

Applications of LIBS for determination of ionic species (NaCl) in electrical cables for investigation of electrical breakdown

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Abstract The formation of water trees in high-voltage cables can wreak havoc to power systems. The water tree is produced within the high voltage cable insulator when impurities like sodium and magnesium present in the insulating material react with moist soil to form chlorides. This water tree causes electrical breakdown by short circuiting the metallic conductor and the earth. In this paper we use laser-induced breakdown spectroscopy (LIBS) to detect the potentially dangerous elements that form the water tree in the insulating cable. The LIBS system used for this work consists of the fundamental (1064 nm) of a Nd:YAG laser, four spectrometer modules that cover the visible and near-UV spectral ranges and an ICCD camera with proper delay and gating sequence. With this arrangement we were able to measure the elemental concentrations of trace metals present in the insulating cable. The concentrations measured with our LIBS system were counter checked by a standard tech-

nique like inductively coupled plasma (ICP) emission spectrometry. The maximum concentrations for ionic species such as Ba (455.40 nm), Ca (393.36 nm), Cr (267.71 nm), Fe (259.94 nm), Cl (542.3 nm), Mg (516.7 nm), Mn (257.61 nm), Na (589.59 nm) and Ti (334.18 nm) are 20.6, 43.2, 1.6, 148.4, 24.2, 22.1, 4.2, 39.56 and 4.35 ppm, respectively. The relative accuracy of our LIBS system for various elements as compared with the ICP method is in the range of 0.03–0.6 at 2.5% error confidence.

1 Introduction

Saudi Arabia has a vast network of underground power transmission systems, laid and managed by Saudi Electric Company (SEC), a quasi-private-sector power company. Although the company has commendable safety track records, there have been sporadic incidents of power breakdown that incurred a huge material, long power interruption and manpower loss. It has been identified that the presence of impurities with penetration of water in the insulating material is one of the few major causes for the electrical breakdown. The penetration of water not only affects the electrical as well as the dielectric properties of the cable material, but also chemically reacts with impurities like Na, K and Mg to form their chlorides and appears as a water tree in the cable. This situation becomes more serious in humid and marine environments like places on the coastlines of the Red Sea and the Arabian Gulf, where the soil is laden with salt and moisture; the status of such breakdown is depicted in Fig. 1.

A power transmission cable is basically a multi-core copper wire shielded with a polymeric compound such as polyvinyl chloride (PVC) or polyethylene, which has high dielectric constant. The formation of water trees in high-voltage cables can cause severe damage and interruption to

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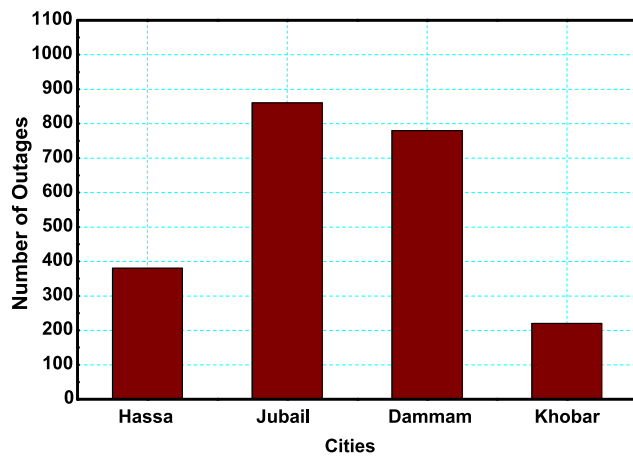


Fig. 1 Outage records for various cities of Saudi Arabia located on the coast of the Arabian Gulf for the 2008–2009 period

