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Study of Drug Bottles Using Laser Induced Breakdown Spectroscopy (LIBS)

Abeer E. Osman[1], Ali A . S. Marouf [1], Mubarak M. Ahmed [2]

[1]Department of Laser Engineering and Industrial Applications, Institute of Laser, Sudan University of Science and Technology, Sudan

[2]Department of Physics, College of Science and Art, Methane, Qassim University, KSA

ABSTRACT

There is a great number of packaging materials, such as bottles and boxes, are made of plastics (polymers). Additives such as plasticizers, antioxidants, antistatic agents, and lubricants may be used for the production of packaging materials. The worst effect of plastic production is deterioration of the environment. Recycling solves this problem and the initial step is identification and classification of the polymers and determination of different elements present in it. In this research, spectroscopic analyzes were performed using Laser Induced Breakdown Spectroscopy (LIBS) for plastic materials samples of drug containers that collected from manufacturing factories located at Khartoum city, Sudan. The samples were collected before and after used, were irradiated by Nd: YAG laser at 266 nm, repetition rate 2Hz, with pulse energy of 80mJ. The recorded spectra of the samples were analyzed using National Institute Standard and Technology (NIST). and Origin Lab Pro 9 data analysis software. All elements present in plastic drug bottles were detected using laser induced breakdown spectroscopy. The elements that detected are (Ca, Cd , Ra, Ac, W, V, Na, Pm, Cl, K, , Ce, Ar, Nd, Ac, Tc, Sc, Li, Pa, and Pb).

Keywords : Laser-induced breakdown spectroscopy (LIBS), Plastic Bottles, Polyethylene terephalate (PET), Drug store, Waste recycling, Health safety.

I. INTRODUCTION

The plastic products and packaging materials are common use in our daily lives [1]. which are generally utilized in various applications such as water and drug bottles, food packaging, medical supplies, and so on [2,3]. Because of the many advantages of plastic materials. most important advantages of plastic material are it is soft, transparent, flexible are lightweight, plastics are cost effective, require little energy to produce, and are and biocompatible, or biodegradable and many other of properties [4]. Plastic bottles for oral pharmaceutical products can be manufactured from a wide range of polymer materials including polyethylene terephalate (PET) being developed to have unique performance properties [5], PET has become from the most favorable packaging material world-wide beverages [6]. The reason for this development is the excellent material properties of the PET material, especially its un-break ability and the very low weight of the bottles compared to glass bottles of the same filling volume, solvent resistant, barrier to gas and moisture [2,7]. In spite of that, plastics are helpful in everyday life, except that it must be observed the toxic chemicals compounds utilized in production, in order to environmental and health safety [8].

Laser-induced breakdown spectroscopy (LIBS) has been developed as a new powerful spectroscopic

technology for chemical detection [9,10]. In the LIBS system an intense pulsed laser beam is focused onto the sample surface to produce luminous hot plasma. Optical emissions from the plasma are the result of the de-excitation events of the electronically excited atoms and ions. These spectral emissions are the characteristics of the sample and are identified using the NIST database. The spectral information can be utilized to determine the qualitative and quantitative elemental compositions [11]. LIBS has advantages over other analytical techniques, because there is little or no need for sample preparation; it can be used for rapid real-time analysis in field operations. Remote sensing of samples is also possible since only the photons need to be in direct contact with the sample [12,13]. The ability to analyze remotely without contact is especially important when dealing with waste plastics of various sizes and shapes placed on fast-moving conveyor belts. LIBS is a well-known technique for the qualitative and quantitative analysis of trace metals [14].

In this study, the laser induced breakdown spectroscopy was applied to characterize the hazardous and other inorganic trace element constituents in pre-and post-used plastics drug containers.

2. Materials and Methods

Preparation of Samples:

The samples used for testing in this study is plastic materials used in Sudanese drug containers from factories in Khartoum District, Sudan. The samples were collected before and after use, such plastic bottle samples were sterilized and washed with distilled water before it has been tested, to avoid any encountered contamination and to guarantee samples purity. The samples were also cut into slices that are around 3cm^2 in area and 2 mm in thickness. The total number of drug bottle samples was 9 specimens. All of these details are shown in table (1) that illustrates bottles of drug.

