

Determination of Starch Content in Potato Powder by Differential Thermal Analysis

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This paper comes up with a method for rapid determination of starch content in potato powder by differential thermal analysis. Resorting to thermogravimetric analysis and infrared absorption spectrum analysis, the author locates the characteristic peaks of starch decomposition by identifying the absorption peaks and optimizes the influencing factors. The results reveal a linear relationship between starch content and the peak area of the differential thermal curve when the heating rate is 5°C/min and the particle size is 100mesh. The linear relationship is expressed by the equation: $y=1.0111x-25.272$ ($R^2=0.99329$). The method is fast, accurate and highly practical.

1. Introduction

According to different processing and drying methods, potato powder is divided into: potato granule powder, potato powder and potato flake powder (Liu et al, 1999). The potato granules (granules) mainly refers to the production through the use of air drying technology and powder particles, the finished product is mainly through powder polymer particles exist in the form of potato powder in potato cell monomer; and potato powder is refers to the use of fresh potato as raw material after cleaning ,then removed the skins and cut, steamed and mashed, and finally produced by using a series of processes ; the potato flakes (flakes) mainly refers to the production process after drying drum, the thickness of 0.11 to 0.25mm at the same time like potato chips with a diameter of 3 ~ 10.0mm size the rules of powder, because of its appearance looks like snowflakes, so called flakes. The dehydrated potato products were crushed to obtain the potato powder, which we referred it as potato fine powder, referred to as "fine powder" (Wang, 2005).

The standard method for the determination of starch content in grain "for the determination of starch content in grains and oils" (GB/T5514-2008), the determination of reducing sugar in "food" (hereinafter referred to as GB/T5009.7-2008) the standard method, the standard method is accurate, but complicated operation, the use of reagents, the analysis speed is slow, and more stringent requirements for operation in recent years and with the development of science and technology, the rapid determination of starch content in grain has been developing rapidly, the near infrared absorption spectroscopy, polarimetry and reducing sugar analyzer which, near infrared absorption spectrometry for the determination of starch content in maize has been identified as the national standard method but because of the complicated modeling, so the application of the scope is limited.

This paper optimizes the conditions of differential thermal analyzer, validates the accuracy and precision of DTA in measuring starch content in potato powder, and identifies the decomposition temperature of the starch in potato powder with the aid of infrared spectrometry. Furthermore, in comparison with the infrared spectrogram of standard starch, the author locates the characteristic peaks of starch on differential thermal spectrograms, and thereby verifies the DTA results, determines the DTA test parameters of starch in potato powder, and proves that it is practical to apply DTA to starch content of potato powder.

2. Research materials and research method

2.1 Research materials

30 kinds of potato powder from different regions are crushed by a pulverizer for later use.

Test materials include standard potato starch (Sigma-Aldrich), sodium sulphate, sodium hydroxide, methyl red,

lead acetate, petroleum ether, sodium sulfate, hydrochloric acid, amylase, ethanol, copper tartrate A, copper tartrate B, and glucose, which are analytically pure, as well as potassium bromide, which is spectrally pure. All of the materials are purchased from the market.

2.2 Equipment

DTG-60 differential thermal analyzer: Shimadzu International Trade (Shanghai) Co., Ltd.; Fourier transform infrared spectrometer (TTIR-650): Tianjin Gangdong Sci. & Tech. Development Co., Ltd.; DF-4 tablet press: Tianjin Gangdong Sci. & Tech. Development Co., Ltd.; electronic balance: Fuzhou Huazhi Scientific Instrument Co., Ltd.; thermostatic water bath: Jintan Jingda Instrument Manufacturing Co., Ltd.

2.3 Research method

2.3.1 Optimization of potato powder DTA conditions

(1) Influence of the Heating Rate of Differential Thermal Analyzer on DTA Results

To start DTA, warm up sample 3 at the rates of 20, 15, 10 and 5°C/min respectively. Get accurate experimental results with reasonably chosen instruments and heating rates (Wu et al, 2006).

