



OPEN Ultra-low energy LIBS for chlorine detection in cement pastes via microwave enhancement using 0.05 mJ laser energy

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This study investigates chlorine detection in cement pastes using microwave-enhanced laser-induced breakdown spectroscopy (MWE-LIBS) under ultra-low laser energy conditions. The motivation is to enable in situ analysis inside core-drilled cement structures, where laser energy must be minimized to reduce fume generation and avoid structural damage. A pulsed nanosecond laser with energy as low as 0.05 mJ was able to generate the initial plasma; however, this energy was insufficient to produce measurable spectral emissions without microwave (MW) assistance. Laser of 1064 nm was used, When MW was applied, plasma formation was dramatically enhanced without the additional surface ablation, with the plasma volume increasing by over 2200 times. This expansion potentially reduces fume generation and enabled multifiber collection, thereby capturing emissions from a larger plasma region. The MW pulse width and power directly influence Cl I peak values at 837.5 nm. Under maximum MW conditions, a linear calibration curve for Cl concentration from 0.11 to 3.0 wt % yielded a limit of detection (LOD) of 0.204 wt % and a limit of quantification (LOQ) of 0.679 wt %. The relative standard deviation (RSD) dropped below 10% at concentrations above 1 wt %, demonstrating acceptable analytical precision.

Keywords MWE-LIBS, Chlorine detection, Cement analysis, Ultra-low energy laser, Plasma enhancement, Rapid spectroscopy

Evaluating the condition of cement-bound structures is an important part of maintaining the safety and durability of infrastructure, such as bridges, tunnels¹. For reinforced concrete structures, the reinforcement corrosion caused by chlorides often becomes decisive degradation mechanism compromising the integrity of the entire structure. Chlorides typically originate from seawater or de-icing salts². In regions with sub-zero temperatures, de-icing salts are widely applied to roads to maintain traffic safety. Under these exposure conditions, chlorides can penetrate into the concrete by means of diffusion and convection transport³. Once the chloride content at the surface of the reinforcement exceeds a critical value, pitting corrosion may be initiated. This localized form of corrosion is particularly dangerous, as it can lead to severe loss of cross-sectional area of the reinforcement, which may result in structural failure if not detected and remediated in time.

Once corrosion has initiated, predicting the rate of cross-sectional loss under in-situ conditions is extremely difficult due to the multitude of influencing factors, many of which cannot be quantified with sufficient accuracy. Therefore, design standards for new structures aim to minimize the risk of chloride-induced corrosion over service life by avoiding the accumulation of chlorides at the reinforcement up to a critical value, hereby prescribing measures such as adequate concrete cover and density of the concrete itself.

However, experience has shown that in harsh exposure conditions, the requirements according to the design standards of the past have not always been sufficient. As a result, the repair and maintenance of reinforced concrete structures play a critical role in ensuring structural safety and have significant economic implications². For existing structures, engineering guidelines define threshold chloride contents to reduce the risk of reinforcement corrosion. According to the German guideline³ the threshold value is 0.5 wt.% chloride by mass of cement for reinforced concrete and 0.2 wt.% for prestressed concrete. To assess the risk of corrosion of existing structures, powder samples are typically taken and analyzed by potentiometric titration, direct potentiometry or photometry to determine the chloride content^{4–6}. Sampling is commonly carried out by drilling powder

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in defined depth layers directly from the structure or by grinding depth-specific layers from concrete cores obtained from the structure.

As the analyses requires homogenization of the extracted samples (concrete powder), valuable information on chloride gradients at the millimeter scale is lost. Furthermore, conventional methods necessitate assumptions regarding the cement content in the analyzed sample to convert results expressed as chloride by mass of concrete into chloride by mass of cement—the parameter required for corrosion risk assessment. For many existing structures, information on the original concrete mix, and thus on the cement content, is not available, which introduces additional uncertainty into the evaluation process.

Because of these drawbacks, a newer technique called Laser-Induced Breakdown Spectroscopy (LIBS) is becoming more widely used^{7–13}. LIBS offers several advantages over traditional methods. It is faster, often requires minimal sample preparation, and provides spatially resolved measurements, which means it can detect localized concentrations of elements like chlorine with high precision¹³. As a result, LIBS is proving to be a more practical and informative tool for assessing chloride contamination in concrete, especially in real-time or on-site applications¹⁴.

To further improve the capability of LIBS, the MW-enhanced LIBS (MWE-LIBS) was first introduced by Illguth et al. into the cement analysis particularly of using the atomic emission Cl I at 837.6 nm in calibration methodology¹⁵. MWs assistance in LIBS would expand the plasma size and prolong the plasma lifetime^{16–19}. As a result, the capture of emission could be set into the longer gate delay, where background noise is lower and longer exposure time to capture more light. The expansion and sustainment of plasma thus results to better SNR^{20,21}.