Table (1) drug bottles

bottles at first	Bottles Before used	Bottles After used	drug in bottle
Sample (S11)	Sample (S21)	Sample (S31)	AMIBUTAMOL
Sample (S12)	Sample (S22)	Sample (S32)	AMIHISTIN
Sample (S13)	Sample (S23)	Sample (S33)	AMIDOL

The experimental setup:

Samples were analyzed using a RT100-B system (Applied Spectra, Inc) as shown in figure 1. The RT100-B consisted of a 266nm Nd:YAG laser operated at 2 Hz repetition rate and 80mJ pulse energy, we were used Andor Echelle Spectrograph and DT334T ICCD, and an x- y -z translational stage. Gate delay = 0.5ns, gate width = 2ns, gain (amplification)= 95, these parameters are controlled within applied spectra's Axiom software. Gate delay, gate width and gain were optimized for best signal/background(S/B) ratio, while keeping good spectral line resolution and signification signal strength for samples.

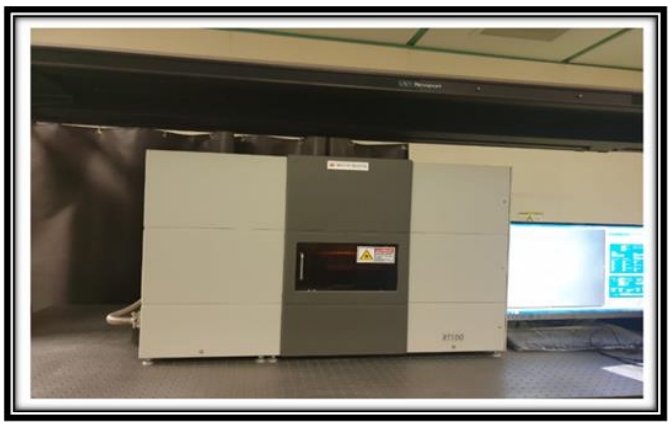

Figure (1). Applied Spectra's RT100 –B/LIBS instrument

The procedure

Each sample was mounted in a sample cell and irradiated by the Nd-YAG laser where the sample plasma spark was collected to the spectrometer via a fiber optic, which was interfaces to a computer. The emission spectra were collected within the range from 200-1100 nm within the range. Several LIBS analyses were conducted at its surface in order to check trace elements in drug bottle samples. The recorded spectra of the samples were analyzed using NIST data.

3. Results and Discussion

Figures (2) to (10) show the LIBS emission spectra for the samples after irradiation with 80mJ pulse energy. Tables (2) to (10) list the analysis of the wavelengths corresponding to different elements and their intensities in the nine samples.

LIBS results of the drug bottles samples at first (group 1):

All of the plastic drug bottles samples divided into three groups. The first group (S11,S12 and S13) that drug bottles at first, the second group (S21,S22 and S23) ,and the last one consists of (S31,S32and S33) were sampled using the LIBS technique to assess the presence and amounts of certain elements (in this case , Ca, Cd , Ra, Ac, W, V, Na, Pm, Cl, K, , Ce, Ar, Nd, Ac, Tc, Sc, Li, Pa, and Pb). Difference in the spectral lines between the different samples, which would be expected, because there are different between the groups (S11, S12 and S13) were before preparation (S21, S22and S23) were the bottles before used and at last (S31, S32 and S33) were the bottles after used. That caused the charred surfaces simply were to add or remove some elements. Figures (2), (3) and (4) show the spectral lines of (S11, S12 andS13) samples. The spectra for all these samples represented in the range (200 - 1150) wavelengths by nanometers.

Figure (2) for sample S11 shows 6 peaks, as tabling in table (2) and they were analyzed to their corresponding elements (Os I, Pt I, Pb I, Nb II, Tc I and W I). Figure 3 for sample S12 shows 17 peaks, as tabling in table (3) and they were analyzed to their corresponding elements (Os I, Pt I, W I, Fe I, Gd II, Nb I, V I, Ca I, Na I, Er II, Eu II, K I, Ne I and Xe I). Whereas, many elements have two peaks because of the different chemical compounds and states of existence these elements can attain (W I, Ca II and Na II) as shown in figure (3). At last samples in first group S13 at figure (4) shows 12 peaks, as tabling in table (4) and analyzed to their corresponding elements (Pt I, Pu I, Cr I, Ce II, Tb II, Ca I, Pr II, Li II, Na I, Eu I, Cd II and F I).

