Instantaneous monitoring of local fuel concentration in a liquid hydrocarbon-fueled flame using a LIBS plug


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A portable device composed of photodiodes and bandpass filters was developed to measure local fuel concentration in a liquid hydrocarbon-fueled spray flame. The plasma emission spectra in and around the flame were selectively captured using such simplified device or plug instead of using a laboratory standard laser-induced breakdown spectroscopy (LIBS) system consisting of an ICCD and a spectrometer. The hydrogen (656 nm) and oxygen (777 nm) atomic lines were selected to determine the fuel concentration in atmospheric pressure. The H/O signal intensity ratio was found to be a strong function of the fuel concentration, and thus a calibration curve for the concentration measurements was established and validated using conventional LIBS. The proposed scheme to measure the local equivalence ratio of spray flames using a bundled layout of multiple LIBS plugs alongside the combustor wall may offer simple and highly robust diagnostics, especially under the harsh combustion conditions within air-breathing engines.

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1. Introduction

The reliable ignition of flammable mixtures is a fundamental issue for combustion science. In various combustion systems — including gas turbines and automotive engines — ignition is an important step for reliable operation, and for such ignition events to take place, the spatial and temporal distributions of the fuel-air mixture must be understood. Specifically, the local equivalence ratio and instantaneous fuel concentration in and around the chemical reaction zone must be known. The equivalence ratio and the fuel concentration have been measured either by using a spark plug ion sensing or oxygen sensor placed in the spark position [1]. Both measurement methods have a very slow response time to detect the fast flow dynamics associated with air-breathing engines. Therefore, they have only been implemented in limited experimental setups associated with static flow without complexity. To overcome such inherent limitations, various laser diagnostics technologies have been introduced to detect the properties of complex, reacting flow conditions.

The present study makes use of laser-induced breakdown spectroscopy (LIBS), which is favorable in terms of detecting various state materials with real time acquisition, minimal system complexity, and applicability in harsh chemical environments compared to other laser diagnostic systems. LIBS is a type of emission spectroscopy that uses a high energy laser pulse as the excitation source. The laser beam is focused with sufficient energy to dissociate and excite molecules to generate a small plasma with a high temperature. The atoms and electrons of the materials in the plasma volume absorb the photon energy, which results in excitation of the electrons in neutral atoms and ions. As the plasma cools, the excited ions and electrons move from their excited state to ground states, emitting light with specific atomic frequencies in the process. The plasma emission is collected and analyzed to obtain atomic information of unknown materials. As a result of these known advantages, LIBS has been previously applied in the analysis of reacting flows to obtain a local equivalence ratio [1–10] and gas concentration [11–13]. Atomic lines of H, C, O, and N and molecular bands of CN and C2 can be effectively acquired in hydrocarbon fuel-air mixtures using LIBS. O and N signals will appear upon dissociation of air molecules as oxidizer while C and H signals will appear upon the dissociation of hydrocarbon molecules as fuel.

Starvropoulos et al. first measured the equivalence ratio of laminar premixed flames using LIBS [2]. The equivalence ratio of the laminar premixed [2–5] and/or diffusion [6,7] methane-air
flames could be determined by the atomic lines of hydrogen and oxygen. The calibration curve between the H/O intensity ratio and the equivalence ratio was established for each case. The equivalence ratio at various points of the flames was obtained using the calibration curve. When LIBS is applied in reacting flows, atomic signal ratios, such as H/O [2–8,13], H/N [12], and other combinations of the H/O/N signals [1,9,10] are quite useful in estimating the equivalence ratio, which is the real-time fuel concentration at the breakdown point in the combustion chamber. In addition, C/N, C/O [14,15], and C2/CN [13] intensity ratios are often used since the C atomic signal and molecular band line that contains C can be observed from fuel plasma. Therefore, the local concentration of air and fuel can be quantitatively measured using each signal intensity at the breakdown location.

