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Thermal Analysis of Sclerotium of *Pleurotus tuber-regium* (*ọsụ*) for Effective Drying

Austin I. Gbasouzor, Klein Ileleji

Department of Agricultural and Biological Engineering, College of Engineering, Purdue University, West Lafayette, IN, USA
Email: ai.gbasouzor@coou.edu.ng, ileleji@purdue.edu

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Abstract

Thermal drying could lead to the deterioration of substance which affect its nutraceutical and chemical properties. Hence, thermal stability of substance is necessary in course of its drying to acertain the degree of temperature it will be subjected to. In this research, sclerotium of *Pleurotus tuberregium* is subjected to thermogravimetric analysis, differential thermal analysis and differential scanning calorimetry. Both thermogravimetric analysis and differential thermal analysis were conducted from ambient temperature to 1000˚C at a rate of 20˚C/min constant heating rate, while differential scanning calorimetry was conducted from ambient temperature to 400˚C at a rate of 10˚C/min constant heating rate. Besides, the oxides contents of sclerotium of *Pleurotus tuberregium* were determined using x-ray fluorescence analysis and the mircostruture was determined with scanning electron microscopy. It was discovered that complete dehydration of the sample ended at about 110.38˚C and oxidation reaction occurred between 233.42˚C to 373.82˚C with release of heat by the sample. Sclerotium of *Pleurotus tuber-regium* is thermal stable up to 233.42˚C with decompostion of steroid which is its second major component at about 400˚C to 480˚C. The x-ray fluorescence analysis of sclerotium of *Pleurotus tuber-regium* revealed that Na_2O, MgO, SiO_2, Al_2O_3, and Fe_2O_3 are the major compound and SEM analysis showed that it is a solid with amorphous structure having some fibrous skeleton. This study revealed that sclerotium of *Pleurotus tuberregium* could be dried to minimal moisture content without the deterioration of its nutritional and medinicnal properties.

Keywords

Bio-Active, Macro-Fungi, Ọsụ, Thermal Behavior, Nutraceutical

1. Introduction

Pleurotus tuber-regium is an edible marcofungi found in tropical and subtropical regions of the world with both the sclerotium and the sporophores being grown for their nutritional and medicinal benefits [1] [2] [3] [4] [5]. Other species of edible marcofungi have been investigated [6] [7]. The sclerotium of pleurotus tuber-regium is used for soup thickening and can be prepared into delicious local cake [8]. The sclerotium contains bioactive compounds that have proven to be effective in treatment and managing of some common ill-health [8]-[12]. Bioactive compounds do not have direct nutritional benefits but they have physiological and immunological effects which are essential in total wellbeing. Most drying processes involve the application of heat. The effect of heat on the bioactive components of sclerotium of pleurotus tuber-regium will aid in its drying. Flavonoid and phenolic contained in Chenopodium quinoa did not lose their content when roasted at 180˚C for 5 minutes [13]. Heat causes enzymatic activity as well as oxidation, which may lead to degradation of phenolic compounds [14].

In engineering, materials are better understood with respect to composition, stability, chemical reactions and dynamic properties. Thermal analysis provides information about the structure, composition, purity and the temperature phase change of a material. Thermal methods of analysis include a group of techniques in which change in physical property of a material is measured as a function of temperature while the substance is subjected to a controlled temperature variation [15]. The change in mass can be measured when a substance is heated or cooled according to temperature or time variation by thermogravimetry [16]. Thermogravimetric analysis (TGA) is the most commonly used tool to obtain experimental kinetic data [17] and it provides information about physical transitions and chemical reactions [18]. TGA is a powerful and robust technique to explore the thermal stability of a material. The differential scanning calorimetry (DSC) measures the heat flow between a sample and a reference material when both are subjected to a controlled temperature change [16]. DSC is a flexible technique to explore thermal transitions within a given sample. By heating a sample and measuring the heat flow as compared to a reference standard, we can access thermoanalytical information on a given material. These methods are also used for the determination of adequate drying temperatures.