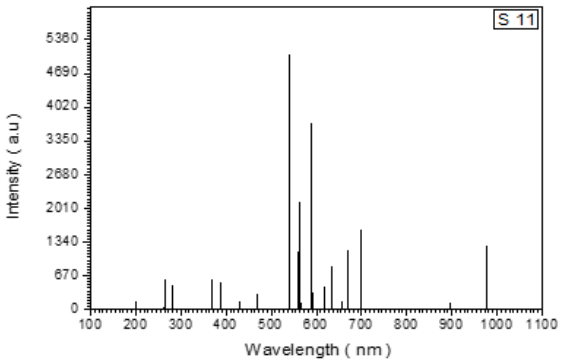

Figure 2. LIBS emission spectrum of sample (S11).

Table (2) The analyzed data of sample S11, irradiated by 80mJ

Measured λ (nm)	Intensity (a.u)	Elements
200.145	1169	Os I
265.415	1609	Pt I
280.108	1507	Pb I
367.357	1645	Nb II
386.824	1574	Tc I
429.461	1306	W I

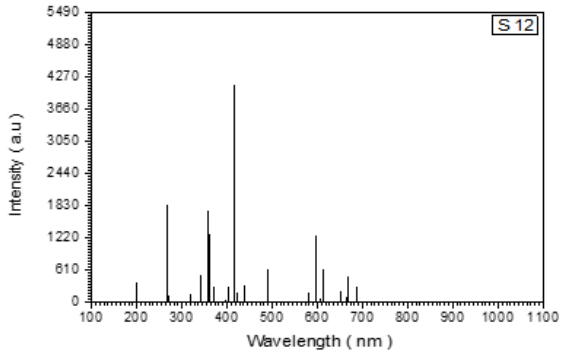

Figure 3. LIBS emission spectrum of sample (S12).

Table (3) The analyzed data of sample S12, irradiated by 80mJ

Measured λ (nm)	Intensity (a.u)	Elements
200.145	381	Os I
265.945	1836	Pt I
269.567	160	W I
319.329	182	Fe I
342.490	554	Gd II
358.946	1783	Nb I
404.551	316	V I
417.117	4142	W I
422.673	201	Ca I
440.512	351	Na I
490.003	600	Er II
585.745	209	Ca I
596.607	1313	Eu II
612.027	580	K I
651.421	235	Na II
667.583	460	Ne I
687.211	316	Xe I

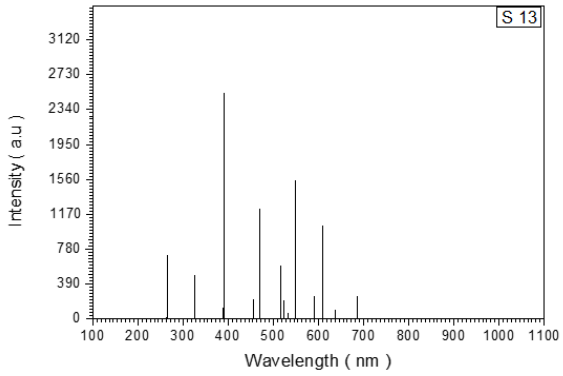

Figure 4. LIBS emission spectrum of sample (S13).

Table (4) The analyzed data of sample S13, irradiated by 80mJ

Measured λ (nm)	Intensity (a.u)	Elements
265.954	730	Pt I
324.520	478	Pu I
390.291	2541	Cr I
456.236	225	Ce II
470.241	1216	Tb II
518.885	596	Ca I
522.011	201	Pr II
548.511	1548	Li II
589.593	243	Na I
601.815	1034	Eu I
635.472	116	Cd II
685.603	277	F I

LIBS results of the drug bottles samples before use (group 2):

The second group was (S21, S22 and S23) are showing the chemical compounds spectra for all of these samples in figures (5), (6) and (7). The spectra for all this samples represented in the range (200 - 1150) wavelengths by nanometers. Figure (5) for sample S21 shows 12 peaks, as tabling in table (5) and analyzed to their corresponding elements (Pt I, Ni I, Ar I, Ra II, Na II, K I, Er II, Ca I and Kr I) but (K I and Na II)

elements have two peaks or more as shown in figure (5). Whereas. Figure (6) sample S22 shows 15 peaks, as tabling in table (6) and analyzed to their corresponding elements (W I, Er II, Bk I, Ra II, Tc I, Pa I, K II, Ca I, He II, Pm I, Xe II, Al I and Eu II) but (Ca I and K I) elements have two peaks as shown in figure (6). The last sample in the second group S23 at figure (7) shows 8 peaks, as tabling in table (7) and analyzed to their corresponding elements (Er II, Tc I, Pa I, K II, He II and Pr I) but (K I) element have three peaks.

