

# Investigation of the Effects of Laser-Based Combustion on Sorghum Bran

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DOI: <https://doi.org/10.56807/buj.v2i2.52>

## Abstract

The aim of this work is to develop a new procedure for production of silica powder from sorghum bran ash via laser-based combustion. The raw material of sorghum bran was obtained from a sorghum mill in Khartoum, Sudan. Nd: YAG laser with output power 60 Watts was used to combust 5 grams of sorghum bran for different combustion times, combustion was non-flaming. The weight of silica in the ash was determined by chemical treatment. In the silica producing process using Nd: YAG laser; silica content percentage can be raised through increasing the time of laser combustion. The mineral contents of the produced ash were characterized by X-ray fluorescence (XRF) analysis. The micro/nanostructures of the produced ash were investigated by X-ray diffraction (XRD), Fourier transform infrared spectroscopy (FTIR) and scanning electron microscope (SEM). XRF results showed the presence of the following elements: Fe, Cr, Ni, Zn, Pb and Mn elements. XRD results showed the presence of a crystalline hexagonal phase of silica and amorphous silica. FTIR showed several absorbance peaks assigned to silica. SEM results showed the micro/nanostructures of silica. The synthetic procedure is environment-friendly, straightforward and inexpensive.

**Keywords:** Agricultural waste ash; Combustion synthesis; Laser-based combustion; Laser-matter interaction; Nanoparticles; Nanosilica; Sorghum; Waste utilization.

## التحقق من اثر الحرق بالليزر على نخالة الذرة الرفيعة

### الملخص

الهدف من هذا العمل هو تطوير إجراء جديد لإنتاج مسحوق السيليكا من رماد نخالة الذرة الرفيعة عن طريق الحرق بالليزر. تم الحصول على المواد الخام من نخالة الذرة الرفيعة من مطحنة الذرة الرفيعة في الخرطوم ، السودان . تم استخدام ليزر Nd: YAG بقوة 60 وات لاحتراق 5 غرامات من نخالة الذرة الرفيعة في ازمته الاحتراق المختلفة. تم تحديد وزن السيليكا في الرماد بالمعالجة الكيميائية. في عملية إنتاج السيليكا باستخدام ليزر Nd: YAG ؛ يمكن زيادة نسبة محتوى السيليكا من خلال زيادة وقت الاحتراق بالليزر. اتسمت المحتويات المعدنية للرماد المنتج بتحليل الأشعة السينية (XRF). تم فحص التركيبات الدقيقة / النانوية للرماد الناتج بواسطة حيود الأشعة السينية (XRD) ، التحليل الطيفي بالأشعة تحت الحمراء (FTIR) ، المجهر الإلكتروني الماسح (SEM). أظهرت نتائج XRF وجود العناصر التالية: عناصر Fe و Cr و Ni و Zn و Pb و Mn. أظهرت نتائج XRD وجود مرحلة سداسية بلورية من السيليكا والسيليكا غير المتبلورة. أظهر FTIR العديد من قمم الامتصاص المخصصة للسيليكا. أظهرت نتائج SEM التركيبات الدقيقة / النانوية للسيليكا. وهذه الطريقة الجديدة صديقة للبيئة ومباشرة وغير مكلفة.

## 1. Introduction

Combustion synthesis of advanced materials is an efficient approach and energy saving, which can occur in the gas, liquid, and solid phases (Mukasyan & Manukyan, 2019, 87). It is an economically viable and simple technique for the preparation of nanomaterials. Combustion synthesis is based on the principle that an exothermic reaction is initiated under heating, which yields self-sustained propagation (C.-L. Yeh, 2016; C. Yeh & Chen, 2009, 165). Heat of combustion assists in crystallization and formation of the desired phase (Tyagi, 2006, 12).