Although numerous LIBS research studies have focused on gas-state reacting flows, only limited work has been performed on the combustion of liquid-state fuels. Gebel et al. [16] captured the breakdown emissions in kerosene spray and quantitatively analyzed both the diameter and shape of the emission. The optical emission spectra, such as H, N, O, CN, and C2 signals, were observed with various time delays. Lee et al. [17] found that the size and concentration of the droplet in the gasoline spray has a linear relationship with the C2 intensity. Since the combustion characteristics in liquid fuels have notable differences compared to hypersonic vehicles, the inlet unstart can cause an in-light engine because it will force the vehicle to stop. Particularly for a LIBS signal.

A general LIBS system has limitations in determining the fuel properties of the environment within the engine. The spectrometer and ICCD camera for the LIBS system are susceptible to damage due to impacts and harsh thermal stimuli, making it impossible to mount these on a vehicle engine. For this reason, we developed a small LIBS plug composed of two photodetectors and bandpass filters that can determine the instantaneous fuel properties to resolve such weaknesses of a real LIBS system. These miniaturized LIBS plugs possess great advantages in terms of weight, size, cost, and complexity when compared to existing LIBS setups.

In the present study, the H and O atomic lines were measured in the gasoline-air mixture using a LIBS plug. The obtained H/O intensity ratio was compared to the equivalence ratio of the gasoline-air mixture. The novelty of the work performed here lies in the design of the LIBS plug, which in principle can be placed as a bundle alongside the combustor wall where the local identification of flow properties of a spray flame are desired in real time. The validity of such photodetectors and filters in place of a full LIBS system (spectrometer/ICCD) was assessed by comparing the signals from both setups.

2. Experimental setup

2.1. Conventional LIBS setup

Fig. 1 shows the experimental setup to monitor liquid-fueled combustion efficiency using conventional LIBS equipment. Ordinary gasoline fuel was purchased from a gas station and was used. The liquid fuel was sprayed with air through a siphon nozzle (Delavan 30609-2). Gasoline was injected through a pump that controls the flow rate, and compressed air was injected through a Mass Flow Controller (MFC). The flow rate of gasoline was fixed at 10 ml/min, and the flow rate of air was fixed at 10 L/min. Nd:YAG laser (Continuum, Surelite I) with wavelength of 1064 nm and pulse duration of 5 ns was used. The laser energy was ~100 mJ with 3.15% coefficient of variation. The laser beam was focused by using a lens with a focal length of 100 mm to generate the plasma. In a conventional LIBS setup, the generated plasma emission was collected using an optical collector or an ICCD camera (Andor iStar) via a spectrometer (Andor iStar 5000). The delay time of the ICCD camera was 1 μs, and the ICCD camera was exposed for 20 μs. Only two peaks (H: 656 nm, O: 777 nm) were identified for the analysis.

2.2. Miniaturizing the LIBS setup: photodiodes and bandpass filter-embedded plug

In this paper, we produced a LIBS plug as part of a novel device setup for combustion diagnosis. Instead of using a spectrometer and ICCD camera with original LIBS equipment, photodiodes and bandpass filters were used for the LIBS plug. The fuel properties were determined using LIBS generally using the equivalence ratio from H and O atomic signals. Accordingly, the LIBS plug was developed using bandpass filters to pass the wavelength corresponding to the H atomic line and O atomic line to determine the equivalence ratio of the fuel. The pictured image of the device is shown in Fig. 2, and the cross-sectional drawing of the plug is depicted in Fig. 3. A laser beam passing through a plug is focused with a lens, generating plasma onto a target point, and the resulting plasma emissions are collected along the same path in the photodiodes with bandpass filters to detect the specific wavelengths of the emission spectrum. The photodiode signals are transmitted to an oscilloscope through a BNC-style cable. The bandpass filters B1 and B2 are utilized to obtain the atomic signals, as shown in Table 2. To obtain the H atomic signal, a bandpass filter with a center wavelength (CWL) of 656 nm and a full width-half-max (FWHM) of 10 nm was used in the device. To obtain the O atomic signal, a bandpass filter with CWL of 780 nm and FWHM of 10 nm was utilized. The diameters of the two bandpass filters are both at 12.5 mm with a minimum transmission of 50%. A UV plano convex fused silica lens was used to focus the laser beam while collecting the plasma emission. The diameter of the convex lens was 15 mm, and the focal length was 25 mm. Si photodiodes (FD5010, Thorlabs) were used to measure the plasma emission. The rise time of the photodiodes was 1 ns, and the peak responsivity was 0.44 A/W at 730 nm. To miniaturize the device, the laser and the plasma paths were aligned into a single path, and the plug was connected to a BNC-style cable to transmit the collected signal.
2.3. Expanded experimental composition in the laboratory

