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Abstract

This study investigated changes in rice starch viscosity under varying irradiation doses, enabling ready discrimination between irradiated and non-irradiated rice through pH control. The results demonstrated that starch viscosity decreased significantly with increasing absorbed irradiation dose. At pH 2.5, the viscosity of irradiated starch was significantly higher than that of non-irradiated starch, exhibiting significant differences between the two groups. The absorbed dose of rice can be estimated from the relationship among peak viscosity, hot pasting viscosity, and absorbed dose.

Full Text

Preamble

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Study on the Rapid Detection of Irradiated Rice Based on RVA

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Abstract

This paper investigates the changes in rice starch viscosity following irradiation at different doses and demonstrates that irradiated rice can be readily distinguished from non-irradiated rice through pH control. The results show that starch viscosity decreases significantly with increasing irradiation dose. At pH 2.5, the viscosity of irradiated starch is apparently higher than that of non-irradiated samples, showing significant differences between the two groups. The

absorbed dose of irradiated rice can be estimated using the relationship among peak viscosity, hot pasting viscosity, and absorbed dose.

Key words: Starch, Irradiation, RVA diagram, Detection

Introduction

Food irradiation processing, involving the controlled application of energy from ionizing radiations such as gamma rays, X-rays, and electron beams, has recently become one of the most convenient and efficient methods for food preservation. Based on the physical, chemical, biological, and microbiological changes that occur in food products during irradiation, these minimal changes can be detected to determine whether food has been irradiated. In 1996, the European Committee for Standardization (CEN) adopted five European standards for detecting irradiation in food commodities (EN-1784 to EN-1788), and in 2004, five additional validated standard methods were introduced: EN-13783, EN-1384, EN-14596, EN-13708, and EN-13751.

Rice is one of the major cereals, and its main component is starch consisting of amylose and amylopectin. Rice storage methods are closely related to food safety and public health. The purpose of rice preservation techniques is to protect grains from insect infestation and microbial contamination during storage, processing, market circulation, and consumption. Gamma irradiation is an economical and effective method for rice preservation and disinfestation due to its high penetrating power and thorough insecticidal effect. It is capable of killing worm eggs inside rice grains without leaving any odor or residual toxicity after irradiation, thus attracting increasing attention.

Currently, detection methods for irradiated food are being studied in China, but no uniform standard has been established yet. Additionally, there is no single technique that can be applied to all food detection scenarios. Only a few reports focus on distinguishing irradiated food or estimating the irradiation dose. In this paper, we selected starch from round-shaped rice as our research target and investigated viscosity changes at different dosages after irradiation. This method does not require reference samples.

2.1 Sample Source

All studied samples were purchased from a local supermarket and consisted of round-shaped rice (specific brand names not detailed). The samples were sealed in polyethylene (PE) bags, with nine bags prepared, each containing 50 g.

3 Results and Analysis

The starch samples were exposed to a ^{60}Co source at the Irradiation Center of Zhejiang University, Hangzhou, China, at ambient temperature. Nine irradiation doses were applied: 0, 0.1, 0.3, 0.5, 0.9, 1, 3, 5, and 7 kGy, with a dose rate of 0.8 kGy/h.

2.3 Analysis of Starch Viscosity

The samples were ground into powders and passed through a 100-mesh sieve. Starch viscosity was determined in duplicate using a Rapid Visco Analyser (RVA, Model-3D, Newport Scientific Inc., Australia) controlled by computer software, Thermo Cycle for Windows (TCW), following the American Association of Cereal Chemists (AACC) method from 1998. For samples with 14% moisture content, 3.0 g of starch was mixed with 25 mL of distilled water. The temperature profile inside the canister during stirring was as follows: maintained at 50°C for 1 min, then raised to 95°C at a rate of 12°C/min, held at 95°C for 2.5 min, then cooled and maintained at 50°C for 1.4 min. The paddle rotated at 960 rpm for the first 10 seconds, then at 160 rpm for the remainder of the test. Viscosity values were expressed in Rapid Visco Units (RVU), with the following parameters used as characteristic values of the RVA profile: peak viscosity (PV), hot viscosity (HV), cool viscosity (CV), breakdown (PV minus HV), and setback (CV minus PV).

2.4 Detection Method

Two parallel samples were selected from the starch samples without gelatinization. One was mixed with an acid solution at pH 2.5, while the other was mixed with distilled water as a blank control. Both samples were stirred uniformly under identical conditions, and their viscosity was measured using the Rapid Visco Analyser while monitoring the dynamic changes in the RVA profile. Samples were determined to be irradiated if the RVA profile at pH 2.5 was higher than that of the samples with distilled water. Conversely, samples were determined to be non-irradiated if the RVA profile at pH 2.5 was lower than that of the samples with distilled water.

3.1 Irradiation Effect on Starch Viscosity at Different Doses

When starch was irradiated at different doses, the peak viscosity (PV), hot viscosity (HV), and cool viscosity (CV) decreased considerably with increasing dose, as shown in [Figure 1: see original paper], though no obvious regularity was observed. The parameters of starch viscosity decline can serve as a reference for determining whether samples have been irradiated. Some reports suggest that starch degradation reactions resulting in decreased molecular weight are responsible for the decline in starch viscosity when amylose and amylopectin are irradiated. Others hold the view that high-dose irradiation causes changes in physicochemical properties, such as decreased optical rotation and alterations in optical properties and granule structure, which can lead to cleavage of polysaccharide chains and generation of degradable dextrin fragments, representing the key reason for the decrease in starch content.