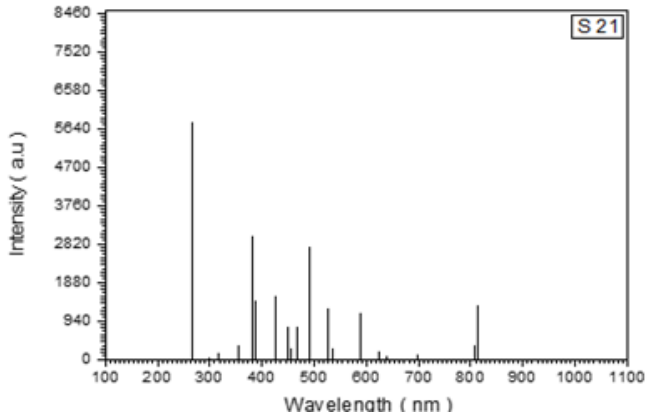

Figure 5. LIBS emission spectrum of sample (S21).

Table (5) The analyzed data of sample S21, irradiated by 80mJ

Measured λ (nm)	Intensity (a.u)	Elements
265.945	5866	Pt I
354.818	383	Ni I
386.492	3062	Ra II
425.936	1512	Ar I
449.087	856	Na II
464.237	774	K II
490.007	2756	Er II
526.556	1304	Ca I
534.294	331	K I
589.592	1162	Na I
807.962	363	K I
813.296	1304	Kr I

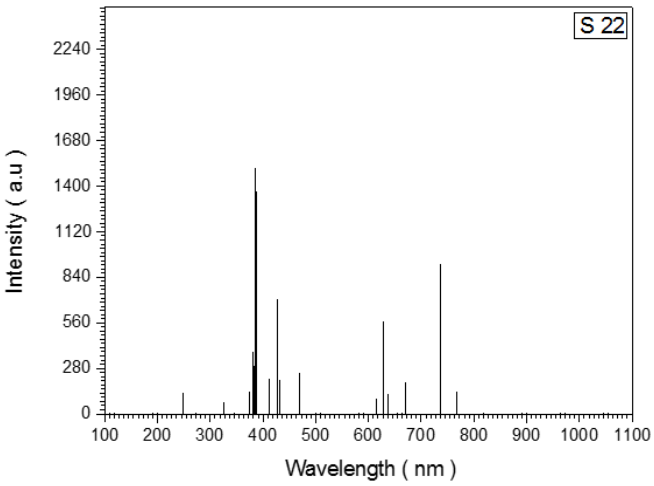

Figure 6. LIBS emission spectrum of sample (S22).

Table (6) The analyzed data of sample S22, irradiated by 80mJ

Measured λ (nm)	Intensity (a.u)	Elements
248.144	143	W I
326.478	71	Er II
372.538	131	Bk I
381.442	336	Ra II
386.824	1472	Tc I
411.762	215	Pa I
426.340	697	K II
430.774	209	Ca I
468.580	263	He II
616.217	95	Ca I
628.606	564	Pm I
637.528	113	Xe II
669.602	198	Al I
737.022	937	Eu II
769.897	145	K I

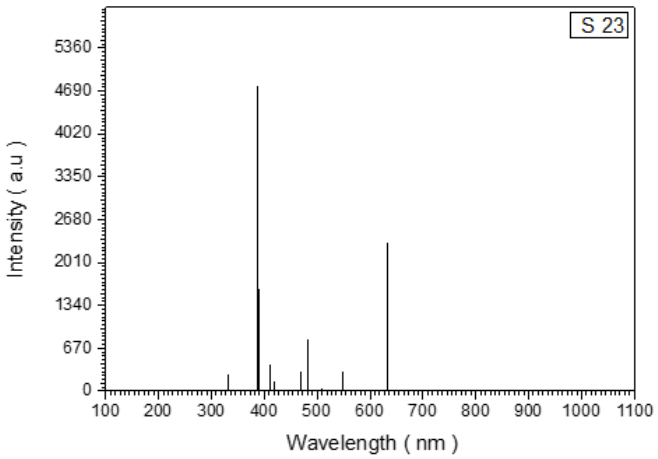

Figure 7. LIBS emission spectrum of sample (S23).

