



Article Characterisation of Pasting, Structural and Volatile Properties of Potato Flour

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Abstract: Potato flour is an important raw material for potato staple food products; nevertheless, the quality and flavor vary significantly due to process changes. In this study, the physicochemical features of fresh and five different dehydration temperature potato samples, including the degree of starch gelatinization (DG), pasting, structure properties and volatile components, were compared to investigate the effect of hot air drying (HAD) on potato flour. The results showed that the degree of pasting, viscosity and volatile aroma components changed significantly with differences in drying temperature. With the increase in drying temperature, the gelatinization degree and peak viscosity of potato powder increased or decreased, the breakdown viscosity of HAD-50 was higher, the setback viscosity of HAD-90 was higher, while the crystallization zone of HAD-90 was destroyed due to the high temperature. The flavor components of potato flour are increased during processing due to lipid oxidation, Maillard reaction and thermal degradation. The level of aldehydes,3,5-Octadien-2-one and E,E)-3,5-Octadien-2-one gradually reduced as the processing temperature increased, while the content of furans grew and then decreased, nonanal and 2-Penty-l-Furan increased. Overall, lower HAD temperatures are beneficial for the quality and flavor of potato flour. The information presented here will be useful for the further development of potato flour products.



1. Introduction

Potato, a highly nutritious agricultural commodity with a rich source of high-quality protein, starch, basic vitamins, minerals and trace elements, is widely used as a food and industrial crop [1]. With a yearly output of more than 370 million tons, potato is among the most important crops, making it the third largest after wheat and rice in total output [2]. Considering the important role of potato as a vital food-security crop, there has been a concerted policy from the Chinese Government that potatoes be promoted as the fourth major staple food in China, next to wheat, rice and corn [3]. Potato flour is an indispensable intermediate raw material in processed potato food; the potato is processed into whole flour and then added to the staple food in a certain proportion (processed into a new, staple potato food product), which is conducive to improving the nutritional value of traditional staple foods, to meet the current demand for nutritious staple foods [4]. However, the research and development of nutritional potato staple food products is confronted with lots of constraints. For instance, the dehydration of fresh potatoes can affect the profile of endogenous compositions, such as moisture, pasting properties and volatile and non-volatile precursors of potatoes, which can significantly affect the quality of the end products [5]. In order to improve the quality of potato flour, it is essential to reduce the drying time by using modern drying technologies to replace traditional natural drying methods [6-8].

Free starch content, microstructure, pasting characteristics and color are often used to characterize the quality of potato flour [9]. To address the problems of high pasting



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Copyright: © 2022 by the authors. Licensee MDPI, Basel, Switzerland. This article is an open access article distributed under the terms and conditions of the Creative Commons Attribution (CC BY) license (https:// creativecommons.org/licenses/by/ 4.0/). and processing performance limitations of traditional whole potato flour, it is necessary to seek some new processing processes to obtain potato flour with lower pasting [10]. In addition, starch crystallinity is an important parameter that characterizes the crystalline nature of starch granules, and its size directly affects the application performance of raw potato powder products [11].

Different processing conditions have been noted to have a great effect on the qualities and volatile compositions of processed potatoes. For example, Yang used SPME GC-MS to identify key volatile compound differences between fresh potato puree and potato puree stored at 4 °C for 1 day and identified more than 30 compounds in both types of puree at varied concentrations [12]. Processing with a microwave oven, which is considered a quick cooking method, has been reported by Jansky to produce the least flavor compounds in potatoes [13]. Such chemical reactions during processing also lead to the development of a blend of volatile and non-volatile chemical compounds that can make food tastier or create desirable or unwanted flavors [14]

Many techniques and methods are available for identifying and quantifying volatile compounds in potatoes, including simultaneous distillation and extraction [15], solvent-assisted flavor evaporation [16], dynamic headspace extraction [17] and solid-phase microextraction (SPME), have been investigated [18]. Others include gas chromatography-mass spectrometry (GC-MS) [19] and gas chromatography-olfactometry (GC-O) [20], whereby volatile compounds are separated by GC, and the human nose is used as a "sniffing port" to describe the various aromas as they exit the GC column. A portion can also be diverted for simultaneous identification by MS (GC-MS-O). Relatively, more research has been performed on the identification of volatiles and reaction products in raw and cooked potatoes, but few on dehydrated potatoes. Therefore, much remains to be learned about the dynamic flavor traits of potatoes and the key components that contribute to them.

Nowadays, most studies focus on the characteristics of potato starch [21], a small number of scholars have begun to pay attention to the properties of potato flour and potato flour added to steamed wheat bread, including rheological and texture properties, viscoelasticity and volatile aroma components [5], but little information is available on the potato flour's properties. Therefore, the aim of the current work is to investigate the influence of the processing temperature on the pasting properties and flavor of potato flour. In this regard, we aim to monitor the viscosity, X-ray diffraction and the profiling of volatile compositions of raw and dehydrated potato flour by means of a rapid visco analyzer, X-ray diffractometer and headspace solid-phase microextraction coupled with gas chromatography-mass spectrometry (HS-SPME GC-MS).

2. Materials and Methods

2.1. Raw Material and Processing Procedures

Fresh potato tubers of the *Longshu* 10 variety were purchased from Gansu province, the main potato-producing area in northwest China. To ensure the uniformity of physical characteristics of the experimental materials, the samples were carefully selected for size uniformity, no rot, no bad odor, no sprouts, no pests or diseases infections and no mechanical damage or greening.

