Research Paper

Identification of Chemical Elements in Soil Contaminated With Paper Waste Using LIBS

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ABSTRACT: *Identification chemical elements in soil contaminated with paper waste has been done by using laser-induced breakdown spectroscopy (LIBS). Emission spectra taken from the waste-contaminated soil and uncontaminated soil were obtained. Plasma emissions are then detected using a multichannel analyzer (OMA) to obtain the emission line spectrum that represents the content of chemical elements in the target soil. The spectrum is recorded and compared with the spectrum of reference standards of atoms, ions and molecules (National Institute of Standards and Technology/NIST) so that the atomic and molecular content in the sample can be known. From the results of the study it was found that there are elements of heavy metals in the form of Fe, Cd, Cu, Cr and Mn in polluted soils.*
KEYWORDS: *Chemical Elements, Soil, Paper Waste, LIBS*

I.   INTRODUCTION

Wastewater that contains suspended or dissolved solids, undergoes physical, chemical and biological changes that will produce toxic substances or create a medium for germ growth. Waste will change color to blackish brown and foul-smelling. This foul odor will cause respiratory problems. If this waste is discharged into the river it will pollute the river and if it is still used it will cause itching, diarrhea, and nausea.

Several analytical techniques are used to identify the element content in the soil to determine the level of environmental pollution [1]. X-ray diffraction (XRD) techniques are used for pollution analysis in soil pollution [2]. Atomic absorption spectroscopy (AAS) technique is used for the analysis of heavy metal contamination in the soil around gold mining exploration in the city of Palu [3]. But these conventional methods require complex and difficult sample preparation in the laboratory. In addition, the XRD and AAS methods require a very long time to analyze pollution on soils with a very large area so that the method is not effective and efficient. Therefore, it is necessary to develop a new method that can be used for analysis of soil pollution with fast time, does not require complicated sample preparation, is able to detect all elements contained in the target sample simultaneously and has a high level of sensitivity for identification of impurities and elements Minor which is dangerous to the soil.

Soil samples are samples that have characteristics that are difficult to analyze using the LIBS method because of the matrix effect in the form of physical and chemical properties. However, we have succeeded in analyzing heavy metal pollution in soils using a new LIPS method [4]. As a result, toxic and heavy metal impurities in the soil can be identified with a high level of sensitivity (ppm order). These results promise the possibility of using the LIPS method for elemental analysis in polluted soils.

Dario Santos et.al. conducted an experiment on LIBS for the determination of cadmium in soil [5]. The soil sample was prepared in the form of pellet prior to analysis. They concluded that the LIBS can be employed to screening of cadmium pollution in soil. Pandhija et.al. demonstrated an experiment on LIBS for the determination of heavy metals Pb in soil [6]. Semi-quantitative analysis

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of Pb in soil can be realized with a detection limit of 45 ppm. Various studies on soil analysis by using LIBS have been made as reported elsewhere [7-12]. There is lack of information dealing with detection of oil pollution in soil by using LIBS.

Recently, laser-induced breakdown spectroscopy (LIBS) has become a tremendous method for qualitative and quantitative analyses of sample target in various kinds of samples including liquid [13,14], metals [15,16] and solid [17,18]. In this technique, a pulse neodymium yttrium aluminium garnet (Nd:YAG) laser is focused on/in a sample to induce a luminous plasma. In the plasma region, molecules and atoms ablated from the sample can be effectively dissociated and excited [19,20]. Compared to other conventional analytical methods, standard LIBS is much superior because rapid analysis can be performed without tedious sample preparation and it has low-cost experimental equipments [20]. Several studies have reported on application of standard LIBS for the analysis of soil target [21,22]. Del`Agglio et al. have detected heavy metals elements such as Cr, Cu, Pb, and Zn in soil. Comparative study has been made by using ICP-OES. The correlation between ICP-OES and LIBS was confirmed by the satisfactory agreement [23]. However, in standard LIBS method using pulse Nd:YAG laser, the soil sample should be prepared in the form of pellet for effective dissociation and excitation process [24,25].