Unlike a conventional LIBS method, laser-induced plasma emission in the droplet was collected using photodetectors with bandpass filters B1 and B2 (656 ± 10 nm and 780 ± 10 nm, respectively, Table 1) to pass through wavelengths corresponding to hydrogen and oxygen atomic lines. The collected emissions were observed using an oscilloscope (Teledyne LeCroy, WAVESURFER 64MXS-B). Fig. 4 shows a schematic illustration of multiple plugs used to detect plasma emissions in gasoline spray. Only a single plug was used in the present study to conduct a proof-of-concept experiment.

The gasoline–air mixture contained in a uniform droplet stream was used to construct a calibration curve with various equivalence ratios. The uniform droplet was generated by the ultrasonic nebulizer with a vibrating ceramic plate at a 1.65 MHz frequency. The droplets were sent to the bunsen burner via an air drying channel. The fuel flow rate into the nebulizer was fixed at 3 ml/min, and the equivalence ratio of the mixture was controlled by using the various air flow rates in the range of 6 L/min to 28 L/min. The mixture conditions are presented in Table 2. The equivalence ratio is defined as:

\[
\phi = \frac{m_{\text{fuel}}}{m_{\text{air}}} / \frac{m_{\text{fuel}}}{m_{\text{air}}}^{\text{stoich}}
\]

The stoichiometric fuel-to-air ratio in the gasoline-air mixture is 0.068, and the inlet velocity of the stream varied from 0.5 m/s to 4 m/s. This specific experimental configuration was derived from our previous research [17].

3. Results

3.1. Spectral lines from conventional LIBS

At first, we performed the original LIBS experiment to investigate the spectra from gasoline in further detail. Fig. 5 shows the signals obtained from a conventional LIBS setup with various equivalence ratios of a gasoline-air mixture. Based on the peak values, H Balmer-α, O I, and N I atomic lines could be identified. O and N signals were detected due to the breakdown of air as an oxidizer. The H signal was generated by the breakdown of gasoline particles (hydrocarbons). Therefore, the H atomic peak increased as the equivalence ratio became higher. However, O and N atomic peaks decreased with a higher equivalence ratio. The H signal had a peak at 656 nm and the O signal peaked at 777 nm (triplet at 777.194 nm, 777.417 nm, 777.539 nm). However, an N signal was generated at both 747 nm (746.831 nm, 744.229 nm, 742.364 nm) and 823 nm (824.239 nm, 821.634 nm). Such small differences in the spectra formed at the same atomic line due to different degrees of degeneracy for each spectrum at the same energy level.

The atomic lines of hydrogen and oxygen with respect to the gate delay time at three different laser energies (50 mJ, 100 mJ, and 150 mJ) with 3.43%, 2.19%, and 1.48% coefficient of variation, respectively) in the spray are shown in Fig. 6 (a) and 6 (b). The gate width was fixed at 20 μs, and the delay time ranged from 100ns to 1μs. Both intensities of the atomic signals increased slightly to about 300 ns, and these signals decreased as time elapsed. The intensity is influenced by various factors, including the laser energy, plasma temperature, number density of fuel droplets in plasma, and the inherent error of the equipment. In particular, the pump...
operated in the pulsation mode, resulting in a non-constant fuel flow rate. Since all spectra are extracted from the same plasma, the intensities of the H and O signals have similar deviations, and the uncertainties in the signal intensities are 18%, 16%, and 12% corresponding to laser energies of 50 mJ, 100 mJ, and 150 mJ, respectively. Kobayashi et al. [18] compared atomic signals according to a delay time in for CH₄, C₂H₄, C₃H₈, and C₄H₁₀ mixtures. They found that the intensities of the H and O atomic signals gradually decreased to about 1 μs while the C atomic signal decayed at approximately 100 ns in all mixtures. However, the atomic signals remained somewhat after 1 μs in the present study because an additional breakdown of hydrocarbon molecules occurred in gasoline droplets rather than in the gas-phase fuel. In addition, many factors, such as the laser energy, exposure time of the camera, and signal amplification of ICCD camera in the present study were different from those in other studies. With a higher laser energy, the signals increased and decayed slower compared to those when a lower laser energy was used. This means that a higher laser energy can generate an intense plasma, subsequently deriving a

