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Synthesis of Silica, Silicon Carbide and Carbon from Wheat Bran and Converting its Crystal Structure Using Nd: YAG Laser

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Abstract: *This paper reports obtaining of useful and highly value materials from wheat bran. For this purpose, wheat bran sample was burned for 30 s using Nd: YAG laser with output power 60 W. The product of synthesis process and non-burned wheat bran were characterized by x-ray diffractometer (XRD), energy dispersive x-ray (EDX) and Fourier transform infrared (FTIR) so as to investigate its crystal structure and chemical components. XRD results of the sample before burning process revealed amorphous silica and cubic phase of silicon carbide. The obtained results showed that burning process using Nd: YAG laser can convert the cubic structure of silicon carbide into hexagonal and rhombohedral structure and convert rhombohedral structure of carbon into hexagonal structure; also it revealed a monoclinic structure of silica after burning. FTIR showed a number of absorbance peaks assigned to silica and silicon carbide.*

Keywords: *Crystal Structure, EDX, FTIR, Laser Matter Interaction, Moissanite, Wheat Husk, Wheat Straw, Silica, XRD.*

1. INTRODUCTION

Critical economic and environmental fettle of the current days hearten researchers and companies to improve and develop technologies purposed to reduce industrial wastes. As a consequence, much effort has been expended in different areas, including the agricultural outputting [1]. As an example, rice husk is an agricultural byproduct whose main constituent is organic material and hydrated silicon [2]. The use of rice husk has the benefit of producing valuable silica and silicon carbide [2, 3] also of reducing disposal and pollution problems [2, 4]. Many researches show that -amorphous silica can be prepared from a rice husk [1, 3, 5, 6] and rice straw [7][3] reported that a 95% silica powder could be produced after heat- treated at 700°C for 6 h. [8]Real et al in 1996 found that by mineral acid leaching silica of >99% purity can be obtained by burning rice husks at 600°C under an inert atmosphere [8]. Furthermore, charring of rice husk produces Nano size silica carbon called (nanosil) intermixed composite which may find newer application because such product is not available synthetically, this may directly be converted to silicon carbide and silicon nitride at high temperature [1-3].

Also wood is a renewable naturally occurring resource; Sene et al in 1994 and Ye et al in 2017[9, 10]found a facile approach is reported to transform wood into hierarchical porous graphene using CO_2 laser scribing [9,10]. They show that laser-induced graphene (LIG) can be formed on the surface of wood to provide high electrical conductivity graphene. They depicted the surface of square of pine wood is converted to 3D porous graphene when irradiated by a 10.6µm CO_2 laser under Ar or H_2 at room pressure.

However wheat husk, which also containing silica, there are two forms of silica after burning process, amorphous silica and crystalline silica [11] and wheat, their ashes contain >90% silica by mass with minor amounts of metallic elements, making it another economical source of nanoscale silica in the future [2, 9].

In this paper, we burned wheat bran by a 1.064µm Nd: YAG laser with output power 60 W. We used it, because of it is high gain and good thermal properties; it is the most important solid-state laser for scientific, medical, industrial, and military applications [12,14]. The laser heat was used instead of the heat of the furnace in burning wheat bran and this method saved time, power and effort. We study wheat bran before and after burning. The physical and chemical characterizations selected in this study included XRD, EDX, FTIR and Digital Microscope. The objective of the recent work is to obtain useful and valuable materials like silica and silicon carbide from wheat bran burning using Nd: YAG laser.