One of the key advantages of MWE-LIBS is its ability to operate effectively even when only ultra-low laser energy is available. This becomes particularly important in applications where device size, energy consumption, or safety constraints limit the use of high-energy lasers, as is often the case in in-situ or portable measurement systems. A notable example is the analysis of nuclear debris during the decommissioning of the Fukushima Daiichi Nuclear Power Plant (FDNPP)^{22–27}. In such environments, several factors necessitate the use of very low laser energy: long optical fibers—often more than 10 m—severely attenuate the transmitted energy^{27–32}; compact, semiconductor-based lasers powered by alternating current and positioned near the radioactive debris typically produce only 1 to 3 mJ of pulse energy³³; and toxic fumes generated by laser ablation must be carefully filtered³⁴, further restricting the allowed energy input and exposure time.

Beyond nuclear applications, ultra-low energy LIBS has proven useful in various other fields. In cultural heritage conservation, it enables non-destructive elemental analysis of fragile artworks and historical artifacts, where even minimal surface damage is unacceptable³⁵. In on-site applications, such as detection of silver nanoparticles, laser energy must be strictly limited for both device miniaturization and mobility³⁶. In space exploration, ultra-low energy LIBS is advantageous due to the need for lightweight, energy-efficient instruments, as demonstrated by LIBS systems deployed on Mars rovers^{37,38}. Furthermore, in environmental monitoring, particularly in confined or sensitive ecosystems, portable LIBS systems benefit from operating at low energy to minimize ecological disturbance³⁹.

In this study, we applied ultra-low energy LIBS using 0.05 mJ laser energy to the analysis of cement samples. Under normal conditions, the spectral emissions generated by such low-energy ablation were weak or entirely absent. However, with the application of MW enhancement, a significant increase in plasma size and emission signal intensity are observed. This plasma expansion could be seen with the naked eye. Taking advantage of the MW enhancement effects, we used a multifiber setup to capture a large area of plasma emission. We also investigated how much MW input was necessary to achieve optimum enhancement. Finally, the detection limit of chlorine was analyzed using the calibration data.

Experimental setup

Experiments were performed to determine chlorine concentrations in cement pastes using MWE-LIBS. A schematic diagram of the experimental setup is presented in Fig. 1. A Quantel pulsed laser (Lumibird, France) operating at 1064 nm wavelength, with a laser pulse width of 8 ns and a repetition rate of 10 Hz, was used as the excitation source. Laser energy used is 0.05 mJ and the laser beam was focused onto the sample surface using a lens of focal length 75 mm, producing a spot diameter of approximately 28 μm , and resulting in power density at approximately 1.01 GW/cm². Each cement sample received 50 laser shots. Optical set up is the same as reported in Ref 14.

Helium gas was introduced at a constant flow rate of 3 L/min, with the tip distance at approximately 7.5 mm from the plasma to effectively stabilize and enhance the plasma emission. MW pulses with power ranging from 500 to 1000 W and pulse widths from 0.005 to 1 ms were generated by a semiconductor-based MW generator (i-Lab Inc, Japan) to the plasma through a silver-coated copper spiral antenna positioned at an optimized angle of approximately 10° relative to the laser propagation.

Plasma emissions were collected via bundle fiber optics (7 $\times$ $\varnothing$ 105 μm core fibers) positioned strategically at an angle optimal and transformed into linear bundle fiber directed to a Shamrock Czerny-Turner spectrometer (Andor Technology, Northern Ireland) equipped with a less than 50 μm entrance slit. The spectrometer utilized an 1800 lines/mm grating with a 500 nm blaze wavelength, providing a wavelength detection range of approximately 17 nm and achieving a spectral resolution of approximately 0.02 nm. This setup enabled accurate detection and quantification of chlorine concentrations in cement, covering a measured range from 0.11% to 3.28%.

Multi-track feature

One of the key features of the optical collection system is the use of a bundle fiber optic collector composed of seven 105 μm -diameter optical fibers, which significantly enhances both the spatial coverage of the plasma

