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Characterization of the Crystal Structure of Sesame Seed Cake Burned by Nd: YAG Laser

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Abstract

This paper reports obtaining of useful and high-value materials from sesame seed cake (SSC). For this purpose, SSC sample was burned for 30 s using Nd: YAG laser with output power 60 W. The products of this process and non-burned SSC 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 SSC before burning process showed amorphous silica, rhombohedral phase of carbon, monoclinic phase of aluminum chloride, the hexagonal phase of moissanite-4H, (yellow, black) and hexagonal phase of graphite-2H, C (black). While the results of the burned SSC sample showed that the burning process using the power of Nd: YAG laser cased in appearing of crystalline hexagonal phase for silica and Carbon Nitride and converting the rhombohedral phase of Carbon into hexagonal phase. FTIR showed a number of absorbance peaks assigned to silica.

Keywords

Crystal Structure, FTIR, Hexagonal Carbon, Laser-Based Combustion, Sesame Seed Cake, Sesame Oil Cake, Silica, XRD

1. Introduction

There are a large number of agricultural wastes and they have become an increasing concern in recent years, as they may cause significant environmental

problems [1]. With appropriate techniques, agricultural wastes can be recycled to produce useful materials, the source of energy, chemical recovery, chemical or dye adsorption and natural fertilizer for crops [2]. Many investigations show that useful and high-value materials can be obtained from a cheap agricultural waste. For example, rice husk contains about 20% of ash which can be recovered as amorphous silica [3]. Many types of research relating to extraction silica from rice husk and rice straw have been reported [4] [5] [6] [7] [8]. Della *et al.* found the relative amount of silica was increased after burning out the carbonaceous material at different times and temperatures. A 95% silica powder could be produced after heat-treating at 700˚C for 6 h [4]. Singh *et al.* discussed synthesis and characterization of rice husk based nano-silica and reported the activated rice husk silica transforms into the crystalline product when burnt above 1000˚C [9].

Also, in wheat husk there are two forms of silica after burning process, crystalline silica and/or amorphous silica [10]. Chen *et al.* prepared nano-silica from wheat straw through combustion and acid leaching [11], Naqvi *et al.* extracted amorphous silica from wheat husk by using $KMNO_4$ [12], and Gawbah *et al.* used Nd: YAG laser to synthesis silica and some valuable materials from wheat bran [13].

Silica is one of the most important components and can be found in many applications such as biotechnology-related materials, medical-related materials [8], raw materials for cement industry [14]. Tailored materials composed of nanoparticles have potential for application in numerous technological fields [6].

In this study, we used sesame seed cake (SSC); it is the residue left after oil extraction which used as cattle feed [15] [16]. This residue can be recovered and value added [17]. We burned SSC by a 1.064 μm Nd: YAG laser with 60 W output power for 30 s. The advantage of Nd: YAG laser comes from its high gain and good thermal properties; it is the most important solid-state laser for scientific, medical, industrial, and military applications [18] [19]. The laser heat was used instead of the heat of the furnace in the burning of SSC and this method saved power, time and effort. Lasers have sufficiently high power with low divergence to be able to focus down to a desired size and to have enough power density to heat samples at high pressure; Lasers with high power stability and beam pointing stability are essential for producing a heating spot at steady temperature and at a constant position [20]. Silica (SiO_2) is a basic raw material that is widely used in electronics, ceramic, and polymer material industries. Due to their small-diameter, silica powders have many technological applications, such as thixotropic agents, thermal insulators, composite fillers, etc. [21]. Silica also has been used as a major precursor for a variety of inorganic and organometallic materials which have applications in synthetic chemistry as catalysts, and in thin films or coatings for electronic and optical materials [22] [23]. Carbon nitrides can applied in the field of catalysis, electrocatalysis, optoelectronics, sensors, separations and others [24].

Utilizing SSC in preparing silica, carbon nitrides and hexagonal carbon will

decrease the cost of waste disposal and also convert this waste into value-added products. We studied SSC before and after burning process. The physical and chemical characterizations selected in this study included EDX, XRD, FTIR and Digital Microscope. The objective of the work is to obtain useful and valuable materials like silica and silicon nitride from SSC burned using Nd: YAG laser.