2. Material and Method

2.1. Material

Fresh sclerotium of *Pleurotus tuberregium* was harvested from a decaying *Treculia africana* (breadfruit) tree. It was identified at the Faculty of Agriculture, Chukwuemeka Odumegwu Ojukwu University, Igbariam Campus. 5.0 kg of the sample was thoroughly washed with distilled water and dried in dust free condition for three weeks. The dark brown back was carefully removed with sterilized knife and inner white component was ground into fine particles for analysis (**Figure 1** & **Figure 2**).

Figure 1. (a) Unpilled and (b) Pilled sclerotium of *Pleurotus tuber-regium*.

2.2. Methods

2.2.1. Determination of XRF Analysis of Sclerotium of *Pleurotus tuber-regium*

The x-ray fluorescence analysis of sclerotium of *Pleurotus tuber-regium* was determined using Shimadzu EDX-720 x-ray fluorescence. The analysis was conducted in vacuum and the system was cooled using nitrogen gas.

2.2.2. Differential Scanning Calorimetry (DSC) Analysis of the Sclerotium of *Pleurotus tuber-regium*

The DSC curves were obtained using a TA Instruments DSC 2920 with controller thermal analyst 2000 system at 10˚C/min constant heating rate following the standard procedure [19]. The hot air flow of 50 mL/min and sample weighing about 2.5 mg were utilized in the experiments [16]. This was aimed at determining the thermal stability property of the sclerotium of Pleurotus tuber-regium.

An empty covered sample pan with lid was weighed using Sartorius BP 410 digital scale and the value recorded. The sclerotium of Pleurotus tuber-regium was collected with spatula and dropped. The sample pan containing the sclerotium of Pleurotus tuber-regium was reweighed to determine the mass of the sclerotium of Pleurotus tuber-regium. The pan containing the sample was placed in the DSC 2920 equipment furnace. The heat flow rate was set at 10˚C/min and maximum temperature the sample was subjected to 400˚C. The machine was activated to run the experiment.

2.2.3. Thermogravimetric Analysis (TGA)

The thermal properties of the samples were measured with TGA 2950 Hi-Res Thermogravimetric Analyzer-TA Instruments with controller Thermal Analyst 2000. The weight of sample was 6.50 mg. Before analysis, the instrument was preliminarily calibrated with standard weight and tested with standard calcium oxalate monohydrate [16]. The sample was heated from ambient temperature to 1000˚C at a rate of 20˚C/min constant heating rate in nitrogen atmosphere following the standard procedure [20]. This was aimed at determining the thermal stability property of the sclerotium of Pleurotus tuber-regium. TGA was used to

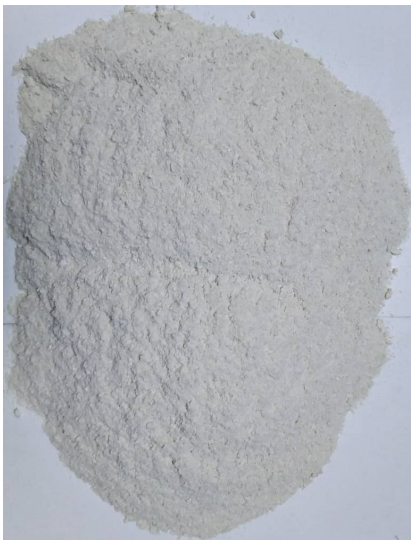

Figure 2. Dried sclerotium of *Pleurotus tuber-regium* (*ọsụ*) grounded powder.

measure the rate change in mass of sclerotium of Pleurotus tuber-regium as a function of temperature in a controlled atmosphere. This analysis indicates material thermal stability with temperature (*i.e.* weight loss or gain) due to decomposition, oxidation or dehydration. It shows change in mass of sclerotium of Pleurotus tuber-regium versus temperature as it is heated at uniform rate of 20˚C/min.

The derivative thermogravimetric curves (DTG) were calculated as the first derivative of the TG curves and were used to determine the mass loss points. All the mass loss percentages were determined using TA-60 data analysis software. The derivatives of TGA thermograms were obtained using origin 8.0 analysis software [21] [22]. The software measures the changes in mass in relation to changes in temperature, with the derivative mass loss curve indicating at which point the mass loss in most apparent.