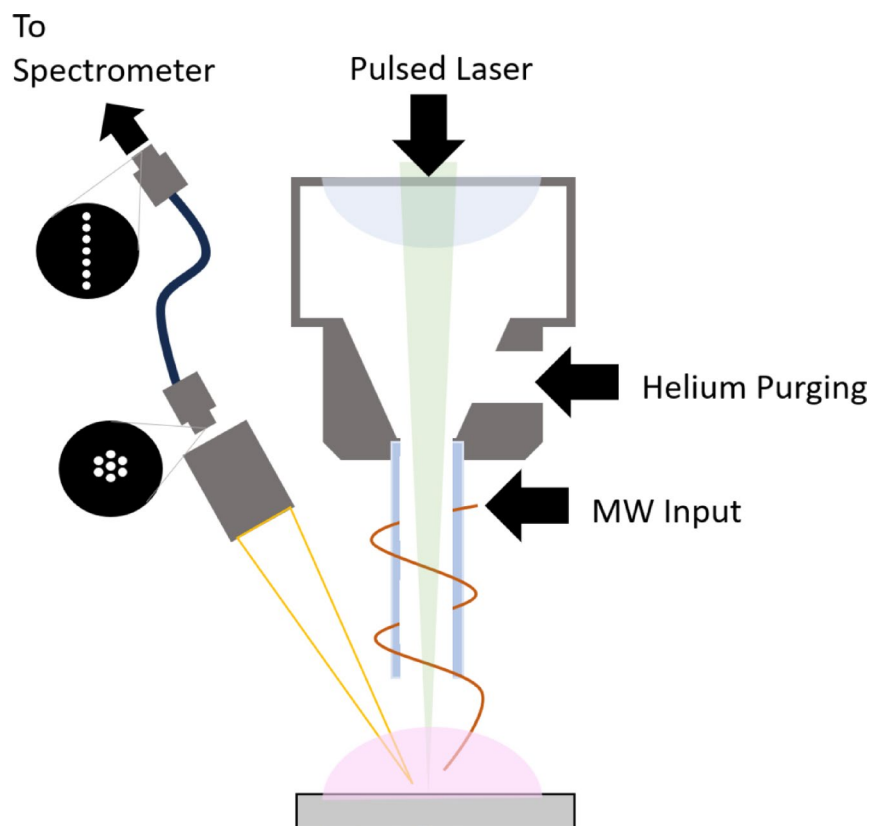


Fig. 1. Experimental setup of MWE-LIBS with a concentric antenna aligned along the laser propagation direction¹⁴.

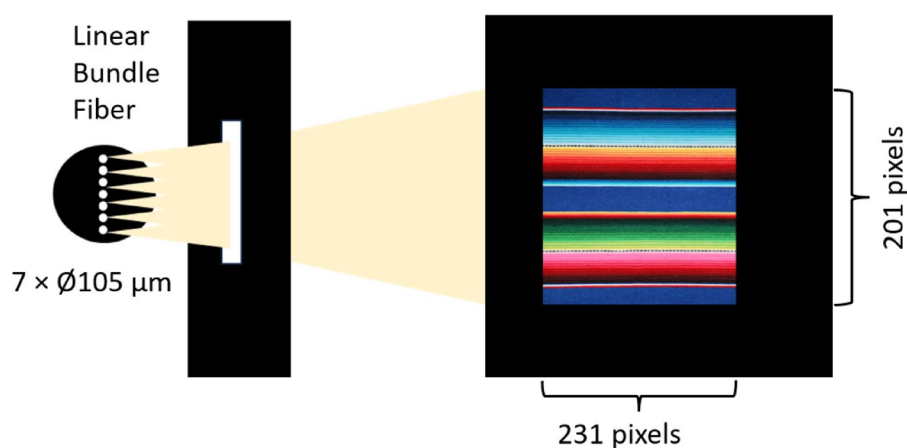


Fig. 2. The linear fiber bundle ($7 \times \text{Ø}105 \mu\text{m}$ core fibers) directs plasma emission into the CCD camera, spreading the collected light over a larger area (231 pixels (X) $\times$ 201 pixels (Y)) to increase spatial coverage and signal collection efficiency.

emission and the efficiency of light delivery to the detector. The bundled fiber head with an effective core diameter of $355 \mu\text{m}$ is designed to collect light from a wide area of the plasma, ensuring that emissions from various regions of the breakdown zone are captured. This light is then reorganized into a linear fiber array ($0.90 \text{ mm} \times 0.13 \text{ mm}$) that aligns at the entrance slit geometry of the Shamrock spectrometer as shown in Fig. 2. By converting a spatially distributed plasma emission into a linear projection, the system maximizes the use of the entrance slit width and improves the intensity distribution across the CCD detector using its multi-track feature. The active emission region is located between vertical pixels (px) $Y = 400$ to 600 , where plasma light is captured, and horizontal pixels $X = 110$ to 340 , where the spectral signal is extracted. This means the signal is concentrated within a 231-pixel-wide spectral range (X) and a 201-pixel-tall spatial range (Y). All other pixels

