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Journal of Applied Spectroscopy

1. Draft manuskrip sebelum proses submission
2. Submission paper ke Journal of Applied Spectroscopy (13 Oktober 2017)
3. Editor and reviewers' comments (22 Januari 2018)
4. Balasan komentar Editor dan Reviewers
5. Paper setelah proses revisi mempertimbangkan masukan Editor dan Reviewers
6. Final recommendation to be accepted (16 Februari 2018)
7. Final proof (20 Desember 2018)
8. Bukti Publish (24 Januari 2019)

1. Draft manuskrip sebelum proses submission

Laser-Induced Gas Plasma Spectroscopy for the Analysis of Carbon in Soil

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Abstract

Analysis of carbon (C) in soil has been carried out by using laser-induced gas plasma spectroscopy utilizing specific characteristics of transversely excited atmospheric (TEA) CO₂ laser. In this study, fine particles of soil sample were painted on a metal surface by adding little amount of moisture, the metal functions to initiate the gas plasma. When a TEA CO₂ laser was focused on a metal surface, high temperature and large volume gas plasma was induced. The fine particles were vaporized and moved into the gas plasma region to be dissociated and excited. By using the technique, semi quantitative analysis of C in soil has been made. A further measurement revealed an excellent linear calibration curve with zero intercept. The detection limit of C in the soil was approximately 23 mg/kg.

Keywords: Laser-induced breakdown spectroscopy; LIBS; Laser-induced gas plasma spectroscopy; Carbon analysis; Soil sample

1. Introduction

Recently, carbon inventories in soils have changed due to the serious global climate change [1-3]. Soil carbon is fundamental determinant of many soil chemical and physical properties such as nutrient availability, soil structure, and water holding capacity, which directly influence ecosystem quality and soil quality [4-5]. The use of more coal and oil for homes, factories, and transportation releases carbon dioxide and other greenhouse gases in atmosphere, resulting in increasing the temperature of atmosphere. This increment of the temperature influences the soil carbon dynamics and reduces the annual change of soil organic carbon [5]. Thus, an efficient technique of measuring soil carbon is required to estimate the terrestrial carbon inventories.

Several analytical techniques have been employed to the study of soil carbon. X.M. Zou et al. estimated soil labile organic carbon by using a sequential fumigation-incubation procedure [6]. Paul et.al. applied the technique to predict the retention time and rates of carbon turnover in soils [7].

Laser-induced breakdown spectroscopy (LIBS) has recently become very popular technique for rapid qualitative and quantitative elemental analysis [8]. In this method, a pulsed neodymium yttrium aluminium garnet (Nd:YAG) laser is usually employed as an excitation source. When the Nd:YAG laser was focused on a sample target, a strong

luminous plasma is produced; the plasma contains the atomic and ionic elements as well as molecules from the target. The plasma was then imaged to the optical spectrometer to obtain the emission spectrum. However, the standard LIBS cannot make direct analysis on soil sample because once the pulse ND:YAG laser was focused on the soil target, the blow off of soil seriously occurs. Therefore, to perform analysis of soil by using this method, the soil is most often pressed into a pellet. We noticed that even though the sample is pressed into the pellets in order to avoid the blowing-off of the samples, the samples is still not as hard as the other solid samples, such as glass or ceramic. Therefore, a strong shock wave, which is necessary to induce a high-temperature plasma [9], cannot be produced due to the lack of repulsion forces on the sample surface. As a result, the atomic emission intensity of the plasma is rather weak for the case of ordinary LIBS using Nd-YAG laser.

On the other hand, we have found that a TEA CO₂ laser is suited to soil sample because the wavelength of the TEA CO₂ laser is longer than that of the Nd: YAG laser, resulting in high absorbance in the soil [10]. Also, the long pulse of the TEA CO₂ laser enables one to produce strong and long-lived laser plasma, in which the dissociation of the soil particles and excitation of the atoms effectively take place. We can produce a long-lived laser plasma even when we focused the TEA CO₂ laser directly on the soil.

However, this method still suffers from low sensitivity.

In this study, we proposed a unique technique for the direct analysis of carbon in soils by means of the specific characteristics of a TEA CO₂ laser and utilizing subtarget effect to induce a high-temperature and large-volume gas plasma. The tiny amount of soil particles faintly painted on a metal plate by adding a moisture. When a TEA CO₂ laser was focused on the metal, on which the thin layer soil painted, a high-temperature gas plasma was produced, leaving the metal plate without any ablation and damage. By using this method, estimating of carbon (C) inventories in soils can be made, thus global climate change can be predicted.

2. Experimental Setup

The experimental setup used in this study was shown in Fig 1(a). The laser employed for radiation source was pulsed transversely excited atmospheric (TEA) CO₂ laser (Shibuya SQ 2000 laser, pulse energy of 3 J, wavelength of 10.6 μm , pulse duration of 200 ns in full width half maximum, beam cross section of 3 mm x 3 mm. The laser energy was fixed at 1.5 J by setting an aperture in the path of laser beam and focused through a ZnSe window on a sample using ZnSe lens of 100 mm in focal length. The spot size of laser beam for the tight focusing condition was 1 mm x 1 mm, which results

in a power density of 0.75 GW/m^2 .

The sample applied in this study was a carbon soil sample collected from the garden. For semi-quantitative analysis, a standard soil sample containing various concentrations of carbon (200, 500, and 1000 mg/kg) was used. Prior to the analysis, the small soil particles with moisture were directly painted on a nickel plate (dimension of 20 mm x 20 mm), which serves as a metal subtarget, as illustrated in Fig. 1(b). During the experiment, the samples were placed in a metal chamber with dimensions of 12 cm x 12 cm x 12 cm, which was filled with nitrogen gas. The pressure of the surrounding gas in the chamber was set at 1 atmosphere. The flowing rate of gas was 3 liters per minute (Lpm). It should be mentioned that the gas flowing is required in this experiment, otherwise gas breakdown takes place in front of the sample due to the interaction between dust and TEA CO_2 laser beam.

The plasma emission spectrum was obtained by detecting laser plasma radiation using optical multi channel analyzer (OMA) system (ATAGO Macs-320) consisting of a 0.32 m focal-length spectrograph with a grating of 1200 groves/mm, a 1024-channel photodiode detector array, and a micro-channel plate image intensifier. The spectral resolution of the OMA system is 0.2 nm. The light emitted from the laser plasma was collected by an optical fiber ($\theta = 27^\circ$ in solid angle) and fed into the OMA system. One

end of the fiber was placed at 15 cm distance from the focusing point of the laser light and set perpendicularly to the path of laser beam. Both OMA systems were set in time-integrated mode for with exposure time of 2 sec under the laser operation of 10 Hz, thus averaging 20 shots.