This research examine the LIBS method for identification of all macro and micro elements, and analysis of the potential for toxic metal contents in soils polluted by the paper industry waste.

II. METHODS

The equipment used in this study is (1) Nd: YAG laser as an energy source (1064 NM, 150 mJ, 7ns), (2) Optical Multichanel Analyzer (OMA) connected to (3) optical fibers, (4) Chamber as a sample container connected to (5) vacuum pumps and (6) pressure measuring equipment, (7) mirrors, (8) lenses for laser beam focusing and (9) computers. Each sample is given the same experimental treatment, which is raised at a low pressure of 5 Torr, 83 mJ energy. The arrangement of equipment used in the study is shown in the following Figure 1.
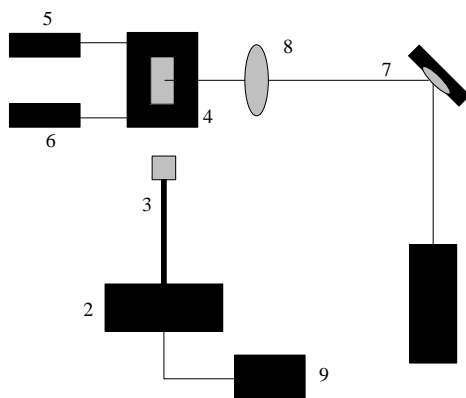
**Figure 1**. The arrangement of LIBS tools in research

The samples used in this research were soil with contaminated waste and soil uncontaminated. Determination of an element in a sample can be known through spectrum graph data. The data for each detected wavelength intensity is matched with the Atomic Spectra Database Line Form reference data from NIST (National Institute of Standards and Technology) Physical Measurement Laboratory.

III. RESULTS AND DISCUSSION

Plasma produced from the mechanism of generation by laser beam shooting has unique characteristics. That is because the ionized and excited gas from the material constituents emits specific elements in the form of wavelengths with laser color and plasma sizes that vary according to the energy level of the transition. Laser interaction with the target will cause an increase in temperature on its surface. This will cause ablation on the target surface. The target will absorb the energy of the laser beam so that the atoms at the base energy level will transition to higher energy levels. The accumulated energy will become vapor and cause the bonding of the material atoms will be broken. This process will produce plasma emissions in accordance with the conditions of the given experiment.

In this research, the content of chemical elements in soil contaminated with waste around the disposal of paper mill liquid waste was identified by the plasma spectrometer method. The results obtained are compared with uncontaminated land that is far away from the waste disposal site. From the spectrum data obtained with OMA and the results compared with NIST, in uncontaminated soils there are several chemical elements such as Fe I, Fe II, Mg, Al, Ca, Si with different intensities. Whereas in polluted soils, several chemical elements are detected such as Cr, Cu, Al, S at certain wavelength ranges.

