Elemental Profiling of Rice and Husk by Laser-Induced Breakdown Spectroscopy (Libs)

By Firoza Kabir & Jannatul Ferdous Ema

Abstract- Laser induced breakdown spectroscopy (LIBS) was used to determine the elemental content of Rice and Husk which were collected from Bangladesh Rice Research Institute (BRRI) in Gazipur, Bangladesh (latitude: 24.0958° N, longitude: 90.4125° E). Samples were collected from one experimental plot. Several elements such as Ni, Cu, Zn, Fe, Ca, Al, Si, Na, K, Ti, Mn, Li, Mo, Co, Mg, C were identified in Rice and husk samples by Laser Induced Breakdown Spectroscopy.

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I. Introduction

LIBS have been established as a fast spectroscopic technique for multi-elemental detection of elements in many diverse situations (Cremers et al., 2006, Miziolek et al., 2006, Singh and Thakur, 2007). In this technique a high power laser pulse is used for the generation of weakly ionized plasma from the samples. The spectral emission lines from the excited atoms/ions of the plasma are then spectroscopically analyzed for both qualitative and quantitative detection of multiple elements in the sample. The technique is extremely versatile and powerful, and is capable of analyzing solid, liquid and gaseous samples. For example, LIBS has been used for the detection of trace elements of soils (Martin et al., 2003 and Acra et al., 1996), geological analysis (Harmon et al., 2006), aerosol analysis (Samuel et al. 2003 and Radziemski et al. 1983), analyses of industrial waste water (Rai and Rai, 2008 and Gondal et al., 2003) etc, to name a few.

In this paper, we used LIBS technique for the analysis of rice and husk collected from the agricultural land in Gazipur in Bangladesh (latitude: 24.0958° N, longitude: 90.4125° E) with a view to detect important elements and to determine their enormous uses in future.

II. Experimental Details

In the present LIBS experiment intense, transient plasma was produced by focusing the fundamental beam at 1064 nm from a Q-switched Nd: YAG laser (Spectra-Physics LAB-170-10) on the sample by a convex lens of 100 mm focal length. The laser pulse had a temporal width of 8 nsec and repetition rate of 10 Hz. The beam has a Gaussian profile in the far-field and has a beam divergence of less than 0.5 m rad. The experiments were performed in air. The laser pulse energy used was 40 mJ. The spot size at the sample position was about 200 μm. The light emitted from the plasma was focused by a fused quartz lens (f=50 mm) and collected by a 3 m long multimode silica optical fiber and was then transmitted through the fiber to the entrance slit of a 750 mm focal length computerized Czerny-Turner spectograph (Acton Model SP-2758). The spectrograph was equipped with three ruled gratings: 2400, 600 and 300 grooves/mm blazed at 240, 500 and 300 nm, respectively, which were interchangeable under computer control. The schematic diagram of the experimental set up and more details are available elsewhere (Haider et al., 2011).

The spectrum was detected by an intensified and gated CCD camera (Unigen II coated Princeton PI-MAX camera with 1024X1024 pixels). The ICCD camera was electrically triggered by the Nd: YAG laser pulse after a software-controlled, adjustable time delay. In this way, the intense background initially created by the high-temperature plasma was largely eliminated, and the atomic/ionic emission lines of the elements were more clearly observed. In the present experiments, a delay time t_d of 1.5 μs and a gate width t_w of 50 μs were selected for the optimum signal. Usually, spectra from a number of laser shots (about 40–80) were acquired and averaged to increase the signal-to-noise ratio. Samples were manually moved between exposures to prevent crater formation and to avoid other deleterious effects. The spectrum, captured by the ICCD camera, was transferred to the personal computer by USB cable. All the functions of the ICCD camera and the Acton spectrograph were fully controlled by the WinSpec/32 software provided by the manufacturer (Haider et al., 2012).

III. Sample Processing

Rice and Husk samples from one experimental plots of Bangladesh Rice Research Institute (BRRI), Gazipur, were collected. Varieties of fertilizers were used in different plots. The fertilizers that were applied to the fields were NPK (Nitrogen, Phosphorous, Potassium), NPKS (Nitrogen, Phosphorous, Potassium, Sulphur, Zinc) and NPKSZN (Nitrogen, Phosphorous, Potassium, Sulphur, Zinc, and Copper).

The collected samples were powdered by hand mortar and pestle. Then the powder of each sample was
passed separately through a 75 micron sieving machine which makes the sample most homogeneous to carry out LIBS experiment. Small pellets were made by using a hand press with sufficient pressure (80 bars).

**IV. RESULTS AND DISCUSSIONS**

For every sample, spectra were acquired in the UV to IR region (190 to 900nm) using two gratings. A 600 grooves/mm grating blazed at 500nm was used to take spectra in the range of 360-880nm. Another grating of 2400g/mm blazed at 240nm was used for 190-360nm spectral range.

Some representative LIBS spectra for the rice and husk samples are shown in the figures (1-20) where different emission lines are labeled with the charge state of the elements. Here, figure (9-11) shows the presence of Silicon in Husk samples.