2. EXPERIMENTAL

Wheat bran sample was collected from (Tuti Island, in Khartoum, Sudan). It was washed with distilled water to remove adhering soil and other contaminants then dried at room temperature after that it was milled. One gram of the sample was placed in high temperature glass beaker (schott duran - Germany) and it was burned on the air by the heat of Nd: YAG laser (Dornier Medilas fibertom 5100) beam with output power 60W for 30 s. The laser beam was delivered by single mode fiber optic with diameter 125 µm. The distance between the sample and the end of the fiber optic was 1 cm. Because of the small spot size of the laser beam the process of burning was done point by point laser was fixed on holder and the high temperature glass beaker was rotated every 30 s carefully for 5 mm displayed in Figure 1, this experiment repeated many times before investigations. Microscopic photograph for the burned wheat bran was done by (Digital Microscope 500X Digital Zoom. Samples were examined before and after burning by XRD (Shimadzu, MAX_X, XRD-7000) using Cu K_α with scanning speed of 1000°/mi. EDX spectrometer was employed to characterize burned and non-burned wheat bran samples. The samples were prepared by grinding before XRD measurements carefully by agate mortar for homogeneity. For this (Shimadzu - EDX- 8000) was operated at 4KV to 50KV. The chemical groups presented in burned and non-burned wheat bran samples were identified by the FTIR spectrometer (Satellite FTIR 5000) in the wavenumber rang of (400-4000) cm^{-1}.

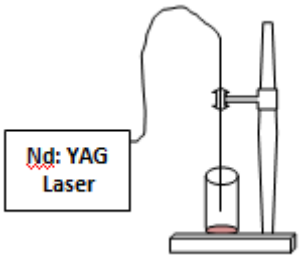

Figure1. *Schematic drawing of the experimental setup*

3. RESULT AND DISSECTIONS

3.1. XRD Results

The X-ray diffraction patterns were shown in Figure 2; it showed amorphous structure (including multi phases), Figures 3 and 4 showed the analyzed spectra for the wheat bran samples before and after burring analyzed by MDI jade 0.5 match program [15], graphs obtain the presence of the amorphous silica in the two samples, at the normal broad peak at 2θ=21.7– 21.8 for the samples before and after burning respectively. It also indicates that burning process using laser is very effective to change the wheat bran from amorphous phase into crystalline structure phase. Therefore, several phases were appeared in the samples. In non-burned wheat bran graph the phases peaks were appeared at 2θ=26.57 refer to Carbon (C) phase, at 2θ=37.8, 43.99, 64.397 and 77.509 these peaks refer to silicon carbide (SiC) structure [16].

Also there are several phases appeared after burning process that shown at 2θ=37.74 Moissanite -33R (SiC) [17], 2θ=43.74, 64.38 and 77.48 silicon carbide (SiC), 2θ=45.75 and 50.19 Carbon C) , 2θ= 59.74 Coesite, syn SiO_2 and at 2θ= 86.03 and 75.74 that refer to the Moissanite -5H SiC (Dark –

blue[18]. That also shown the peaks intensity and appearance was obtained with burn process. Tables 1 and 2 showed the details of the XRD result. Digital microscope image in Figure 5 confirms the presence of moissanite.

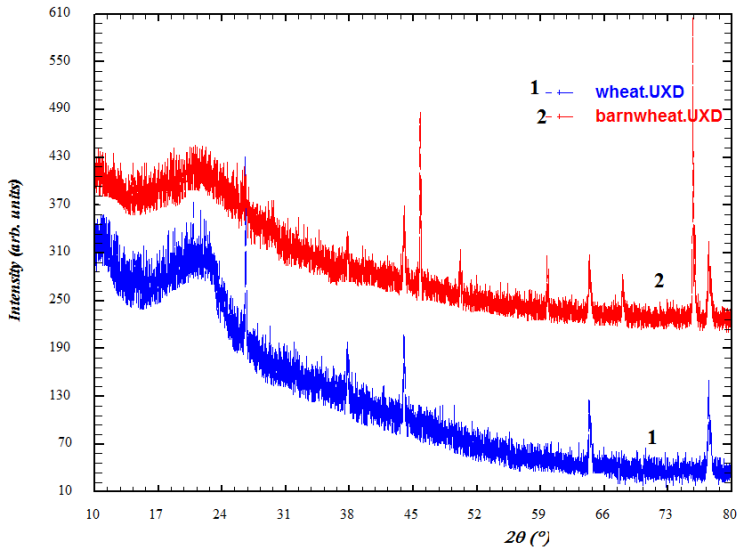

Figure2. *X-ray powder diffraction patterns of 1- non- burned, 2- burned wheat bran samples*