3. Results and Discussion

First, the soil carbon assessment was made by using our present technique, meta-assisted laser-induced gas plasma spectroscopy. To this point, a small amount of soil fine particles (around 500 mg) with moisture were spread on the nickel plate, which serves as a metal subtarget. Figure 2 shows the emission spectrum taken from the soil sample. It is seen that a strong emission intensity of C I 247.8 nm clearly appears with low background intensity. Furthermore, many typical atomic lines of Si showing characteristics of the soil sample can readily be observed in the right side of the carbon line. Carbon atom is contained in garden soil at several thousands ppm (part per million or mg/kg). This result indicated that our present technique offers high possibility to measure the carbon concentration in soil influenced by the global climate change. Furthermore, the present technique is superior to ordinary LIBS technique for conducting rapid and efficient measurements of total soil carbon. For the case of

ordinary LIBS technique, in which Nd:YAG laser is usually used as an energy source, the soil sample must be prepared in the form of hard pellet to produce a strong shock wave that is necessary to induce a high-temperature plasma. Otherwise, the strong shock wave cannot be produced due to the lack of repulsion force on the sample surface. Thus, the ordinary LIBS technique is tedious sample preparation. While, in our present technique, the analysis can be performed without sample pretreatment because we only directly painted the soil sample with moisture on the metal subtarget prior to the analysis.

It is assumed that the mechanism of plasma generation for the case of our present technique is as follows: when a pulsed TEA CO₂ laser was focused on the nickel plate as displayed in Fig.1(b), on which the thin layer soil painted, a strong plasma was produced leaving the metal plate without any ablation and damage because the power density of the laser beam impinging on the metal is lower than the ablation threshold of the metal due to the fact that most part of laser energy is absorbed by the strong laser plasma (plasma shielding effect). It is hypothesized that when the metal subtarget is placed in tight contact on the backside of the thin soil sample, the forward momentum of the strongly propelled atoms is enhanced by the repulsion force supported by the subtarget, generating a strong shock wave. Based on our previous studies related to the

excitation mechanism through shock wave model [11], the propelled atoms gushing out with very high speed are supposed to move in a manner somewhat like a piston pushing the surrounding gas to form a shock wave. During this stage, atoms are excited by heat arising from the compression between the shock front and the propelling atoms. The most important point in the excitation via shock wave is that the energy required to excite atoms is the gas plasma induced by the shock wave, which is converted from the kinetic energy of propelled atoms. Therefore, a hard sample is really necessary to induce high kinetic energy of propelled atoms. It is assumed that the fine particles of soil sample were vaporized by the pulsed laser and moved into the gas plasma to be dissociated and excited. For the case of soft sample, it is supposed that the expulsion of atoms by the surface is weakened because the soft sample surface absorbs the recoil energy and the forward-moving atoms do not acquire sufficient speed to form a shock wave.

The method described above was adopted for a study on semi-quantitative analysis of the company soil sample containing various concentrations of C. Taking advantages of the fact that the ratio intensity between C I 247.8 nm and Si I 251.9 nm is almost constant, the Si emission line was employed as a standard. An excellent linear calibration curve with a zero intercept was successfully made for the soil sample, as

presented in Fig. 3. It should be noted that the use of silicon (Si) for standardization should be evaluated with regard to soil mineralogy because the Si content in soils varies with the mineralogy of soils.

In order to examine the sensitivity of the present technique, an analysis of carbon in soil was conducted. Figure 4 shows the spectral emission taken from a company soil sample containing 500 mg/kg of carbon. The emission line of C I 247.8 nm can clearly be seen along with many lines of the Si in the right side of the carbon. The detection limit of carbon was estimated to be around 23 mg/kg; the detection limit was derived by calculating what concentration of the signal yielded 3 times of the noise level is because the 3 times of the noise is clearly identified as a signal that can be distinguished from the noise [12]. This result is superior to the case of ordinary LIBS, which the detection limit of C was around 300 mg/kg as reported by other researchers [4]. Therefore, our present technique is very potential to perform C analysis in soil carbon influenced by the global climate change.

4. Conclusion

It was demonstrated that the analysis of carbon in soil sample can be carried out by using metal-assisted laser-induced gas plasma spectroscopy utilizing specific

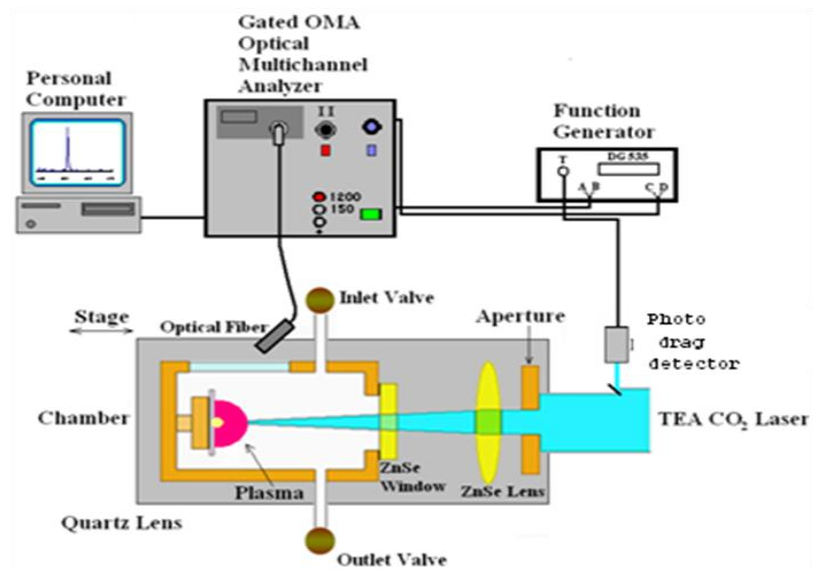
characteristics pulsed TEA CO₂ laser. When a pulsed TEA CO₂ laser was focused on the metal surface, on which soil fine particles painted, a high-temperature and large-volume gas plasma was produced. The fine particles was vaporized and moved into the gas plasma region to be dissociated and excited. Using the present technique, semi-quantitative analysis of carbon in soil sample was made. A nice linear calibration curve with zero intercept was obtained for the soil samples containing different concentration of carbon. The detection limit of carbon was approximately 23 mg/kg. Even though further study is required, this technique has high possibility to be applied for carbon analysis in soil to measure the dynamics of carbon due to the global climate change.

References

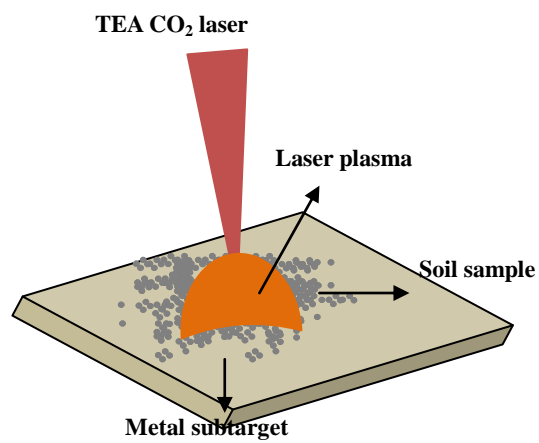
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Figure captions

- Fig. 1. (a) Experimental setup used in this study, and (b) lustration of the sample holder employed in this study.
- Fig. 2. Emission spectrum taken from the garden soil by using metal-assisted laser-induced gas plasma spectroscopy.
- Fig. 3. Calibration curve of carbon content in the soil containing various concentration of carbon by using metal-assisted laser-induced gas plasma spectroscopy.
- Fig. 4. Emission spectrum taken from the soil containing 500 mg/kg of C using metal-assisted laser-induced gas plasma spectroscopy.