2.2.4. Determination of Scanning Electron Microscopy

The scanning electron microscopy (SEM) was carried out with a TESCAN Vega 3 XMU scanning electron microscope. A field emission gun and an accelerating voltage of 15kV and magnification of 500X were used.

3. Results and Discussions

3.1. Components of Sclerotium of *Pleurotus tuber-regium*

The x-ray fluorescence analysis of sclerotium of *Pleurotus tuber-regium* is presented in **Table 1**. The analysis indicated that Na_2O, MgO, SiO_2, Al_2O_3, and Fe_2O_3 are the major compound in the sclerotium with traces of Cl, ZnO, SrO SO_3, P_2O_5, CaO, TiO_2, K_2O, Mn_2O_3, and Cr_2O_3. Sodium oxide has the highest concentration of 40.07%, followed by magnesium oxide and silicon dioxide with concentration of 20.23% and 19.24% respectively. Aluminum oxide has concentration of 8.49% while the concentration of iron III oxide is 5.49%.

Table 1. Table type styles (Table caption is indispensable).

S/N	Component	Concentration (wt. %)	S/N	Component	Concentration (wt. %)
1	Na_2O	40.069	9	SO_3	0.754
2	MgO	20.232	10	P_2O_5	0.438
3	SiO_2	19.243	11	CaO	0.008
4	Al_2O_3	8.493	12	TiO_2	0.033
5	Fe_2O_3	5.491	13	K_2O	0.032
6	Cl	2.042	14	Mn_2O_3	0.003
7	ZnO	2.000	15	Cr_2O_3	0.002
8	SrO	1.004			

Sodium oxide is ionically bonded ceramic oxide that has many applications which include but not limited to binder, adsorbent, cleaner, degreaser, mouthwash, body wash, toner, and fragrance. It is utilized by petrochemical, pharmaceutic, construction, plastics, paint, and coating industries. Sodium oxide can be used in the production of alkali-activated materials [23] [24] [25]. Such applications' include alkali-activated slag (AAS) concrete, which is produced by using an alkali activator on ground granulated blast furnace slag, thereby reducing the effect of CO_2 emission by convectional cement industries. Therefore, sclerotium of *Pleurotus tuber-regium* can serve as alkali activator since it contains pronounced percentage of sodium oxide and silicon dioxide.

Magnesium Oxide is an incombustible material with high melting point of 2270˚C. The oxide in magnesium oxide is converted to hydroxide in water and therefore, the properties are very similar to those of magnesium hydroxide. Magnesium oxide gives some exciting health benefits and can relief the symptoms of a variety of ill-health conditions. It has a high thermal conductivity coupled with a low electrical conductivity [26]. It can serves as excellent filler in polylactic acid composites used in orthopedic implants due to good biocompatibility and non-toxic biological activity [27]. Besides, Magnesium oxide undertakes the function of alkaline degradable material and could improve the mechanical properties of the polymer matrix [28].

Silicon dioxide, also known as silica is a natural compound of oxygen and silicon, though it can be produced in the laboratory. It has worthy use in medicine and nutrition such as in tablet-making, including as an anti-caking agent, adsorbent, disintegrant, or glidant to allow powder to flow freely when tablets are processed. As anti-caking additives, it has the ability to help prevent ingredients from becoming moist and clumping together. In tableted foods for special diets, it can aid as an absorbent for dl-a-tocopheryl acetate and pantothenyl alcohol. Also, it serves as a stabilizer that prevents chill haze during beer production and is filtered out of the end product. This give indication why sclerotium of *Pleurotus tuber-regium* may therefore be used as an alternative to maize starch BP as a tablet disintegrant.

3.2. Thermal Behavior of Sclerotium of *Pleurotus tuber-regium*

The thermogravimetric (TGA) diagram of the sample of the sclerotium with is shown in Figure 3. The green curve represented the thermogravimetric plot while its derivative is represented with the blue curve. The sample was heated from ambient temperature of 27.32˚C to 1000˚C at 20˚C/min. The initial mass of the test sample was 6.50 mg. It could be observed that three mass losses occurred, as shown in Figure 3. The first mass loss was related to sample dehydration, followed by a period of stability. The second loss was the oxidation of the organic matter, and the third loss was related to the decomposition of organic material [22].