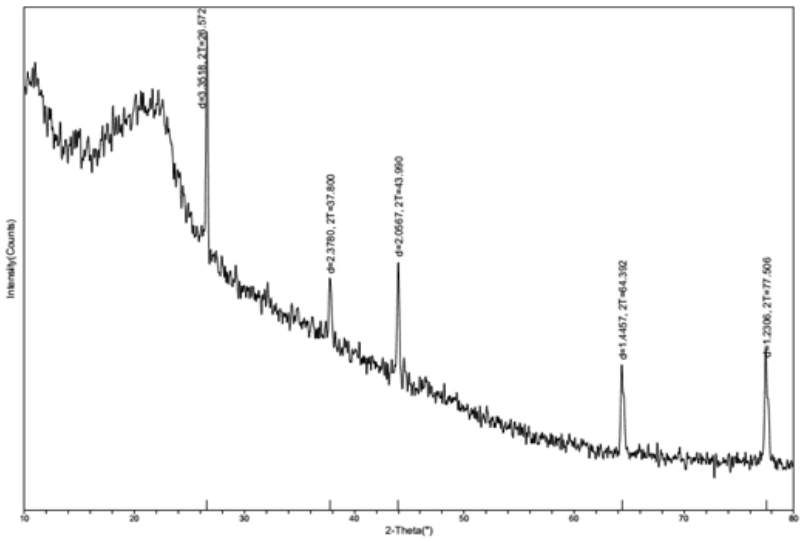

Figure3. *X-ray powder diffraction of nuture wheat bran*

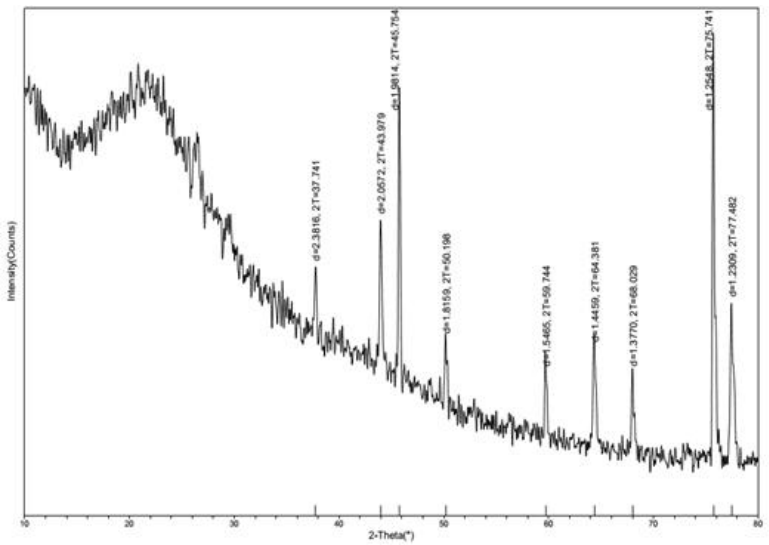

Figure4. *X-ray powder diffraction of burn wheat bran*

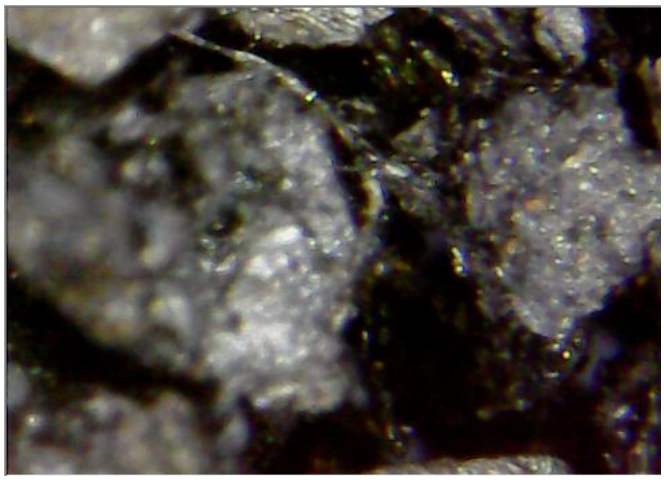

Figure5. *Microscopic photograph for the burned wheat bran (4X Digital Zoom)*

Table1. *X-ray diffraction parameters of nature wheat bran*

2-Theta	d(A)	BG	Height	I%	Area	I%	FWHM	Crystal structure	Phase
26.572	3.3518	195	165	100.0	2756	100.0	0.203	Rhombohedral - R	Carbon (C)
37.800	2.3780	124	49	29.7	1571	57.0	0.390	Cubic - Fm-3m (225)	Silicon carbide(SiC)
43.990	2.0567	94	92	55.8	2430	88.2	0.321		Silicon carbide(SiC)
64.392	1.4457	40	68	41.2	1826	66.3	0.326		Silicon carbide(SiC)
77.506	1.2306	33	77	46.7	2604	94.5	0.411		Silicon carbide(SiC)

Table2. *X-ray diffraction parameters of burn wheat bran*

2-Theta	d(A)	BG	Height	I%	Area	I%	FWHM	Crystal structure	Phases
37.741	2.3816	84	38	18.1	1208	29.4	0.386	Rhombohedral - Powder Diffraction, R3m (160)	Moissanite-33R, syn (Si)C
43.979	2.0572	69	76	36.2	1963	47.7	0.314	Cubic - Powder Diffraction, Fm-3m (225)	Silicon carbide(SiC)
45.754	1.9814	63	147	70.0	2413	58.7	0.200	Hexagonal - Powder Diffraction, P3 (143)	Carbon (C)
50.198	1.8159	51	35	16.7	734	17.8	0.255	Rhombohedral , R-3m (166)	CarbonC)
59.744	1.5465	35	35	16.7	921	22.4	0.320	Monoclinic - Powder Diffraction, P21/a (14)	Coesite, syn SiO2(Corles)
64.381	1.4459	31	51	24.3	1594	38.7	0.380	Cubic - Powder Diffraction, Fm-3m (225)	Silicon carbide(SiC)
68.029	1.3770	29	41	19.5	982	23.9	0.291	Hexagonal - Single Crystal, P	Moissanite-5H SiC(Darkblue)
