcium value of N and U samples with Libyan standard

3.2.3 Magnesium analysis

Figure (12) presents the comparison of magnesium value of all samples with the Libyan standard.

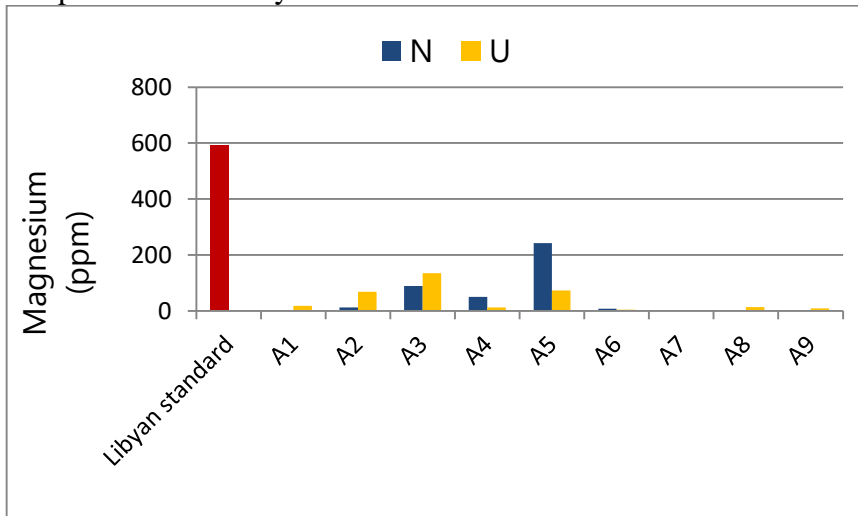


Fig. 12 Comparison the results of the magnesium value of N and U samples with Libyan standard

It is clear from the figure that the value of magnesium in the Libyan standard is very high comparing to all collected samples. The reason

is that magnesium is very expensive, in addition, magnesium and calcium have the same function and do nearly the same effect, which explain why the oil manufacturers focus on the use of calcium more than the magnesium to remove oil from the sediments and take them to the oil filter. However, The N and U samples number (A4,A7,A8 and A9) have a values close to zero of magnesium and calcium elements, so the cleaning and dispersion additives are almost non-existent, subsequently this also leads to the accumulation of dirt and blockages inside the engine and thus the engine fails completely.

3.2.4 Sodium analysis

In Figure (13), the value of sodium amount in the samples has been presented. Because this value is not mentioned in the Libyan standard, we would compare the values of sodium before and after being used with each other, which may affect keeping the surface free of deposits. And the increase in sodium amount in all samples after being used could be explained from the presence of contamination from soot or fuel [33,35].

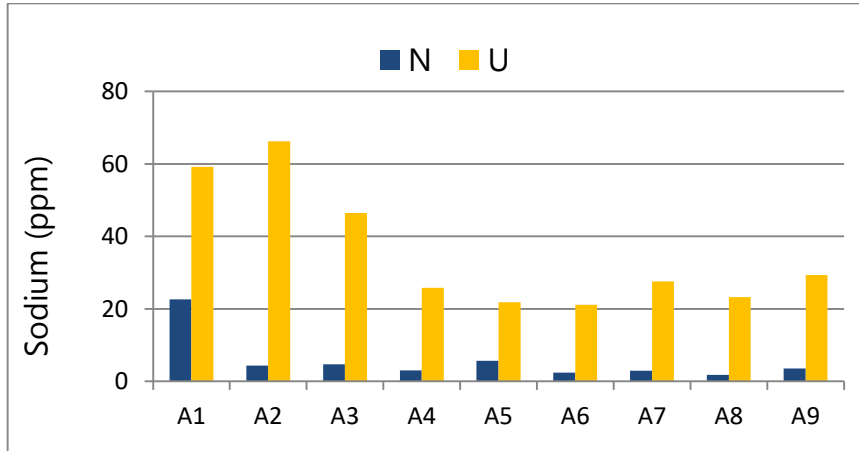


Fig. 13 Comparison the results of the sodium value between N and U samples

3.2.5 Total Acid Number analysis

Figure 14, shows the total acid number analysis of the samples before used and comparing the results between each other, there is a slight rise in the acidity of the samples number (A1,A2 and A3) but it

is still at the normal range. In fact, high concentration of acidic compounds in a lubricant can lead to corrosion of machine parts and clogged oil filters and the acid concentration of a lubricant depends on the presence of additive package.

