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Original research article

## Quantitative analysis of Fe and detection of multiple elements in the coal ash by laser-induced breakdown spectroscopy

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### ARTICLE INFO

#### Keywords:

Laser-induced breakdown spectroscopy  
Coal ash  
Quantitative analysis  
Fe  
Aerosols  
Internal standard method

### ABSTRACT

Sulfates produced by the reaction of metal oxides in coal ash with the acid gases in the atmosphere are the main causes of haze-fog formation. In this study, laser induced breakdown spectroscopy (LIBS) was used for elemental analysis in coal ash. A new method was adopted to conduct an accurate qualitative analysis of the four elements (Al, Ca, Cu and Fe) that easily form sulfates in coal ash. An internal standard method was used to quantitatively analyze the typical Fe element of the four elements with a calibration curve that had a linear correlation coefficient ( $R^2$ ) of 0.986. In order to test the accuracy of the curve, three kinds of coal ash with unknown composition were selected and analyzed by LIBS and X-ray fluorescence spectrometry (XRF). The relative differences of the two methods were around 7.42%, 3.37% and 4.34%, respectively, which are all in the acceptable range. The experimental results indicate that LIBS can be employed for the rapid detection and analysis of metal elements in coal ash and provide a new classification method for coal ash based on the content of Fe element.

### 1. Introduction

Coal ash, as a mineral dust, is the main component of a variety of metal oxides. It is the product of the complete burning of coal. Since the Second Industrial Revolution, coal, as a significant energy material, were widely used in people's production and life. At the same time, a large amount of mineral dust (coal ash) from coal combustion were released into the atmosphere, reacting with the acid gases in the atmosphere to form sulfates [1], which forms a haze-fog in the atmosphere and brings great threat to human health. Several significant air pollution events happened due to coal ash, such as the Great Smog of London [2], the Yokkaichi asthma incident [3] and the Belgian Meuse Valley incident [4], and all caused a large number of fatalities.

Till now, there are still a lot of developing countries in the world which have faced the similar environmental challenge such as China [5], India [6], Iran [7] and so on. Therefore, there is urgent need for developing sufficient tools to detect haze-fog so that timely actions may be taken to prevent more people from harms. We propose the rapid detection and analysis of sulfate-forming elements in coal ash, that is believed to be the main cause of haze-fog.

Laser induced breakdown spectroscopy (LIBS) [9] is an element detection technique developing in recent years. Due to its advantages of real-time, fast, non-contact and simultaneous detection of multiple elements, LIBS has been widely applied on detection

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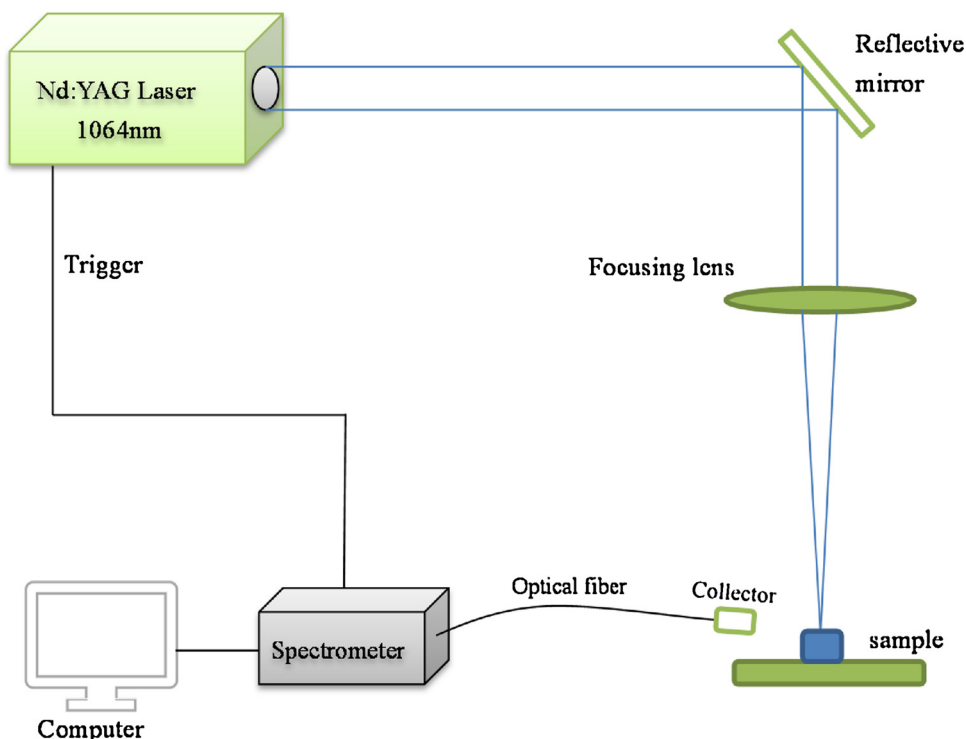


Fig. 1. Schematic diagram of the LIBS setup.

and analysis of elements in soils [10–15], aerosols [16–20], liquid [21,22] and rocks [23]. It has become an important medium in the field of environmental monitoring.

In this article, seven kinds of coal ash samples from a steel company were detected based on laser-induced breakdown spectroscopy. The qualitative analysis of four elements Al, Ca, Cu and Fe, which are easy to form sulfates, was carried out. The Fe content was chosen as an example for quantitatively analysis and a calibration curve was obtained. Three kinds of samples with unknown composition were selected to test the predictive ability of the curve. In addition, it is proposed that the content of Fe element in coal ash can be used to provide a new method for the classification of coal ash.

## 2. Experimental

### 2.1. Experimental setup

The schematic of the LIBS experiment is shown in Fig. 1. The laser used in the experiment was a Q-switched Nd-YAG laser, which was operated at a fundamental wavelength of 1064 nm. The maximum energy is 600 mJ in a single laser pulse, and the pulse energy for the employed laser beam in the current measurement is around 100 mJ per pulse with 10 ns duration at a frequency of 5 Hz. The laser beam was focused onto the sample surface using a focusing lens ( $f = 150$  mm), of which the irradiation energy was collected with a fibre-optical probe. The emission signal from the plasma was transferred via a fiber to a spectrometer system. The spectrum was recorded on a personal computer, and the PLSUS software was utilized to identify emission lines and presented the possible elements according to the spectrum. The effective spectral resolution of this spectrometer system was around 0.03 nm, depending on the wavelength. In order to increase the stability and reduce the standard deviation of the spectral intensities, 10 measured spectra were averaged. The spectrometer and wavelength shift were calibrated via the pure metal sample.

### 2.2. Sample preparation

In this experiment, seven kinds of coal ashes from a steel company were prepared as samples, which were labeled with Sample-1#, Sample-2# to Sample-7#, respectively. The accurate elemental concentration was obtained by X-ray Fluorescence Spectrometry [24–26] (As shown in Table 1). Since the sample is powder, in order to obtain a better LIBS signal, the tablet machine was used to press the coal ash into coal lump of 10 mm in diameter and 5 mm in thickness.

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