(a)



(b)

Figure 1

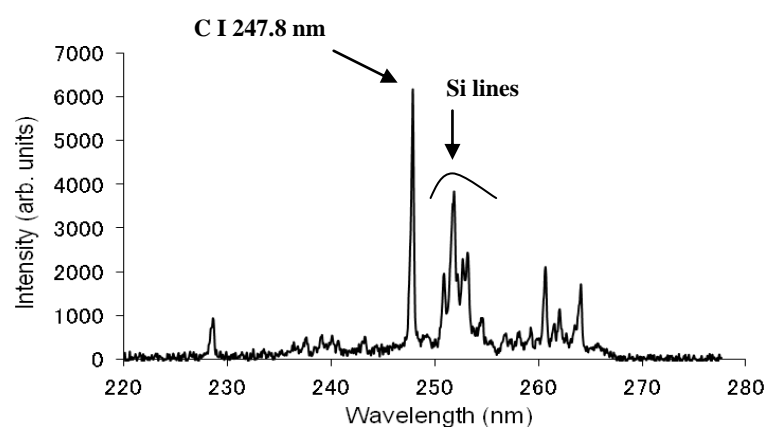


Figure 2

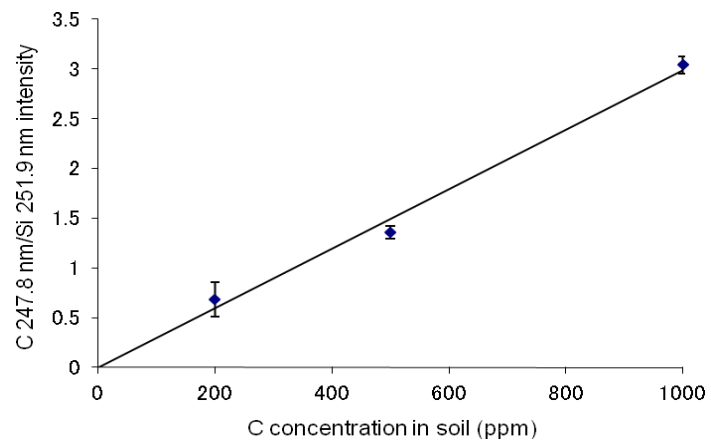


Figure 3

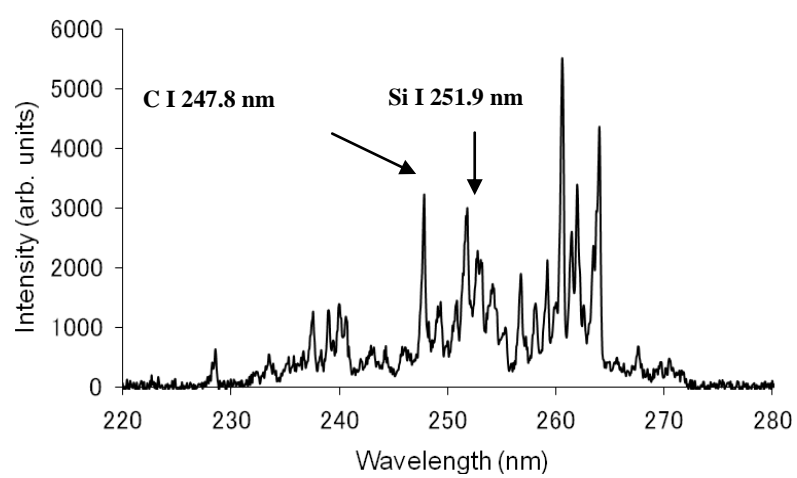


Figure 4

2. Submission paper ke Journal of Applied Spectroscopy (13 Oktober 2017)

Submission of manuscript to Journal of Applied Spectroscopy

Ali Khumaeni <khumaeni@fisika.undip.ac.id>

Fri, Oct 13, 2017 at 8:06 AM

To: jas on Dragon <jas@dragon.bas-net.by>

Dear Professor V. A. Tolkachev
Editor in Chief
Journal of Applied Spectroscopy

Good morning,

I am Ali Khumaeni, from Department of Physics, Diponegoro University, Indonesia. I would like to submit a final manuscript entitled "Laser-induced gas plasma spectroscopy for the analysis of carbon in soil" to Journal of Applied Spectroscopy. Please kindly find the manuscript as in attachment.

Thank you very much for your kindness.

Best regards
Ali Khumaeni
Assistant Professor
Department of Physics
Diponegoro University
Semarang, Indonesia
email: khumaeni@fisika.undip.ac.id

2 attachments**FINAL FIGURES.pdf**
146K**MANUSCRIPT.pdf**
147K

252-17 Re: Submission of manuscript to Journal of Applied Spectroscopy

jas on Dragon <jas@dragon.bas-net.by>
Reply-To: jas on Dragon <jas@dragon.bas-net.by>
To: Ali Khumaeni <khumaeni@fisika.undip.ac.id>

Fri, Oct 13, 2017 at 11:00 PM

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Friday, October 13, 2017, 4:06:36 AM, you wrote:

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email: khumaeni@fisika.undip.ac.id

--

Best regards,
jas

<mailto:jas@dragon.bas-net.by>

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3. Editor and reviewers' comments (22 Januari 2018)

Re: 252-17 Re: Submission of manuscript to Journal of Applied Spectroscopy Review

jas on Dragon <jas@dragon.bas-net.by>
Reply-To: jas on Dragon <jas@dragon.bas-net.by>
To: Ali Khumaeni <khumaeni@fisika.undip.ac.id>

Mon, Jan 22, 2018 at 4:56 PM

I support the acceptance of this work but before publication I would recommend the following revisions:

1. Introduction "However, the standard LIBS cannot make direct analysis on soil sample because once the pulse Nd:YAG laser was focused on the soil target, the blow off of soil seriously occurs". There are many pub-lications where standard LIBS is used for the direct analysis of carbon in soil samples (see for example Belkov, M.V., Burakov, V.S., De Giacomo, et al Spectrochimica Acta B, 64, 899-904, (2009), Burakov, V.S., Raikov, S.N., Tarasenko, N.V., Belkov, M.V. and Kiris, V.V. Journal of Applied Spec-troscopy, 77, 595-608,(2010).

Of course, the samples are usually pressed into the pellets. The proposed in the paper LIBS approach contains sample preparation stage as well. The authors need to explain why they consider that painting of fine soil parti-cles on a metal surface with adding moisture is more simple sample preparation procedure. The authors should specify the thickness of the soil layer and give more explanations and evidences to support their conclu-sion, in particular, how the layer thickness is controlled as well as how its identity for the samples of the differ-ent concentrations is ensured and how the layer non-homogeneity can affect the results of analysis.

2. Introduction, last sentence "By using this method, estimating of carbon (C) inventories in soils can be made, thus global climate change can be predicted". It is not clear in result of which processes (mechanisms) the at-mospheric carbon dioxide and other greenhouse gases can influence the soil carbon content. I suggest to exclude this conclusion or the authors should give evidences to support it.