(2) Influence of Potato Powder Mesh Number on DTA Results

In a DTA quantitative analysis, the analysis results are affected by the particle size of the sample. If the sample is poorly crystallized, it has an even heavier influence on DTA, resulting in a more complex situation (Xu and Li, 2011). To arrive at accurate results, the author carries out DTAs on sample 3 at 40mesh, 60mesh, 80mesh and 100mesh respectively and selects appropriate mesh number according to the analysis.

2.3.2 The accuracy of starch content determined by DTA

The accuracy of the method is measured by relative standard deviation (Wang, 1991). In order to validate the accuracy of DTA in measuring the starch content in potato powder, the author obtains the peak area and starch content of different samples respectively by DTA and GB (Chinese national standard), repeats the process three times, and calculates the relative standard deviations (Pignon et al, 2010)

2.3.3 The precision of starch content determined by DTA

In order to validate the precision of DTA in measuring the starch content in potato powder, the author measures the starch content and peak area of potato powder respectively by GB and DTA, calculates the standard deviations and relative standard deviations, and conducts t-test and variance analysis.

2.4 Validation method

2.4.1 Pre-treatment of raw materials

The author crushes the purchased potato powder by a pulverizer, takes a 100g sample by coning and quartering, places the sample into a drying oven, and set the temperature at 105°C to dry the sample. Each time, the author takes 3-7mg of sample to perform DTA.

2.4.2 DTA of potato starch content

The author takes about 5mg of standard sample and 5mg of the sample to be tested, and places them in separate ceramic crucibles. To get the DTA curves, the author increases the temperature at 5°C/min from room temperature to 600°C with air as the experimental gas (Agrawal and Ghoshadastidar, 2007)

2.4.3 FTIR

The author takes about 300mg of potassium bromide and 1mg of starch to be tested, mixes the two and grinds the sample for 10min, and presses it into pellets. After pressing, the author scans the potato starch with an infrared absorption spectrometer 32 times. The results are compared to infrared spectrograms of standard starch with Origin.

2.4.4 Determination of starch content in potato powder

According to GB/T 5009.9-2008, the author measures the starch content of the sample three times in parallel and takes the average value.

3. Results analysis

3.1 TGA

In order to study the thermal decomposition process of potato starch, the author conducts DTA of three different kinds of standard potato starch by DTA-60 (Wang, 2008). Based on the results, there is no significant difference in the differential thermal spectrograms of different potato varieties. The author then screens potato starch through a 100-mesh sieve and obtains a differential thermal spectrogram according to the method in Section 2.4.2 (See Figure 1):

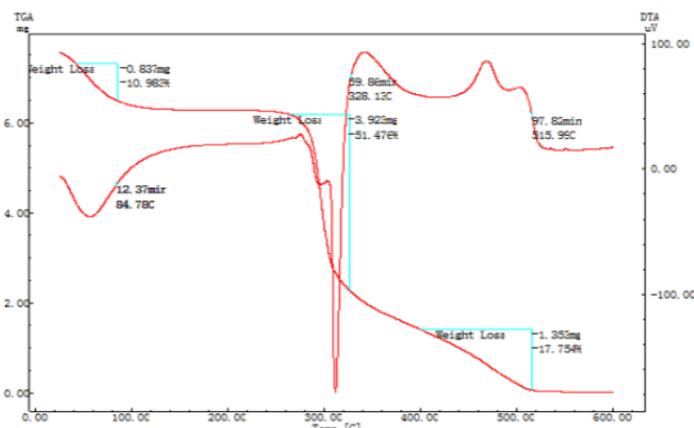


Figure 1: Differential thermal curves of potato starch

As shown in Figure 1, the thermal decomposition of potato starch can be divided into 4 stages: Stage 1: ($t \leq 100^\circ\text{C}$) mass loss (about 10.98%) due to the weightlessness caused by the evaporation of volatile substances, mainly the evaporation of water; Stage 2: ($280^\circ\text{C} \leq t \leq 350^\circ\text{C}$) mass loss (about 51.47%) largely resulted from the decomposition of organic matters like starch; Stage 3: ($400^\circ\text{C} \leq t \leq 500^\circ\text{C}$) mass loss (about 17.75%) mostly because of carbonization of organic matters; Stage 4: ($500^\circ\text{C} \leq t \leq 600^\circ\text{C}$) gradual flattening of thermogravimetric mainly attributable to the complete carbonization of organic matters, leaving nothing but inorganic matters.