2. Material and Method

2.1. Experimental

SSC sample was collected from Omdurman, Sudan. It was washed with distilled water many times to remove adhering soil and other contaminants then dried at room temperature after that it was milled. One gram of the sample was placed into a high-temperature glass beaker (Schott Duran—Germany) and it was burned on the air by the heat of Nd: YAG laser (Dornier Medilas fiber to 5100) with an output power of 60 W 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, the laser was fixed on a holder while the high-temperature glass beaker was rotated every 30 s carefully for approximately 5 mm see Figure 1, this step repeated many times before investigations for accuracy.

2.2. Characterization

Samples were examined before and after burning process by XRD (Shimadzu, MAX_X, XRD-7000) using Cu K_{α} with scanning speed of 1000˚/min and the data were collected for (2θ) range from 10˚ to 80˚ at a step size of 0.0002˚. The samples were prepared by grinding carefully before XRD measurements by agate mortar for homogeneity. The data were analyzed by MDI jade 0.5 match program. EDX spectrometer (Shimadzu - EDX-8000) was employed to characterize burned and non-burned SSC samples. It was operated at 4 KV to 50 KV with

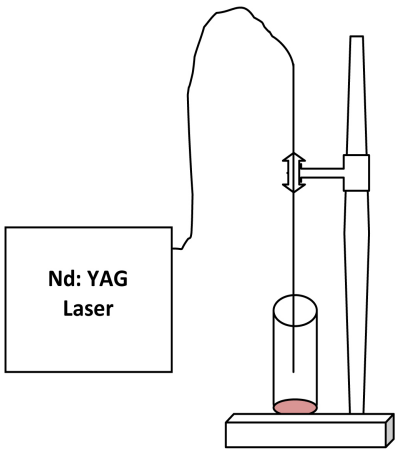

Figure 1. Schematic diagram of the experimental setup.

software quantitative analysis. The chemical groups presented in burned and non-burned SSC samples were identified by the fourier transform infrared spectrometer. Samples were mixed with dry potassium bromide powder KBr with a ratio of 1:100, by applying sufficient pressure, the mixture was prepared to scan. FTIR spectra of samples were collected in the wavenumber range of (400 - 4000) cm^{-1} using (FTIR) spectrometer (Satellite FTIR 5000). Microscopic photograph of the burned SSC was done by (Digital Microscope 400X Digital Zoom).

3. Results and Discussion

3.1. XRD Results

The X-ray diffraction patterns were shown in **Figure 2**, it showed amorphous structure (including multi-phases) of the two samples, **Figure 3** and **Figure 4** showed the analyzed spectra for the SSC samples before and after burning process analyzed by MDI jade 0.5 match program, graphs showed 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 laser burning process is very effective to form the crystalline structure phases of silica. Therefore, some of the silica phases appeared in the samples. In non-burned SSC graph; the phases peaks were appeared at 2θ = 15.009 refer to rhombohedral carbon C (Transparent) phase, at 2θ = 24.484 refer aluminum chloride acetate $C_{10}H_{15}A_{l2}ClO_{10}$, at 2θ = 30.159 refer to a monoclinic aluminum chloride hydroxide hydrate $Al_{10}C_{l3}(OH)_{27}!13H_2O$, at 2θ = 38.234 refer to hexagonal moissanite-4H, synSiC (yellow, black) and at 2θ = 77.457 refer to hexagonal graphite-2H C(black). Different phases were appeared after burning