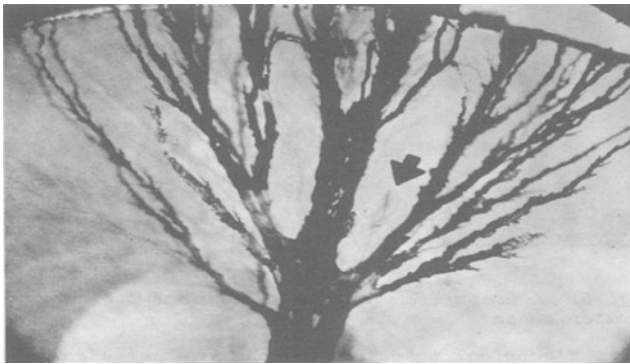


Fig. 2 Microscopic view of electrical tree formation causing the electrical breakdown [7]

the power distribution. The water tree is produced within the high voltage cable insulator when impurities like sodium and magnesium present in the insulating material react with moist soil to form chlorides. Figure 2 shows a SEM picture of an electrical tree which initiated at the conductor shield and caused the failure inside the cable under a laboratory test. The follow-through current was limited, thus preserving the tree from total destruction. The dark tree branches depicted in Fig. 1 are small hollow channels. The tree appears black because transmitted light was used and the walls of the channels are rough.

This water tree causes electrical breakdown by short circuiting the metallic conductor and the earth. Although the shielding material is highly water resistive, the high voltage or weathering can crack the insulator of the power cable that lets water enter the insulator and chemically react with the impurities to form water trees. Such cables have more probability to become faulty and undergo degradation over a certain period and trigger electrical breakdown between the main conductor and the earth shield, which leads to power outage [1–8].

In order to test the quality of electrical cables, a special setup based on laser-induced breakdown spectroscopy (LIBS) has been developed at our laboratory. Laser-induced breakdown spectroscopy depends on focusing of a pulsed laser beam onto a sample surface to generate plasma composed of excited species which emits light. The detection of the emitted light from the plasma after spectral dispersion could permit the identification of the elements present in the sample by using their characteristic spectral lines. This technique could be applied for analysis of many samples of different phases such as gas, liquid and solid samples [9–27]. The unique features of the LIBS technique are: little or no sample preparation requirements, remote analysis possibility, rapid analysis and multi-elemental detection [10–14].

The LIBS sensor was tested for monitoring of the ionic species content present inside faulty cables, newly manufactured cables and even PVC materials used for manufacturing these cables by Saudi Electrical Manufacturing Industries. There are some recent papers published on polymers using LIBS. The LIBS technique has been applied to identify various kinds of polymers using different ways such as correlating the LIBS spectra of the unknown polymers with a spectral library [28, 29], by using ratios of elemental emission lines and molecular bands [30] or by using artificial neural networks [18]. A variety of statistical methods were applied for identification of polymers using LIBS. Most commonly used are linear parametric correlation or nonparametric correlation methods [31–34].