75.741	1.2548	27	210	100.0	4114	100.0	0.238	Hexagonal - Single Crystal, P	Moissanite-5H SiC(Darkblue)
77.482	1.2309	25	79	37.6	2283	55.5	0.351	Cubic - Powder Diffraction, Fm-3m (225)	Silicon carbide(SiC)

3.2. FTIR Analysis

FTIR spectra were investigated for the burned and non-burned wheat bran samples that showed graphene and silicon bonds (see Figure 6). The absorbance peak around the 3434 cm^{-1} was due to the adsorbed water in the wheat bran samples after and before burning process [2], Also it may be refer to the OH stretching and vibration. The peak around 2940 cm^{-1} can be assigned to the C-H stretching modes and the CH_2 group was presented at around 940 cm^{-1} as asymmetric stretching. The absorbance peak around 2344.31 cm^{-1} obtained to -C≡N- (Nitrites) and -C≡C- (Alkynes) compounds [19]. A strong peak at 1630 cm^{-1} assigned to the Aromatic stretching in wheat bran structure and it became higher after burning process [2, 9, 20]. The absorbance bond at 1422 cm^{-1} peak refers to C-O vibrations or to C-H deformation in the sample [21, 22]. In generally, the 1422 cm^{-1} trough appear due to C-O stretching of carbonate [19]. The absorbance peak around 1110 cm^{-1} assigned to the Si-O-Si anti-symmetric stretching mode and the peaks around 660 cm^{-1} and 420 cm^{-1} correspond to siloxane bonds (Si-O-Si) stretching and bending vibrations [2, 21]. The FTIR results came to confirm the XRD results when the (SiC) synthetic moissanite is characterized by the peaks at range between 2400 – 1200 cm^{-1} [23].