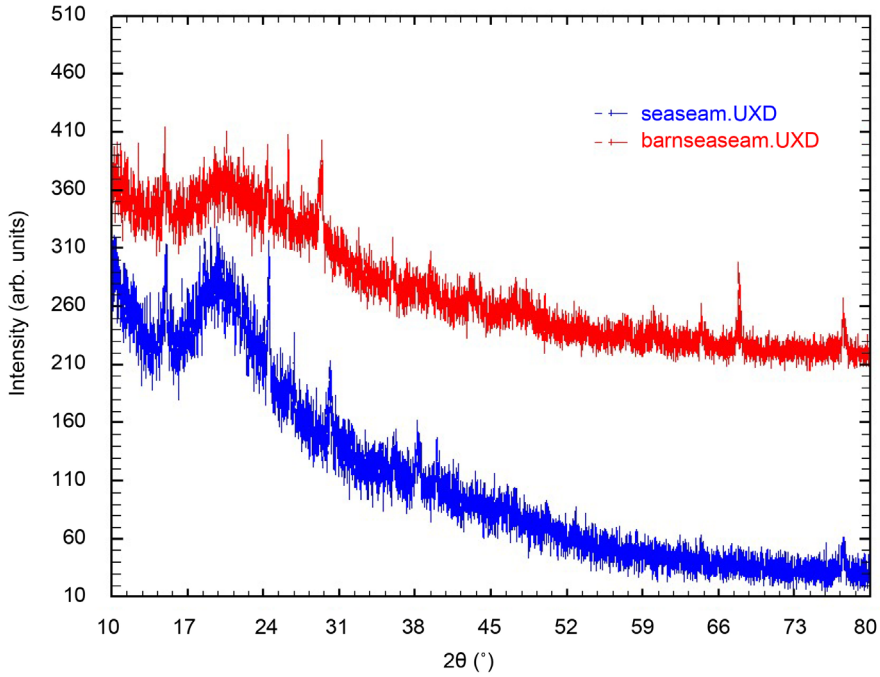

Figure 2. X-ray powder diffraction patterns of 1-nature, 2-burned SSC sample.

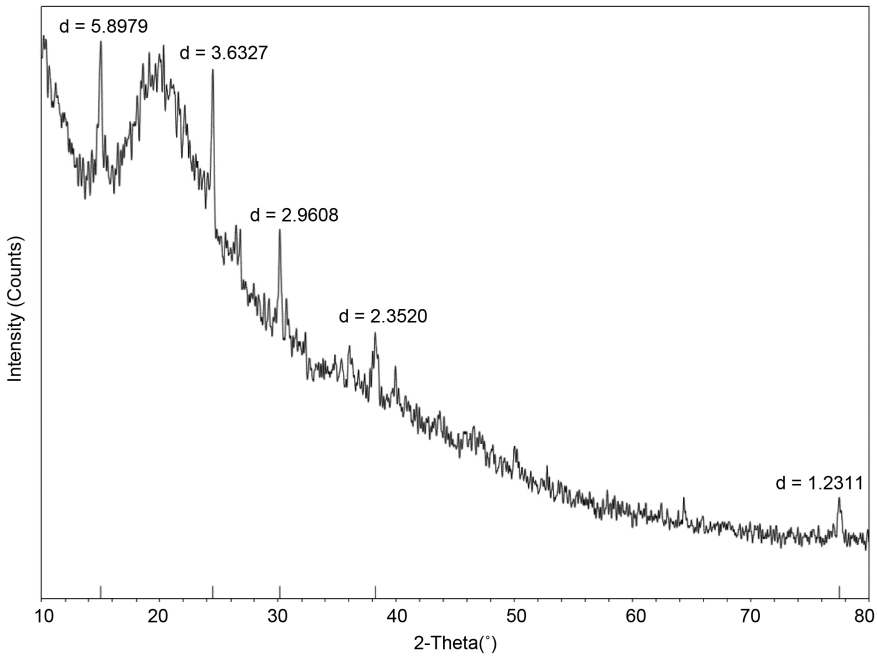

Figure 3. X-ray powder diffraction of nature SSC.