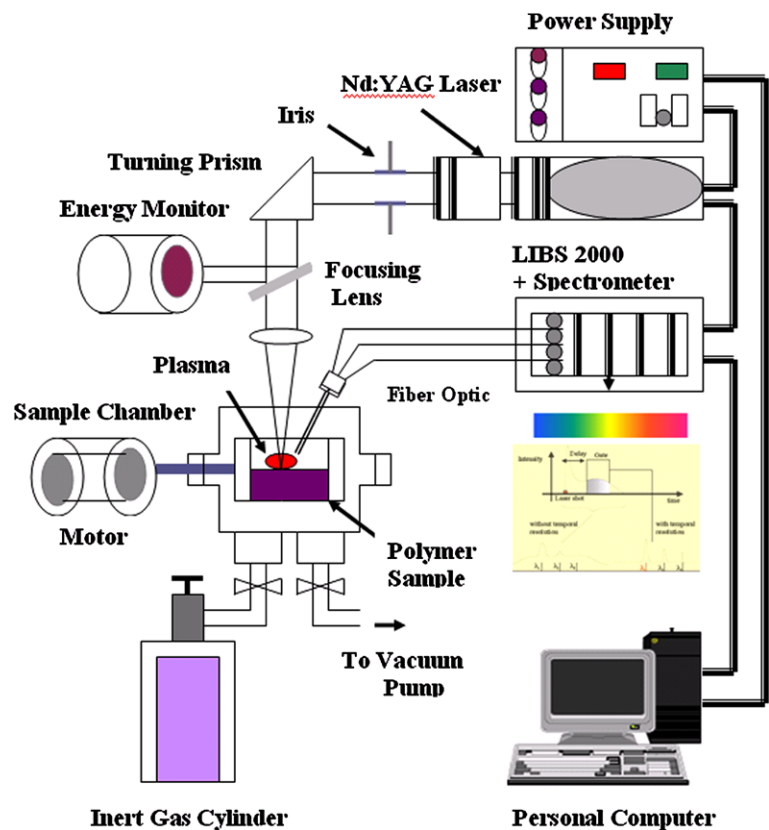
Other analytical techniques, such as Raman, infrared and near-infrared (NIR) spectroscopy, have also been applied for the identification of different families of plastics. Raman spectra provide structural fingerprint information that can be studied to identify plastics [35]. Infrared spectroscopy has also been applied as an important technique to identify plastics [36, 37]. LIBS techniques can be applied to complement NIR spectroscopy, for the identification of polymers, as mentioned previously [38, 39]. Most of the work reported in the literature on polymers using LIBS has been focused on the identification of different kinds of polymers and, to the best of our knowledge, there is no report about the application of LIBS for electrical cable analysis. Hence, our work toward such application of LIBS could be a significant contribution.

The detected amount of these ionic species using our LIBS setup was much higher as compared with the world standards for electrical cables, which may be a major reason for outages of cables and electrical breakdown in the Kingdom of Saudi Arabia around its coastal region.

2 Experimental procedure and sample preparation

Figure 3 depicts the schematic of the LIBS system employed in this work as referred to in our earlier works

Fig. 3 A schematic of the LIBS experimental setup applied for the analysis of underground cables



[15–25]. The excitation source for this work is the fundamental (1064 nm) of a pulsed Nd:YAG laser (Spectra Physics model GCR 100), with the laser energy of 1 J/pulse, the repetition rate of 10 Hz and the pulse width of 8 ns. The laser beam is focused on the cable surface with an appropriate IR lens. The light from the plasma spark is collected by a collimating lens using UV-grade fused silica 1-m multi-mode sampling fiber and is transferred to a LIB2000+ spectrometer (Ocean Optics). The resolution of our spectrometer is 0.1 nm in the 200–620 nm wavelength range. The detector used in this spectrometer is a gated CCD camera having 14 336 pixels. The software present in the spectrometer could read the data from the chip and reconstruct the spectrum. The emission was recorded at a 45° angle. For each LIBS analysis, a cable sample was placed on a stainless steel disc shaped (20-mm diameter by 10-mm long) platform mounted on a rotary table. Thirty laser pulses were shone onto the sample to have a complete measurement. The table is rotated using a stepper motor having 12 revolutions per minute. The major elements detected in the samples are Na, Mg, Ba, Ca, Cr, Fe, Mn and Ti. All the spectral lines for the above-mentioned elements recorded with our LIBS setup were identified using the NIST atomic spectral database and also using a standard reference data book [27, 28, 40, 41]. A set of measurements were carried out on electrical cable samples, with the aim to determine the optimum time delay between the laser pulse and the start of the de-