Silica nanoparticles have gained greater attention because of their highly reactive surface area to volume ratio, physical and chemical stability, low toxicity and straightforward surface chemistry that allows them to be functionalized or combined with a variety of functional molecules or species (Ghorbani, Sanati, & Maleki, 2015, 223).

Many researchers have studied the utilization of agricultural wastes and agriculture by-products to obtain useful and valuable materials, including the synthesizing, preparation and extraction of nanostructured materials such as silica, silica gel, soluble sodium silicate or other materials by different extraction methods. Some methods were environmentally friendly techniques. There is a growing interest in the extraction of silica nanoparticles and valuable materials from agriculture by-product and waste; a variety of routes has been reported in literature for this purpose; for example, the extraction of biogenic silica from samples of some alpine plant species (Carnelli, Madella, & Theurillat, 2001, 426).

Bulk of research has been conducted to synthesize silica in diversified manners using rice hull and husk. For example; the preparation of pure silica from rice hull ash using a recyclable method (Ma, 2012, 498), optimizing of silica extraction

procedure from rice husk as agricultural waste by environmentally friendly method (Ghorbani, 2015, 58), extraction of soluble sodium silicate from rice husk ash (Owoeye & Isinkaye, 2017, 8), production of silica powders from rice husk ash using green procedure via a carbonation method (Liu, 2011, 1315), preparation of activated carbon and precipitated white nano-silica from rice husk ash (Rahim, Ismail, & Mageed, 2015, 493) and separation of silica gel from rice husk ash by microwave heating (Rungratnimitchai, Phokhanusai, & Sungkhaho, 2017, 45).

There have been numerous research studies on the extraction of amorphous hydrated silica in Gramineae plant (Chen, 2010, 2784), preparation of pure silica from sedge as agricultural residue (Ghorbani, 2013, 824) and obtaining of highly value and useful materials from wheat bran and sesame seed cake (Gawbah, Elbadawi, Alsabah, Orsod, & Marouf, 2018, 121; Gawbah, Marouf, Alsabah, Orsod, & Elbadawi, 2017, 9) and some researchers have studied the manufacture of chemical pulp from sorghum waste to (Belayachi & Delmas, 1995, 413). Fabrication of micro/nanostructures on alloys and metals in different environments has been reported by several researchers; for example; K. M. Tanvir Ahmed *et al.* reviewed the utilization of direct femtosecond laser micromachining on the fabrication of these structures on metals (Ahmed, Grambow, & Kietzig, 2014, 1229). Also, Marouf *et al.* utilized a fast laser texturing method to fabricate micro/nano surface textures in Silicon photovoltaic cell using UV femtosecond laser pulses (Marouf, Abdallah, Abdulrahman, & Al Naimee, 2014, 95). The objectives of the present paper were to describe a simple combustion method for the preparation of micro/nanosilica from agricultural waste and to investigate the characteristics of the obtained micro/nanosilica. Overall, we present a new alternative approach to synthesize micro/nanosilica from agricultural waste.

## 2. Materials and Methods

### 2.1 Sorghum bran

Sorghum bran (a by-product from sorghum milling) used in this study was obtained from a local sorghum mill, Khartoum, Sudan. It was washed three times by distilled water to remove dust and other impurities, then dried by direct exposure under sunlight, and then crushed using a milling machine.



Fig. 1. Laser-combustion setup, using Nd: YAG laser.

### 2.2 Laser combustion

Five grams from the dried powder was burned using of 1.064 $\mu$ m Nd: YAG laser with output power 60Watts with continuous mode in a high-temperature crucible for 10 sec at atmospheric pressure. The laser beam was delivered by single

mode fiber optic with diameter 125  $\mu$ m; so the value of its spot size is equal to 12265.625  $\mu$ m<sup>2</sup>. The combustion process was done point by point every 10 sec because of the small spot size of the laser beam. The crucible was rotated every 30 sec, while the laser was fixing in a holder. Combustion was non-flaming and carbon dioxide escalation has been observed during the burning process. The setup of the burning process is arranged in Fig. 1. This experiment was repeated twice with 20 s and 30 s exposure times. The produced ash was grinding carefully by agate mortar for the characterization.