Table (7) The analyzed data of sample S23, irradiated by 80mJ

Measured λ (nm)	Intensity (a.u)	Elements
331.639	242	Er II
386.824	4774	Tc I
411.762	458	Pa I
418.624	198	K II
468.581	328	He II
482.923	834	K I
598.614	344	Pr I
630.729	2307	K I

LIBS results of the drug bottles samples after use (group 3):

The chemical compound spectra of the last group (S31, S32 and S33) are shown in figures (8), (9) and (10). The spectra for all of these samples represents in the range (200 - 1150) wavelengths by nanometers. Figure (8) for sample S31 shows 13 peaks, as tabling in table (8) and analyzed to their corresponding elements (Cd II, Ra II, Ac I, V I, Pm I, Ca I, Cl I, and K I) but both (Na II and W I) shows two peaks. Whereas, figure (9) for sample S32 shows 11 peaks, as tabling in table (9) and analyzed to their corresponding elements (Ar II, Nd II, Ac I, Pm I, Ca I, Tc I, Cl II, Na II, Sc I, Pb I and Pa I). The last sample in the third group S33 at Figure (10) shows 9 peaks, as tabling in table (10) and analyzed to their corresponding elements (Tc I, Nd II, V I, K I, Li II, Pm II, Ce I and Cl I) but only (K I) shows two peaks.

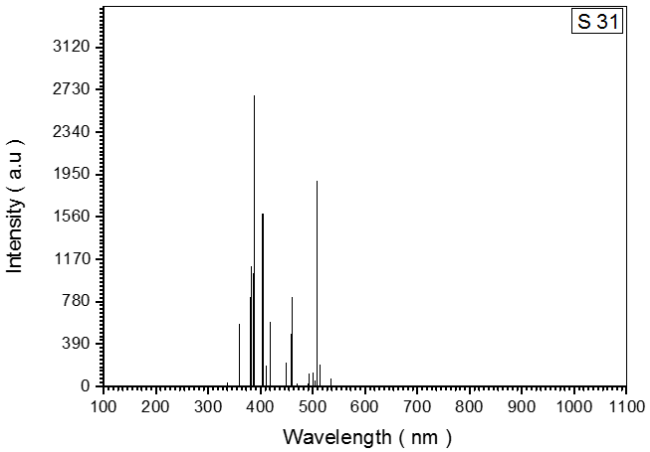

Figure 8. LIBS emission spectrum of sample (S31).

Table (8) The analyzed data of sample S31, irradiated by 80mJ.

Measured λ (nm)	Intensity (a.u)	Elements
358.496	604	Cd II
381.442	1143	Ra II
388.556	2677	Ac I
404.559	1598	W I
409.978	167	V I
417.117	588	W I
449.087	217	Na II
459.755	815	Pm I
487.817	133	Ca I
499.547	125	Cl I
514.883	225	Na I
533.969	65	K I

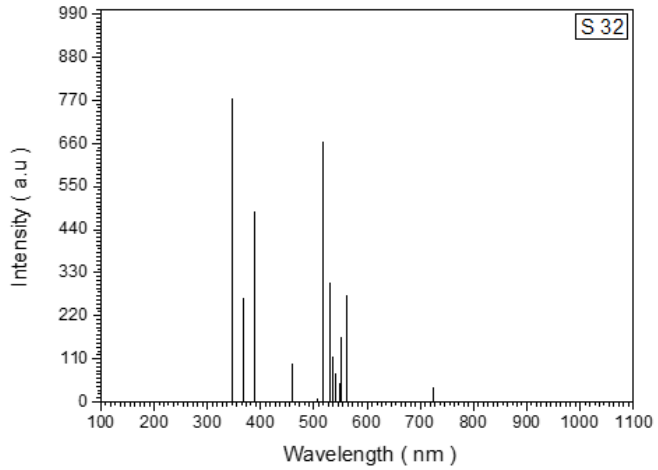

Figure 9. LIBS emission spectrum of sample (S32).