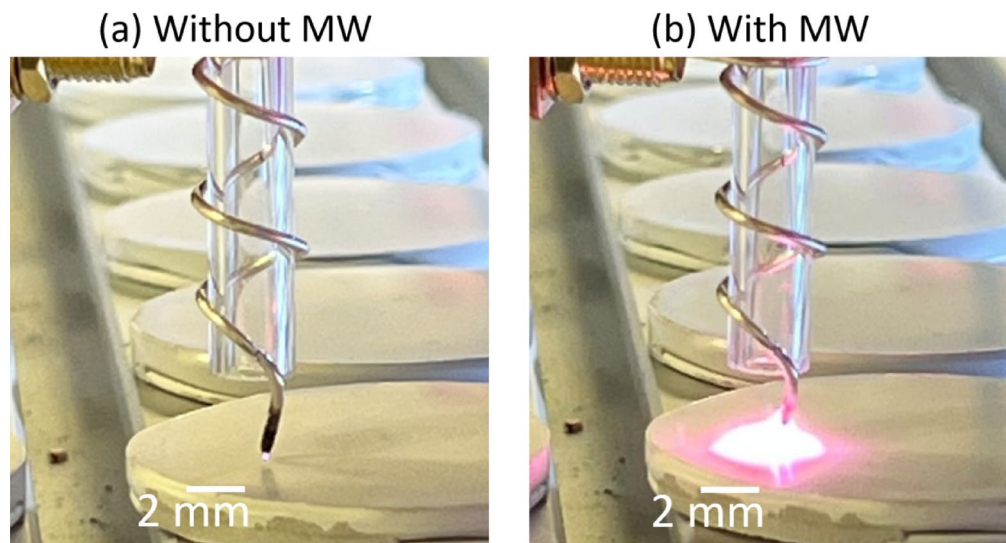


Fig. 3. Comparison of plasma size (a) with and (b) without MW enhancement using ultra-low laser energy of 0.05 mJ. The images were captured by an iPhone 13 Pro.

	Without MW	With MW
Radius (mm)	0.20 ± 0.01	2.59 ± 0.21
Area (mm ²)	0.13 ± 0.01	21.04 ± 3.45
Volume (mm ³)	0.02 ± 0.01	36.30 ± 8.93

Table 1. Plasma radius, area, and volume comparison in ultra-low laser MWE-LIBS. The measurements were extracted from python-based image processing. The error bars were determined by adjusting the threshold range for dark pixel values within the 0–255 RGB scale.

outside these ranges—specifically $X = 1\text{--}109$ and $341\text{--}1024$, and $Y = 0\text{--}399$ and $601\text{--}1023$ —can be considered black or background pixels and are typically omitted from analysis. This arrangement results in higher signal collection efficiency and improves spectral resolution for analysis.

Cement sample preparation

The cement paste samples were prepared using Ordinary Portland Cement (CEM I 42.5R) in accordance with DIN EN 197–1 standards⁴⁰. Chlorine levels were controlled by dissolving specific amounts of salt in deionized water, with concentrations calculated based on the salt mass. The cement was mixed with water at a ratio of 0.5, sealed in polyethylene bags, and cured at 23 °C for 28 days. After curing, samples were dried at 105 °C to a constant weight, then ground to a powder with particle sizes below 90 μm. The resulting powder was pressed into circular pellets (4.0 cm diameter, 1.0 mm thickness) at 100 N/mm² for 2 min. A total of 10 Cl concentrations between 0.11 and 3.28 wt % were prepared. For most experiments, the 3.28 wt % sample was used, except for calibration procedures. Further preparation details are available in reference¹⁶.

Results

The addition of MWs can dramatically enhance plasma formation in ultra-low energy LIBS, as shown in the images captured with a smartphone camera (iPhone 13 Pro, Fig. 3). Both images were taken under the same conditions: a silver-coated copper helical antenna is placed above the sample, and helium gas flows steadily at 3 L/min. An ultra-low-energy pulsed laser (0.05 mJ, 1064 nm) is used to generate the initial plasma. The energy used in this ablation is 1.01 GW/cm which created a minute plasma ablation which is almost invisible to the eye. With MW application, a 1 kW MW pulse lasting 1 ms is applied together with the laser. The MW-enhanced plasma is strong and enlarged significantly.

Table 1 shows the radius, area, and volume measurements of the plasma plume with and without MW, obtained using image processing in Python. The image was converted to 8-bit and analyzed using pixel-based measurement techniques. The difference is clear: the plasma remains small without MW, with a radius of 0.20 ± 0.01 mm, an area of 0.13 ± 0.01 mm², and a volume of just 0.02 ± 0.01 mm³. In contrast, the plasma grows significantly with MWs, reaching a radius of 2.59 ± 0.21 mm, an area of 21.04 ± 3.45 mm², and a volume of 36.30 ± 8.93 mm³. This means that the plasma volume increased by more than 2200 times, which is significantly greater than the expansion effects of 18–50 times reported for 1–3 mJ laser energy in the references^{16–18}.