3.2 FTIR analysis

To study the changes in the samples at 280°C - 350°C , the author carries out FTIR analysis on standard starch respectively at room temperature, after burning at 280°C , and after burning at 350°C (Rouduit et al, 2013).

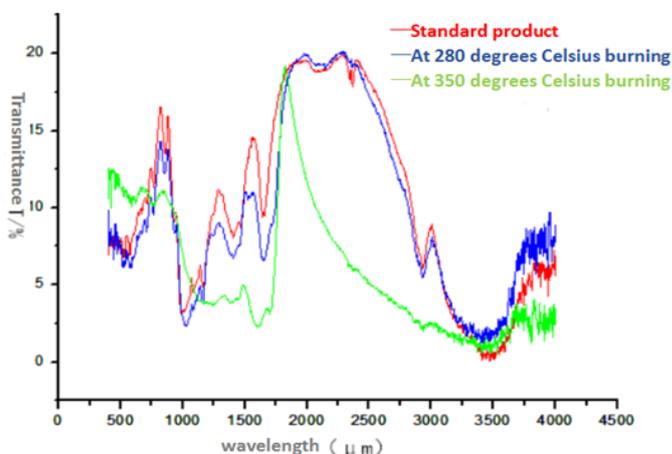


Figure 2: Infrared spectrograms of residues of potato starch after burning at 280°C and 350°C

Figure 2 shows that when the temperature rises to 280°C , i.e. prior to decomposition, the peak shape of the infrared spectrum is similar to that of the starch. However, when the temperature rises to 350°C , i.e. after decomposition, the characteristic peak of the starch is quite different from that of the starch prior to decomposition, indicating that the starch has been decomposed. The results further prove that the decomposition temperature of starch falls between 280°C and 350°C , which is consistent with the results of DTA.

3.3 Optimization of differential thermal conditions

3.3.1 Optimization of instrument heating rates

In order to study the effect of heating rate on the DTA results, the author conducts DTAs with the heating rate of the instrument controlled at $20^\circ\text{C}/\text{min}$, $15^\circ\text{C}/\text{min}$, $10^\circ\text{C}/\text{min}$ and $5^\circ\text{C}/\text{min}$, respectively. See Figure 3 for the results.

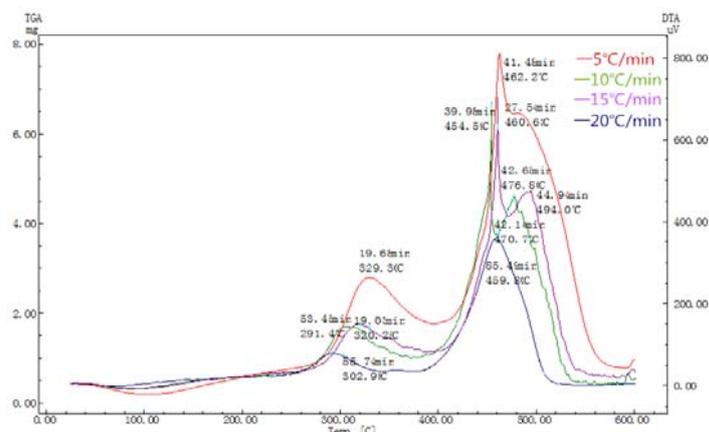


Figure 3: Influence of different heating rates on starch peak of potato powder

Figure 3 displays the differential thermal curves of different heating rates, which demonstrate that: the starch peak of sample 3 is obviously affected by different heating rates. When the heating rate is set at 20°C/min, 15°C/min and 10°C/min, respectively, the rate of resolution is decreased with the increase of heating rate. At this time, the peak shape is broadened, so the symmetry is not strong. When the heating rate is 5°C/min, the starch peak is high, and the peak shape symmetry is better. That is why the author uses the heating rate of 5°C/min.