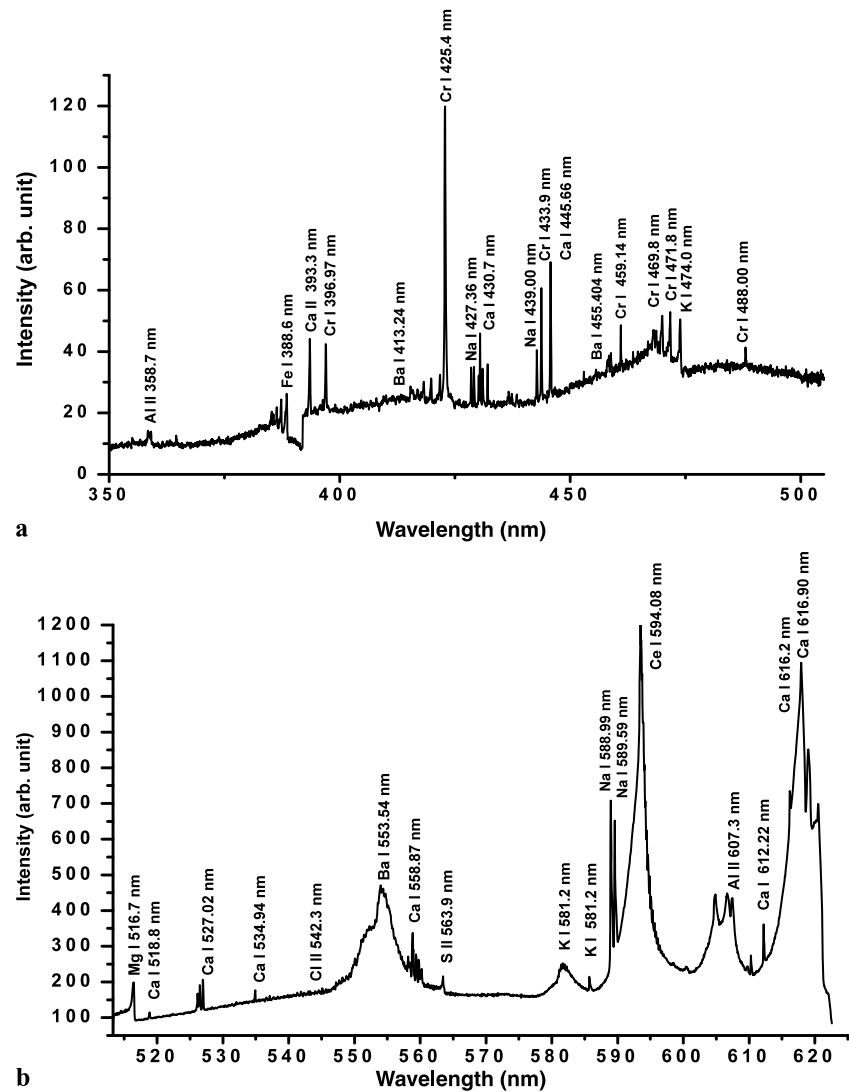
tection system (ICCD camera). Four sets of plastic materials employed in this study were supplied by electrical cable manufacturers located in Jeddah and Riyadh, Saudi Arabia. These model polymers were polyvinyl chloride (PVC) (mixture) [$M_n = 20,000$ – $100,000$], polypropylene (PP) [$M_n = 75,000$], polyvinyl chloride (PVC) [$M_n = 30,000$ – $95,000$], low-density polyethylene (LDPE) [$M_n = 80,000$] and high-density polyethylene (HDPE) [$M_n = 85,000$], in granular and powder forms.

The concentrations of different trace metals present in these polymer samples were also measured with a calibrated inductively coupled plasma (ICP) spectrometer to verify the results achieved with our calibrated LIBS setup. For each LIBS analysis, a fresh polymer test sample was kept at the LIBS platform. For the construction of the calibration curves, pure metals in powder form were thoroughly mixed using a special grinder with polymer samples in powder form in known concentrations and pressed into the form of pellets. All these metals were in powder form and were of high purity (99.99%) purchased from Fisher Scientific, USA.