Potato tubers were washed to remove residual sediment and other impurities prior to peeling. Upon peeling, the potato tubers were sliced into uniformly thick (1 mm) slices. In order to prevent the browning of potatoes during processing, a phenomenon that reduces the color attributes of potato flour, slices were treated with color protectant by dipping into 2 g/L citric acid solution and maintained for 10 min. The potato slices were then put on a sieve to drain the color fixative and then thin-layer hot air drying (HAD). The dryer, which was engineered and built at Jiangsu University prior to the commencement of the experiment, is equipped with a temperature adjustment ranging from 0 to 99.9 °C. The drying temperatures used for this experiment were set at 50 (HAD-50), 60 (HAD-60), 70 (HAD-70), 80 (HAD-80) and 90 °C (HAD-90), and HAD-0 means raw potato materials that are not dried. Once the dryer had reached a steady state for the set points (at least

30 min), the single slices were uniformly distributed on a perforated sieve tray. Sample weight was recorded every 20 min until the moisture content was less than 8% or a constant value was achieved. The dried slices were ground, screened through a 120 mesh sieve and packed in low-density polyethylene bags.

2.2. Degree of Gelatinization

The degree of starch gelatinization (DG) was determined according to the Chemical Industry Standards of the People's Republic of China (HG/T 3932-2007). The DG value of potato starch was determined by means of enzymatic hydrolysis. Gelatinized starch is hydrolyzed to glucose by glucose amylase and can be determined by titration of iodine-thiosulphate [22]. The principle is: the glucose is oxidized by iodine in an alkaline solution to gluconic acid, and the excess acidified iodine is then titrated with sodium thiosulfate.

2.3. Rapid Visco Analyzer (RVA) Pasting Properties

Pasting properties were analyzed using a Brabender Amylograph (Brabender-803201, Micro Visco-Amyl-Graph, Germany), which was carried out according to Xu [23]. Each sample was suspended with deionized water (6% (w/w, dry basis) in an RVA aluminum can, and a heating and cooling program was used where the initial equilibrium temperature was 40 °C, heating from 40 to 95 °C, holding at 95 °C for 5 min, cooling from 95 to 50 °C and holding at 50 °C for 10 min with a heating/cooling rate of 7.5 °C/min while stirring at 250 rpm. The peak, breakdown, hot paste, cold paste, setback viscosities, peak time and pasting temperature, were recorded.

2.4. X-ray Diffraction Analysis

The crystalline structure of the sample powder (200 mesh) was investigated using a Bruker D8-Advance X-ray powder diffractometer (Bruker, Germany), according to the method of Yang [10]. The diffractograms were collected under the conditions of 40 kV, 30 mA, with a scanning angle (2 θ) set from 5 to 45°, with a 0.02° step interval, at a scanning rate of 1°/min and Cu K α radiation source (λ = 0.154 nm). The data were analyzed by Jade 5.0 software (Materials Data Inc., Livermore, CA, USA).

2.5. HS-SPME Sampling

An SPME extraction fiber (50/30 μ m) coated with divinylbenzene/carboxen/ polydimethylsiloxane (Supelco, Inc., Bellefonte, PA, USA) was used for headspace analyses of potato sample volatiles. This fiber is commonly used for flavor analysis and is especially useful for pyrazines [24,25]. The SPME fiber was aged in the GC inlet port at 270 °C for 1 h to ensure the removal of residual gas. Then, 2 g of potato flour sample was weighed and placed in a 20 mL glass sample vial to make way for the extraction of volatile components.

The sealed vial was placed in a 50 °C constant temperature water bath with thermal equilibrium for 10 min. The SPME extraction head was inserted into the headspace of the sample through the cap, and the fiber was exposed for 30 min. After the extraction, the SPME fiber head was removed from the headspace bottle, inserted into the GC inlet and was thermally desorbed for 5 min and transferred to the GC system.

2.6. Identification of Volatile Compounds Based on GC–MS Analysis

GC condition: Compounds were separated on a DB-Wax column (30 m \times 0.25 mm inside diameter, 0.25 µm film thickness, Agilent Technologies). The injection was performed in the splitless mode, and the injector temperature was 250 °C. Helium (99.999%) was used as the carrier gas with a constant flow rate starting at 1.0 mL/min. The oven temperature was programmed as follows: 40 °C for 1 min, 5 °C/min to 100 °C, 3 °C/min to 130 °C, 10 °C/min to a final temperature of 220 °C, with a final holding time of 3 min.

MS condition: The detector adopted an electron impact ion source with the ionizing potential of 70 eV set at 230 $^{\circ}$ C. The quadrupole temperature was set to 150 $^{\circ}$ C, and the

transfer line temperature was kept at 250 $^\circ \rm C.$ Total ion chromatograms were acquired by scanning from 30 to 450 u.

The GC-MS experimental data were processed by Jade 6.0 software (Materials Data Inc., Livermore, CA, USA). Volatile components of the potato flour samples were identified by comparison of the mass spectra in the commercial computer library as NIST (107k compounds) and Wiley (320k compounds, version 6.0), and only volatiles with matching degrees of more than 800 was recorded.

2.7. Statistical Analysis

The experiments were performed in triplicate, and the values are represented as mean \pm standard deviation, using one-way ANOVA in SPSS 21.0 (SPSS Inc., Chicago, IL, USA), and comparisons were made using Duncan's multiple-range test at a significant level of p < 0.05. A clustered heat map of the volatile compounds obtained following GC-MS analysis was created using the Heat Map Dendrogram App in OriginLab 2021(Northampton, MA, USA).

3. Results and Discussion

3.1. The Effect of Drying Temperature on Gelatinization Degree

Figure 1 shows the DG (%) of potato flour processed at different drying temperatures. In general, the DG (%) of raw potato was around 15%, and the pasting degree of cooked powder was almost 95% or higher. It could be said, as shown in Figure 1, that the potato flour has not yet gelatinized (a value of 15.79-22.10%) when the temperature was below 50 °C, and a slight gelatinization began to occur at 60 °C, but at 70 °C, almost half of the starch in the potato flour has been gelatinized (the value is 48.72%). The results were consistent with the DG (%) of low gelatinization potato four (hot air drying at 65° C) by Zhang [26]. The DG (%) was already very high when the drying temperature reached 80 and 90 °C, approaching full gelatinization (the value is 86.51-90.57%). Different hot air temperatures change the temperature, moisture and particle structure of potato starch, resulting in different DGs of the product starch. According to the observation of the microstructure of starch granules [6], it was found that a high temperature would destroy the structure of starch granules, which may be due to serious gelation.