<table>
<thead>
<tr>
<th>Air volume flow rate (L/min)</th>
<th>Gasoline volume flow rate (ml/min)</th>
<th>Air mass flow rate (g/min)</th>
<th>Gasoline mass flow rate (g/min)</th>
<th>Equivalence ratio</th>
</tr>
</thead>
<tbody>
<tr>
<td>28</td>
<td>3</td>
<td>34.3</td>
<td>2.16</td>
<td>0.93</td>
</tr>
<tr>
<td>20</td>
<td>3</td>
<td>24.5</td>
<td>2.16</td>
<td>1.3</td>
</tr>
<tr>
<td>12</td>
<td>3</td>
<td>14.7</td>
<td>2.16</td>
<td>2.16</td>
</tr>
<tr>
<td>8</td>
<td>3</td>
<td>9.8</td>
<td>2.16</td>
<td>3.24</td>
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<tr>
<td>6</td>
<td>3</td>
<td>7.35</td>
<td>2.16</td>
<td>4.32</td>
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Fig. 5. LIBS signals in gasoline-air mixture at height of 10 mm from a nozzle with three different equivalence ratios (0.6, 1, and 4.3).

Fig. 6. LIBS signals of (a) H and (b) O according to the delay time at different laser energies (50 mJ, 100 mJ, and 150 mJ).
longer decay time. Furthermore, the plasma volume that contains gasoline droplets is dependent on the laser energy.

Air-breathing engines require a fast diagnostic method to monitor the state of a chemical reaction. For example, the characteristic time of flow inside an SR-71 or X-51A engine is only 10 ms, and this is an extremely short time to determine the properties of a reacting flow using standard LIBS sensors, not to mention issues with size, cost, and complexity associated with a standard LIBS setup. Instead, the necessary flow measurement can be achieved in principle using an improved and reliable setup associated with multiple LIBS plugs arranged in a combustion chamber.

### 3.2. Temporal evolution of plasma emission from photodetectors

Photodetector signals of the plasma emission generated by a laser pulse (100 mJ) from an average of 30 single shots in air with B1 and B2 are shown in Fig. 7(a) and (b), respectively. The signal measured at the beginning of the plasma generation indicates a plasma background continuum [19]. Since the continuum is from a broadband emission, a strong signal can be detected even if a filter is placed in front of the photodetector. However, the plasma emission passing through the B1 filter decayed within about 250 ns after the continuum disappeared, as shown in Fig. 7(a), due to a near zero concentration of hydrogen in air. On the other hand, some extent of the plasma emission passing through the B2 filter remained until after the continuum disappeared because oxygen was present in air (Fig. 7 b). In other words, filters and photodetectors were used to obtain the lifetime of the atomic signals generated from the plasma. In addition, a temporal evolution of plasma emission with photodetectors at various pressures was also observed [20]. At 1 atm, the plasma emission lasted for more than 200 ns. Initially, the photon energy absorption is less under low pressure conditions, but quenching is slower at the same temperature.

The signals of the plasma emissions obtained by using a plug passing through B1 and B2 in the gasoline-air mixture are shown in Fig. 8(a) and 8(b), respectively. Each signal was obtained from an average of 30 single shots. In Fig. 5, the hydrogen signal had a peak at 656 nm and the oxygen signal had a peak at 777 nm, which indicates that plasma emission passing through B1 contained the hydrogen atomic line while the plasma emission passing through B2 contained the oxygen atomic line. Therefore, the emission intensities passing through B1 and B2 represented a hydrogen atomic signal and oxygen atomic signal, respectively.