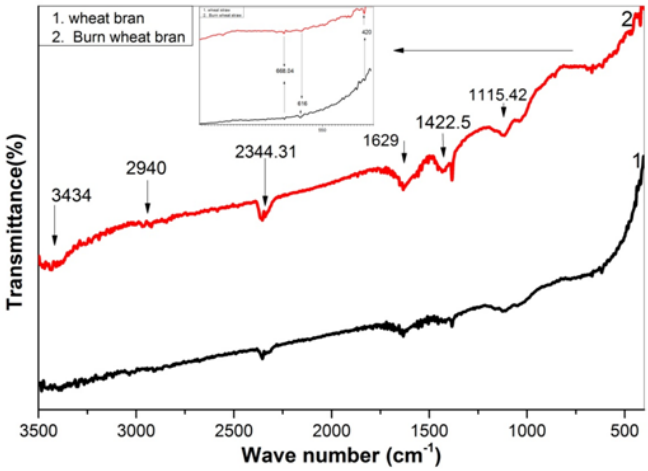

Figure6. *FTIR transmittance of wheat bran samples befor and after burn*

3.3. EDX Results

The EDX result was investigated using (Shimadzu, EDX- 8000), table 3 showed the weight of the elements in the samples of wheat bran before and after burning respectively. The X-ray passed through particles of the wheat bran samples to detect the presence of element specially the concentration of the carbonate [24]It was observed that the concentration of some elements in the sample increased after burning of wheat bran, this is because a quantity of carbon evaporated in the form of carbon dioxide during combustion (see Figures 7).

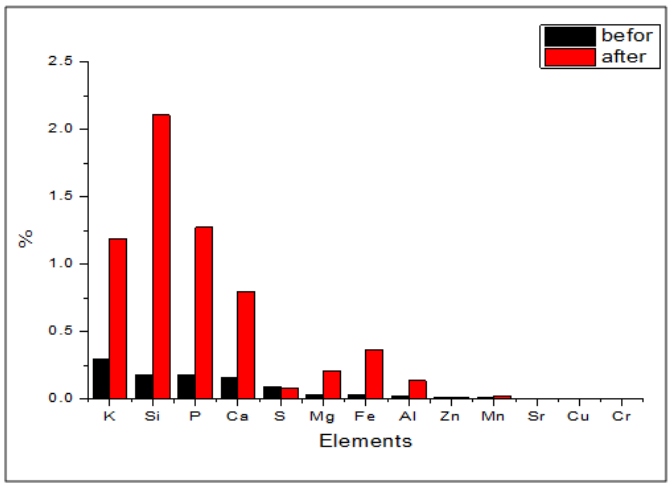

Figure7. *EDX result comparison between samples*

Table3. *The EDX result of samples*

Elements	Wheat	Burned wheat
K	0.301	1.190
Si	0.182	2.106
P	0.181	1.273
Ca	0.162	0.797
S	0.092	0.084
Mg	0.035	0.211
Fe	0.033	0.369
Al	0.021	0.136
Zn	0.010	0.016
Mn	0.008	0.023
Sr	0.003	0.006
Cu	0.002	0.003
Cr	0.000	0.001
Br	0.000	0.000
O	0.000	0.000
C	98.968	93.755

4. CONCLUSION

Wheat bran has silica contents which can be utilized to produce various useful materials. The possibility of producing silica and silicon carbide from wheat bran was achieved in this study by burning it by Nd: YAG laser. XRD results showed amorphous silica and silicon carbide before burning. It was found that this burning process caused in converting the cubic structure of silicon carbide into hexagonal and rhombohedral structure, and it converted rhombohedral structure of carbon into hexagonal structure, a monoclinic structure of silica also was found after burning. FTIR showed a number of absorbance peaks assigned to silica and silicon carbide.

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