Figure 1. Degree of gelatinization at different drying temperatures.

3.2. XRD Analysis

The various temperature treatments applied in this study exerted a significant impact on the functional properties of the starch in the potato flour. Figure 2 shows the X-ray diffraction patterns of the diverse potato starches. The X-ray diffraction patterns revealed the typical B-type diffractions characteristic of potato starch with a doublet peak at 15° and a very strong reflection at $2\theta = 17$ and 22° [27]. There were also some minor reflections at $2\theta = 20$, 24 and 35° .



Figure 2. Structural characteristics of the potato flour. X-ray diffractograms showing the effects of drying conditioning of samples in X-ray diffraction.

The intensity of peaks for treated samples HAD-50, HAD-60, HAD-70 and HAD-80 were on par or very similar, implying that the molecular arrangement of the starch under these treatment conditions was not adversely affected, the crystalline shape of starch was not altered and the diffraction intensity of HAD-50 was the highest. On the contrary, after high-temperature dehydration processing, the crystal diffraction peak of HAD-90 completely disappeared, and the diffraction curve showed a typical amorphous structure diffraction curve, indicating that the crystalline structure of starch granules had been destroyed by heating and shear force.

3.3. Pasting Properties

Starch's pasting behavior is a mixture of complex processes that occur after gelatinization, including starch granules transitioning from swelling to rupturing, amylose leaching and high-energy gel development [28,29]. Figure 3 presents the pasting properties of potato starch under different temperature regimes. As shown in Figure 3, the peak viscosity and valley viscosity of HAD-50 and HAD-60 were higher, whereas those for HAD-90 and HAD-80 were lower, which may be attributed to the following potential explanations. The HAD-90 samples may have undergone varying degrees of heat treatment during the production process, and some of the starch has undergone gelatinization and aging, causing the starch crystals to be partially damaged; the aged starch after gelatinization cannot be re-gelatinized at high temperatures, which also results in a decrease in the starch gelation temperature [30]. Secondly, because low-temperature drying can significantly reduce the loss of heat-sensitive nutrients and prevent starch crystals from being destroyed, it is clear that HAD-50 and HAD-60 are difficult to gelatinize, while the gelatinization temperature of HAD-90 was low [31].

According to the characteristic value of the gelatinization characteristic curve, the breakdown viscosity of HAD-50 is large, which indicates that the viscosity stability is not good, and the setback viscosity of HAD-90 is large, which indicates that the gel ability formed after cooling is poor and easily subject to aging.



Figure 3. Pasting curves of potato flour dried at 50, 60, 70, 80 and 90 °C.

In addition, in potato flour, a certain proportion of protein on the surface of starch granules can also inhibit the swelling of starch granules, according to Bharti [32] and Regina [33], which can also effectively reduce the gelatinization degree of HAD-50 and HAD-60. However, with the increase in HAD temperature, protein bond breakage may occur, resulting in the rapid transition of starch particles from expansion to decomposition. This claim is based on the fact that proteins are denatured during cooking, making them inactive [34,35].

Researchers have recently reported on the impact of various processing techniques on the quality of whole potato powder. The findings demonstrate that low-temperature hot-air drying and freeze-drying improve various aspects of potato powder [26,36]. However, there is no further study on the effect of different hot-air drying temperatures, which is supplemented by the results of this study. According to our findings, potato flour produced by low-temperature HAD had better processing characteristics, including low levels of gelatinization, internal starch particles that were completely intact and good gel stability, which was consistent with the findings of Zhang [26] and Shen [37].

3.4. Identification of Volatile Compounds in Different Potato Samples

The volatile compounds in raw and processed potato samples were extracted at different temperatures by HS-SPME and then analyzed by GC-MS. A total of 52 compounds were tentatively identified using the NIST and Wiley MS Library Database (Table 1). These included 9 alcohols, 12 hydrocarbons, 15 aldehydes, 7 ketones, 2 furans and 7 additional compounds. Hexanal, a major constituent of processed potato (above 30% of the total area), was not found in the raw samples.