Comparing Figs. 7(a) and 8(a), one could see that the shape of the signals generated in air was different from that generated in the mixture. The H signal, defined here as the photon signal passing through B1, had a peak at 656 nm, while the O signal, defined here as the photon signal passing through B2, had a peak at 777 nm.

![Fig. 7. LIBS plug signals of (a) H and (b) O in the air.](image)

![Fig. 8. LIBS plug signals of (a) H and (b) O in gasoline-air mixture at various equivalence ratios (0.93, 1.3, 2.16, 3.24, and 4.32).](image)
through the B1 filter in the mixture, remained for a long time unlike that (mostly from the broadband emission, not from H atom) in air, as shown in Fig. 8(a). This occurs because hydrogen is contained in gasoline as fuel, and the hydrogen atomic signal from fuel is added to the background signal. Therefore, a genuine hydrogen atomic signal can be extracted by subtracting the signal obtained in air from that in the fuel mixture. On the other hand, as shown in Figs. 7(b) and 8(b), the O signal from the air did not change significantly. Although the intensity of the signal and the decay time varied depending on the equivalence ratio of the mixture, the overall shape of the signals did not change much due to the constant concentration of oxygen in air. Fig. 8(b) shows that the O signal passing through B2 decreases as the equivalence ratio increases because the amount of oxygen contained per unit volume of the gasoline-air mixture decreased as the equivalence ratio increases.

### 3.3. Analysis of signal intensity

As shown in Fig. 7(a), the plasma background continuum remains for about 250 ns. Atomic signals in gasoline have been reported to disappear within about 1 μs [17]. The hydrogen and oxygen signal intensities were calculated based on the signals in Fig. 8(a) and (b) in the range of 250 ns to 1μs, respectively. The H signal intensity was constant at about 110 mV regardless of the equivalence ratio (Fig. 9). However, the O signal decreased linearly from about 47 mV to 14 mV as the equivalence ratio increased because the equivalence ratio of the mixture was controlled by changing the air flow rate with a constant fuel flow rate. Therefore, the H signal was constant while the O signal decreases with an increase in the equivalence ratio.

To verify the feasibility of using the plug to monitor the combustion efficiency, a conventional LIBS method was conducted to compare a simultaneous detection of the optical spectra under the same environment. Plasma emissions in the gasoline-air mixture of five equivalence ratios (0.93, 1.08, 1.3, 1.44, and 1.62) were detected using conventional LIBS and plug simultaneously by taking 30 laser shots in each composition for a shot-to-shot comparison. The results of the shot-to-shot comparison between the H (656 nm) atomic signal of conventional LIBS and plug with B1 are shown in Fig. 10(a). The results of a comparison of the O (777 nm) atomic signal using the two methods are shown in Fig. 10(b). Both the LIBS signal and plug signal varied according to the intensity of plasma emission regardless of the filter that was used. As shown in Fig. 10(a), although the H (656 nm) intensity measured using the conventional method had the same value with different compositions, the photodetector signals changed slightly. Since the H signal was obtained from fuel, the LIBS signal fluctuated depending on whether a droplet was present at the position where the plasma occurred. In addition, the broadband emission passing through B1 increased as the equivalence ratio increased. Therefore, the data distribution is slightly spread. On the other hand, the broadband emission passing through B2 did not change much regardless of the equivalence ratio, and its data distribution was concentrated as shown in Fig. 10(b). However, the average value of the LIBS signal was lower as the equivalence ratio increased because the O atom in the mixture decreased gradually.