3 Results and discussion

In order to improve the limit of detection (LOD) and to increase the sensitivity and quality of the LIBS system, the

Fig. 4 Typical LIBS spectra showing different trace metals present in PVC sample. (a) Depicts the LIBS spectrum recorded in the 350–510 nm region while (b) shows the spectrum recorded in the 510–625 nm region



optimal experimental conditions were obtained prior to testing actual electrical cable polymer samples. To achieve these objectives, different parameters like delay time, laser energy, focusing of the laser beam on the sample and optimization of the collection lens for plasma-emitted light were varied to maximize the level of the LIBS signal.

3.1 LIBS analysis of raw materials

LIBS spectra in the 200–620 nm spectral region were recorded with our setup for different kinds of polymers manufactured in Saudi Arabia and spectra due to PP, PVC and LDPE types of polymers used in electrical cables. Typical spectra of a PVC sample recorded in the 350–505 and 510–620 nm regions are depicted in Fig. 4a and b. The trace metals present in these samples were identified and are listed in Table 1. The major elements detected in the polymer samples are Ba, Ca, Cr, Fe, Mg, Mn, Na, Cl and Ti and are marked in Fig. 4a and b. The Cl/Na intensity ratio of these

lines was estimated in different polymers, namely: LDPE, PP and PVC. The Cl/Na ratios estimated from LIBS spectra were estimated as 1.47, 109, 1.20 and 0.097, respectively, for HDPE, LDPE, PP and PVC samples. Polyethylenes, HDPE and LDPE, have justifiably shown the highest Cl/Na intensity ratios. It is clear from the intensity ratio that LIBS was able to differentiate between the least branched groups in HDPE with a Cl/Na intensity ratio of 1.47 and LDPE with a ratio of 1.29. The sensitive lines in LIBS for each element present in the test samples were recorded in the 200–620 nm region. The characteristic atomic emission wavelengths of all identified elements were marked. The atomic emission line at 455.4 nm is a characteristic line for Ba; so are the 393.3-nm emission line for Ca, the 425.5-nm emission line for Cr, the 259.9-nm emission line for Fe, the 516.7-nm emission line for Mg, the 257.6-nm emission line for Mn and the 334.1-nm emission line for Ti. Similarly, the prominent 589.5-nm atomic emission line for Na has been selected for quantitative analysis. The selected atomic emission lines

Table 1 Trace elements detected in electrical cable by LIBS and ICP techniques

Element	λ (nm)	PP as a powder		PP as a sheet		PVC as a powder		PVC Jeddah as a powder		PE as a powder		PE as a sheet	
		LIBS (ppm)	ICP (ppm)	LIBS (ppm)	ICP (ppm)	LIBS (ppm)	ICP (ppm)	LIBS (ppm)	ICP (ppm)	LIBS (ppm)	ICP (ppm)	LIBS (ppm)	ICP (ppm)
Ba	455.4	7.08	2.04	0.09	0.01	20.6	27.50	13.20	11.38	0.19	8.23	6.91	
Ca	393.3	43.23	29.47	37.56	29.87	9.50	7.30	219.3	214.77	28.45	37.56	32.70	
Cr	425.5	1.6	0.9	1.4	0.8	1.7	0.9	1.6	0.8	1.5	1.5	0.53	
Fe	259.9	10.78	7.54	24.23	29.67	8.22	7.47	95.2	91.10	7.32	148.43	140.36	
Cl	542.3	2.5	–	2	–	7.2	–	24.2	–	–	–	–	
Mg	516.7	9.17	4.51	7.86	4.97	7.34	5.70	22.10	19.44	8.32	9.72	7.79	
Mn	257.6	0.08	0.05	0.62	0.10	0.45	0.22	4.21	2.81	0.43	0.74	0.56	
Na	589.6	39.56	25.75	32.45	10.15	34.78	26.91	36.52	30.47	33.45	27.87	25.55	
Ti	334.1	1.01	1.92	4.35	1.95	3.21	1.31	4.21	2.43	4.32	1.92	1.50	