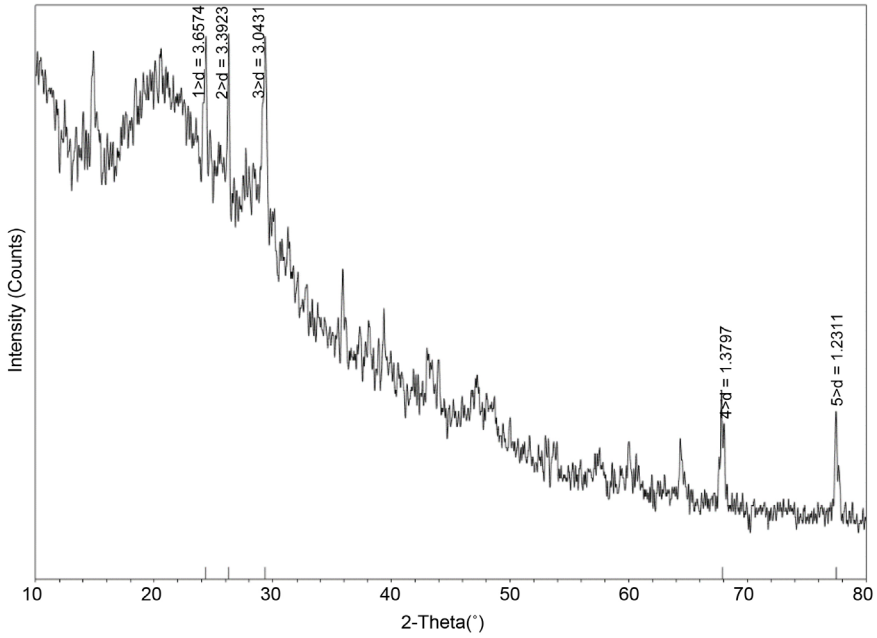

Figure 4. X-ray powder diffraction of burned SSC.

process that shown at $2\theta = 29.325$ refer to hexagonal carbonate (C) phase, at $2\theta = 77.462$ refer to hexagonal carbon nitride (CN) phase, at $2\theta = 24.316$ refer to orthorhombic silicon oxide, at $2\theta = 26.249$ this peak refer to hexagonal silicon oxide SiO_2 and at $2\theta = 67.876$ refer to hexagonal silicon oxide. That also shown the peaks intensity and appearance was obtained with burning process. Table 1 and Table 2 showed the details of the XRD results. Digital microscope image in Figure 5 confirms the presence of white silica.

Table 1. X-ray diffraction parameters of SSC.

2-Theta	d(A)	Height	Area	I%	FWHM	Crystal structure	Phase
15.009	5.8979	77	2712	100.0	0.428	Rhombohedral	Carbon C (Transparent)
24.484	3.6327	84	1983	73.1	0.287	Unknown	Aluminum Chloride Acetate $C_{10}H_{15}Al_2ClO_{10}$
30.159	2.9608	59	1913	70.5	0.394	Monoclinic	Aluminum Chloride Hydroxide Hydrate $Al_{10}Cl_3(OH)_{27}H_2O$
38.234	2.3520	37	1394	51.4	0.458	Hexagonal	Moissanite-4H, syn SiC (Yellow, black)
77.467	1.2311	23	781	28.8	0.413	Hexagonal	Graphite-2H C (Black)

Table 2. X-ray diffraction parameters of burned SSC.

2-Theta	d (A)	Height	Area	I%	FWHM	Crystal structure	Phase
24.316	3.6574	49	1365	66.9	0.339	Orthorhombic	Silicon oxide $Si_{64}O_{128}$
26.249	3.3923	35	768	37.6	0.176	Hexagonal	Silicon oxide SiO_2
29.325	3.0431	61	2041	100.0	0.407	Hexagonal	Carbon (C)
67.876	1.3797	27	1066	52.2	0.480	Hexagonal	Quartz, syn SiO_2 (White)
77.462	1.2311	37	1115	54.6	0.366	Hexagonal	Carbon Nitride CN_x