Page 7, Line 14 "This result indicated that our present technique offers high possibility to measure the carbon concentration in soil influenced by the global climate change." How can you distinguish what part of the measured carbon concentration is related to the global climate change.

3. Page 8, Line 11. "...strong plasma was produced leaving the metal plate without any ablation and damage..." What is an optimal thickness of the soil layer to be satisfied this condition.

4. "*meta-assisted laser-induced gas plasma spectroscopy*" should be replaced by "*metal-assisted laser-induced gas plasma spectroscopy*"

Reviewer

Friday, October 20, 2017, 6:23:50 AM, you wrote:

Dear Editorial Office of ZhPS

Thank you very much for your kind reply. I would like to submit the revised manuscript registered under No. 252-17 and consent as in attachment. Thank you very much for your kindness.

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Ali Khumaeni

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Editor in Chief
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Best regards,
jas

<mailto:jas@dragon.bas-net.by>

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4/25/22, 12:32 PM

FISIKA UNDIP Mail - Re: 252-17 Re: Submission of manuscript to Journal of Applied Spectroscopy Review

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jas

<mailto:jas@dragon.bas-net.by>

4. Balasan komentar Editor dan Reviewers

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Of course, the samples are usually pressed into the pellets. The proposed in the paper LIBS approach contains sample preparation stage as well. The authors need to explain why they consider that painting of fine soil particles on a metal surface with adding moisture is more simple sample preparation procedure. The authors should specify the thickness of the soil layer and give more explanations and evidences to support their conclusion, in particular, how the layer thickness is controlled as well as how its identity for the samples of the different concentrations is ensured and how the layer non-homogeneity can affect the results of analysis.

We have additional explanation as shown in introduction and experimental setup.

It should be considered that the present preparation method is much convenient compared to the case of conventional LIBS by pressing the sample into the pellet. In case of pellet, some additional agent is necessary such as KBr in order to make a hard sample pellet to produce a strong plasma. Therefore, pellet preparation is much complicated compared to that of present technique.

2. Introduction, last sentence “By using this method, estimating of carbon (C) inventories in soils can be made, thus global climate change can be predicted”. It is not clear in result of which processes (mechanisms) the atmospheric carbon dioxide and other greenhouse gases can influence the soil carbon content. I suggest to exclude this conclusion or the authors should give evidences to support it.

We have erased last sentence following your suggestion as shown in revised manuscript.

Page 7, Line 14 “This result indicated that our present technique offers high possibility to measure the carbon concentration in soil influenced by the global climate change.”

How can you distinguish what part of the measured carbon concentration is related to the global climate change.

We have erased the sentence because it not directly relates to global climate change.

3. Page 8, Line 11. "...strong plasma was produced leaving the metal plate without any ablation and damage..." What is an optimal thickness of the soil layer to be satisfied this condition.

The suitable thickness of sample was 1 mm. We have added this explanation in the revised manuscript, experimental procedure.

The suitable thickness of the sample on the subtarget was 1 mm. Based on experiment, if the sample is too thick, the plasma quenching happens and therefore reducing the plasma temperature and plasma lifetime.

5. Paper setelah proses revisi mempertimbangkan masukan Editor dan Reviewers

Laser Plasma Spectroscopy using Pulsed CO₂ Laser for the Analysis of Carbon in Soil

Ali Khumaeni^{1*}, Wahyu Setia Budi,¹ Asep Yoyo Wardaya,¹ Koo Hendrik Kurniawan,² and Kiichiro Kagawa

¹*Department of Physics, Faculty of Mathematics and Natural Sciences,
Diponegoro University, Tembalang, Semarang, Indonesia*

²*Research Center of Maju Makmur Mandiri, Kembangan, Jakarta Barat, Indonesia*

³*Fukui Science Academy, Fukui, Japan*

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Abstract

Analysis of carbon (C) in soil has been successfully made by laser plasma spectroscopy using a pulsed CO₂ laser. Fine particles of soil sample were attached on a surface of metal subtarget by adding little amount of moisture; the metal is used to initiate the gas plasma. Experimentally, a pulsed laser was irradiated and focused on the subtarget to induce a luminous plasma. The particles were vaporized and entered to the plasma region. Dissociation and excitation happens in the high-temperature plasma region. The result certified that analysis of C in soil can be demonstrated. A further measurement revealed that a calibration curve of C was successfully carried out. The limit of detection of C in the soil was around 23 mg/kg.

Keywords: LIPS; Laser-induced plasma spectroscopy; LIBS; Carbon analysis; Soil sample

1. Introduction

Recently, carbon inventories in soils have changed due to the serious global climate change [1-3]. Soil carbon is fundamental matrix of soil chemical and physical properties such as nutrient contains, structure of soil , and capacity of water in the soil, which affect ecosystem and soil quality [4-5]. The use of more coal and oil for homes, factories, and transportation releases carbon dioxide and other greenhouse gases in atmosphere, resulting in increasing the temperature of atmosphere. This increment of the temperature influences the soil carbon dynamics and reduces the annual change of soil organic carbon [5]. Thus, an efficient technique of measuring soil carbon is required to estimate the terrestrial carbon inventories.

Several analytical techniques were employed to the analysis of carbon in soil. X.M. Zou et al. conducted a sequential fumigation-incubation for estimating carbon in soil [6]. Paul et.al. used the method to predict the retention time and rates of carbon turnover in soils [7].

Emission spectroscopy by using a laser, which people usually call laser-induced breakdown spectroscopy (LIBS), has been attracting many scientists for applications to rapid elemental analysis in various materials [8]. A Nd:YAG laser is usually employed as an excitation source. When the laser was directed and focused on a sample target, a

high-temperature plasma is produced; the plasma contains the atomic and ionic elements as well as molecules from the target. The plasma was then imaged to the optical spectrometer to obtain the emission spectrum. However, the standard LIBS cannot make direct analysis on soil sample because once the laser was irradiated on the soil target, the blow off of soil seriously occurs. Therefore, to perform analysis of soil by using LIBS, the soil must be changed in the pellet. However, the pelletized sample is not hard like ordinary solid sample. Therefore, a plasma with high-temperature cannot be induced [9].

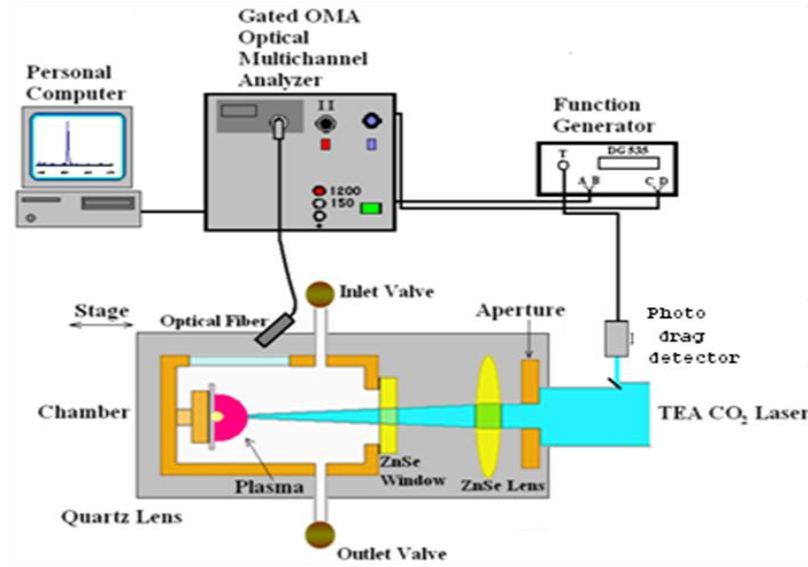
In other direction, we have found that a pulsed CO₂ laser is very favorable for soil analysis due to the fact that the laser has long wavelength, which is high absorbance in the soil [10]. We proposed a sampling technique for the analysis of C in soils by using a pulsed CO₂ laser. A subtarget technique was suggested for sampling the sample. The tiny amount of soil particles faintly painted on a metal plate by adding a moisture. When a pulse laser was irradiated on the subtarget, on which the thin layer soil painted, a high-temperature plasma was induced and the metal subtarget is not ablated and damaged. It should be considered that the present preparation method is much convenient compared to the case of conventional LIBS by pressing the sample into the pellet. In case of pellet, some additional agent is necessary such as KBr in order to make