Figure 4 presents the corresponding chlorine emission spectra under the same experimental conditions described in Fig. 3. The spectra were acquired using 30 μs gate width and 1 μs gate delay, over a narrow 15 nm

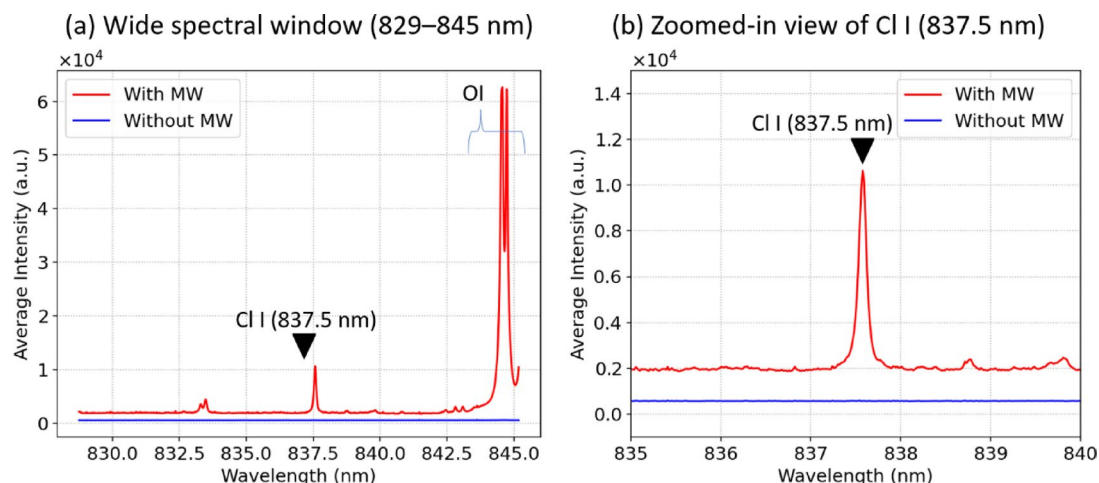


Fig. 4. Chlorine emission spectra with and without MW enhancement. **(a)** Spectra over a limited 15 nm range (829–845 nm) showing clear MW-induced enhancement of Cl I (837.5 nm) and O I lines. **(b)** Zoomed-in view centered on the Cl I line at 837.5 nm, highlighting the significant intensity increase due to MW application.

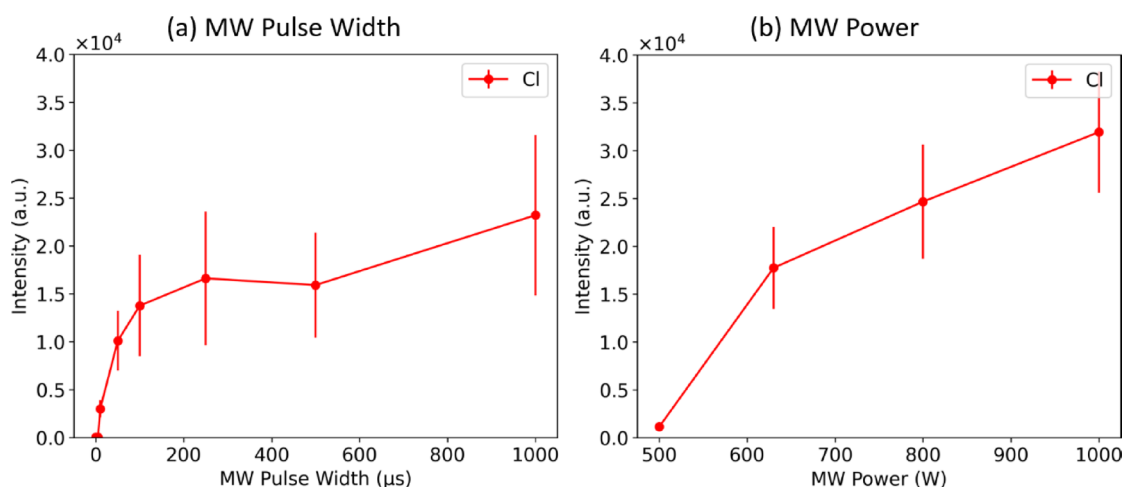


Fig. 5. **(a)** Effect of MW pulse width on the emission intensities of Cl I in cement samples. MW pulse width was varied from 5 to 1000 μ s. **(b)** Effect of MW power on emission intensity. MW power varied from 500 to 1000 W. Error bars represent the standard deviation from 64 replicates.

window (829–845 nm), to focus on the Cl I emission. In panel (a), the spectrum with MW enhancement (red line) reveals clear emission peaks at 837.5 nm (Cl I) and 844.6 nm (O I), which are not detectable in the absence of MWs (blue line). Panel (b) shows a zoomed-in view of the Cl I line where the baseline also increased with MWs.

Spectral acquisition was performed using 50 accumulated shots. A single shot showed only a minimal emission peak, which was not clearly distinguishable against the background (data not shown). The expansion and sustainment of the plasma by MWs directly translate into stronger spectral emission in ultra-low energy LIBS. This effect was previously observed only under conditions with a delayed gate delay^{21,22}.

