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THE INFLUENCE OF SALINITY LEVEL ON THE THERMOGRAVIMETRIC RESPONSE OF BLACK BEAUTY ZUCCHINI

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Abstract
Thermogravimetric analysis (TGA) is a fundamental laboratory instrument applied for investigation of the material properties in various fields such as pharmaceutical, environmental, food and petrochemical applications. The differential thermal analysis (DTA) is common thermal analysis method in which an analyte and an inert reference are heating at a certain heating rate while any temperature change is recorded. In this method, changes in the weight of a specimen are measured while its temperature is increased. Moisture and volatile contents of a sample can be measured by TGA. In our research, it provides qualitative and quantitative data for kinetics of thermal weight losses of the 'Black Beauty' *zucchini samples due to the influence of three salinity levels: 1g/l, 2g/l and 4g/l NaCl. The equipment used in this experiment is Shimadzu DTG-60H. The TGA thermograms for the fruits and leaves of the germinated zucchini in different salinity conditions were studied. For the zucchini leaves, an increase in the temperature at which certain reactions take place was noted, along with the increase in the salinity level. For the determination of structural chemical components, complementary methods such as IR and UV Vis spectroscopy are necessary.*

Keywords: salinity level, thermogravimetric analysis, zucchini.

1. INTRODUCTION
Differential Thermal Analysis (DTA) is a powerful technique for analyzing the thermal behavior of materials. The principle of DTA is based on the measurement of the difference in temperature between a sample and a reference material as they are subjected to controlled heating or cooling. During heating or cooling, the sample and the reference material undergo thermal transformations that result in the absorption or release of heat.
These transformations can be detected by measuring the temperature difference between the sample and reference materials as a function of time or temperature (Nassar, 2023).
DTA can be used to determine the thermal properties of materials, such as the specific heat capacity, thermal conductivity, and thermal diffusivity. This is particularly useful for phase-change materials and the study of organic materials using analytical precision (https://ctherm.com).
In the specialized literature, there are few studies on plant materials using thermal analysis, generally in the field of pharmaceuticals or energy sources (biomass) (Yi et al., 2013; Xinjie et al., 2021; Tariq et al., 2022; Si et al., 2024).

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Zucchini (*Cucurbita pepo*) are recognized for their nutritional and medicinal benefits as well as their economic impact (Paris and Lust, 2020). According to studies, these plants are sensitive to salt stress, one of the factors limiting production, especially in arid and semi-arid regions of the world (Ahmed, 2009). The salt stress negatively affects physiological and biochemical processes essential in growth and development processes, especially in the germination, seedling or flowering phase (Khoshsokan et al., 2012).
Therefore, we aimed to use thermogravimetry and Differential Thermal Analysis (DTA) to study the behavior of different 'Black Beauty' zucchini samples (cotyledons, leaves, fruits) subjected to various levels of salt stress.

2. MATERIALS AND METHODS

In order to observe the response of 'Black Beauty' zucchini to salinity stress, we put to germinate five uniform and healthy seeds (for each experimental variants) in 9-cm sterile Petri dishes on a dual filter paper. Then were moistened with 10 ml distilled water (control - BBM) or saline water solution at 1g/l (BB1), 2g/l (BB2), 4g/l (BB4) NaCl.
We monitored the germination process, for seven days and then, we collected samples from each experimental variant (Figure 1)

***Figure 1.** Germinated seeds of* **'Black Beauty'** *(a); Collected samples (b)*

To continue the observations, at the moment of the appearance of true leaves, the seedlings were transferred to 14 cm pots where they were kept until the climatic conditions allowed their planting in the field. After one month, samples were collected from leaves and fruits of 'Black Beauty' zucchini (Figure 2).

Figure 2. Leaves for sample collection (a); Fruit for sample collection (b); Dried leaves prepared as samples (c)

The leaf mass from the seed that did not receive any salinity (left side of the figure 2c) is 1.02g and the leaf from the seeds that were growing in the salinity of 4mg/l NaCl has a mass of 4.57g (right side of the picture). Before being processed, the fruit samples were kept for 10 minutes at 110 °C.