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No.	Compounds	Retention Time (min)	The Relative Peak Area (%)					
			HAD-0	HAD-50	HAD-60	HAD-70	HAD-80	HAD-90
	Alcohol							
1	Ethyl alcohol	10.16	ND	1.63 ± 0.10	2.48 ± 0.09	1.74 ± 0.06	3.98 ± 0.14	2.75 ± 0.11
2	1-Pentanol	20.52	ND	1.66 ± 0.12 1.02 ± 0.04	1.3 ± 0.07	2.17 ± 0.09	ND	1.09 ± 0.15 2.18 ± 0.07
3	1-Octen-5-01 1-Methoxy-2-	26.93	9.56 ± 0.11	1.92 ± 0.04	1.8 ± 0.09	2.21 ± 0.08	ND	2.18 ± 0.07
4	propanol	15.52	ND	ND	2.69 ± 0.10	ND	2.82 ± 0.12	2.48 ± 0.05
5	2-Ehyl-1-hexanol	28.37	ND	ND	1.16 ± 0.11	1.73 ± 0.06	1.71 ± 0.13	1.14 ± 0.05
6	1-Pentanol	15.9	6.3 ± 0.08	ND	ND	ND	ND	ND
7	Benzyl alcohol	25.71	2.58 ± 0.10	ND	ND	ND	ND	ND
8	3-Methyl-1-butanol	8.39	2.55 ± 0.07	ND	ND	ND	ND	ND
9	1-Penten-3-ol	14.08	2.32 ± 0.06	ND	ND	ND	ND	ND
10	Hexane	5 72	1.14 ± 0.07	ND	0.7 ± 0.04	1.12 ± 0.04	1.6 ± 0.03	ND
10	Methylene Chloride	9.88	ND	1.77 ± 0.07	1.75 ± 0.04	ND	1.0 ± 0.00 1.56 ± 0.09	0.96 ± 0.08
	Octamethyl-	11.00	0.51 + 0.05	1.0 + 0.12	100 ± 0100		1.00 ± 0.07	1.02 + 0.05
12	cyclotetrasiloxane	11.08	0.71 ± 0.05	1.69 ± 0.12	ND	0.97 ± 0.06	1.24 ± 0.07	1.02 ± 0.05
13	Decamethyl-	16.1	ND	0.83 ± 0.08	0.7 ± 0.03	1.78 ± 0.09	0.62 ± 0.03	0.85 ± 0.05
15	cyclopentasiloxane	10.1		0.05 ± 0.00	0.7 ± 0.05	1.70 ± 0.09	0.02 ± 0.05	0.05 ± 0.05
14	Dodecane	18.43	ND	1.89 ± 0.06	2.06 ± 0.05	2.39 ± 0.09	2.99 ± 0.10	3.02 ± 0.07
15	Tridecane	21.16	ND	ND		ND 175 0.11	2.7 ± 0.09	ND
10	Trichloromethane	11.88	ND	ND	1.8 ± 0.05	ND	2.03 ± 0.04 ND	1.00 ± 0.03 0.87 ± 0.04
18	3-Methyl-tridecane	22.64	ND	ND	ND	ND	1.38 ± 0.06	0.07 ± 0.01 ND
10	Hexamethyl-	7 71	NID	ND	NID	NID	1 61 1 0 05	ND
19	cyclotrisiloxane	7.71	ND	ND	ND	ND	1.01 ± 0.05	ND
20	cis-1-Ethyl-2-Methyl-	18.62	5.5 ± 0.03	ND	ND	ND	ND	ND
	cyclopentane							
21	hexadiene	19.08	1.03 ± 0.10	ND	ND	ND	ND	ND
22	Aldehydes	11 21	NID	2 28 1 0 08	170 0.08	174 0.08	1.05 0.00	1 22 L 0.0E
22	Heyanal	11.51	ND	2.30 ± 0.00 51 73 + 1 23	1.79 ± 0.06 43.99 ± 0.77	1.74 ± 0.06 37.7 ± 0.92	1.05 ± 0.09 31.26 ± 0.95	1.52 ± 0.05 28.63 + 1.16
24	(Z)-2-Heptenal	23.24	ND	1.1 ± 0.06	1.12 ± 0.26	1.78 ± 0.06	1.32 ± 0.08	2.08 ± 0.04
25	Nonanal	25.43	ND	2.58 ± 0.08	5.84 ± 0.05	6.58 ± 0.17	7.13 ± 0.19	7.32 ± 0.47
26	Decanal	28.27	ND	2.65 ± 0.06	3.53 ± 0.05	1.53 ± 0.07	5.26 ± 0.08	ND
27	Octanal	22.16	0.31 ± 0.04	ND	ND	ND	ND	2.56 ± 0.12
28	Heptanal	18.24	ND	2.18 ± 0.03	2.42 ± 0.04	2.38 ± 0.04	2.35 ± 0.10	ND
29	(E) 2 Octornal	29.07	ND 1 22 \pm 0.06	2.54 ± 0.08	2.38 ± 0.05	2.49 ± 0.08 1.67 \pm 0.10	2.7 ± 0.06	2.73 ± 0.05
31	2-Dodecenal	20.02	1.22 ± 0.00	ND	ND	1.07 ± 0.19 1.95 ± 0.07	ND	ND
22	4-Ethyl-	29.62	ND	ND	ND	1.50 ± 0.07	ND	
32	Benzaldehyde	33.62	ND	ND	ND	ND	ND	0.85 ± 0.05
33	2-Methy-l-Butanal	8.32	6.36 ± 0.10	ND	ND	ND	ND	ND
34	Benzeneacetaldehyde	22.77	4.65 ± 0.11	ND	ND	ND	ND	ND
35	3-Methy-I-Butanal	8.39	2.84 ± 0.57	ND	ND	ND	ND	ND
30	2-Metnyi-Propanai Ketone	6.48	2.7 ± 0.10	ND	ND	ND	ND	ND
37	Acetone	7.62	ND	1.24 ± 0.06	1.63 ± 0.06	2.41 ± 0.13	2.7 ± 0.11	5.84 ± 0.14
28	6-Methyl-5-Hepten-2-	23.01	ND	1.78 ± 0.08	2.89 ± 0.11	2.14 ± 0.06	3.22 ± 0.09	241 ± 0.03
50	one	25.01		1.70 ± 0.00	2.07 ± 0.11	2.14 ± 0.00	0.22 ± 0.07	2.10 ± 0.03
39	3-Octen-2-one	25.97	ND	4.62 ± 0.17	4.4 ± 0.12	3.82 ± 0.14	3.74 ± 0.17	3.18 ± 0.11
40	5,5-Octaolen-2-one (FF)-3 5-Octadion-2-	28.84	ND	7.04 ± 0.15	0.83 ± 0.10	5.71 ± 0.16	5.2 ± 0.10	4.75 ± 0.14
41	one	29.96	ND	4.12 ± 0.01	3.57 ± 0.15	2.71 ± 0.07	1.98 ± 0.10	1.73 ± 0.18
42	1-Penten-3-one	10.76	7.66 ± 0.10	ND	ND	ND	ND	ND
43	2,3-Octanedione	16.96	0.82 ± 0.06	ND	ND	ND	ND	ND
44	Furan 2-Ethy-l-Europ	10.08	ND	ND	ND	3.21 ± 0.09	3.7 ± 0.06	3.88 ± 0.11
44	2-Eury-1-Furan	18.96	ND	1.66 ± 0.60	3.31 ± 0.08	3.21 ± 0.09 8 29 ± 0.13	3.7 ± 0.00 3.2 ± 0.16	5.00 ± 0.11 6.0 ± 0.16
15	Additional volatiles	10.70		1.00 ± 0.00	0.01 ± 0.00	0.27 ± 0.10	0.2 ± 0.10	0.0 ± 0.10
46	Toluene	12.62	0.44 ± 0.09	ND	ND	ND	1.29 ± 0.02	1.42 ± 0.14
47	15-Crown-5	35.67	ND	ND	ND	ND	0.89 ± 0.07	ND
48	1-Methyl-	25.99	ND	ND	ND	ND	0.87 ± 0.06	0.87 ± 0.08
40	naphthalene Triothylamina	710	NID	NID	NID	ND	ND	1.02 - 0.04
49 50	A cetic acid	7.10 27.33					ND	1.95 ± 0.04 3.87 ± 0.10
51	1.3-dichloro-Benzene	19.43	1.14 ± 0.07	ND	ND	ND	ND	ND
50	2-Methyl-7-	24.07		ND	ND	ND	ND	ND
52	phenylindole	24.07	0.88 ± 0.03	ND	ND	ND	ND	ND

Table 1. Volatiles and their relative content (%) in raw and dehydrated potatoes at different drying temperatures.