In the variation of MW pulse width and power, Fig. 5 shows the range of MW enhancement effects on Cl I intensity. The MW pulse width was varied from 5 to 1000 μ s, during which Cl I emission intensities increased significantly with longer pulse widths. It is apparent that extended MW pulses enable sustained re-excitation of atoms, as expected, since plasma sustainment is proportional to the pulse width. In this experiment, we used a relatively long exposure time of 1000 μ s to allow more light to be captured. We expected to observe the point where enhancement effects begin to diminish, which occurred below 100 μ s. Between 100 and 1000 μ s, the enhancement effects significantly plateaued, especially when considering the error bars.

Figure 5b shows the emission intensity as a function of MW power, which was varied from 500 to 1000 W. The intensities steadily increased with increasing MW power, suggesting that higher MW power improves plasma sustainment and excitation efficiency. Finally, a MW power of 600 W and a pulse width of 100 μ s were sufficient to significantly enhance the Cl emissions.

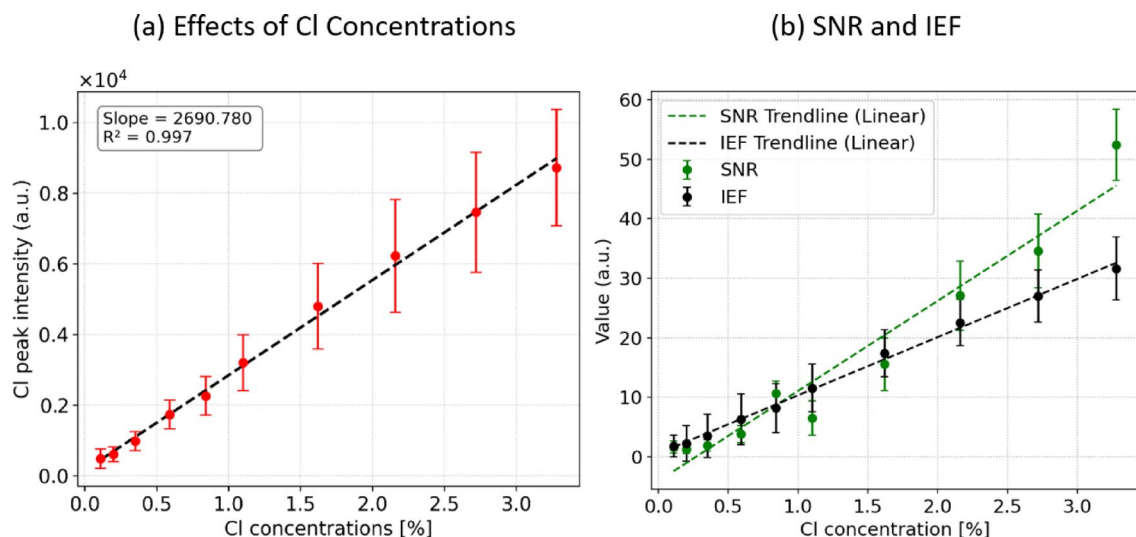


Fig. 6. Calibration and enhancement behavior of chlorine emission in MW-LIBS. **(a)** Linear calibration plot of Cl I intensities (with error bars) as a function of Cl concentration [%] in cement. The strong correlation ($R^2 = 0.997$) demonstrates excellent linearity of the LIBS signal response. **(b)** Signal-to-noise ratio (SNR) and intensity enhancement factor (IEF) versus Cl concentration. Error bars represent the standard deviation from 64 replicates.