a hard sample pellet to produce a strong plasma. Therefore, pellet preparation is much complicated compared to that of present technique.

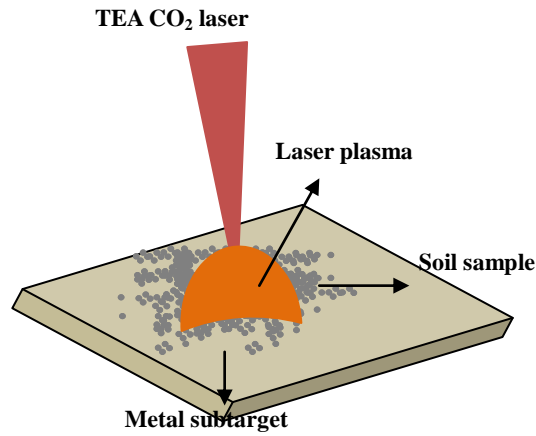
2. Method

Figure 1(a) displays the basic setup for experiment. A pulse CO₂ laser (Shibuya SQ 2000, 10.6 μm , 200 ns, 3 J) was used as an energy source. The pulsed laser was directed and focused onto the sample by ZnSe lens (100 mm in focal length). The spot size of laser on the sample was $1 \times 1 \text{ mm}^2$, which the laser fluence is 0.75 GW/cm^2 ..

A carbon soil sample collected from the garden was used as a sample in this research. For semi-quantitative analysis, a standard soil sample that contains various concentrations of carbon (200, 500, and 1000 mg/kg) was used. Prior to the analysis, the fine soil particles with moisture were directly attached on a nickel plate ($0.1 \times 20 \times 20 \text{ mm}^2$), which serves as a subtarget, as shown in Fig. 1(b). The suitable thickness of the sample on the subtarget was 1 mm. Based on experiment, if the sample is too thick, the plasma quenching happens and therefore reducing the plasma temperature and plasma lifetime.



(a)



(b)

Fig. 1. (a) Basic setup, and (b) lustration of the sample holder employed in this study.

The sample was put in a steel chamber ($12 \times 12 \times 12 \text{ cm}^3$). The pressure inside chamber was kept constant at 1 atm of nitrogen gas.

The emission spectrum from the laser plasma was obtained by using an optical multichannel analyzer system (OMA), which consists of spectrograph and photodiode

detector. An optical fiber connected to OMA system was used to collect the plasma emission. During experiment, time integrated mode of OMA was operated.

3. Results and Discussion

First, the soil carbon assessment was made by present method. To this point, a small amount of soil fine particles (around 500 mg) with moisture were spread on the nickel plate, which plays a role as a subtarget. Figure 2 displays C spectrum obtained from the soil sample. High-intensity C I 247.8 nm clearly occurs with low background intensity. Furthermore, many typical atomic lines of Si showing characteristics of the soil sample can readily be observed in the right side of spectrum.

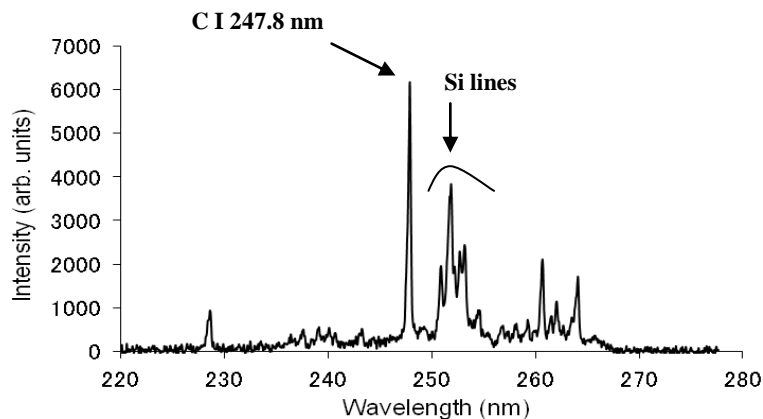


Fig. 2. C spectrum obtained from the garden soil

Carbon atom is contained in garden soil at several thousands ppm (part per million or mg/kg). The present technique is superior to ordinary LIBS technique for conducting

rapid and efficient measurements of total soil carbon. For the LIBS case, the soil sample should be made as a hard pellet in order to obtain high-temperature plasma. In the sample preparation, a chemical agent such as KBr is commonly used. Therefore, the ordinary LIBS required tedious sample pretreatment for soft sample or powder analysis. While, in our present method, the analysis can be made without sample pretreatment because we only directly painted the soil sample with moisture on the metal subtarget prior to the analysis.

An analysis of the company soil sample, which contains various concentrations of C was further made by using a present method. Considering intensity ratio between C I 247.8 nm and Si I 251.9 nm is almost constant, the Si line near C was used as a standard. Curve with linear calibration was made from the soil, as demonstrated in Fig. 3. It should be noted that the use of silicon (Si) for standardization should be evaluated with regard to soil mineralogy because the Si content in soils varies with the mineralogy of soils.

To examine the sensitivity by using present technique, a carbon analysis in soil was conducted. Figure 4 shows a spectrum obtained from a company soil sample containing 500 mg/kg of carbon.

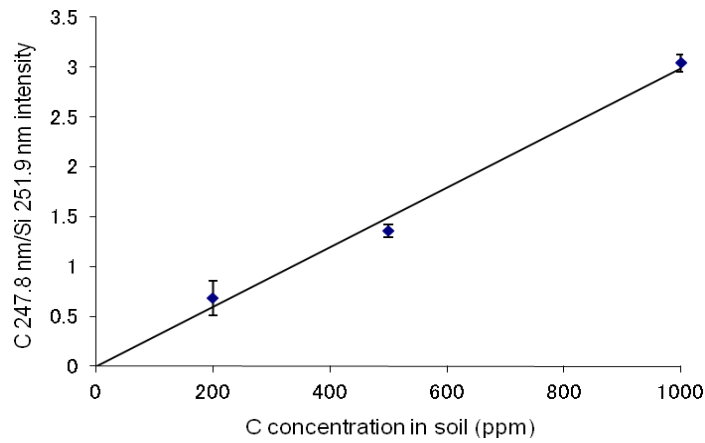


Fig. 3. Carbon curve obtained from the soil containing various concentration of carbon by using metal-assisted laser-induced gas plasma spectroscopy.