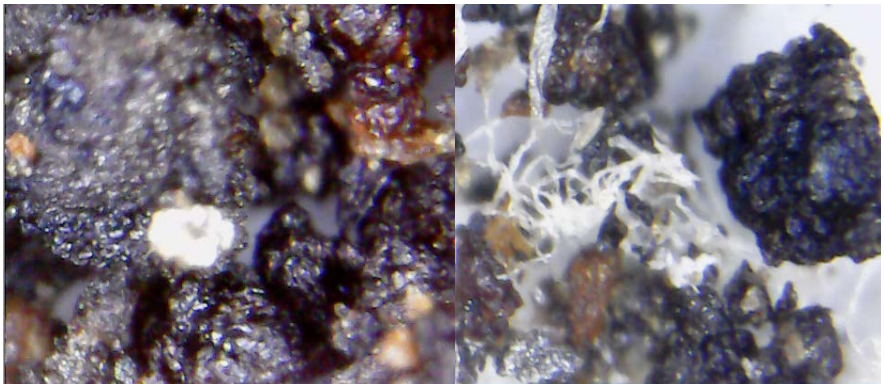

Figure 5. Microscopic photograph of the burned SSC (400× Digital Zoom).

3.2. FTIR Analysis

FTIR spectra were investigated for the burned and non-burned SSC samples that showed in **Figure 6** and **Table 3**. The absorbance peak at 3430 cm^{-1} was due to the adsorbed water in the SSC samples after and before burning process, also that maybe refer to the OH stretching mode [11] [13]. The peak around 2926.55 cm^{-1} may be assigned to the asymmetric and symmetric vibrations of C-H, [25].

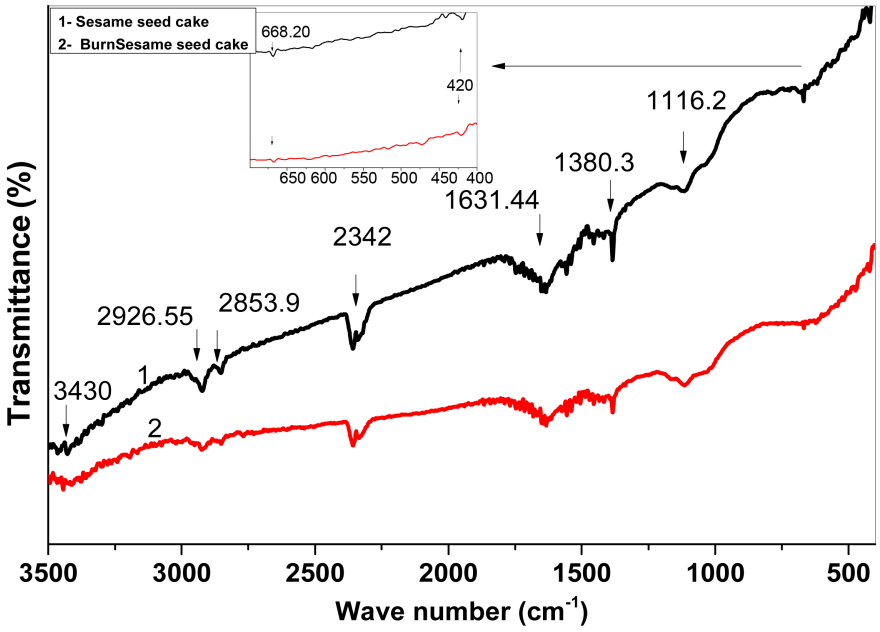

Figure 6. FTIR transmittance of SSC samples before and after burning process.

Table 3. FTIR Results of samples.

Absorption Peak Position (cm^{-1})	Function group/modes of vibration	reference
3430	OH stretching and vibration	[11] [13]
2926.55	the asymmetric and symmetric vibrations of C-H	[25]
2853.9	stretching vibration C-H	[26]
2342	$-C\equiv N-$ (Nitrites) and $-C\equiv C-$ (Alkynes) compounds	[13] [28]
1631.44	the aromatic stretching	[11] [13]
1380.3	stretching vibration C-N	[26] [28]
1116.2	the Si-O-Si anti-symmetric stretching mode	[3] [13]
668.20 - 420	to siloxane bonds (Si-O-Si) stretching and bending vibrations	[11] [13] [29]