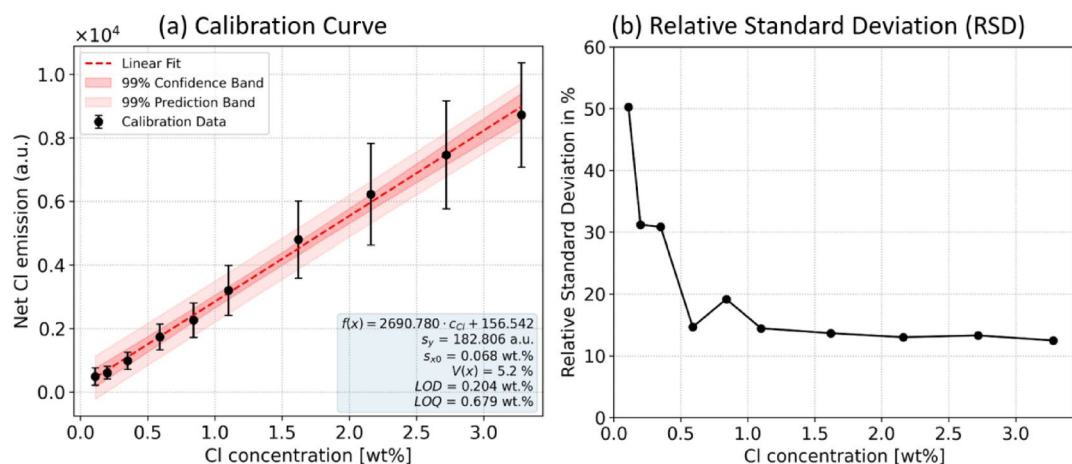


Fig. 7. **(a)** Calibration curve for Cl I detection in cement using ultra-low energy MWE-LIBS using 0.05 mJ. The linear regression line (red dashed) is shown with 99% confidence and prediction bands. Error bars represent the standard deviation of repeated measurements. **(b)** Relative standard deviation (RSD) of Cl intensity across concentrations, showing improved precision at higher concentrations.

Figure 6a shows a linear calibration plot of Cl I peak intensity versus Cl concentration in cement. The measured intensities exhibit a strong linear relationship with concentration, as indicated by the regression coefficient $R^2 = 0.997$ and a slope of approximately 2690.78. Error bars represent the standard deviation of repeated measurements, accounting for experimental variability due to plasma fluctuations and sample heterogeneity. This linearity confirms that MW-LIBS is suitable for quantitative chlorine analysis and can be further used for detection limit quantification.

Figure 6b shows the evolution of the signal-to-noise ratio (SNR) and the intensity enhancement factor (IEF) with increasing Cl concentration. Both metrics show a linear trend, with the SNR increasing more sharply than the IEF. The SNR is defined as the ratio of the emission signal to background noise, and its increase indicates improved clarity and detectability of the Cl signal at higher concentrations. The IEF, which compares the Cl peak intensity with and without MW application, also increases, reflecting more efficient energy coupling and excitation under higher analyte densities.

The quality of the calibration model for chlorine detection in cement using ultra-low energy MWE-LIBS was evaluated using standard statistical indicators⁹, as shown in Fig. 7. The net Cl emission intensity increases linearly with Cl concentration from 0.11 to 3.0 wt %. The primary parameter to assess detection capability is the

limit of detection (LOD), which represents the lowest measurable concentration that can be distinguished from the background. For this calibration, the LOD was calculated to be 0.204 wt %, while the limit of quantification (LOQ), indicating the lowest concentration that can be quantified with acceptable precision, was 0.679 wt %. Moreover, the calibration curve for Cl emission intensity versus Cl concentration results in the linear equation:

$$f(x) = 2690.780 \cdot \text{Cl I (1/wt \%)} + 156.542 \tag{1}$$

The standard deviation of the residuals, $S_y = 182.806$ au., was used to calculate the standard deviation of the calibration $S_{x0} = 0.068$ wt %. These values reflect how well the measured LIBS signals fit the regression line. The relative standard deviation of the calibration, denoted as $V(x)$, was determined to be 5.2%, which indicates the overall precision of the model over the tested concentration range. The 99% confidence and prediction intervals are also plotted to visualize the uncertainty of the fit and the expected variability in future measurements.

The precision of individual measurements at each concentration was assessed by evaluating the relative standard deviation (RSD), as presented in Fig. 7b. At low Cl concentrations, particularly below 0.5 wt %, the RSD was high—exceeding 50%—due to the low signal-to-background ratio. As the concentration increased, the RSD decreased sharply and stabilized below 10% for concentrations above 1.0 wt %, demonstrating improved reproducibility and signal quality at higher concentrations.

Discussions

The detection of chlorine in cement has been successfully demonstrated using ultra-low energy MWE-LIBS. The plasma dynamics follow the same principles as in standard MWE-LIBS, where the re-excitation of electrons by MWs increases the probability of collisions over a sustained period. This leads to an enlarged plasma, visible optical emission (in contrast to the invisible emission observed in conventional LIBS under the same laser conditions), and improved calibration curve quality.

However, there is still significant room for improvement, particularly in detection performance when compared to previous studies, as shown in Table 2. In this work, the system achieved a limit of detection (LOD) of 0.204 ± 0.068 wt % and a limit of quantification (LOQ) of 0.679 ± 0.204 wt % for chlorine. Although these values are slightly higher than those using higher energy standardized systems—such as the standard MWE-LIBS method (LOD: 0.17 ± 0.02 wt %) or the German DIN 32645 standard (LOD: 0.03 wt %, LOQ: 0.11 wt %)—they are still competitive given that only 0.05 mJ of laser energy was used in our system.

In comparison, conventional LIBS setups report LODs in the range of 0.87 ± 0.07 wt %, with some interlaboratory efforts achieving as low as 0.01–0.10 wt %, with higher energy conditions. The LOD results in this manuscript is much lower than from reference 14 but is higher with reference 13. However dual-pulsed LIBS (DP-LIBS) study from Jiaotong University, for instance, reported a range of 0.19–0.26 wt %, which is on the same range.