Table (9) The analyzed data of sample S32, irradiated by 80mJ

Measured λ (nm)	Intensity (a.u)	Elements
347.675	779	Ar II
367.354	269	Nd II
388.556	486	Ac I
459.755	98	Pm I
518.885	666	Ca I
528.507	308	Tc I
539.212	117	Cl II
541.455	72	Na II
552.052	156	Sc I
560.885	274	Pb I
722.713	45	Pa I

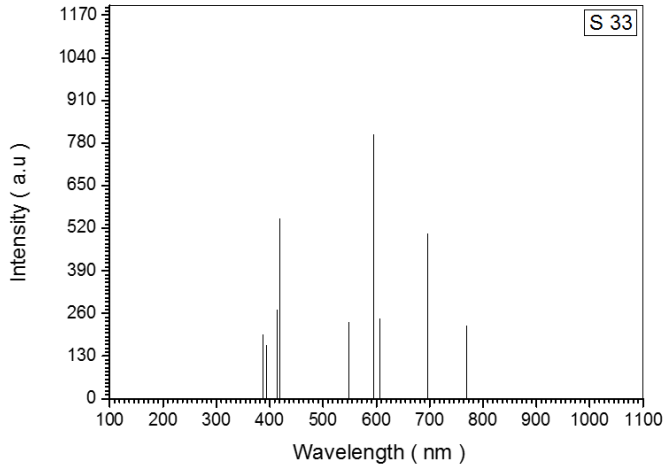

Figure 10. LIBS emission spectrum of sample (S33).

Table (10) The analyzed data of sample S33, irradiated by 80mJ

Measured λ (nm)	Intensity (a.u)	Elements
386.824	198	Tc I
393.888	172	Nd II
413.448	271	V I
418.624	554	K II
548.511	230	Li II
594.649	808	Pm II
607.201	245	Ce I
696.418	502	K I
767.242	224	Cl I

For all samples the detection limit is (200 – 1150 nm) depending on the sensitivity of the detector, it was out of the range of the main elements that the drug containers polyethylene terephthalate (PET) made of. (C and H) elements in the range before 200 nm for this rezone these two elements were not appear in the results.

The LIBS spectra showed that the elements of (S11, S21and S31) samples that drug bottles (AMIBUTAMOL DRUG) are (Os I, Pt I, Pb I, Nb II, Tc I and W I) for sample S 11 (bottle at first Bottle), (Pt I, Ni I, Ar I, Ra II, Na II, K II, Er II, Ca I and Kr I) for sample S21 (Before used Bottle) and (Cd II, Ra II, Ac I, V I, Pm I, Ca I, Cl I, and K I, NaI, W I) for sample S31 (After used). Some elements in sample S31 were not found in in sample S11 and sample S21 like (Cd II, Ac I, V I, C l I, and Pm I). This group is bad recycling samples.

For LIBS spectra (finger print wavelength) for (AMIHISTIN DRUG) are (Os I, Pt I, W I, Fe I, Gd II, Nb I, V I, Ca I, Na I, Er II, Eu II, K I, Ne I and Xe I) for S 12 sample (bottle at first), (W I, Er II, Bk I, Ra II, Tc I, Pa I, K II, Ca I, He II, Pm I, Xe II, Al I and Eu II) for S 22 sample (Before used Bottle) and element (Ar II, Nd II, Ac I, Pm I, Ca I, Tc I, Cl II, Na II, Sc I, Pb I

and Pa I) for S32 sample (After used). There are some strange elements like (Ar, Nd, Ac, Cl, Sc, and Pb) some of these elements are agree with the results of previous studies [15,16), also all elements for above samples of this group is bad recycling samples.

At the last group (AMIDOL DRUG) the finger print elements of LIBS spectra are (Pt I, Pu I, Cr I, Ce II, Tb II, Ca I, Pr II, Li II, Na I, Eu I, Cd II and F I) for sample S 13 (bottle at first Bottle) , (Er II, Tc I, Pa I, K II, He II and Pr I) for sample S 23 (Before used Bottle) and (Tc I, Nd II, V I, K I, Li II, Pm II, Ce I and Cl I) for S 33 sample (After used). There are some strange elements like (Tc I, Ce I, Nd I, V I , Li II, Pm , and K I). For above elements this group is bad recycling samples also.

II. CONCLUSIONS

- ✓ All elements present in plastic drug bottles were detected using laser induced breakdown spectroscopy. The elements that detected are (Ca, Cd , Ra, Ac, W, V, Na, Pm, Cl, K, , Ce, Ar, Nd, Ac, Tc, Sc, Li, Pa, and Pb).
- ✓ Element that present in plastic drug bottles samples were estimated and results achieved are in good agreement and all of these elements were normal and this group is not useful for recycling.
- ✓ The sensitive lines for the above-mentioned heavy elements were identified using standard data published by NIST for the elemental analysis in different plastic and drug bottles samples.
- ✓ This study reveals that the plastic drug bottles samples collected from manufacturing factories located at Khartoum city, Sudan. They could cause a lot of problems in recycling, it is not recommended recycling this samples.

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