### 2.3 Determination of Silica in sorghum bran ash

15ml of water was added to 0.4g of the finely powdered ash into a porcelain dish and then it was decomposed in 25ml of 1:1 hydrochloric acid. The liquid has evaporated to dryness and placed in an air oven at 100-110 °C for one hour to dehydrate the silica. The residue was moistened with 5mL of concentrated hydrochloric acid. 75mL of water was added and heated on a steam bath for 10-20 minutes to assist in the solution of soluble salts. The separated silica was filtered off on a what man No. 41. The precipitate was washed with warm, dilute hydrochloric acid, and then with hot water until forming chlorides. The filtrate was washed and evaporated to dryness with steam bath and heat in an air oven at 100-110 °C for one hour. The residue was moistened with 5mL concentrated hydrochloric acid, 75mL water was added warm to extract soluble salts, then filtered through a fresh smaller filter paper. The filtrate was washed with warm dilute hydrochloric acid, and finally, with a little hot water, the moistened filters were folded up and placed in a weighed platinum crucible. The paper was dried with a small flame, charred, and the carbon was burned off over a low flame until all the carbon had been oxidized, the crucible was covered, and heated for an hour at the full temperature of a Meker- type burner to complete dehydration, then it was cooled in desiccators, and weighed. Each measurement was repeated three times, and the

average value was calculated until the weight is constant. To determine the exact Silica content, the residue was moistened with 1 mL water; two drops of concentrated sulphuric acid and about 5ml of the purest available hydrofluoric acid were added. The crucible was placed in an air bath and the hydrofluoric acid was evaporated in a fume cupboard with a small flame until the acid was completely expelled. Heat was increased to volatilize the sulphuric acid and finally heated with a Meker-type burner for 15 minutes, cooled in a desiccator, weighed and re-heated to constant weight. The loss in weight represents the weight of the silica.

#### 2.4 Characterization

Mineral composition of the sorghum bran ash powder (combusted for 30 seconds) was determined by X-ray fluorescence (XRF), using oxford X-MET5000 Handheld XRF Analyzer. The crystalline phase of the produced silica powder was characterized by X-ray diffraction (XRD), using Cu  $K_{\alpha}$  ( $\lambda=1.540600\text{\AA}$ ) radiation on a Shimadzu, MAX\_X, XRD-7000 diffractometer. With scanning speed of  $1000^{\circ}/\text{min}$ , tube voltage 60kV and current 50mA. The data were collected for  $(2\theta)$  rang  $10^{\circ}$  to  $80^{\circ}$  at step size of 0.014 for with 2theta correction  $0.09^{\circ}$ . Fourier transform infrared spectroscopy (FTIR) analyses were carried out on Satellite FTIR 5000 spectrometer in the wavenumber range of  $(400-4000)\text{ cm}^{-1}$  employing the KBr pellet method. The structure and morphology of the nano/microstructure distributions of the produced sorghum bran ash were examined by scanning electron microscope (SEM) (Tescan, Brno, Czech Republic).

### 3. Results and Discussions

#### 3.1. The analysis of sorghum bran ash

XRF elemental analysis of sorghum bran ash before and after combustion, respectively, is presented in table 1. It showed the presence of the

following elements: Fe, Cr, Ni, Zn, Pb and Mn elements; removing of this metallic impurities and producing silica quite white in color could be achieved by preliminary leaching with boiled solutions before combustion (Chakraverty, Mishra, & Banerjee, 1988, 23; James & Rao, 1986; Patel, Karera, & Prasanna, 1987, 2460).