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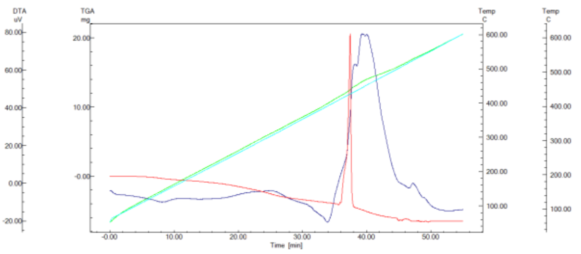
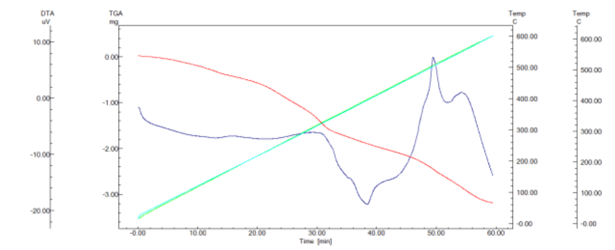
The thermogravimetric curves were plotted for each of the samples, using Shimadzu DTG-60H (https://www.shimadzu.com). This instrument varies the sample temperature in accordance with a program and simultaneously measures the change in mass of the sample (TG) and the temperature difference between the sample and a standard substance (DTA). The thermogravimetric (TG) measurement range is broad at ±500 mg, with a weighing capacity of 1 g (including tare weight), so it can be used for diverse measurements. In our case, the standard substance was Al_2O_3 (α-Alumina) and the weight of α-Alumina and the aluminum capsule was 28.55 grams. The atmosphere inside the oven was air. Thermogravimetric experiments are carried out on the samples with 0.18 mm particle size at the heating rate of 20°C/min. To generate the graphics from data collected from our equipment, we had to make a software that was able to synchronize all the values according to temperature, not by time. The first step is to export data from Shimadzu software as text. All the values are saved on columns, for time one column, the oven temperature another column, the sample temperature another column, and another two columns for TDA and TGA. The software is able to read up to three files in the same time, so we can have up to three data values on the same time. The synchronization can done manual or by the computer. After this stage the computer it will generate the new data set of value for temperature axis, this is necessary because all the temperature for the samples presents variation due to heat absorption for different phases.

3. RESULTS AND DISCUSSIONS

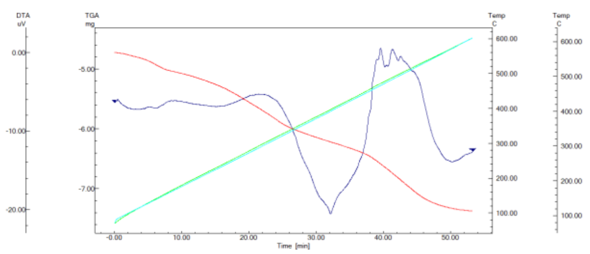
For temperature between 100°C and 600°C (green curve) the sample mass (red curve) and differential thermal analysis DTA (blue curve) were recorded (Figure 3).



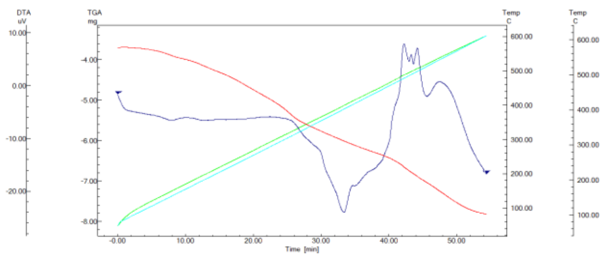
a) BBM



b) BB1



c) BB2



d) BB4

Figure 3. Images from Shimadzu graphical interface, for seeds growth in different salinity concentration

From the figures above, we notice a decrease in the mass of the samples (BB1, BB2, BB4), with the increase in temperature. This is a general characteristic, due to the evaporation of water at 100°C or other substances at 200°C. Sometimes these substances do not evaporate, but recombine (by reduction or oxidation), causing a slight increase in mass, due to the absorption of oxygen from the atmosphere (BBM). Regarding the thermogravimetric response of 'Black Beauty' zucchini, we note

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in all cases, the appearance of sudden variations on the graph; they indicate a decrease in the temperature difference between the α-Alumina and the sample, due to the evaporation of the substances. The data obtained from the DTA (blue curve) allow the determination of the caloric capacity of the substance that has evaporated, and even their identification by comparing with a database.
The next step in our experiment was to study the thermogravimetric response of leaves and fruits samples obtained from 'Black Beauty' zucchini seeds subjected to various salinity levels.