![Figure 1: LIBS spectrum of rice sample in the spectral range of 240 nm to 250 nm](image1)

![Figure 2: LIBS spectrum of rice sample in the wavelength limit 276 nm-286 nm](image2)
**Figure 3:** LIBS spectrum of rice sample in the wavelength limit 286-296 nm

**Figure 4:** LIBS spectrum of rice sample (NPK) in the wavelength limit 390-435 nm

**Figure 5:** LIBS spectrum of rice sample in the wavelength limit 430-470 nm
Figure 6: LIBS spectrum of rice sample in the wavelength limit 580-620 nm

Figure 7: LIBS spectrum of rice sample in the wavelength limit 655-700 nm

Figure 8: LIBS spectrum of rice sample in the wavelength limit 730-775 nm
Figure 9: LIBS spectrum of husk sample in the wavelength limit 203-215 nm

Figure 10: LIBS spectrum of husk sample in the wavelength limit 240-250 nm

Figure 11: LIBS spectrum of husk sample in the wavelength limit 248-260 nm
Figure 12: LIBS spectrum of husk sample in the wavelength limit 276-286 nm

Figure 13: LIBS spectrum of husk sample in the wavelength limit 311-322 nm

Figure 14: LIBS spectrum of husk sample (NPK) in the wavelength limit 329-340 nm
Figure 15: LIBS spectrum of husk sample in the wavelength limit 385-435 nm

Figure 16: LIBS spectrum of husk sample in the wavelength limit 425 - 475 nm

Figure 17: LIBS spectrum of husk sample in the wavelength limit 500-545 nm
The standard database of atomic emission lines of the US National Institute of Standard and Technology (NIST, http://physics.nist.gov/PhysRefData/ASD/lines_for m.html) was used for the identification of the elements.
from the observed spectrum (Abedin et al., 2011). Table 1 summarizes the elements detected in the samples of rice and husk from the experimental plot NPK.

Table 1: Elements found in the samples of rice and husk. A tick indicates the presence of the element in the sample whereas a cross indicates the absence of the element in the sample.

<table>
<thead>
<tr>
<th>Elements</th>
<th>Rice</th>
<th>Husk</th>
</tr>
</thead>
<tbody>
<tr>
<td>Fe</td>
<td>✓</td>
<td>✓</td>
</tr>
<tr>
<td>Cu</td>
<td>✓</td>
<td>✓</td>
</tr>
<tr>
<td>Na</td>
<td>✓</td>
<td>✓</td>
</tr>
<tr>
<td>Ca</td>
<td>✓</td>
<td>✓</td>
</tr>
<tr>
<td>Ti</td>
<td>✓</td>
<td>✓</td>
</tr>
<tr>
<td>Si</td>
<td>✓</td>
<td>✓</td>
</tr>
<tr>
<td>Co</td>
<td>✓</td>
<td>✓</td>
</tr>
<tr>
<td>Ni</td>
<td>✓</td>
<td>✓</td>
</tr>
<tr>
<td>Al</td>
<td>✓</td>
<td>✓</td>
</tr>
<tr>
<td>Zn</td>
<td>✓</td>
<td>✓</td>
</tr>
<tr>
<td>Li</td>
<td>✓</td>
<td>✓</td>
</tr>
<tr>
<td>K</td>
<td>✓</td>
<td>✓</td>
</tr>
<tr>
<td>Sn</td>
<td>×</td>
<td>×</td>
</tr>
<tr>
<td>Mn</td>
<td>✓</td>
<td>✓</td>
</tr>
<tr>
<td>Sr</td>
<td>✓</td>
<td>✓</td>
</tr>
<tr>
<td>Mo</td>
<td>✓</td>
<td>✓</td>
</tr>
<tr>
<td>P</td>
<td>✓</td>
<td>✓</td>
</tr>
<tr>
<td>C</td>
<td>✓</td>
<td>✓</td>
</tr>
<tr>
<td>Mg</td>
<td>✓</td>
<td>✓</td>
</tr>
</tbody>
</table>

From table 1, it is found that the elements Ni, Cu, Zn, Fe, Ca, Al, Si, Na, K, Ti, Mn, Li, Mo, Co are present in Rice and Husk samples in addition to Sr, P, C and Mg.

This experiment has detected the presence of micronutrients (such as Ni, Cu, Zn, Fe, Mn, Na) as well as some macro-nutrients (such as Ca, K, Si) in all samples. However no quantitative determination of these nutrients was attempted in the present study.

V. Conclusion

The present LIBS technique has been applied to get an elemental profile of the rice and husk samples of agricultural lands.

Rice is a staple food for more than one third of the world’s population. Every year, rice growers produce about 422 million metric tons. Rice husks, a waste product produced during cultivation, make up about 20 percent of this. Because these husks are very abrasive, agricultural manufacturers use them in cheap items, such as fertilizer additives, stockbreeding rugs and bed soil, which take advantage of this quality.

But rice husks could have a more valuable use. They contain large amounts of silica, which engineers can convert to silicon for use in LiBs. Silica comprises between 15 and 20 percent of a rice husk’s weight. This silica has evolved to be Nano porous, so that air and moisture can enter rice kernels but bacteria and insects cannot.

Pure silicon can be extracted from rice husk silica by adding acid and heat to remove metallic impurities and organic components and then using magnesium to reduce the silica to silicon. This treatment preserved the three-dimensional porous nanostructure. They then coated this silicon with carbon and used it in anodes in lithium coin cells.

Silicon extracted from rice husks could help meet the increasing demand for silicon in batteries used to run portable electronic devices and hybrid electric vehicles. This would allow a waste product from one of the world’s most popular crops to contribute to the development of advanced technologies.
REFERENCES  Références Referencias