Ultra-low energy MWE-LIBS has its ups and downs, and Fig. 8 summarizes its strengths. This graph illustrates the trade-off between device simplicity and the sophistication and cost of the spectrometer and optical system. In traditional LIBS, as shown by the blue curve, maintaining relative detection sensitivity typically requires more complex and expensive optical systems, and the device typically becomes heavier. However, the red curve representing ultra-low energy MWE-LIBS shows that introducing MW enhancement allows for significant improvements in sensitivity even with less expensive, lightweight spectrometer devices. This means that MWE-LIBS can achieve strong spectral signals and lower limits of detection without relying on high-specification or costly optics, making it a more efficient and practical solution for applications that require portability and cost-effectiveness.

Conclusions

This study demonstrated the successful detection of chlorine in cement using ultra-low energy MWE-LIBS. By combining a 0.05 mJ laser pulse with a 1 kW MW pulse, stable and visibly enhanced plasma something not possible using LIBS alone at this energy level. The MW sustainment mechanism enabled prolonged excitation and increased collision probability, resulting in a larger plasma volume and significantly stronger optical emission.

The system achieved a limit of detection (LOD) of 0.204 ± 0.068 wt % and a limit of quantification (LOQ) of 0.679 ± 0.204 wt %, which are competitive with or better than conventional LIBS methods despite the radically

	Condition	LOD/LOQ, wt %
This Manuscript	Ultra-Low Energy MWE-LIBS, 0.05 mJ	0.204 ± 0.068 / 0.679 ± 0.204
From References	LIBS, 3 mJ ¹⁴	0.87 ± 0.07 / **
	Standard Method (MWE-LIBS), 3 mJ ¹⁶	0.17 ± 0.02 / **
	German standard DIN 32645*, 3 mJ (MWE-LIBS) ¹⁴	0.03/0.11
	Global Interlaboratory Experiments (LIBS), 3.8–25.5 GW/cm ²¹³	0.01–0.10/0.03–0.33
	Jiaotong University, China (DP- LIBS) 15 mJ + 30 mJ ⁴¹	0.19–0.26/ **

Table 2. Comparison of chlorine detection performance (LOD/LOQ) between the ultra-low energy MWE-LIBS system developed in this work and previously reported LIBS and MWE-LIBS methods. *Similar method with DIN 32645. **LOQ was not measured.

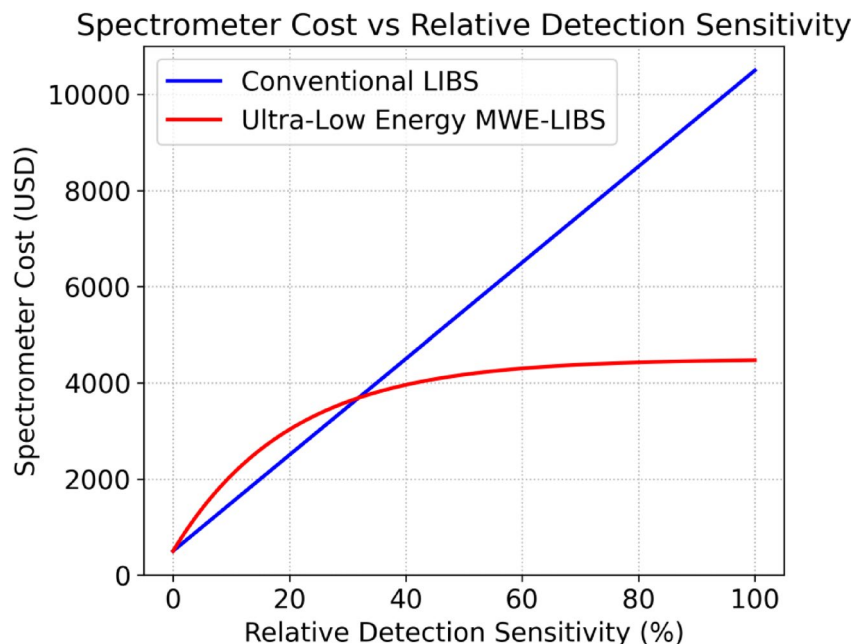


Fig. 8. Spectrometer cost versus relative detection sensitivity for traditional LIBS (blue) and ultra-low energy MWE-LIBS (red). Relative detection sensitivity is normalized such that 100% corresponds to the highest achievable sensitivity in the system. This figure is a conceptual representation and does not correspond to exact experimental data.

reduced laser energy. We confirm from this study that MW enhancement effectively compensates for low laser power, enabling high-sensitivity detection in compact and energy-efficient configurations.

Data availability

The data underlying the results presented in this paper are not publicly available at this time but may be obtained from the authors upon reasonable request.

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Declarations

Competing interests

The authors declare no competing interests.

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