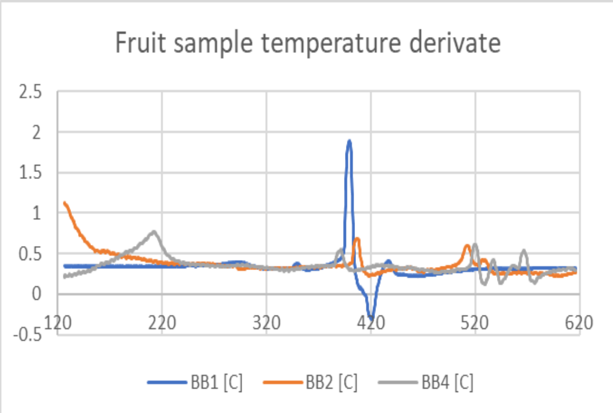

Figure 4. Temperature derivate for fruits sample

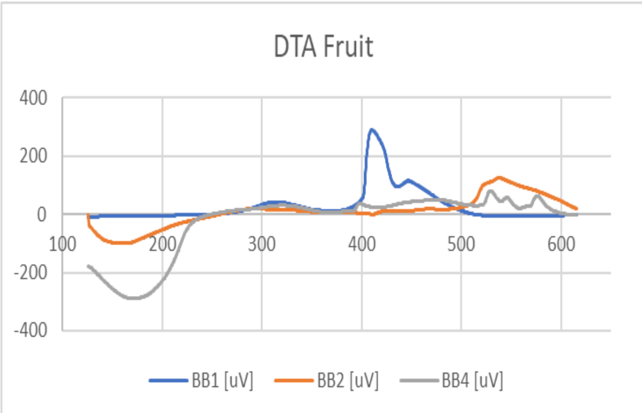

Figure 5. Differential thermal analysis for fruit sample

In figure 4, the speed of temperature variation over time is represented vertically, and the temperature in degrees Celsius horizontally, for fruits sample. We note the strongest variation for fruit samples obtained from seeds germinated at 1g/l NaCl (BB1 – blue curve) and similar behavior of the temperature derivative for fruit samples subjected to salinity levels of 2g/l (BB2 – orange curve), respectively 4g/l (BB4 – gray curve).
Figure 5 shows the potential difference (μV) depending on the temperature (°C), for the studied fruit samples. The minimum potential noted on the gray curve (BB4 – 4g/l NaCl), for 180°C, indicates the fact that this sample contains substances that have not yet evaporated, unlike the samples corresponding to the low salinity levels of 1g/l NaCl (BB1 – blue curve) and 2g/l NaCl (BB2 - orange curve).

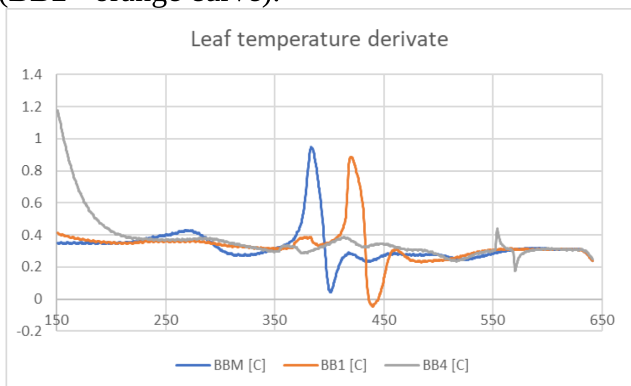

Figure 6. Temperature derivate for leaves sample

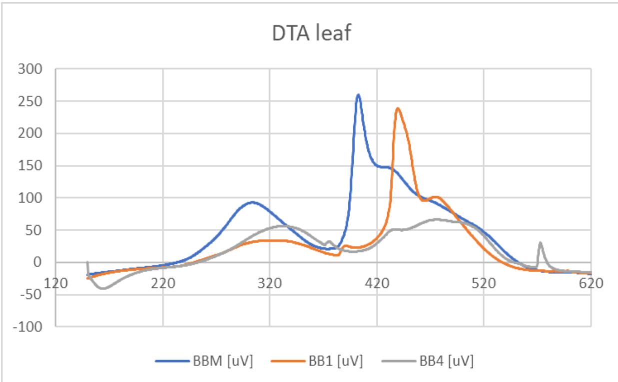

Figure 7. Differential thermal analysis for leaves sample

In figure 6, the derivate of temperature in relation to time is represented vertically, and the temperature in degrees Celsius horizontally, for leaves samples. We notice a similar thermal

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behavior of the leaf samples subjected to low values of NaCl (1g/l - BB1) with those of the control group (0 g/l BBM).

Figure 7 is the most relevant, the value for peak position is 404^0C for BBM (0 g/l NaCl), 441^0C for BB1(1 g/l NaCl) and 568^0C for BB4 (4 g/l NaCl). The difference between BBM peak position and BB1 position is 37^0C. The difference between BBM and BB4 is 164^0C. From this difference presented above we can see that ratio between these two values, 37^0C and 164^0C is almost 4 time, corresponding to salinity ratio.

4. CONCLUSIONS

Differential Thermal Analysis (DTA) is a versatile technique that can be used to study a wide range of materials, including vegetal materials. The creating a database for the plant material, associated with this type of analysis, allows the precise identification of the chemical reactions that occur during the heating process. But for the determination of structural chemical components, complementary methods such as IR and Vis spectroscopy are necessary.

The thermogravimetric curves plotted for 'Black Beauty' zucchini cotyledons did not provide conclusive data regarding the influence of the salinity level. Therefore, further analysis and repetition of the experiment are necessary.

From DTA curves for 'Black Beauty' zucchini leaves, it was noted that the temperature at which certain reactions take place increases with the increase of the salinity level, in the same proportion.

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