

Fibers from grape leaves: thermo-gravimetric and differential scanning calorimetry analysis

Obada Nayef Al-leimon*, Ahmad Nayef Al-leimon

King Abdullah II School for Excellence, Al Marj, Al karak, 61112, Jordan

*obada-nayef@outlook.com

ABSTRACT: We prepared fibers from an alternative source, dry grape leaves, using a simple and low-cost procedure. The thermal analysis of the prepared fibers obtained from dry grape leaves using thermal gravimetric analysis (TGA) and differential scanning calorimetry (DSC) shows similar characteristics to that of standard cotton. Furthermore, chemical analysis of the remaining grape leaves' biomass after cotton fiber preparation indicated its potential use as biofertilizer.

KEYWORDS: Agro-waste; Grape leaves; Cotton fibers; TGA; DSCA; Biofertilizer

Introduction. Agro-waste is defined as waste that is produced from various agriculture activities that includes different parts of plants that are usually considered useless and are ultimately discarded.¹ The agro-waste of grape farms and related industries includes grape leaves and the pomace. Several reports have documented the beneficial use of grape waste produced from wine and juice production. During wine production, approximately 25% of the grape weight results in by-product/waste, termed 'pomace', which is comprised of skins and seeds.² Various applications for grape pomace have been reported including functional food production, biosurfactants, cosmetics, pharmaceuticals, supplements, gold nanoparticles, dye removal and as an effective adsorbent for Cr(VI).^{2,3,4,5,6} Meanwhile, very limited work has been done to investigate the potential use of the other components of grape farm waste including grape leaves.

Deciduous plants are those plants that lose their leaves at certain times of the year in a process called abscission. These plants include trees, perennial herbs, and shrubs. Huge quantities of leaf biomass fall to the ground every year and help to recycle natural carbon, nitrogen, phosphorous, sulfur and several other mineral nutrients of the soil when decayed. Plant leaves contain many derivatives of the four major biological molecules in their cellular structure. Among the four major biological molecules, the derivatives of polysaccharides called cellulose are considered the most abundant biological molecule on earth.

Cotton produced by plants of the genus *Gossypium* is produced as a seed hair or white fibrous pulp composed largely of cellulose and other non-cellulosic materials. Cellulose is a complex molecule of glucose polymers linked by glycosidic covalent bonds. It has very important mechanical and physical properties that lead to greater resistance to chemical effects when compared with other polysaccharides. Cotton is considered the most important fibrous material used in the textile industry. However, due to shortages in cotton production and

many other economical and agricultural reasons, the industry began to replace cotton with crude oil derivatives and polymers.⁷ Recently, the fluctuating and increasing crude oil prices, political issues, and oil shortages related to their unrenewable nature has led researchers to find other alternatives that are ecofriendly, renewable, as well as degradable to substitute for crude oil textile polymers and the shortage in the natural cotton supplies., The folk application of fallen grape leaves, treated or untreated, as bio-fertilizer is already known, while other applications have been rarely reported. Therefore, the aims of this project were to recycle fallen grape leaves and to evaluate their potential as a source of cotton fiber using a simple and cheap approach. Furthermore, we will evaluate grape biomass as a soil amendment to support the growth of plants.

These include functional foods, biosurfactants, cosmetics, pharmaceuticals, supplements, production of gold nanoparticles, adsorbent for Cr(VI) and dye removal.^{2,3,4,5} The potential of grape leaves as agro-waste has been poorly studied. In 2013 and 2016, respectively reported grape leaves as a rich source of phenolics compounds and resveratrol as an important stilbene that benefits human health.^{8,9} However, the idea of extracting cellulosic fibers and cotton fibers from the dry grape leaves was never reported.