Table 2 Trace elements and their spectral assignments along with the fingerprint wavelengths of elements detected in polymer samples

Trace metal	Measured wavelength (nm)	LIBS signal intensity	Spectral assignment of observed transitions, upper state to lower state
Na I	588.9	2120.1	$2p^63s \rightarrow 2p^63p$
Na I	589.6	2748.3	$2p^63s \rightarrow 2p^63p$
Cl II	542.3	185.74	$3p^3(^4S)^5D \rightarrow 3p^3(^4S)^5P$
Ti I	517.3	273.2	$3d^24s^2 \rightarrow 3d^2(^3F)4s4p(^3P^0)$
Cr I	558.8	351.3	$3d^44s^2 \rightarrow 3d^5(^4G)4p$
Cr I	534.6	275.2	$3d^5(^6S)4p \rightarrow 3d^5(^6S)4d$
Ca I	527.1	250.3	$3p^63d4s \rightarrow 3p^63d4p$
Mg I	518.3	225.4	$3s3p \rightarrow 3s4s$
Cr I	493.6	64.3	$3d^44s^2 \rightarrow 3d^5(^4G)4p$
Ba II	455.4	25.3	$6s \rightarrow 6p$
Cr I	425.5	52.4	$3d^5(^4D)4s \rightarrow 3d^5(^4G)4p$
Ca II	393.3	170.2	$3p^64s \rightarrow 3p^64p$
Ti I	334.2	6.2	$3d^24s^2 \rightarrow 3d^2(^1G)4s4p(^3P^0)$
Fe II	259.9	14.3	$3d^6(^5D)4s \rightarrow 3d^6(^5D)4p$
Al I	208.3	12.1	$3s^23p \rightarrow 3s^224d$

have no interference from other atomic emission lines. The assignment of spectral lines for all trace elements present in polymers recorded with our LIBS setup was done by using the NIST atomic spectral database and Refs. [27, 28, 40, 41], which is presented in Table 2. Table 1 lists the trace elements detected in different polymer samples by our LIBS system and verified by the ICP technique. The ionic species concentrations detected in the electrical cable polymer samples are quite high and could cause the tree formation and thus lead to electrical breakdown.

Most of the ionic species are present in electrical cables in the form of chlorides and nitrates. In addition to the formation of chlorides like NaCl, from the impurities present in the insulating material, this can directly enter the cable from the soil itself, as NaCl is highly hydrophilic and attracts moisture content. Hence, in addition to ionic species present in primary material used for shielding of electrical cables, the contents of Na and Cl were detected using our LIBS setup in the polymeric raw material employed in manufacturing of electrical cables. Table 1 presents the Na and Cl content levels along with other trace (ionic species) elements like Ba, Ca, Cr, Fe, Mn and Ti. In principle, these elements in the form of NaCl are not supposed to exist in the PVC raw material that is being used in the cable insulation manufacturing process. However, different amounts of Na and Cl were found in each sample as clear from Table 1 and from LIBS spectra (Fig. 4). The amounts of Na and Cl vary from one cable sample or raw material to another, i.e. in samples nos. 3 and 4 (PVC) the levels of both Na and Cl were high, implying a more salty environment surrounding the electrical cable conductor and more hazard for absorbing water inside the cable insulation and faster degradation to happen, while the amounts of Na and C in sample

no. 2 (PP) are very low, meaning less absorbing water and thus better quality cable. However, one cannot rule out that an increased amount of Cl in the PVC sample can also be (perhaps primarily) due to the Cl contained in and liberated upon breakdown from the initial structure of the PVC polymer. The samples manufactured from PP have more insulation and are more resistive to water absorption and recommended to be used as insulating material in electrical cables. The concentration of the prepared samples was also counter checked by using a calibrated ICP spectrometer.