3.3.2 Optimization of the mesh number of potato powder

In order to study the effect of particle size on DTA results, the author carries out DTAs on 3mg~5mg of sample 3 at 40mesh, 60mesh, 80mesh and 100mesh respectively. See Figure 4 for the results.

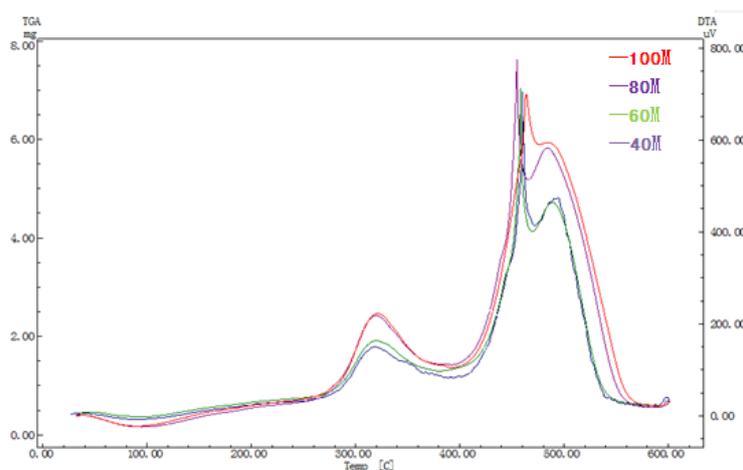


Figure 4: Influence of different particle sizes on starch peak of potato powder

Figure 4 shows the differential thermal curves of sample 3 with different particle sizes, which demonstrates that: When the particle size of the sample is 40mesh, 60mesh and 80mesh, the larger the particle size, the lower the resolution. However, when the sample passes 100mesh, the peak height of the starch peak is larger and the peak shape is better (Jarušková and Kučerová, 2001). Based on the above results, the author decides to measure the starch content in potato powder at the heating rate of 5°C/min, and to carry out DTA on 100mesh samples.

3.4 DTA

In the optimized test conditions, the author conducts DTAs on samples made from different kinds of potato powder. Two parallel determination tests are performed. See the figure below for the correlation curve between average starch peak area and the starch content measured by GB (Chinese national standard) method.

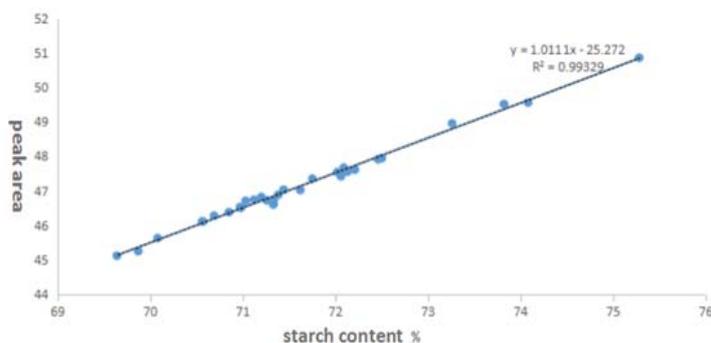


Figure 5: Correlation between starch content and starch peak area in differential thermal spectrogram

As shown in Figure 5, the potato starch content is positively correlated with the corresponding peak area of the DTA curve, and the linearity equation is $y = 1.0111x - 25.272$, $R^2 = 0.99329$.

3.5 The accuracy of starch content determined by DTA

The author obtains the peak area and starch content of different samples respectively by DTA and GB (Chinese national standard), repeats the process three times, and calculates the relative standard deviations (Lorenzini and Saro, 2003).