Results and Discussion. Cotton fibers from fallen grape leaves were successfully prepared through a simple and cheap procedure as illustrated in the experimental methodology. Figure 1 shows the biomass of the prepared white cotton fibers from dry grape leaves. However, the utilization of grape winery waste known as pomace (grape waste comprised of skins and seeds) in a vast array of applications has been previously reported.²

The resulting cotton fibers obtained from the dry grape leaves were further characterized and compared with normal cotton using thermal gravimetric analysis (TGA) and differential scanning calorimetry (DSC). The results of thermal analysis appear in Figure 2 and 3 as a comparison between grape cotton

(Figure 2) and natural cotton (Figure 3) and shows that they have the same characteristics.



Figure 1. White biomass of cotton fibers prepared from dry grape leaves.

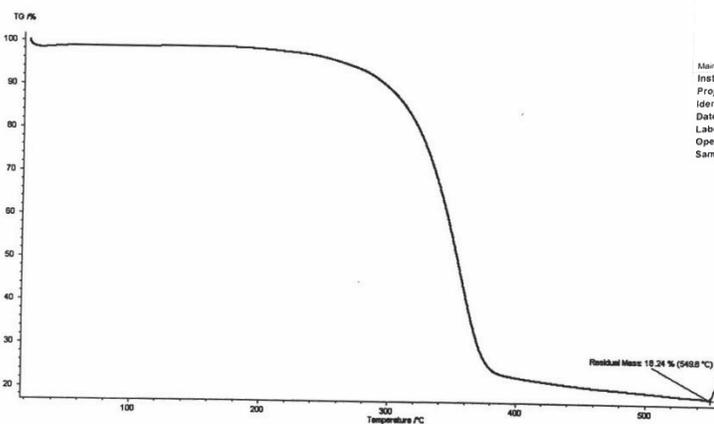


Figure 2. TGA analysis of grape cotton.

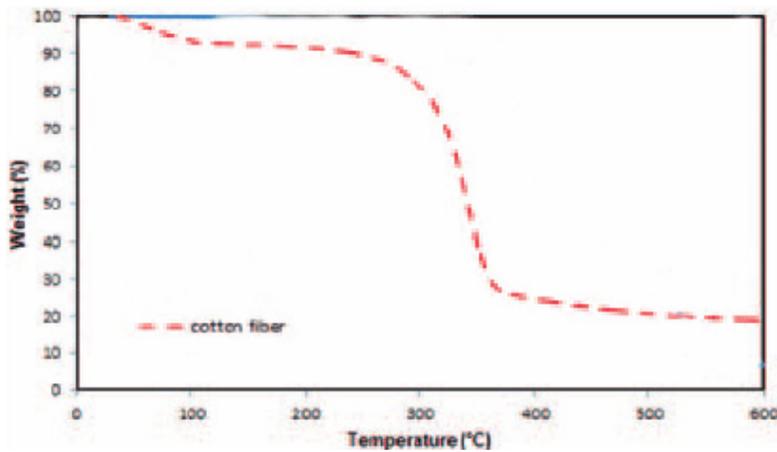


Figure 3. TGA Analysis of natural cotton.

Differential scanning calorimetry, or DSC, is a thermos-analytical technique in which the difference in the amount of heat required to increase the temperature of a sample and a reference is measured as a function of temperature. When test grape cotton was compared with normal cotton using differential scanning calorimetry, or DSC, the results (Figure 4) showed that, the sample (grape cotton) and the reference (standard cotton) were maintained at nearly the same tempera-

ture throughout the experiment. As a result, our product (grape cotton) as analyzed by TGA and DSC tests appears to have the same physical and chemical properties.