The peak at 2853.9 cm^{-1} attributed to stretching vibration C-H [26]. The absorbance peak around 2342 cm^{-1} obtained to $-C\equiv N-$ (Nitrites) and $-C\equiv C-$ (Alkynes) compounds [13] [27]. The peak near 1631.44 cm^{-1} assigned to the aromatic stretching in SSC structure [11] [13] [23]. The peak at 1380.3 cm^{-1} attributed to stretching vibration C-N [26]. The peak around 1116.2 cm^{-1} was due to the Si-O-Si asymmetric and symmetric stretching modes [3] [13]. The peaks around (668.20 and 420) cm^{-1} correspond to siloxane bonds (Si-O-Si) stretching and bending vibrations [11] [24] [13].

3.3. EDX Results

The EDX result was investigated using (Shimadzu, EDX-8000), **Table 4** showed the weight of the elements in the samples of SSC before and after burning re-

spectively. The X-ray passed through particles of the samples SSC to detect the presence of element especially the concentration of the carbonate, that noticed It was observed that the concentration of elements in the sample increases after burning for the carbon element shown in Figure 7, this is because a quantity of carbon evaporates in the form of carbon dioxide during combustion.

Table 4. EDX result of samples.

Elements	Sesame seed cake (%)	Burned Sesame seed cake (%)
Ca	1.759	5.476
K	1.381	3.770
S	0.517	0.517
P	0.511	1.554
Si	0.116	0.269
Mg	0.071	0.206
Fe	0.032	0.119
Zn	0.016	0.041
Ag	0.014	0.000
Mn	0.009	0.031
Sr	0.009	0.021
Ti	0.008	0.005
Cu	0.005	0.014
Br	0.001	0.002
O	0.000	0.000
C	95.550	87.894

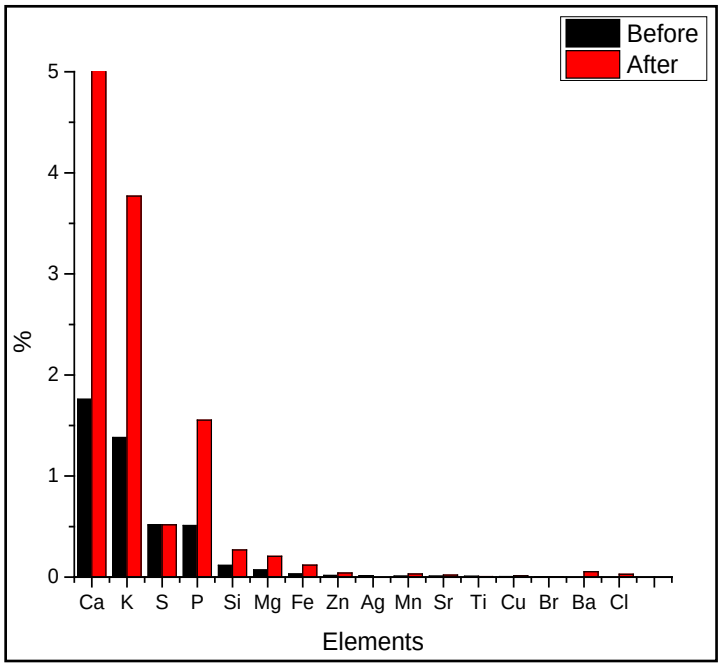

Figure 7. EDX result comparison between samples.

4. Conclusion

Sesame seed cake (SSC) has silica contents which can be utilized to produce various useful materials. The possibility of producing silica and silicon carbide from SSC was achieved in this study by burning it by Nd: YAG laser. XRD results of the SSC before burning process showed amorphous silica, the rhombohedral phase of carbon, monoclinic phase aluminium chloride, the hexagonal phase of moissanite-4H, synSiC (yellow, black) and hexagonal phase of graphite-2H, C (black). The obtained results showed that burning process using Nd: YAG laser cased in appearing of crystalline hexagonal phase for silica and carbon nitride and converting the rhombohedral phase of Carbon into hexagonal phase. FTIR showed a number of absorbance peaks assigned to silica.

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