3.2 pH Effect on the RVA Profile

According to the method described in section 2.4, samples were divided equally into two portions: one mixed with distilled water (pH 7) and the other with acid solution (pH 2.5). The RVA profile was obtained using the Rapid Visco Analyser. For non-irradiated rice, as shown in [Figure 1: see original paper], starch viscosity was higher at pH 2.5 than at pH 7. However, for irradiated samples, starch viscosity was lower at pH 7 than at pH 2.5, which contrasts with the behavior of non-irradiated counterparts. The higher the irradiation dose, the lower the viscosity observed. These results can be explained by the fact that hydrogen ions can coalesce small polysaccharide chains cleaved by irradiation into larger molecules, resulting in increased molecular weight that compensates for the viscosity decrease. Therefore, by controlling pH to markedly alter RVA performance, we can quickly evaluate whether starch from round-shaped rice has been exposed to irradiation treatment.

3.3 Relationship Between Viscosity and Irradiation Dosage

Based on the data displayed in , the relationship between peak viscosity (PKV), hot paste viscosity (HPV), cool paste viscosity (CPV), and irradiation dosage shows that the logarithm of viscosity (Y) exhibits exponential correlation with absorbed dosage (D) after exponential fitting, with viscosity as the ordinate and absorbed dosage as the abscissa. The absorbed dosage can thus be calculated. As shown in , the theoretically calculated value deviates significantly from the actual irradiation dose when the irradiation range is 0–0.1 kGy. Negative values represent two cases: either the sample is non-irradiated, or the estimated absorbed dosage is less than the actual absorbed dosage. In practice, we should first determine whether samples are irradiated: if the RVA value is higher under acidic conditions than under neutral conditions, the samples have been exposed to irradiation treatment. Conversely, if the RVA profile is higher under neutral conditions than under acidic conditions, the samples are not irradiated.

Table 1. Relationship Between Viscosity and Irradiation Dose

Dose (kGy)	Peak Viscosity	Hot Viscosity	Cool Viscosity	Breakdown	Setback	Pasting Time
0	8.25	8.42	10.42	5.92		
0.1	2.03	1.87	2.05	0.32		
0.3	$2.04e^{-0 \cdot 032}$	$1.87e^{-0 \cdot 064}$	$2.05e^{-0 \cdot 046}$			
R^2	0.9062	0.9703	0.9212			

Table 2. Relationship Between Viscosity and Irradiation Dosage

D (kGy)	Lg(RVU/kGy)	Destimate (kGy)	Relative Error (%)	Lg(RVU/kGy)	Destimate (kGy)	Relative Error (%)	Lg(RVU/kGy)	Destimate (kGy)	Relative Error (%)
0.1	-0.04	-3.28	-4.85	-6.70					
1	-0.13	-4.89	-6.84						
3	-0.22	-4.99	-6.65						

4 Discussion

4.1 Mechanism for Starch Viscosity Decrease Caused by Irradiation

The starch in round-shaped rice consists of amylose and amylopectin. Amylose, commonly called linear starch, is composed of D-anhydroglucose units linked by α-1,4 glycosidic bonds. Amylopectin is composed of D-anhydroglucose units connected by α-1,6 glycosidic bonds and α-1,4 glycosidic bonds, with branches positioned at the α-1,6 glycosidic bonds. Experimental analysis shows that rice viscosity decreases with increasing irradiation dose. Gamma irradiation degrades starch through cleavage of glycosidic linkages and peptide bonds into smaller fragments, such as short-chain aldehyde, ketone, and acid peptide derivatives, as well as monosaccharides (glucose, maltose, dextrin), amino acids, and small molecular weight fragments, resulting in decreased starch viscosity.

Water in the samples can generate free radicals and hydrated electron free radicals, which react with groups in glycosidic bonds and peptides as well as radicals generated from proteins after irradiation. The fact that starch can be further cleaved through a chain reaction may accelerate the viscosity decrease. H radicals from water can also react with groups on aldehydes, ketones, and acids, leading to polymerization of short chains that can compensate for starch viscosity decrease to some degree, as shown in [Figure 1: see original paper].

4.2 Error Analysis for Estimated Irradiation Dosage

Analysis of the data in reveals that the theoretically calculated value deviates significantly from the actual irradiation dose when the irradiation range is 0.1–1 kGy, but the error is relatively small (0.2–9.33%) when the irradiation range is 3–7 kGy. The relative error between the theoretically calculated value and actual irradiation dose is within 2.5% at 7 kGy. The main sources of error include: (1) irradiation-related errors, as the experimentally designed dose may not match the actual dose received by the sample due to sample volume versus point measurement; (2) theoretical calculation based on the formula D = $(A\times\Gamma\times T)/R^2$ assumes dose concentration at a single point in air, creating a 0.873$\times10^{-3}$ Gy difference; (3) inaccuracies in the actual distance between samples and the irradiation source compared to calculated distance; (4) failure

to correct the radioactivity of the irradiation source immediately during sample irradiation and ensure samples are at the same horizontal level as the source; (5) sampling time differences for detection, as irradiation dose varies if sampling occurs earlier or later by one hour; and (6) systematic errors from viscosity measurement.

All these factors contribute to discrepancies between theory and practice. Based on experimental data analysis, lower irradiation doses produce larger errors, while higher doses produce smaller errors. It is recommended that irradiation dose should not be less than 1 kGy when using viscosity for dose verification, as results become more convincing above this threshold. A chemical dosimeter should be placed in the packaging bag to track the dose during sample irradiation, ensuring accurate measurement of the actual absorbed dose.

In actual irradiation operations, the intensity of the ^{60}Co source, dose heterogeneity of the radiation field, dose rate value, heterogeneity of irradiation treatment toward samples, uncertainty in sample selection, environmental factors, and systematic errors can all affect irradiation performance and detection results. This research provides a method for estimating irradiated dose that can serve as a reference for related studies.

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Note: Figure translations are in progress. See original paper for figures.