The emission line of C I 247.8 nm can clearly be seen. Other lines of Si in the right side of the carbon was also detected. The detection limit of carbon was around 23 mg/kg; the calculation of the detection limit follows the equation reported in this work [11]. This result is superior to the case of ordinary LIBS, which the detection limit of C was around 300 mg/kg as reported by other researchers [4]. Therefore, our present technique is very potential to perform C analysis in soil carbon influenced by the global climate change.

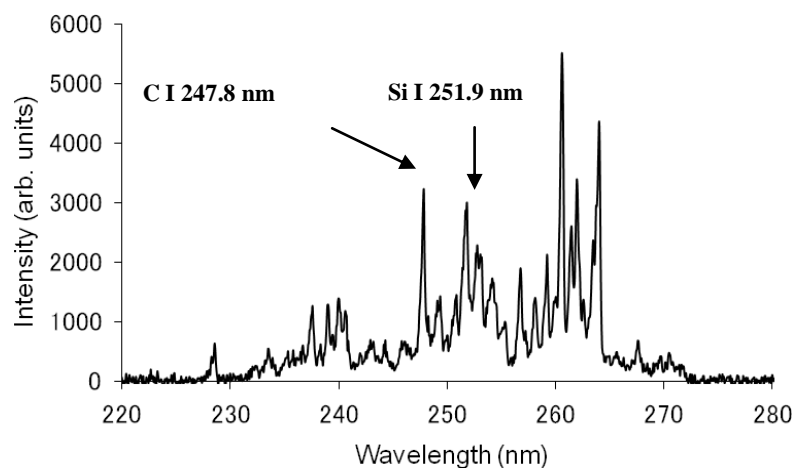


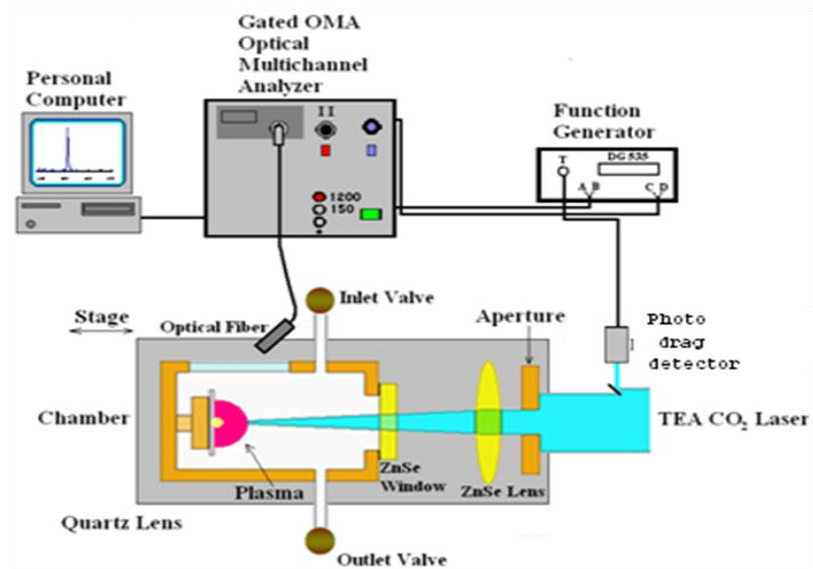
Fig. 4. Emission spectrum taken from the soil containing 500 mg/kg of C using metal-assisted laser-induced gas plasma spectroscopy.

4. Conclusion

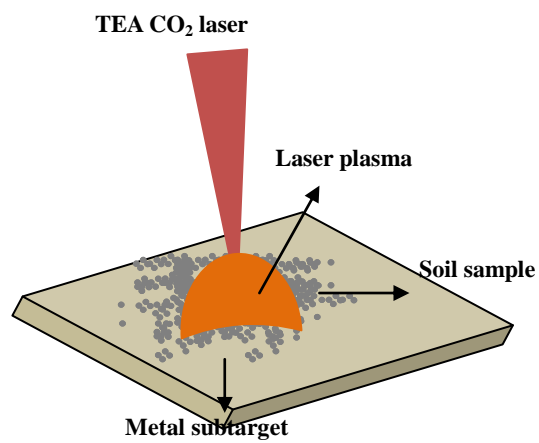
Carbon analysis in soil sample was demonstrated by laser-induced plasma spectroscopy using a pulsed CO₂ laser. In experiment, fine particles of soil mixed with moisture were painted on a subtarget. A luminous plasma was produced when the laser beam was irradiated on the subtarget. The soil particles then moved and enter the plasma region. Dissociation and excitation processes of particles takes place in the plasma region. A semi-quantitative analysis was demonstrated for C element in soil sample. The limit of detection of C in soil was 23 mg/kg.

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11. W. Setiabudi , H.Suyanto, H. Kurniawan, M. O. Tjia , and K. Kagawa, *Applied Spectroscopy*, **53**, 719-730 (1999).



(a)



(b)