Figure 2 is the spectrum difference between polluted soils and uncontaminated soils in the wavelength range of 250 nm -290 nm. In polluted soils there are heavy metal elements in the form of Cr II at wavelengths of 267.71nm and 283.57nm. Whereas in Figure 3, it shows the presence of chemical elements in polluted soils in the form of Ca II (315.88 nm, 317.94nm), Cu I (324.76nm, 327.39nm), and Cr I 357.87nm in the wavelength range of 290nm - 370nm. In the range of wavelength 370nm - 450nm (as shown in figure 4), in the contaminated soil detected elements of Mn I 370.61nm and Cr I 425.43nm. Figure 5 shows the heavy metal elements detected in polluted soils in the form of Ca I (558.88nm, 559.43nm, 559.87nm), S II (616.18nm), Al II (649.38nm) in the wavelength range of 450nm - 700nm .
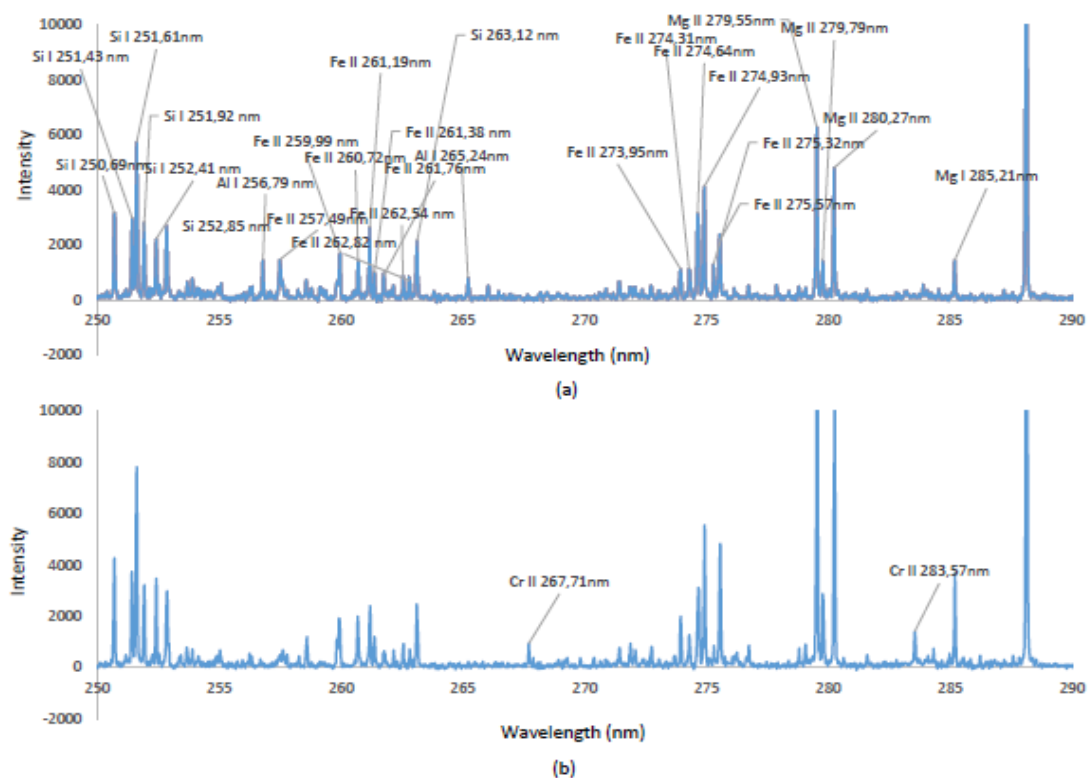
Figure 2. the spectrum difference between polluted soils (b) and uncontaminated soils (a) in the wavelength range of 250 nm -290 nm
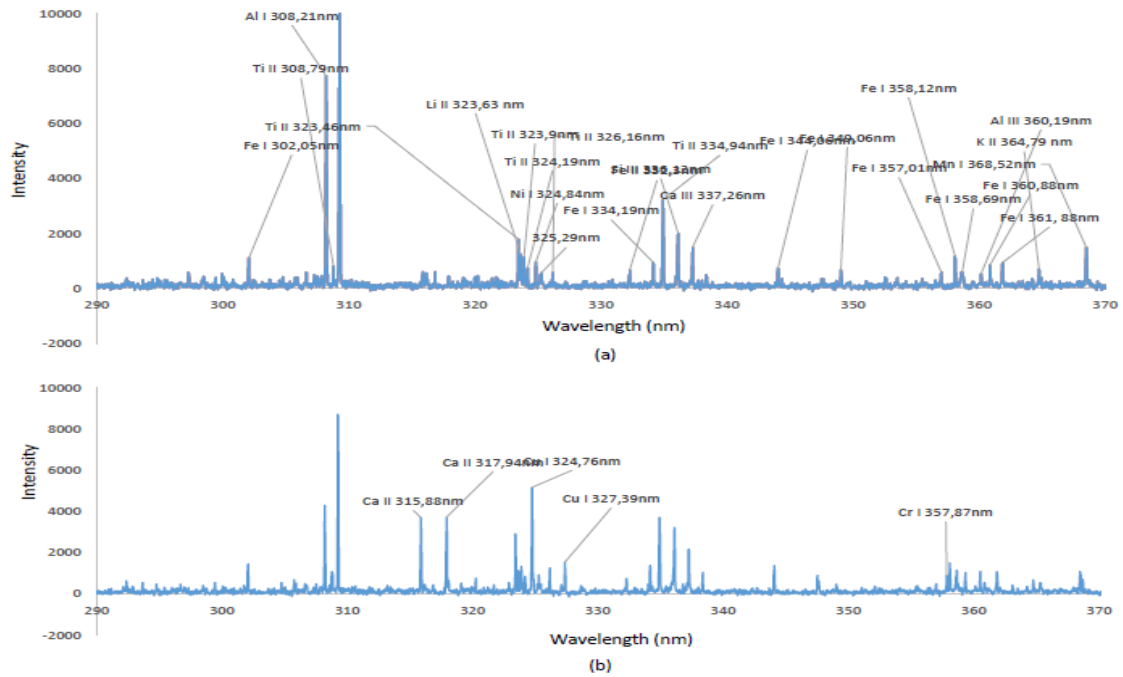
Figure 3. the spectrum difference between polluted soils and uncontaminated soils in the wavelength range of 290 nm -290 nm
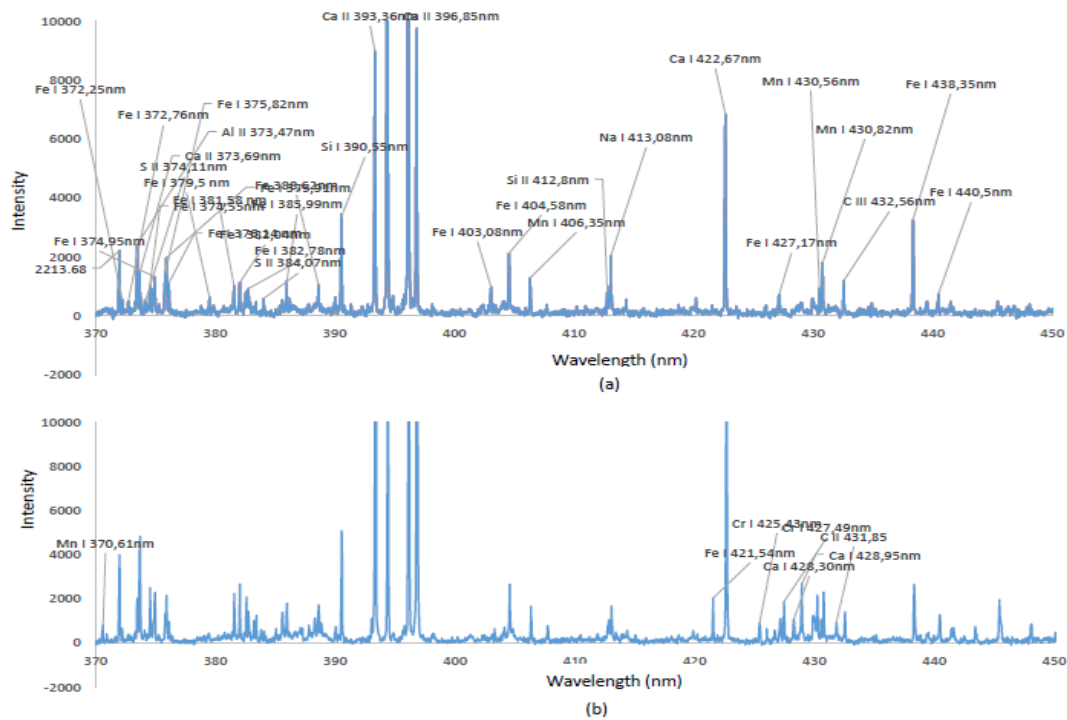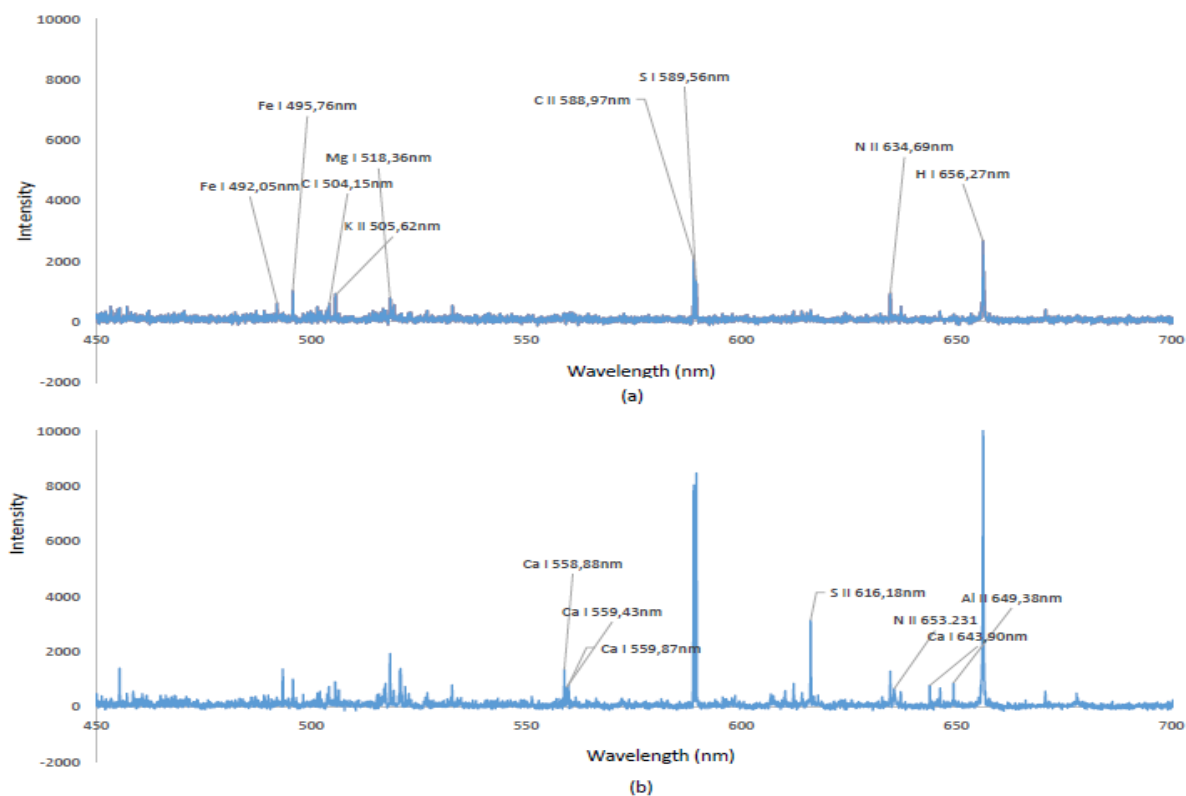

Figure 4. the spectrum difference between polluted soils and uncontaminated soils in the wavelength range of 370 nm -450 nm

Figure 5. the spectrum difference between polluted soils and uncontaminated soils in the wavelength range of 450 nm -700 nm

Spectrum characteristics that represent elemental content have varying intensities. Each element has a different energy level from each other. The intensity of the spectrum resulting from the spectroscopic method is influenced by three factors, namely transition probability, population at each level, and sample concentration. The higher the intensity of the spectrum of an atom, it can be concluded the greater the concentration of atoms in the sample.

IV. CONCLUSION

The spectrum confirms that the LIBS method is able to detect heavy metal elements in contaminated soil. The LIBS method clearly detects the presence of elements Fe, Cu, Cr, Mn, Ca, Al with varying wavelengths which are waste paper products that pollute the soil.

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