The dehydration of the sample ended at about 110.38˚C with mass loss of 17.70%. After the dehydration the sample was thermally stable until oxidation occurred. It implies that sclerotium of *Pleurotus tuber-regium* is thermally stable up to 233˚C. The mass loss plateau of the TGA curve occurred between 233.42˚C and 373.82˚C as shown in the derivative of the thermogravimetric curve. The major component in the sample which is Sapogenin was decomposed at about 315˚C that represented the peak of the derivative curve. Oxidation reaction occurred within this temperature range (233.42˚C and 373.82˚C) with release of heat by the sample. About 56.65% of the sample was lost during this process. Steroid which is the second major component of the sample was decomposed at about 400˚C to 480˚C as shown in the TGA curve. The decomposition of most of the organic materials stopped at about 531.55˚C with 9.65% of the sample remaining. The ash content that contain majorly magnesium oxide remained after the end of the analysis with a mass 0.6907 mg.

Figure 4 presented the differential thermal analysis of sclerotium of *Pleurotus tuber-regium* with its derivative. The initial mass of the test sample was 2.0 mg and the sample was subjected to maximum temperature of 1000˚C. The curve

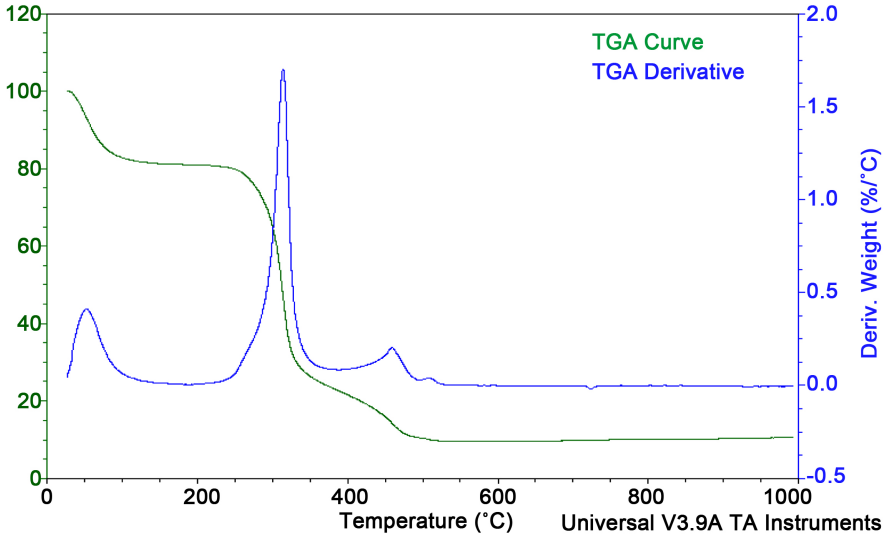

Figure 3. Thermogravimetric diagram of sclerotium of *Pleurotus tuber-regium.*

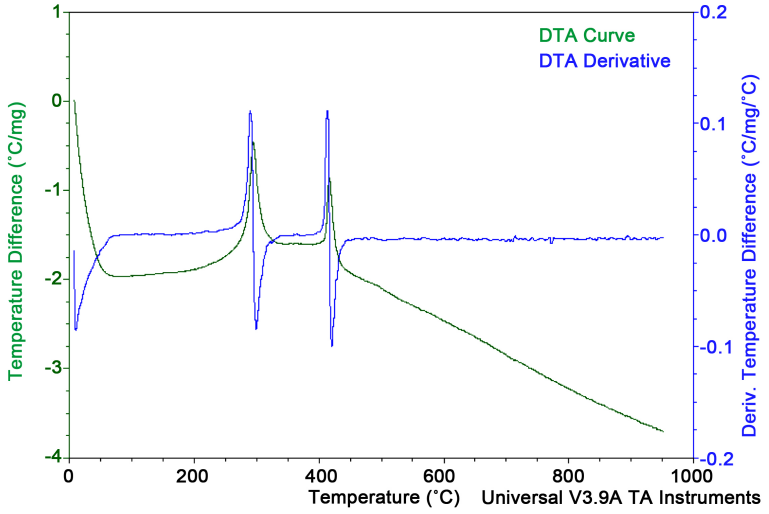

Figure 4. Differential thermal analysis of sclerotium of *Pleurotus tuber-regium.*