Note: The relative peak area (%) of each compound was mean value \pm SD. Abbreviations: ND, not found.

Based on the GC-MS results in Table 1, a stacking histogram was created and displayed in Figure 4. As shown in Figure 4, the volatile flavor components of raw potato samples differed significantly from those of dehydrated potatoes, while in dehydrated potato samples, some substances tended to change regularly with the drying temperature course. The content of aldehydes gradually decreased with increasing processing temperature, while furans first rose and then decreased, with the highest content in the HAD-70 samples. Alcohols and ketones were not linearly correlated to the grades but were obviously rich in HAD-0, and hydrocarbons were rich in HAD-50 and HAD-60 samples.



Figure 4. Comparison of the types and contents of volatile substances in different processed samples.

3.4.1. Volatile Composition of Raw Potato Samples

In total, there were 20 volatile compounds identified in the raw potato samples prior to processing; they include: five alcohols, four hydrocarbons, six aldehydes, two ketones and three additional compounds. Alcohols have been considered the main odorants of raw potato aroma, higher concentrations of which were detected in varieties of potatoes such as Longshu 11, Atlantic and Shepody [38]. Among the alcoholic compounds identified, 1-octen-3-ol, which is a degradation product of hydroperoxide in linoleic acid, was predominant. 3-Methyl- 1-butanol was also present, which is common in plant materials resulting from enzymatic deamination and the decarboxylation of amino acids [39]. It was reported that most unsaturated aldehydes have a pleasant odor; for instance, 3-methyl-butyraldehyde has a pleasant fruit aroma [40], and 2-methyl-butyraldehyde has a sweet and fruity flavor [41]. Chloride is present in fresh potatoes at room temperature and reacts with starch to produce ethers [42]. Methoxyphenyl-oxime, which is a kind of nitrogen-containing compound with a musty taste and meaty flavor, was also detected in the raw potato.

3.4.2. Volatile Composition of Dehydrated Potato Samples

The results of this study, as presented in Table 1, revealed that the types and concentrations of volatile compounds in potato flour changed at varying processing temperatures. From Table 1, the samples treated at 80 and 90 °C had the highest concentrations of volatile compounds with three and five alcohols, nine and six hydrocarbons, seven aldehydes, five ketones, two furans and three and four additional non-identified compounds, respectively. The rest of the samples yielded 20, 23 and 25 kinds of main volatiles at the processing temperature of 50, 60 and 70 °C, respectively.

Aldehydes, alcohols and furans are key components of dehydrated potato aromas. For instance, hexanal is the basic product of linoleic acid oxidation [43], playing a major role in the formation of the characteristic flavors of potato flour. Among the aldehydes detected in the present work, hexanal recorded the highest relative contents at all levels of processing (Table 1). According to Pérez [44], Linoleic acid containing double bonds easily oxidizes in the air to produce peroxide and aldehydes. They also serve as a precursor to many other aldehydes and alcohols, including (E)-2-heptenal and nonanal. Regarding the characteristic aromas of the various aldehydes detected in this study, hexanal has a nutty

and roasted odor, and benzaldehyde has a roasted peanut or almond aroma and fruity flavor. Nonanal is known to have a strong aroma of sweet orange and can be similar to fried peanuts, decanal has a sweet floral aroma, while heptaldehyde has a strong smell of grease [45], and phenylacetaldehyde has a rich aroma of Oriental hairpin. 1-amyl alcohol and 2-ethyl-1-hexanol, known to originate from linoleic acid oxidation, have a mushroom aroma, while 2-undecanone is considered to be the main compound responsible for the fruity aroma.

3.4.3. The Effect of Drying Temperature on Volatile Compounds during Processing

The relative peak area matrix of the GC-MS results for volatile potato components at different temperatures as also analyzed by heat map clustering analysis, as shown in Figure 5. The results show that the distance between 50 and 60 °C is the closest, and the component similarity is higher, then the distance increases at 70, 80 and 90 °C. The difference between all dried materials and fresh materials was the most significant. This can correspond to the result of gelatinization of Figure 5, because at low temperatures, the gelatinization degree of potato is lower, and the flavor component is close, but after high-temperature processing, the potato is basically gelatinized and the flavor substances produced have changed greatly.



Figure 5. Heatmap of volatile matter in different processed samples.

High concentrations of 1-octen-3-ol in raw tubers, which decreased significantly in dehydrated potatoes from 9.55% to 1.72%, and 1-penten-3-one, 1-pentanol were found only in raw samples. This may be from sample tissue degradation as a result of cutting or because the compounds were generated through lipid oxidation and lipoxygenase-initiated reaction. Several new volatile components were generated as a result of Maillard reaction and lipid oxidation during dehydration processing.

This exhibited an obvious difference in the variety of aldehyde compounds between raw and processed samples. Hexanal was found only in the processed potato, of which the concentration reached 51.73% with a 50 °C drying process. The formation of hexanal, reported in previous potato studies, depends on the time and opportunity for lipoxygenase to be in contact with the substrate [18]. It has been observed that high lipoxygenase activity at a lower temperature (50 °C) with a longer dehydration period results in a relatively high aldehyde content. On the contrary, higher temperatures lead to the rapid dehydration of potato slices and also to a reduction in lipoxygenase activity and oxidation of linoleic acid, which reduces the concentration of aldehydes.