3.2 Dependence of LIBS signal intensity on incident laser energy

The incident laser energy is a key parameter in generation of plasma and plays an important role in the sensitivity of the LIBS system, since high incident laser energy produces optically thick plasma. Therefore, for the optimization of the LIBS signal intensity, an energy dependence study is important. Due to this reason, the dependence of the LIBS signal generated from test samples on incident laser energy was also studied. For this purpose, the intensities of the Cr emission line at 267.7 nm from polymer samples at incident laser pulse energies of 20, 30, 50, 70 and 90 mJ/pulse were recorded at a fixed concentration of Cr (0.02% Cr in polymer) and at a time delay between the trigger laser pulse and the beginning of the LIBS spectra acquisition fixed at 4 μ s. Figure 5 depicts the dependence of laser-produced plasma on incident laser energy for elemental Cr present in a Cr-contaminated polymer sample. Figure 5 shows that the LIBS signal increases with rise in the incident laser energy from 20 to 90 mJ per pulse. The line intensity (for Cr) exhibits a linear dependence on the laser energy, which is clear by the

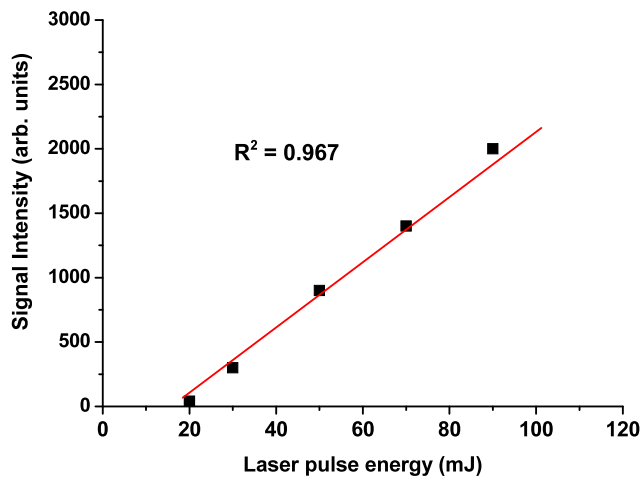


Fig. 5 The dependence of the LIBS signal intensity for the Cr (267.7-nm) emission line on the incident laser energy

least-square fit, $R^2 = 0.967$. It is worth noting that at higher incident laser energies, more critical plasma self-absorption effects appear [19, 20]. At higher concentration, greater self-absorption will take place.

3.3 Calibration of LIBS system

The calibration of our LIBS system was carried out by using standard samples prior to application of the LIBS system for analysis of cable samples. For this task, known concentrations of 100, 75, 50, 20, 10 and 5 ppm of Na, Ba, Cr and Ti and other metals under investigation were prepared in a polymer matrix and LIBS spectra were recorded for six different concentrations of each element. The LIBS calibration curve for measurement of Cr metal is depicted in Fig. 6. The curve was plotted by recording the LIBS signal intensity of the Cr 267.7-nm emission line at different concentrations in standard samples. The spectra were averaged for 20 laser shots for each data point to reduce the background noise as compared with the single-shot spectrum of the same sample. The LIBS signal intensity showed a linear dependence on the concentration of Cr. The line interference poses a serious problem for the identification of emission lines; therefore, we chose this line for analysis which is free from interference and has minimal effects to obtain accurate results.

3.4 Comparison of the data from two detection systems

The accuracy of the elemental concentration evaluated from the LIBS system was verified using the ICP data for the samples under investigation and the comparison of these two detection systems (LIBS and ICP) is listed in Table 1. In order to achieve better accuracy in elemental analysis using LIBS, the sample surface was pre-ablated before taking real measurements, the fluctuation of laser power was minimized by