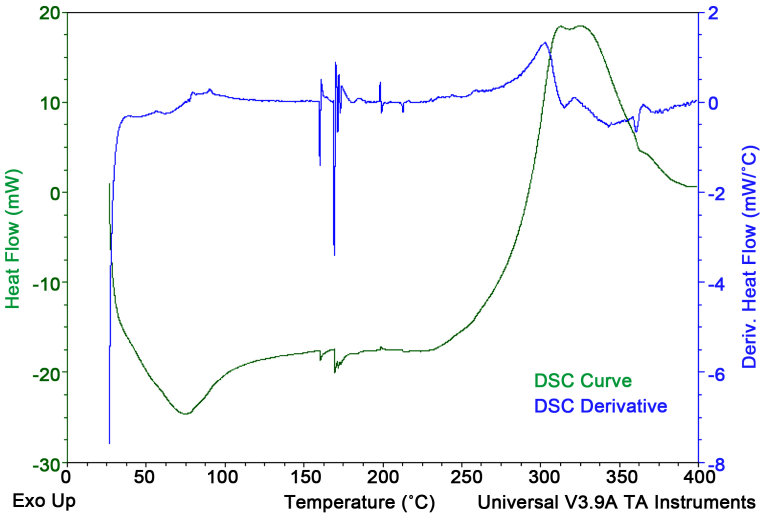

Figure 5. Differential scanning calorimetry analysis of sclerotium of *Pleurotus tuber-regium.*

indicated that three stages of thermal stability in the sample: 70.08˚C - 251.69˚C, 327.92˚C - 395.18˚C, and 445.63˚C - 1000˚C. The result furthermore confirmed that sclerotium of *Pleurotus tuber-regium* is stable up to 233˚C. Also, it could be seen that two major oxidation reactions occurred with peak values at temperatures of 295.41˚C and 419.85˚C.

Differential scanning calorimetry (DSC) analysis of sclerotium of *Pleurotus tuber-regium* with its derivative is shown in **Figure 5**. The sample was heated from ambient temperature of 27.32˚C to 400˚C at 10˚C/min. A mass of 2.50 mg was used for the analysis. The oxidation temperature range observed was between 228.92˚C to 305.15˚C with change in heat flow rate from −17.58 mW to 14.38 wW. **Figure 6** presented the overlay comprising the three curves. It could be observed that sclerotium of *Pleurotus tuber-regium* is thermally stable up to 233˚C.

3.3. Microstructure of Sclerotium of *Pleurotus tuber-regium*

The SEM mircoimaging of sclerotium of *Pleurotus tuber-regium* is shown in Figure 7. The image was taken at 15 kV with magnification of 500. It indicated