The total amount of lipid degradation products formed by different processing methods are quite different. Oruna-Concha reported that different cooking methods (boiling, conventional baking and microwave baking) resulted in a unique profile of flavor compounds and a relatively high concentration of lipid oxidation products in boiled tubers [18]. More opportunities were provided for the interaction of lipoxygenase with the substrate during the slicing and boiling, while gradual heating of the tuber provides more time for oxidizing reaction. The flavor compounds of boiled potatoes are mainly caused by lipid degradation and Maillard reaction and/or sugar degradation, while those in roasted potatoes are formed by thermal degradation. Another oxidative product contributing to the flavor of the boiled potato is c4-heptanal, which produces a soil aroma at low concentrations [46], while high concentrations cause a stale flavor of potato tubers [47].

The concentration of aromatic compounds and furan in processed potatoes was higher. Benzaldehyde is an aromatic aldehyde with a pleasant aroma resulting from the enzyme breakdown of the diglucoside amygdalin [48]. Furans are formed during heating by Maillard sugar-amine reactions and thermal degradation of sugars, such as fructose and glucose [49]. The furans detected in the present work were 2-ethy-l-furan and 2-Penty-l-Furan. 2-ethy-l-furan was formed in potatoes processed at a higher temperature (above 70 °C). They have a very strong meat flavor and a low aroma threshold; in almost all food, 2-pentylfuran has a ham flavor, making a special contribution to the flavor of cooked potato [50].

According to Figure 6, new volatile compounds produced formed via several chemical reactions that occurred during drying, and the relative content changed according to the temperature. The concentration of hexanal dropped dramatically from 51.73% to 28.63%, 3,5-Octadien-2-one and E,E)-3,5-Octadien-2-one also decreased from 7.64% and 4.12% to 4.75% and 1.73%, respectively. Significant increases in Maillard reactions and thermal degradation as a result of lipid oxidation were noted for two volatiles: nonanal and 2-Penty-l-Furan. Some bad flavors were produced, while some of the aromas were lost. It has been reported that the temperature of the gelatinization of potato starch is approximately 57–69 °C [51]; hence, a processing temperature higher than 70 °C will result in starch gelatinization, which is unfavorable for the preparation of raw potato flour. It is, therefore, important to note that high temperatures reduce the processing properties of staple foods, such as water absorption, kneading properties, gluten-like strength, viscosity, amylase activity and regenerative properties. Therefore, 60 °C is a moderate HAD temperature, which is beneficial to the formation of comprehensive properties of potato powder.

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Figure 6. Changing trend of feature volatile component contents in different processed samples.

4. Conclusions

Potato is an important raw food material, but the limited storage and transportation of fresh potato lead to fewer types of processed products, while after drying and milling, potato can retain most of the nutrients, is easy to use, has stable storage and can provide special tastes and aromas. Compared with potato starch, potato flour has more comprehensive nutrition and superior processing performance and is an important raw material for staple potato food products. The variation in physicochemical properties, including viscosity, X-ray diffraction and volatile compositions, of HAD potato flour were observed using enzyme hydrolysis, rapid visco analyzer, X-ray diffractometer and headspace solid-phase microextraction coupled with gas chromatography-mass spectrometry (HS-SPME GC-MS). The results indicate that HAD temperature is an important index affecting the quality of potato powder. Higher temperatures lead to an increase in DG value, a decrease in peak viscosity, poor gel ability and aging, the destruction of the crystal structure and the loss of bad flavor and key flavors of potato powder. However, low-temperature HAD for a long time will also reduce the viscosity stability; HAD-60 has more comprehensive nutrition and flavor and better processability. The results provide more detailed data for the raw material processing technology of staple potato foods and can effectively guide the process optimization and quality classification of potato flour. However, there are other ways to process potato flour, such as infrared drying, microwave drying, etc. The influence of different drying methods needs to be further analyzed in future research.