Table 1: The accuracy of starch content determined by DTA

No.	Sample No.	Peak Area 1	Peak Area 2	Peak Area 3	Average Peak Area	Calculated Content	GB Content (g/100g)	Accuracy %
1	No. 1	46.26	47.18	46.42	46.62±0.56	70.56	70.64	0.1
2	No. 8	47.91	49.19	47.74	48.28±0.91	72.50	72.48	0.2
3	No. 17	50.88	48.76	49.04	49.56±1.32	74.08	74.13	0.6
4	No. 23	49.56	48.02	46.81	48.13±1.43	76.12	76.08	0.5

Table 1 shows that the relative standard deviations of data by DTA method is 0.1%, 0.2%, 0.6% and 0.5% respectively. The relative standard deviations are within 10% of the GB, indicating good accuracy of DTA method.

3.6 The precision of starch content determined by DTA

In order to validate the precision of DTA in measuring the starch content in potato powder, the author measures the starch content and peak area of potato powder respectively by GB and DTA.^[14] See Table 2 for the results.

Table 2: Measured results by GB and DTA ($t_{0.05(8)}=2.306$)

Method	Measured Values						X	S (Standard Deviations)	RSD (Relative Standard Deviations)
GB	71.70	71.64	72.19	72.04	71.97	71.91	71.91	0.23	0.32
DTA	71.70	71.82	72.08	71.91	71.90	71.88	71.88	0.14	0.19

According to Table 2, the relative standard deviations by GB (national standard method) and DTA are 0.23 and 0.14 respectively. In order to verify the precision of starch content determined by DTA, the author has to carry out t-test of the results. It is calculated that $t=0.74$ is smaller than $t_{0.05(8)}=2.306$, indicating that there is no significant difference between the two groups of data.

The author processes the results in Table 2 via SPSS and generates a variance test table (Table 3):

Table 3. Variance Test Table ($F_{0.05(1, 8)}=5.32$)

	SS	df	MS	F	Significance
Intergroup	0.002	1	0.002	0.046	0.835
Intra-group	0.293	8	0.037		
In total	0.295	9			

According to Table 3, $F=0.046$ is smaller than $F_{0.05(1, 8)}=5.32$, and the significance $P=0.835$ is greater than 0.05, indicating that there is no significant difference between the two methods in detecting starch content in potato powder.

4. Conclusion

The test optimizes the conditions of the mesh and the heating rate, proved that the mesh number is 80 and the heating rate is 5 DEG /min for optimum experimental conditions, on this basis, the author uses far- and near-infrared spectroscopies to explore the possibility and reliability of identifying starch content in potato powder by differential thermal analysis. The results showed that the differential thermal analysis method was used to detect the changes of starch content of potato powder in this process will take place in 4 stages: the first stage: ($t \leq 100^{\circ}\text{C}$) quality loss is mainly due to the evaporation of water; the second stage: ($280^{\circ}\text{C} \leq t \leq 360^{\circ}\text{C}$) mass quality loss (about 51.47%) is mainly due to the quality of starch decomposition losses; the third stage: ($400^{\circ}\text{C} \leq t \leq 500^{\circ}\text{C}$) quality loss (about 17.75%) is mainly due to carbonization of organic matter; the fourth stage: ($500 \leq t \leq 600^{\circ}\text{C}$) thermogravimetric curve flattened, and it is mainly due to the third stage that organic compound completely carbonization, only induced by inorganic decomposition (Roduit et al, 2013).

In this test, the author determines the optimized DTA test parameters of starch content in potato powder, and makes detailed analysis of the test performance and reliability of the method. The results are as follows: The standard curves generated by DTA are expressed by the linear equation: $y = 1.0111x - 25.272$ ($R^2 = 0.99329$, which is close to 1). The relative standard deviations of DTA are 0.1%, 0.2%, 0.6% and 0.5%, respectively, which are within 10% of the GB. Moreover, the DTA method has the advantages of short examination time, easy operation, fewer samples (only 3~7mg), and economical efficiency.

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