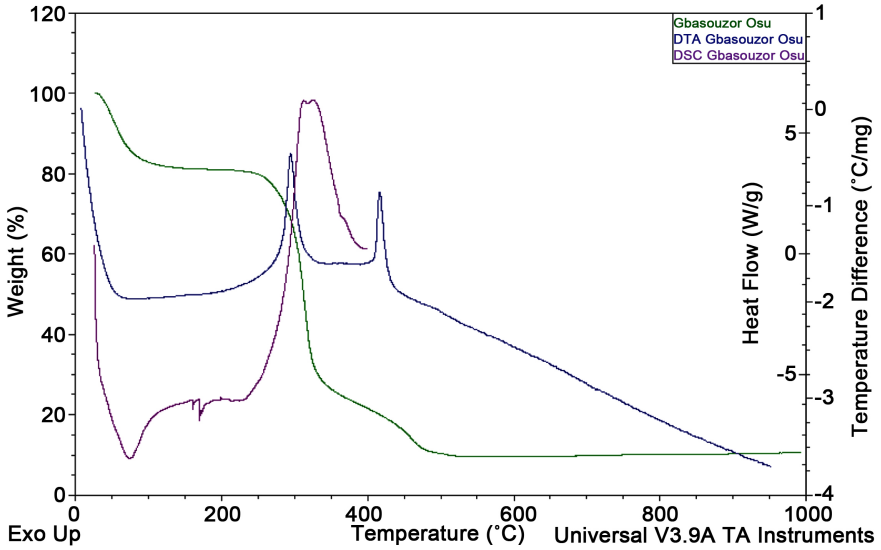

Figure 6. Thermal analysis overlay of sclerotium of *Pleurotus tuber-regium.*

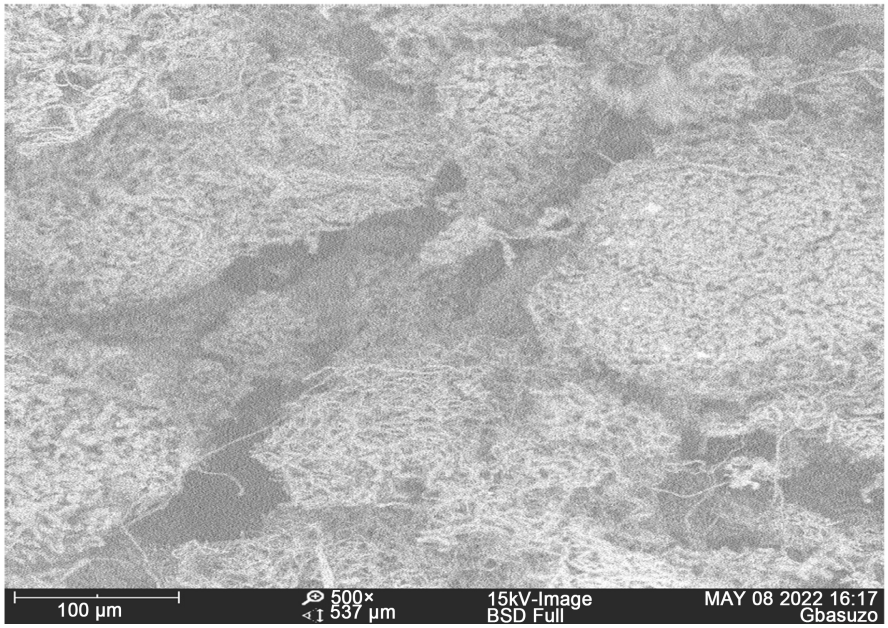

Figure 7. Scanning electron microscopy (SEM) of sclerotium of *Pleurotus tuber-regium.*

that sclerotium of *Pleurotus tuber-regium* is a solid with amorphous structure. It has threadlike fibers with spherical particles. The particles were closely packed and relatively distributed.

4. Conclusion

The x-ray fluorescence analysis of sclerotium of *Pleurotus tuber-regium* showed that Na_2O, MgO, SiO_2, Al_2O_3, and Fe_2O_3 are the major compound in the sclero-

tium with traces of Cl, ZnO, SrO SO_3, P_2O_5, CaO, TiO_2, K_2O, Mn_2O_3, and Cr_2O_3. Thus sclerotium sclerotium of *Pleurotus tuber-regium* contained some hard oxides. Moreover, the SEM analysis revealed that it is a solid with amorphous structure having some fibrous skeleton. The thermal analysis showed that it is thermal stable up to 233˚C. Although three stages of thermal stability were observed upon heating the sample to 1000˚C: 70.08˚C - 251.69˚C, 327.92˚C - 395.18˚C, and 445.63˚C - 1000˚C. Oxidation reaction occurred between 233.42˚C to 373.82˚C with release of heat by the sample. About 56.65% of the sample was lost during the oxidation reaction. Steroid which is the second major component of the sample was decomposed at about 400˚C to 480˚C as indicated in the TGA curve. The decomposition of most of the organic materials stopped at about 531.55˚C with 9.65% of the sample remaining. The ash content that contain majority of magnesium oxide remained after the end of the analysis with a mass 0.6907 mg. From the study, the sclerotium of *Pleurotus tuber-regium* could be totally dehydrated at about 110.38˚C without deterioration of its nutritional and medicinal properties.

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Conflicts of Interest

The authors declare no conflicts of interest regarding the publication of this paper.

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