The calibration curve of the H/O intensity ratio with the equivalence ratio of the mixture using the plug (bandpass filters and photodetectors) is shown in Fig. 11(a). The two variables had a nearly linear relationship, with a coefficient of determination (R²) of 0.9958. The spectral signal intensity of the species should vary linearly with the number density of corresponding species, and the equivalence ratio (Φ) is defined as a mass of fuel and air. Then the equivalence ratio can be expressed regarding the atomic number densities of hydrogen and oxygen. Thus, the H and O intensity ratio ended up varying linearly with the equivalence ratio, as has been well described in Ref. [3]. Lee et al. [17] performed an analysis for the LIBS signals in the gasoline spray using a spectrometer and an ICCD camera, and other studies have also demonstrated that the H/O intensity ratio has a linear relationship with an equivalence ratio [2–8]. Therefore, one can confirm the validity of analysis using the plug developed in this study with two-phase flow. During the measurements of the laser focused position, temporal and spatial fluctuations of a two-phase flow might occur. To verify the accuracy of the experiment using a filter and photodetector, it is necessary to study the shot-to-shot variation for a single laser pulse, and the results of a single-shot H/O intensity ratio based on 30 single shots at different equivalence ratios of the mixture are shown in Fig. 11(b). Each data point corresponds to a single shot with an indicated composition of the mixture. As shown in Fig. 11(b), the
The equivalence ratio and ignition probability obtained by 40 laser pulses at three different heights in a gasoline spray are shown in Fig. 12. The success or failure of ignition in a two-phase flow might have been affected by various factors, such as temperature, atomization of the droplets, flow velocity gradient, and local equivalence ratio at locations where a breakdown occurred. The probability of ignition was presented together with the equivalence ratio, and the equivalence ratio according to the position of the spray was calculated using the calibration curve shown in Fig. 11(a) with the probability of the laser ignition measured at the same position. The ignition probability was defined as the number of successful ignitions divided by the total number of laser sparks. At higher locations, further away from the spray nozzle, the gasoline fuel droplets further evaporate, and the fuel vapor diffuses outward to increase the equivalence ratio away from the center of the flow. This increase in equivalence ratio, and likewise for the fuel concentration, results in an overall increase in the number of fuel droplets in the plasma, leading to the generation of a more eminent and brighter flame kernel. The flame kernel lasting for an extended time can transfer its energy to nearby droplets, leading to an overall combustion in the spray. The distribution of the ignition probability that is shown supports this explanation regarding the equivalence ratios that are considered, and such a tendency in the equivalence ratio and ignition probability has also been shown in a methane jet diffusion flame [7].

The LIBS plug can be used to rapidly determine the properties of the reacting flow, and this novel technology can be used to

![Figure 10](image1.png)

**Fig. 10.** Shot to shot comparison between conventional LIBS signals and plug voltage signals of (a) H and (b) O in gasoline-air mixture at various equivalence ratios (0.93, 1.08, 1.3, 1.44, and 1.62).

![Figure 11](image2.png)

**Fig. 11.** (a) Calibration curve for the H/O ratio as a function of the equivalence ratio in the range of 0.92–5.18. (b) H/O ratio obtained in the gasoline mixture for 30 single shot spectra in the cases of $\Phi = 0.93$, 1.3, 2.16, 3.24, and 4.32.
A thorough examination of the effect of the flow properties on the spray to complement the flame stabilization/reliability in an internal engine. Due to the advantages of the device compared to the standard LIBS, a flight vehicle can achieve a reliable engine thrust under all possible harsh flight conditions. Therefore, quantitative measurements of several operating conditions of the air-breathing engines can fully provide practical guidance for innovative applications and device optimization.

4. Conclusions

A compact LIBS plug equipped with photodiodes and bandpass filters was devised to detect selective plasma emission in this study. To verify the feasibility of using plasma spectroscopy with this new device consisting of photodetectors and bandpass filters, the fuel concentration fields were measured in two-phase spray flames. Bandpass filters with $656 \pm 10$ nm and $780 \pm 10$ nm were used to observe the H and O signals, respectively, and the profile of the H signal time history in air showed a different shape and decay rate compared to that in a two-phase fuel-air mixture flow while the O signal showed no such difference. Since the plasma background continuum diminishes with about 250 ns of delay and the atomic emission lines survive until about 1 µs, the photodetector signals from 250 ns to 1 µs were analyzed to extract ideally pure H and O atomic emission signal intensities. The intensity ratios of H (656 nm)/O (777 nm) were found to be linear with the local equivalence ratio of a gasoline-air mixture. Using the calibration curve, the local equivalence ratio in a two-phase spray flame was successfully measured. Therefore, the onboard device with the novel scheme proposed here can be used in principle to characterize the flow properties in real time at various locations within a combustor of air-breathing engines.

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