Figure 1

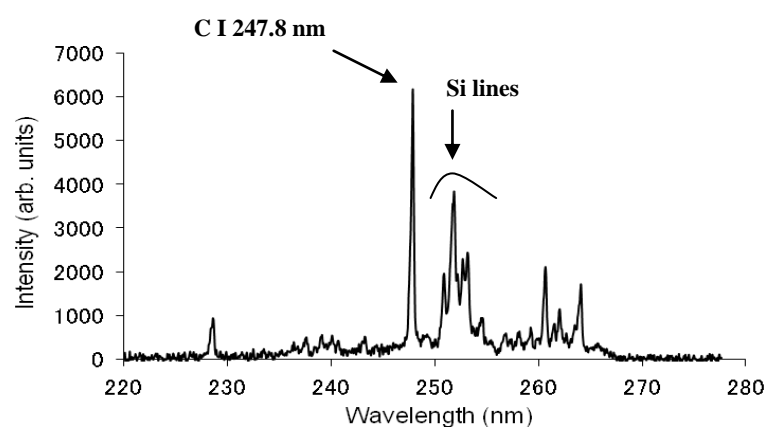


Figure 2

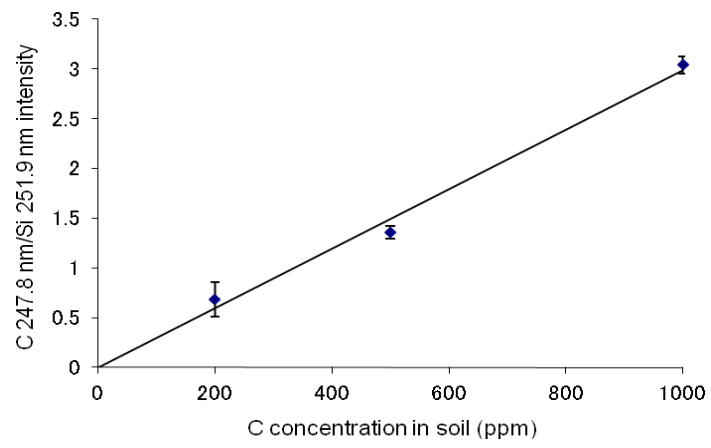


Figure 3

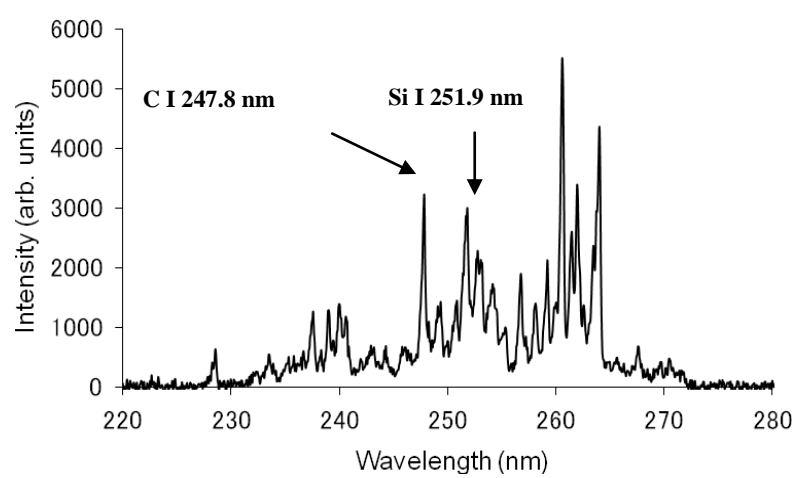


Figure 4



EDITORIAL

CERTIFICATE

Authors:

Ali Khumaeni

Document title:

**Laser Plasma Spectroscopy Using a
Pulsed CO2 Laser for the Analysis of
Carbon in Soil**

Date Issued:

2018-11-20

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Cambridge Proofreading Worldwide LLC is a registered company headquartered in Chicago, Illinois, USA with a global presence. All of our editors are native speakers from USA and the UK. Our Certificate of Good Standing can be found in the Illinois state business database by searching our name here.

7. Final recommendation to be accepted (16 Februari 2018)



Ali Khumaeni <khumaeni@fisika.undip.ac.id>

Re: 252-17 Re: Submission of manuscript to Journal of Applied Spectroscopy Review

jas on Dragon <jas@dragon.bas-net.by>
Reply-To: jas on Dragon <jas@dragon.bas-net.by>
To: Ali Khumaeni <khumaeni@fisika.undip.ac.id>

Fri, Feb 16, 2018 at 8:06 PM

Dear Authors,

The paper No. 252-17 accepted for consideration

for publication in ZhPS Vol. 86, No. 1 (January-February) 2019.

The final decision about the publication will be made in April, 2018

at a meeting of the Editorial Board for the approval the contents of No. 1, 2019.

Sincerely yours,

Editorial office of ZhPS

Thursday, February 8, 2018, 7:37:03 AM, you wrote:

[Quoted text hidden]

8. Final proof (20 Desember 2018)

Re: Fwd: Re: 252-17 Re: Review ZhPS proof urgently !!!

jas on Dragon <jas@dragon.bas-net.by>
Reply-To: jas on Dragon <jas@dragon.bas-net.by>
To: Ali Khumaeni <khumaeni@fisika.undip.ac.id>

Thu, Dec 20, 2018 at 8:37 PM

Dear Authors,

Herewith, we send you the proofs of your paper No. 252-17 to be published in "Zhurnal Prikladnoi Spektroskopii". After checking the proofs, please send us a list of corrections indicating:

- the title of the paper;
- list of the authors;
- page number; line number (from page top or bottom), and the corresponding correction in the following form: "<error> should be replaced by <right variant>."

Yours sincerely,
Editorial office of ZhPS

Wednesday, November 21, 2018, 9:00:53 AM, you wrote:

Dear Editor in Chief
Journal of Applied Spectroscopy

I would like to send a revised manuscript corrected by Proofread company as in attachment. We do hope that the manuscript can be published in Journal of Applied Spectroscopy. Thank you very much for your kindness.

Best regards
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Dear Authors,

It will be best of all if the final variant will be made by a professional firm (for example, Edanz) with the attachment of the copy of the Certificate.

If the manuscript is edited in a professional firm, with certificate, we may include it in an upcoming issue of the journal, where the place appear, because Springer removed

some manuscripts due to bad English.

Sincerely yours,
Editorial office of ZhPS

Wednesday, November 14, 2018, 5:12:29 PM, you wrote:

Dear Editor Office Journal of Applied Spectroscopy

Thank you very much for your latest email. I am so sorry to know that our paper cannot be published in Journal of Applied Spectroscopy. Surely we forget to revise the manuscript following your suggestion. Is it possible to revise our manuscript by checking the Professional company and submit it again to Journal of Applied Spectroscopy. We do hope that the present manuscript can be published to Journal of Applied Spectroscopy.

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Best regards
Ali Khumaeni

On Tue, Nov 13, 2018 at 5:18 PM jas on Dragon <jas@dragon.bas-net.by> wrote:
Dear Authors,
Your paper No.252-17 will not be published in Vol. 86, № 1 (January-February) 2019, because we received neither your answer to the letter dated April 3, 2018 nor the manuscript 252-17 after improvement of English by Edanz or be any other firm.
Please, see item 11 of the Rules.
Sincerely yours,
Editorial Office of ZhPS

This is a forwarded message
From: jas on Dragon <jas@dragon.bas-net.by>
To: khumaeni@fisika.undip.ac.id
Date: Tuesday, April 3, 2018, 2:26:24 PM
Subject: Fwd: Re: 252-17 Re: Submission of manuscript to Journal of Applied Spectroscopy Review

===8<=====Original message text=====

Dear Authors,
Your paper reg. No.252-17 accepted for publication in russian version ZhPS Vol. 86, No.1 (January-February) 2019.
The Editorial Board asks to improve English (see item 11 of the Rules).
Please send the revised version not later than May 10, 2018.
The final decision about the publication in english version JAS will be made in January 2019 at the Springer for the approval the article of No.1, 2019.
Sincerely yours,
Editorial Office of ZhPS

This is a forwarded message
From: jas on Dragon <jas@dragon.bas-net.by>
To: Ali Khumaeni <khumaeni@fisika.undip.ac.id>
Date: Friday, February 16, 2018, 4:06:45 PM
Subject: 252-17 Re: Submission of manuscript to Journal of Applied Spectroscopy Review

===8<=====Original message text=====

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Journal of Applied Spectroscopy

Thank you very much for your kind reviewing our manuscript. We are very pleased to know that our paper is acceptable to be published in Journal of Applied Spectroscopy. We have made a revision following your suggestion and reviewer comments. Please kindly find the answer for reviewer and revised manuscript as in attachment.

Thank you very much for your kindness and we are looking forward to hearing your positive respond.

Best regards

Ali Khumaeni

On Mon, Jan 22, 2018 at 4:56 PM, jas on Dragon <jas@dragon.bas-net.by> wrote:

I support the acceptance of this work but before publication I would recommend the following revisions:

1. Introduction "However, the standard LIBS cannot make direct analysis on soil sample because once the pulse Nd:YAG laser was focused on the soil target, the blow off of soil seriously occurs". There are many publications where standard LIBS is used for the direct analysis of carbon in soil samples (see for example Belkov, M.V., Burakov, V.S., De Giacomo, et al Spectrochimica Acta B, 64, 899-904, (2009), Burakov, V.S., Raikov, S.N., Tarasenko, N.V., Belkov, M.V. and Kiris, V.V. Journal of Applied Spectroscopy, 77, 595-608, (2010).