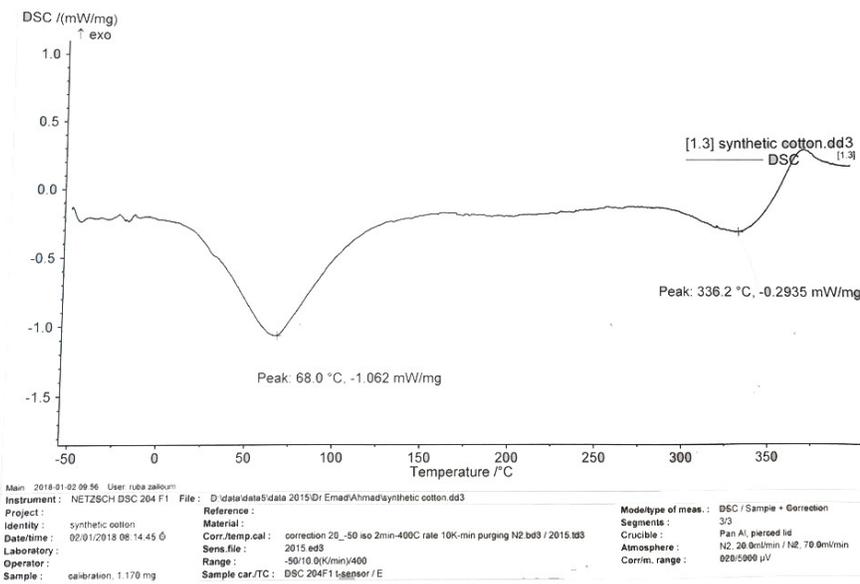


Figure 4. (DSC) - For grapes cotton.

Furthermore, the resulting biomass after grape cotton preparation as well as the huge biomass of the fallen grape leaves seems to be interesting agro-waste product. The chemical analysis shown in Table 1 indicated the usefulness of the remaining biomass of the dry grape (Figure 5) leaves after fiber preparation as potential fertilizer. The chemical analysis results showed that several mineral salts as well as potassium, phosphorus and nitrogen are present in considerable amounts. The presence of such nutritional minerals in grape leaves makes them a potential organic ecofriendly amendment to the soil to support the growth of several cultivated crops.



Figure 5. Grape leaves material remaining after fiber preparation.

Experimental Methods. The fallen grape leaves were collected in the months of October and November. Grape cotton was prepared as follows: approximately 200 g of dry grape leaves were collected and ground using an electrical grinder. The ground grape leaf biomass was then sieved using a soft strainer to separate the fibers from the rest of biomass. A total

of 100 mL sodium hypochlorite (6% - 6.25%) were added to the fibers prepared from the dry grape leaves. The fibers were washed two times with 200 mL water to remove the sodium hypochlorite. Finally, samples of the washed cotton fibers prepared from the grape leaves were subjected to thermal gravimetric analysis (TGA) and differential scanning calorimetry (DSC). The TGA and DSC analyses were performed in the laboratories of the National Center for Agricultural Research and Extension (NCARE), Alraba, Jordan. In addition, the remaining grape leaves biomass after fiber preparation was evaluated as a potential organic fertilizer through an NPK and other minerals analyses. The NPK analysis was performed in the laboratories of the National Center for Agricultural Research and Extension (NCARE), Alraba, Jordan.¹⁰

P	K	N	Ca	Mg
%	%	%	%	%
0.122	0.50	1.39	5.61	0.59
Mn	Zn	Cu	Fe	
Ppm	Ppm	Ppm	Ppm	
123	19.7	13.8	1534	

Table 1. Chemical composition of the remaining biomass from grape leaves.

Lab. No.	Field No.	P	K	N	Ca	Mg
		0.02	0.50	1.39	5.61	0.59
Lab. No.	Field No.	Mn	Zn	Cu	Fe	
		123	19.7	13.8	1534	

Figure 6. Chemical composition certificate obtained from NCARE

TGA is a thermal analysis method in which the mass of a sample is measured over time as the temperature changes. This measurement provides information about physical phenomena, such as phase transitions, absorption and desorption; as well as chemical phenomena including chemisorption, thermal decomposition, and oxidation or reduction.

Conclusion. The present work reported the use of the dry grape leaves as a source of cotton fibers and as organic fertilizer. The results revealed that the thermal characteristics of the prepared grape leaf cotton was similar to the thermal properties of the standard cotton. In addition, the chemical composition of the leftover grape leaf biomass after preparation of cotton fibers can be used as an ecofriendly fertilizer to support the growth and production of cultivated crops as well as be used for filtration, production of different textiles, and for natural industries.

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