#### 3.2. Effect of the duration of combustion on silica content

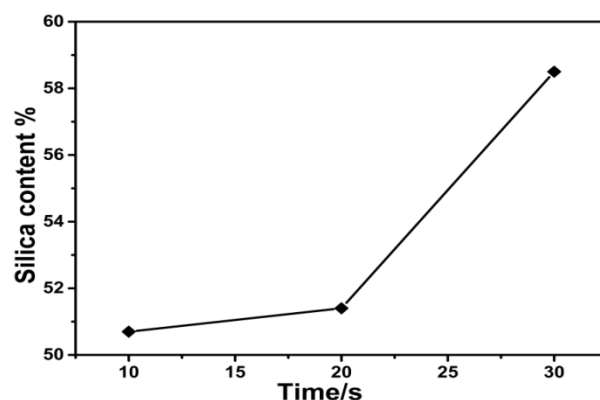


Fig. 2. The effect of combustion using Nd: YAG laser duration on silica content.

The effect of combustion duration on the content of silica was investigated. It was affected by different combustion durations as shown in Fig. 2. The content of silica gradually increased with increasing combustion duration, and reached the value 58.5% at the duration of 30 seconds. It shows that further increasing of combustion duration will give an increase in silica content.

#### 3.3 XRD Results

The X-ray diffraction patterns of sorghum bran before and after combustion were presented in figures 3a and 3b, respectively. The obtained results were analyzed by MDI jade 0.5 match program. The obtained results showed that combustion process using Nd: YAG laser helps in formation of crystalline hexagonal phase for silica and carbon



nitride and converting rhombohedral phase of carbon into hexagonal phase.

Table 1 Mineral content of sorghum bran ash before and after combustion using Nd: YAG laser

Treatment	Minerals (%)					
	Fe	Cr	Ni	Mn	Zn	Pb
Before combustion	0.25	0.05	0.01	0.03	0.01	0.03
After combustion	0.30	0.04	0.02	0.03	0.01	0.04

The diffraction pattern showed two broad peaks at about  $2\theta=15-25^\circ$  these broad peaks are confirming the amorphous nature. In the combusted sorghum bran pattern additional sharp peak at  $2\theta=26.57^\circ$  indicated the presence of the ordered crystalline structure of carbon C70(Kawamura, 1992, 564). Therefore, laser combustion process is effective to lead the sorghum bran from the amorphous phase into the crystalline structure phase (Rhombohedral), because of the high pressure and high temperature generated by infrared laser interaction with sorghum.

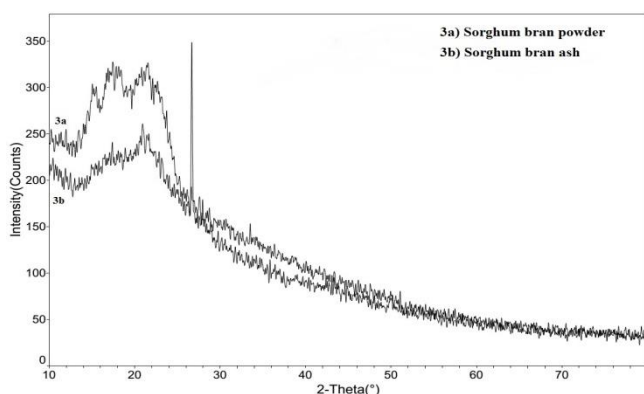


Fig. 3. X-ray diffraction pattern of a) sorghum bran powder, b) sorghum bran ash after combustion using Nd: YAG laser for 30 sec

The diffraction pattern of the sorghum bran ash revealed that there is a little crystalline structure and

that the majority of the particles are amorphous. It revealed that silica obtained from the sorghum bran ash was in the amorphous form. Crystallization is not preferred toward preparing materials from silicon because silica is inactive in its crystalline form (Payá, Monzó, Borrachero, Mellado, & Ordoñez, 2001, 229).