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References

- Dreyer, H. Towards Sustainable Potato Production: Partnering to Support Family Farmers in Africa. *Potato Res.* 2017, 60, 237–238. [CrossRef]
- Zhang, H.; Fen, X.; Yu, W.; HU, H.; Dai, X. Progress of potato staple food research and industry development in China. J. Integr. Agric. 2017, 16, 2924–2932. [CrossRef]
- 3. Huang, G. China to grow and eat more potatoes. Front. Ecol. Environ. 2015, 13, 68. [CrossRef]
- 4. Liu, X.; Mu, T.; Sun, H.; Zhang, M.; Chen, J. Influence of potato flour on dough rheological properties and quality of steamed bread. *J. Integr. Agr.* 2016, *15*, 2666–2676. [CrossRef]
- 5. Zeng, F.; Liu, H.; Yu, H.; Cheng, J.; Gao, G.; Shang, Y. Effect of potato flour on the rheological properties of dough and the volatile aroma components of bread. *Am. J. Potato Res.* **2018**, *96*, 69–78. [CrossRef]
- 6. Raigond, P.; Singh, B.; Gupta, V.K.; Singh, B. Potato flavour: Profiling of umami 5'-nucleotides from indian potato cultivars. *Indian J. Plant Physiol.* **2014**, *19*, 338–344. [CrossRef]
- Bai, J.; Cai, J.; Tian, X. Crust Formation and Microstructural Changes of Gingko Biloba Seeds During Drying. *Food Bioprocess Technol.* 2019, 12, 1041–1051. [CrossRef]
- Wang, H.; Zhang, M.; Adhikari, B. Drying of shiitake mushroom by combining freeze-drying and mid-infrared radiation. *Food Bioprod. Process.* 2015, 94, 507–517. [CrossRef]
- 9. Krystyjan, M.; Gumul, D.; Areczuk, A.; Khachatryan, G. Comparison of physico-chemical parameters and rheological properties of starch isolated from coloured potatoes (*Solanum tuberosum* L.) and yellow potatoes. *Food Hydrocoll.* **2022**, 131, 107829. [CrossRef]
- 10. Yang, S.; Dhital, S.; Zhang, M.; Wang, J.; Chen, Z. Structural, gelatinization, and rheological properties of heat-moisture treated potato starch with added salt and its application in potato starch noodles. *Food Hydrocoll.* **2022**, *131*, 107802. [CrossRef]
- 11. Warren, F.J.; Gidley, M.J.; Flanagan, B.M. Infrared Spectroscopy as a Tool to Characterise Starch Ordered Structure—A Joint FTIR–ATR, NMR, XRD and DSC Study. *Carbohydr. Polym.* **2016**, *139*, 35–42. [CrossRef] [PubMed]
- 12. Zhao, Y.; Wang, X.; Liao, W.; Xu, D.; Liu, G. Study on nutritional quality and volatile aroma compounds of the stir-fried shredded potatoes. *Am. J. Potato Res.* **2022**, *99*, 191–205. [CrossRef]
- 13. Jansky, S.H. Potato flavor. Am. J. Potato Res. 2010, 87, 209-217. [CrossRef]
- 14. Diez-Simon, C.; Mumm, R.; Hall, R.D. Mass spectrometry-based metabolomics of volatiles as a new tool for understanding aroma and flavour chemistry in processed food products. *Metabolomics* **2019**, *15*, 41. [CrossRef] [PubMed]
- 15. Oruna-Concha, M.; Duckham, S.; Ames, J.M. Comparison of volatile compounds isolated from the skin and flesh of four potato cultivars after baking. *J. Agric. Food Chem.* **2001**, *49*, 2414–2421. [CrossRef] [PubMed]
- 16. Engel, W.; Bahr, W.; Schieberle, P. Solvent assisted flavour evaporation—A new and versatile technique for the careful and direct isolation of aroma compounds from complex food matrices. *Eur. Food Res. Technol.* **1999**, 209, 237–241. [CrossRef]
- 17. Oruna-Concha, M.; Bakker, J.; Ames, J.M. Comparison of the volatile components of two cultivars of potato cooked by boiling, conventional baking and microwave baking. *J. Sci. Food Agr.* **2002**, *82*, 1080–1087. [CrossRef]
- 18. Xu, D.; Chen, C.; Zhou, F.; Liu, C.; Tian, M.; Zeng, X.; Jiang, A. Vacuum packaging and ascorbic acid synergistically maintain the quality and flavor of fresh-cut potatoes. *LWT-Food Sci. Technol.* **2022**, *163*, 113356. [CrossRef]
- Hou, F.; Mu, T.; Ma, M.; Blecker, C. Optimization of processing technology using response surface methodology and physicochemical properties of roasted sweet potato. *Food Chem.* 2019, 278, 136–143. [CrossRef]
- Majcher, M.; Jelen, H.H. Comparison of suitability of SPME, SAFE and SDE methods for isolation of flavor compounds from extruded potato snacks. J. Food Compos. Anal. 2009, 22, 606–612. [CrossRef]
- Sarker, M.Z.I.; Yamauchi, H.; Kim, S.J.; Matsumura-Endo, C.; Takigawa, S.; Hashimoto, N. A farinograph study on dough characteristics of mixtures of wheat flour and potato starches from different cultivars. *Food Sci. Technol. Res.* 2008, 14, 211–216. [CrossRef]
- 22. Zhang, W.; Shan, C.; Jiang, H.; Liu, Y.; Zhang, J. Enzymatic Hydrolysis Treatment for Determination of Polysacchrides in Chinese Yam. *Food Sci.* 2009, 20, 385–387, (In Chinese with English abstract).
- 23. Xu, B.; Zhou, S.L.; Miao, W.J.; Dong, Y. Microstructure and pasting characteristics of wheat germ treated by microwave radiation. *Trans. Chin. Soc. Agric. Mach.* **2012**, *43*, 151–157, (In Chinese with English abstract).
- 24. Bail, S.; Stuebiger, G.; Unterweger, H.; Buchbauer, G.; Krist, S. Characterization of volatile compounds and triacylglycerol profiles of nut oils using SPME-GC-MS and MALDI-TOF-MS. *Eur. J. Lipid Sci. Technol.* **2009**, *111*, 170–182. [CrossRef]