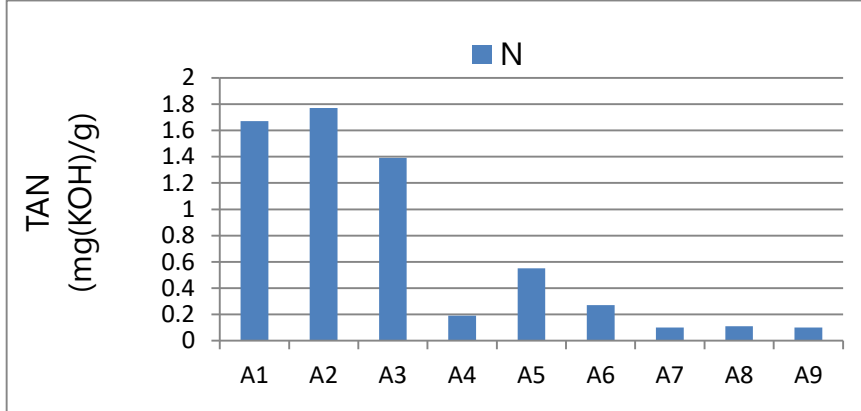


Fig. 14 The results of the Total Acid Number of N samples

3.2.6 Iron analysis

From Figure (15), the values of iron in the samples after being used are presented. The higher value of iron in the samples shows the engine is suffering from high rate of erosion or wear. In other words, it can be said that the more value of iron in the oil samples means the more rate of erosion and wear in internal parts of engine.

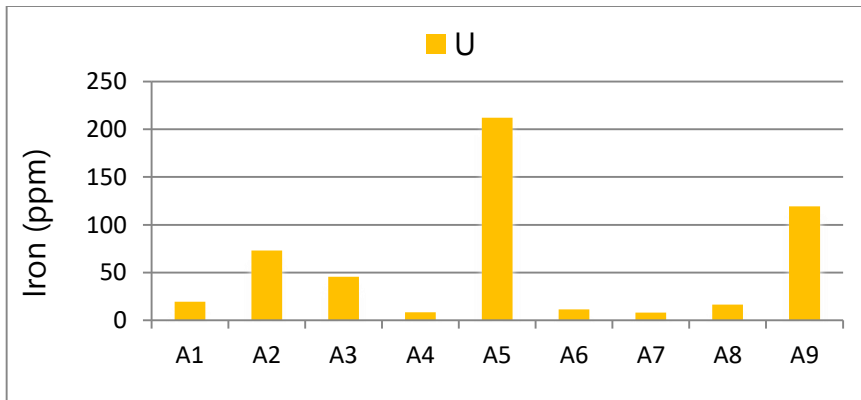


Fig. 15 The results of the iron value of U samples

3.2.7 Cupper analysis

Finally from figure (16), the value of copper in samples after being used reflects what is happening inside engine from friction on connected rod bearing, where the more value of copper means that the connected rod bearing has been put under a higher rate of wear.

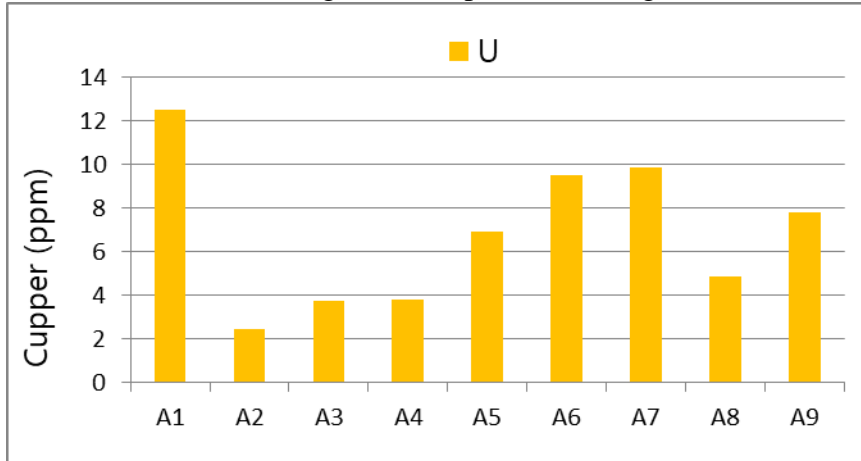


Fig. 16 The results of the copper value of U samples

4. CONCLUSIONS

In this study, physical properties and chemical compositions are measured for new and used oils, nine samples of engine oils that in scattered in the local market have been collected and used in different engines. The samples have been tested physically and chemically before and after being used and to be compared with the Libyan standard. The tests and comparisons showed the next conclusions:

1. The samples number (A1,A2 and A3) agree with the Libyan standard for all results, this means that these samples are very good for use in the engine.
2. The sample number (A5) agrees with the Libyan standard for all results, except for the value of zinc and sodium. This means that this sample is not good in use in the engine in terms of anti-wear and removal the sediment.
3. The samples number (A4,A6,A7,A8 and A9) agrees with the physical properties, but for the chemical additives values,

they disagree with the Libyan standard. This means that these samples are not suitable for use in the engine.

4. Oil quality cannot be detected by using the physical properties tests only.
5. The chemical additives analyses are a major step in the detection of oil quality and it's suitability to use.

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