Of course, the samples are usually pressed into the pellets. The proposed in the paper LIBS approach contains sample preparation stage as well. The authors need to explain why they consider that painting of fine soil particles on a metal surface with adding moisture is more simple sample preparation procedure. The authors should specify the thickness of the soil layer and give more explanations and evidences to support their conclusion, in particular, how the layer thickness is controlled as well as how its identity for the samples of the different concentrations is ensured and how the layer non-homogeneity can affect the results of analysis.

2. Introduction, last sentence "By using this method, estimating of carbon (C) inventories in soils can be made, thus global climate change can be predicted". It is not clear in result of which processes (mechanisms) the atmospheric carbon dioxide and other greenhouse gases can influence the soil carbon content. I suggest to exclude this conclusion or the authors should give evidences to support it.

Page 7, Line 14 "This result indicated that our present technique offers high possibility to measure the carbon concentration in soil influenced by the global climate change." How can you distinguish what part of the measured carbon concentration is related to the global climate change.

3. Page 8, Line 11. "...strong plasma was produced leaving the metal plate without any ablation and damage..." What is an optimal thickness of the soil layer to be satisfied this condition.

4. "meta-assisted laser-induced gas plasma spectroscopy" should be replaced by "metal-assisted laser-induced gas plasma spectroscopy"

Reviewer

Friday, October 20, 2017, 6:23:50 AM, you wrote:

Dear Editorial Office of ZhPS

Thank you very much for your kind reply. I would like to submit the revised manuscript registered under No. 252-17 and consent as in attachment. Thank you very much for your kindness.

Best regards
Ali Khumaeni

On Fri, Oct 13, 2017 at 11:00 PM, jas on Dragon <jas@dragon.bas-net.by> wrote:

Dear Authors,

Your paper has been received and registered under No. 252-17.

In the correspondence, please refer to the specified number (in Subject).

The paper is not prepared in accordance with "Rules of Preparation of Papers Submitted in English".

See attachments, please.

Please, send manuscript in a form pdf-file (full version (for Reviewer): figures, tables should be place in the text just after their first mentioning.

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At request of the publishing house Springer, which publishes the English version of our journal, we must submit them "Consent to publish" signed by all the authors or one author responsible of the paper. In keeping with this "Consent", you assure that your paper was not sent to other journals. If your signature is lacking, the paper will not be published. Consent must be presented as separate file with one of the following formats: wmf, tif, pcx, bmp, jpg.

Sincerely yours,
Editorial office of ZhPS

Friday, October 13, 2017, 4:06:36 AM, you wrote:

Dear Professor V. A. Tolkachev
Editor in Chief
Journal of Applied Spectroscopy

Good morning,

I am Ali Khumaeni, from Department of Physics, Diponegoro University, Indonesia. I would like to submit a final manuscript entitled "Laser-induced gas plasma spectroscopy for the analysis of carbon in soil" to Journal of Applied Spectroscopy. Please kindly find the manuscript as in attachment.

Thank you very much for your kindness.

Best regards
Ali Khumaeni
Assistant Professor
Department of Physics
Diponegoro University
Semarang, Indonesia

email: khumaeni@fisika.undip.ac.id

--

Best regards,
jas

<mailto:jas@dragon.bas-net.by>

--

Best regards,
jas

<mailto:jas@dragon.bas-net.by>

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Best regards,
jas

<mailto:jas@dragon.bas-net.by>

===8<=====End of original message text=====

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Best regards,
jas

<mailto:jas@dragon.bas-net.by>



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9. Bukti publikasi (24 Januari 2019)

Re: Fwd: Re: 252-17 Re: Review ZhPS

jas on Dragon <jas@dragon.bas-net.by>
 Reply-To: jas on Dragon <jas@dragon.bas-net.by>
 To: Ali Khumaeni <khumaeni@fisika.undip.ac.id>

Thu, Jan 24, 2019 at 10:19 PM

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According to Par.7 of the Rules of Manuscript Preparation and Submission,
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 (Abstract is published in ZhPS V. 86, No. 1 (January-February) 2019
<http://imaph.bas-net.by/JAS>),
 full text is published in JAS V. 86, No.1 (<http://springer.com/10812>)).
 Sincerely yours,
 Editorial Office of ZhPS

This is a forwarded message

From: jas on Dragon <jas@dragon.bas-net.by>
 To: Ali Khumaeni <khumaeni@fisika.undip.ac.id>
 Date: Thursday, January 24, 2019, 6:09:12 PM
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(<http://imaph.bas-net.by/JAS>),

full text is published in JAS V. 86, No.1 (<http://springer.com/10812>)).

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Sunday, December 23, 2018, 5:35:22 AM, you wrote:

Dear Editor in chief
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Diponegoro University, Faculty of Science and Mathematics.

Thank you

Best regards
Ali Khumaeni

On Thursday, December 20, 2018, jas on Dragon <jas@dragon.bas-net.by> wrote:
Dear Authors,
Herewith, we send you the proofs of your paper No. 252-17 to be published
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"Zhurnal Prikladnoi Spektroskopii". After checking the proofs, please send
us a list of corrections indicating:

- the title of the paper;
- list of the authors;
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Best regards
Ali Khumaeni

On Fri, Oct 13, 2017 at 11:00 PM, jas on Dragon <jas@dragon.bas-net.by> wrote:

Dear Authors,

Your paper has been received and registered under No. 252-17.

In the correspondence, please refer to the specified number (in Subject).

The paper is not prepared in accordance with "Rules of Preparation of Papers Submitted in English".

See attachments, please.

Please, send manuscript in a form pdf-file (full version (for Reviewer): figures, tables should be place in the text just after their first mentioning.

Please, send manuscript in format . doc (or .docx).

At request of the publishing house Springer, which publishes the English version of our journal, we must submit them "Consent to publish" signed by all the authors or one author responsible of the paper. In keeping with this "Consent", you assure that your paper was not sent to other journals. If your signature is lacking, the paper will not be published. Consent must be presented as separate file with one of the following formats: wmf, tif, pcx, bmp, jpg.

Sincerely yours,
Editorial office of ZhPS

Friday, October 13, 2017, 4:06:36 AM, you wrote:

Dear Professor V. A. Tolkachev

Editor in Chief

Journal of Applied Spectroscopy

Good morning,

I am Ali Khumaeni, from Department of Physics, Diponegoro University, Indonesia. I would like to submit a final manuscript entitled "Laser-induced gas plasma spectroscopy for the analysis of carbon in soil" to Journal of Applied Spectroscopy. Please kindly find the manuscript as in attachment.

Thank you very much for your kindness.

Best regards

Ali Khumaeni
Assistant Professor
Department of Physics
Diponegoro University
Semarang, Indonesia
email: khumaeni@fisika.undip.ac.id

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Best regards,
jas

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