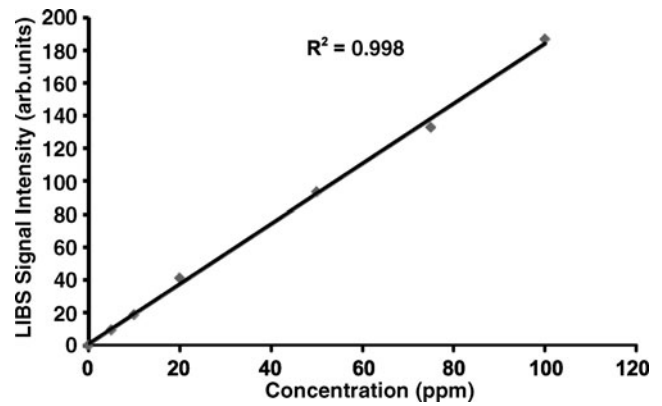


Fig. 6 A calibration curve for the Cr (267.7-nm) emission line obtained by using standard samples of chromium with known concentrations. Here the laser energy was 100 mJ

giving enough instrumental warm-up time and the LIBS signal was averaged for 20 laser shots to get a better average of the data. The variation in LIBS signal intensity was stabilized by averaging for different laser pulses while the relative standard deviation (R.S.D.) decreased with increasing number of laser pulses. The precision of any analytical instrument is defined as the repeatability of the measurement, and is usually estimated in LIBS [15–22] with the relative standard deviation.

The relative accuracy (RA) is calculated as follows [8]:

$$RA = \frac{|d| + S.D. \times t_{0.95386}/\sqrt{n}}{M}, \quad (1)$$

where d is the difference between the LIBS measurement and the ICP (standard) method. S.D. is the standard deviation of the LIBS measurement, M is the measurement from the standard ICP method, n is the number of measurements and $t_{0.975}$ is the t value at 2.5% error confidence. The relative accuracy of our LIBS system for different elements as compared with the ICP method was calculated using the above equation (1) and was in the range of 0.03–0.6 at 2.5% error confidence.

The procedure adopted for calculation of relative accuracy is discussed in our earlier publications [15–17]. For polymer samples, the LIBS measurement precision also depends upon the complexity and homogeneity of the sample and the reproducibility of the laser pulses.

3.5 Target surface topography and electrical tree formation

Water trees are basically tiny hollow channels which are branched and have the general appearance of a tree, as explained by McMahon [7]. Now the partial discharges inside these channels occur and thereby damage the polymer, causing the tree to grow till the failure stage. This can be triggered by imperfections at the conductor shield, at the ground shield, at contaminants (due to presence of ionic species) within the volume of the insulator or wherever a diverging

field is present. Main causes for electrical trees to initiate at these sites are switching surges or lightning impulses.

4 Conclusions

The LIBS technique was applied for studying the quality of raw materials used in manufacturing of cable insulation such as polyvinyl chloride (PVC) (mixture), polypropylene (PP), polyvinyl chloride (PVC), polyethylene (LDPE) and polyethylene (PE). In addition, the manufactured and faulty cables showed the existence of noticeable amounts of ionic species like Na, K, Mg, Cl and other undesired elements. In raw material, the levels of both Na and Cl are very high, which implies that this cable is more vulnerable to absorb water inside the cable insulation, which could lead to faster degradation and water-tree formation. Sensitive emission lines for different ionic species were identified for the elemental analysis. Calibration curves were effective in quantifying the concentrations of different elements present in the contaminated polymer samples. Parametric dependence studies were carried out for the optimization of the sensitivity of the LIBS spectrometer. The accuracy of our system (qualitative as well as quantitative) is obvious from the comparison of LIBS results with the standard inductively coupled plasma (ICP) spectrometry technique and the LIBS results are in good agreement with ICP. This work also demonstrated that the LIBS technique could be applied for online analysis of cable samples by building a portable system. This study revealed that the major cause of insulation failure in electrical cables along the coastal region of Saudi Arabia is the progressive degradation process called water-tree formation due to the presence of ionic species in the insulating layer.

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