- Beltran, A.; Ramos, M.; Grane, N.; Martin, M.L.; Garrigos, M.C. Monitoring the oxidation of almond oils by HS-SPME-GC-MS and ATR-FTIR: Application of volatile compounds determination to cultivar authenticity. *Food Chem.* 2011, 126, 603–609. [CrossRef]
- Zhang, K.; Tian, Y.; Liu, C.; Xue, W. Effects of temperature and shear on the structural, thermal and pasting properties of different potato flour. *BMC Chem.* 2020, 14, 20. [CrossRef]
- 27. Hu, H.; Li, S.; Pan, D.; Wang, K.; Qiu, M.; Qiu, Z.; Liu, X.; Zhang, J. The Variation of Rice Quality and Relevant Starch Structure during Long-Term Storage. *Agriculture* **2022**, *12*, 1211. [CrossRef]
- 28. Schirmer, M.; Jekle, M.; Becker, T. Starch gelatinization and its complexity for analysis. Starch-Stärke 2015, 67, 30–41. [CrossRef]
- 29. Zhu, L.; Zhang, Y.; Wu, G.; Qi, X.; Dag, D.; Kong, F.; Zhang, H. Characteristics of pasting properties and morphology changes of rice starch and flour under different heating modes. *Int. J. Biol. Macromol.* **2020**, *149*, 246–255. [CrossRef]
- 30. Ahmed, M.; Akter, M.S.; Lee, J.C.; Eun, J.B. Encapsulation by spray drying of bioactive components, physicochemical and morphological properties from purple sweet potato. *LWT-Food Sci. Technol.* **2010**, *43*, 1307–1312. [CrossRef]
- 31. Tian, J.; Zhang, Y. Progress on the effects of pretreatment and drying on quality of potato flour. *Sci. Technol. Food Ind.* **2018**, *39*, 347–351, (In Chinese with English abstract).
- Bharti, I.; Singh, S.; Saxena, D.C. Exploring the influence of heat moisture treatment on physicochemical, pasting, structural and morphological properties of mango kernel starches from Indian cultivars. *LWT-Food Sci. Technol.* 2019, 110, 197–206. [CrossRef]
- 33. Regina, A.; Kosar, H.B.; Ling, S.; Li, Z.; Rahman, S.; Morell, M. Control of starch branching in barley defined through differential RNAi suppression of starch branching enzyme IIa and IIb. *J. Exp. Bot.* **2010**, *61*, 1469–1482. [CrossRef] [PubMed]
- Corzo-Ríos, L.J.; Sánchez-Chino, X.M.; Cardador-Martínez, A.; Martínez-Herrera, J.; Jiménez-Martínez, C. Effect of cooking on nutritional and non-nutritional compounds in two species of Phaseolus (*P. vulgaris* and *P. coccineus*) cultivated in Mexico. *Int. J. Gastron. Food Sci.* 2020, 20, 100206. [CrossRef]
- 35. Divekar, M.T.; Karunakaran, C.; Lahlali, R.; Kumar, S.; Chelladurai, V.; Liu, X.; Borondics, F.; Shanmugasundaram, S.; Jayas, D.S. Effect of microwave treatment on the cooking and macronutrient qualities of pulses. *Int. J. Food Prop.* **2017**, *20*, 409–422. [CrossRef]
- Yadav, A.R.; Guha, M.; Tharanathan, R.N.; Ramteke, R.S. Changes in characteristics of sweet potato flour prepared by different drying techniques. *LWT-Food Sci. Technol.* 2006, 29, 20–26. [CrossRef]
- Shen, C.; Wang, L.; Wang, R.; Luo, X.; Li, Y.; Chen, Z. Influence of drying techniques on the physicochemical properties of potato flours. *Food Ferment Ind.* 2016, 42, 117–121. [CrossRef]
- 38. Wu, Y.; Zhou, J.; Ming, T.; Tang, S.; Bu, H.; Chen, Y.; Jiang, J.; Tian, W.; Su, X. Analysis of volatile components of potato from different habitats by electronic nose and GC-MS. *Food Sci.* **2016**, *37*, 130–136, (In Chinese with English abstract).
- Tieman, D.; Taylor, M.; Schauer, N.; Fernie, A.R.; Hanson, A.D.; Klee, H.J. Tomato aromatic amino acid decarboxylases participate in synthesis of the flavor volatiles 2-phenylethanol and 2-phenylacetaldehyde. *Proc. National. Acad. Sci. USA* 2006, 103, 8287–8292. [CrossRef]
- 40. Akyol, H.; Riciputi, Y.; Capanoglu, E.; Caboni, M.F.; Verardo, V. Phenolic Compounds in the Potato and Its Byproducts: An Overview. *Int. J. Mol. Sci.* **2016**, *17*, 835. [CrossRef]
- Tang, Q.; Liu, X.; Chi, J.; Chen, Z.; Li, S.; Yang, C. Effects of different drying methods on quality and volatile components of *Pleurotus eryngii*. *Food Sci.* 2016, 37, 25–30, (In Chinese with English abstract).
- 42. Xu, L.; Yang, L.; Zhang, B. A Study on Starch Etherification with 2-Chloroethanol. *Mat. Rev.* 2007, 21, 152–154. (In Chinese with English abstract)
- 43. Kotsiou, K.; Tasioula-Margari, M. Changes occurring in the volatile composition of Greek virgin olive oils during storage: Oil variety influences stability. *Eur. J. Lipid Sci. Technol.* **2015**, *17*, 514–522. [CrossRef]
- 44. Li, K.; Yin, Y.; Wang, Q.; Lin, T.; Guo, H. Correlation analysis of volatile flavor components and metabolites among potato varieties. *Sci. Agric. Sin.* **2021**, *54*, 792–803, (In Chinese with English abstract).
- 45. Iglesias, J.; Medina, I.; Bianchi, F.; Careri, M.; Mangia, A.; Musci, M. Study of the volatile compounds useful for the characterisation of fresh and frozen-thawed cultured gilthead sea bream fish by solid-phase microextraction gas chromatography-mass spectrometry. *Food Chem.* **2009**, *115*, 1473–1478. [CrossRef]
- Josephson, D.B.; Lindsay, R.C. C4-heptenal: An influential volatile compound in boiled potato flavor. J. Food Sci. 1987, 52, 328–331. [CrossRef]
- 47. Pérez, A.G.; Sanz, C.; Olías, R.; Olías, J.M. Lipoxygenase and hydroperoxide lyase activities in ripening strawberry fruits. J. Agr. Food Chem. 1999, 47, 249–253. [CrossRef]
- Sanchez-Perez, R.; Jorgensen, K.; Olsen, C.E.; Dicenta, F.; Moller, B.L. Bitterness in almonds. *Plant Physiol.* 2008, 146, 1040–1052. [CrossRef]
- 49. Vazquez-Araujo, L.; Enguix, L.; Verdu, A.; Garcia-Garcia, E.; Carbonell-Barrachina, A.A. Investigation of aromatic compounds in toasted almonds used for the manufacture of turron. *Eur. Food Res. Technol.* **2008**, 227, 243–254. [CrossRef]
- 50. Qiao, L.; Wang, H.; Shao, J.; Lu, L.; Tian, J.; Liu, X. A novel mitigator of enzymatic browning-hawthorn leaf extract and its application in the preservation of fresh-cut potatoes. *Food Qual. Saf.* **2021**, *5*, fyab015. [CrossRef]
- 51. Xu, Z.; Xu, Q.; Wang, S.; Wang, Z.; Zhao, D. Comparison of microstructure and thermodynamic properties of starch from different varieties of potato. *Sci. Technol. Food Ind.* **2014**, *38*, 132–136. (In Chinese with English abstract)