### 3.4 FTIR Results

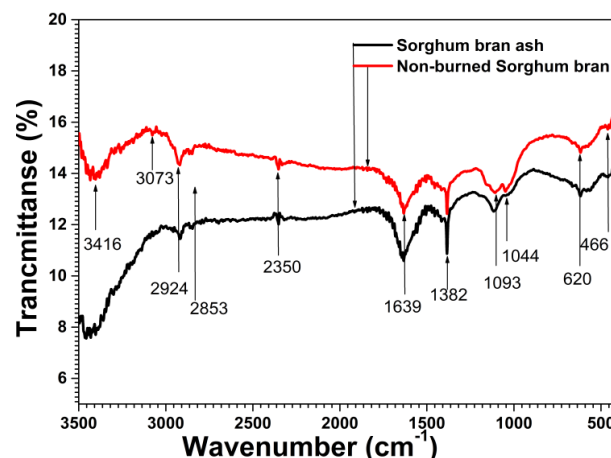


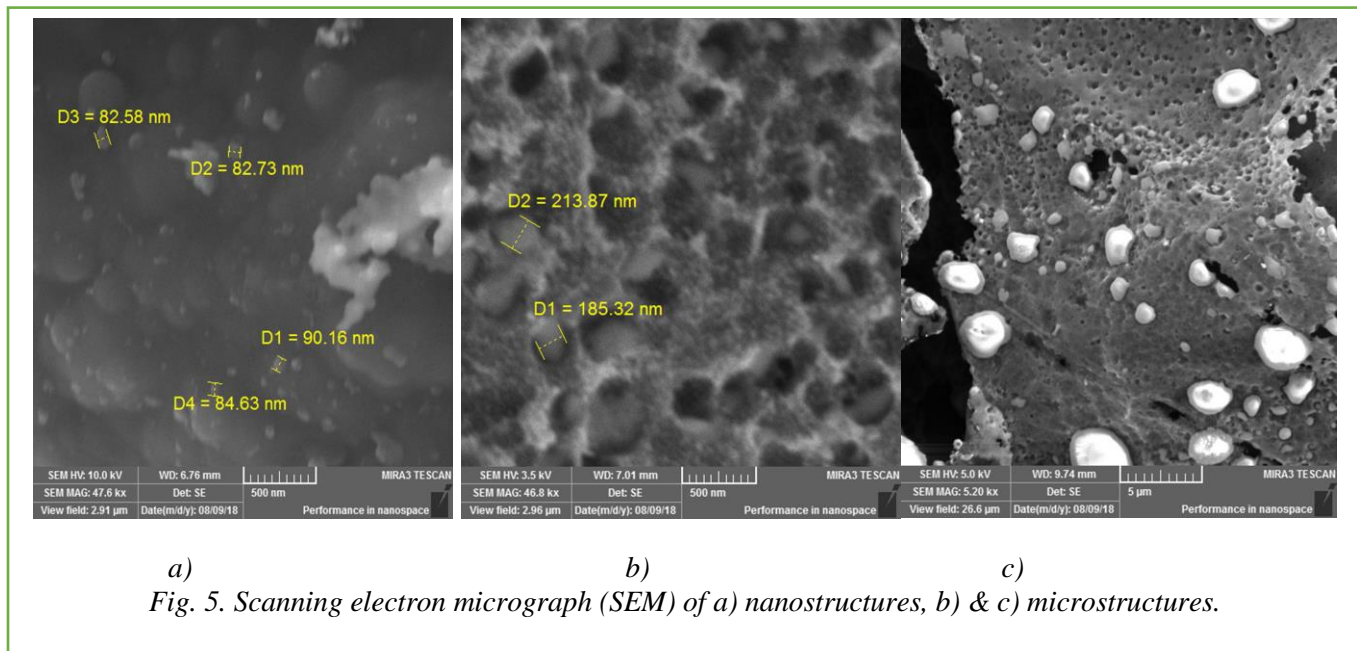
Fig. 4. Fourier transform infrared spectra of sorghum bran samples before and after combustion using Nd: YAG laser for 30 sec.

The sorghum bran ash was confirmed by FTIR examination in Fig. 4. Some observed absorption bands indicate the presence of organic groups; the peak at  $1382\text{ cm}^{-1}$  is attributed to the stretching vibrations of the carbonyl group (Karthik, Dhanuskodi, Gobinath, Prabukumar, & Sivaramakrishnan, 2019, 313). Organic carbon is present in the two samples, which have two peaks at  $2921\text{ cm}^{-1}$  and  $2853\text{ cm}^{-1}$ ; they are attributed to C-H symmetric stretching mode vibration (Senthil Kumar & Rajkumar, 2013, 22230). Broad absorption peaks at  $1639$  and  $3416\text{ cm}^{-1}$  were due to the H-O-H bending and stretching modes of the adsorbed water, respectively (An, Guo, Zhu, & Wang, 2010, 511); whereas the absorbance peak around  $2350\text{ cm}^{-1}$  was due to the formation of some  $\text{CO}_2$  (Guerrero-Ruiz & Rodriguez-Ramos, 2001). Whereas some

observed absorption bands indicate the presence of silica; such as the peaks at  $466\text{ cm}^{-1}$  and  $1093\text{ cm}^{-1}$  were due to the bending modes and the asymmetric stretching vibration of Si-O-Si respectively (Hegde & Rao, 2006, 1569). These two peaks are the main indices of silica materials that represent the successful production of silica nanoparticles. The band at  $620\text{ cm}^{-1}$  is assigned to the Si-Si bonds vibrations (Young, Chen, Liou, Yang, & Chang, 2000, 341). A comparison of the pattern between sorghum ash and un-combusted sorghum is identified around the new peak at  $1044\text{ cm}^{-1}$  attributed to the Si-O-Si mode (Koželj & Orel, 2008, 5074), and the other new peak at  $3073\text{ cm}^{-1}$ ,

Fig. 5a) depicts the image of silica nanostructures at a magnification of 47600X. It can be observed that silica nanoparticles have a shape with an average particle size of about 86 nm. It was noticed that a few amount of the nanostructures are aggregated. Silica microstructures are presented in figures 5 b) and c) at a magnification of 46800X and 5200X respectively, with an average particle size of about  $200 \pm 15\text{ nm}$ . The aggregation of silica nanostructures and the presentation with an average particle size of about  $200 \pm 15\text{ nm}$ ; this is agreeing with the previous studies (Ghorbani, 2015, 60; Ghorbani, 2013, 825)

The SEM images revealed an unburned



which was caused by a telescopic vibration of  $\nu$  (CH)(Li, Wang, & Zhu, 2015, 10).

### 3.5 SEM Results

The SEM images of sorghum bran ash are shown in Figures 5 a, b and c. They show the formation of laser-induced micro/nanostructures of silica from the sorghum bran combusted using Nd: YAG laser.

carbon that can be eliminated from the ash by increasing the combustion duration.

### 4. Conclusion

Sorghum bran has silica contents that can be utilized to produce various useful materials. In this study the potential of producing silica from sorghum bran was achieved through combustion by Nd: YAG laser. The content of silica gradually

increased with increasing combustion duration. The XRD results of the sorghum bran ash showed amorphous silica and crystalline structure of carbon C70. FTIR showed several absorbance peaks assigned to silica. SEM images showed the formation of laser-induced micro/nanostructures of silica.

This work provides a new application of laser in preparation of micro/nanosilica from the agricultural waste, and it demonstrates the possibility of transforming agricultural waste into useful raw materials, averting damage to the environment.

## 5. Acknowledgements

First, we would like to express our heartily appreciation to Almighty God. Secondly, Thanks are extended to the Institute of Laser, Sudan University of Science and Technology in accessing the instrumentation.

**Conflict of interest:** The authors declare that they have no conflict of interest.

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