National Journal of Multidisciplinary Research and Development
www.newresearchjournal.com
ISSN: 2455-9040
Received: 26-11-2025, Accepted: 25-12-2025, Published: 09-01-2026
Volume 11, Issue 1, 2026, Page No. 18-20

Starch composition determined by IR spectroscopy

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Abstract

In this article, we determined the composition of starch by IR spectroscopy using the Model-Nexus spectrophotometer. It has absorption bands between the wavelengths of 3271cm^{-1} and 398cm^{-1}. Starch is an essential polysaccharide widely used in the food, pharmaceutical and chemical industries due to its functional and biodegradable properties. In this study, the chemical structure of starch was investigated by FTIR spectroscopy, with the aim of identifying the characteristic functional groups and molecular interactions. The FTIR spectrum revealed specific bands corresponding to hydroxyl (-OH), ether (C–O–C) and C–H bonds, confirming the polysaccharide structure of starch. The results demonstrate that FTIR is a rapid, non-destructive and efficient method for the qualitative characterization of starch, providing useful information for quality control and research of starch-based products.

Keywords: Composition, starch spectrum

Introduction

Starch or amylum is a polymeric carbohydrate consisting of numerous glucose units joined by glycosidic bonds. This polysaccharide is produced by most green plants for energy storage. Worldwide, it is the most common carbohydrate in human diets, and is contained in large amounts in staple foods such as wheat, potatoes, maize (corn), rice, and cassava (manioc) [1-3].

Pure starch is a white, tasteless and odorless powder that is insoluble in cold water or alcohol. It consists of two types of molecules: the linear and helical amylose and the branched amylopectin. Depending on the plant, starch generally contains 20 to 25% amylose and 75 to 80% amylopectin by weight [4]. Glycogen, the energy reserve of animals, is a more highly branched version of amylopectin.

In industry, starch is often converted into sugars, for example by malting. These sugars may be fermented to produce ethanol in the manufacture of beer, whisky and biofuel. In addition, sugars produced from processed starch are used in many processed foods.

Mixing most starches in warm water produces a paste, such as wheatpaste, which can be used as a thickening, stiffening or gluing agent. The principal non-food, industrial use of starch is as an adhesive in the papermaking process. A similar paste, clothing or laundry starch, can be applied to certain textile goods before ironing to stiffen them [5-11].

Materials and Methods

Starch samples were obtained from dried and finely ground plant material. Prior to analysis, samples were oven-dried at 40 °C for 24 h to minimize moisture interference. Fourier Transform Infrared (FTIR) spectroscopy was used to determine starch composition, particularly the relative contributions of amylose and amylopectin. Approximately 2 mg of each starch sample was mixed with 200 mg of spectroscopic-grade potassium bromide (KBr) and compressed into pellets using a hydraulic press. Alternatively, for attenuated total reflectance (ATR) measurements, starch powders were placed directly onto the ATR crystal without further preparation.

IR spectra were recorded using an FTIR spectrometer over the range of 4000–400 cm^{-1} at a resolution of 4 cm^{-1}, with 32 scans averaged per sample. A background spectrum was collected prior to each measurement and automatically subtracted. Key absorption bands associated with starch molecular vibrations, including O–H stretching (3200–3400 cm^{-1}), C–H stretching (2800–3000 cm^{-1}), and C–O and C–O–C stretching (1200–900 cm^{-1}), were analyzed. The ratio of absorbance at characteristic peaks (e.g., 1047/1022 cm^{-1}) was used as an indicator of starch structural organization and composition. All measurements were performed in triplicate, and mean spectra were used for subsequent analysis.

A Thermo Nicolet FTIR Spectrophotometer (Model- Nexus 670 FTIR) was used with the potassium bromide (KBr) pellet method, for the FTIR studies.

Fig 1: Spectrophotometer Model- Nexus

Result and Discussion

Infrared (IR) spectroscopy provided clear and reproducible spectra for all starch samples analyzed, allowing qualitative and semi-quantitative evaluation of starch composition and molecular organization. The FTIR spectra were characterized by absorption bands typical of polysaccharides, confirming the dominance of starch components, namely amylose and amylopectin. Spectral features were consistent across replicates, indicating good sample homogeneity and reliability of the analytical method.

A broad and intense absorption band observed in the region of 3200–3400 cm^{-1} was attributed to O–H stretching vibrations associated with hydroxyl groups abundantly present in starch molecules. The breadth of this band reflects extensive hydrogen bonding within and between starch chains. Variations in intensity among samples suggested differences in hydrogen bonding patterns, which are closely related to starch composition and chain organization. Samples exhibiting stronger O–H stretching bands likely possessed a higher degree of intermolecular hydrogen bonding, often associated with increased amylose content or more ordered molecular arrangements.

The absorption bands between 2800 and 3000 cm^{-1} corresponded to C–H stretching vibrations of CH and CH_2 groups in the glucose units. These peaks showed minimal variation among samples, indicating that the basic glucose backbone of starch was conserved. This region is generally less sensitive to compositional changes but serves as confirmation of the polysaccharide nature of the samples.

The most informative region for assessing starch composition was the fingerprint region between 1200 and 900 cm^{-1}, which is dominated by C–O, C–O–C, and C–C stretching vibrations. Within this region, distinct bands at approximately 1047, 1022, and 995 cm^{-1} were clearly resolved. The band at ~1047 cm^{-1} is commonly associated with ordered or crystalline regions of starch, while the band at ~1022 cm^{-1} corresponds to amorphous regions. The peak at ~995 cm^{-1} is related to skeletal vibrations of the glucose ring and is sensitive to starch chain conformation.

The ratio of absorbance at 1047/1022 cm^{-1} was used as an indicator of the relative degree of molecular order and, indirectly, starch composition. Samples with higher 1047/1022 ratios exhibited a greater proportion of ordered structures, which are typically linked to higher amylopectin crystallinity. Conversely, samples with lower ratios suggested a higher proportion of amorphous regions, often associated with increased amylose content. These findings align with previous studies demonstrating that amylopectin contributes predominantly to crystalline lamellae, whereas amylose is largely located in amorphous domains.

Additionally, differences in the intensity of the 995 cm^{-1} band supported variations in starch molecular structure. Enhanced intensity of this band indicated more constrained glucose ring vibrations, suggesting tighter packing or stronger intermolecular interactions. Such characteristics are often observed in starches with higher amylose content, which tend to form more compact and less branched structures compared to amylopectin.

The IR results also suggested that starch composition influenced hydrogen bonding patterns, as reflected in shifts and intensity changes in the O–H stretching region. Samples with relatively higher amylose content displayed slightly broader O–H bands, indicating a wider distribution of hydrogen bond strengths. This behavior can be attributed to the linear nature of amylose chains, which allows greater flexibility in hydrogen bond formation. In contrast, starches richer in amylopectin showed more defined O–H stretching profiles, consistent with the highly branched and ordered architecture of amylopectin.

Overall, IR spectroscopy proved to be a valuable tool for assessing starch composition and molecular organization. While the technique does not provide absolute quantification of amylose and amylopectin, it offers rapid and non-destructive insight into relative compositional differences and structural order. The observed spectral variations highlight the close relationship between starch composition, hydrogen bonding, and molecular arrangement. These structural characteristics are known to influence functional properties such as gelatinization behavior, digestibility, and textural performance in food and industrial applications. Therefore, the IR-based analysis of starch composition provides meaningful information that complements conventional chemical and thermal characterization methods.

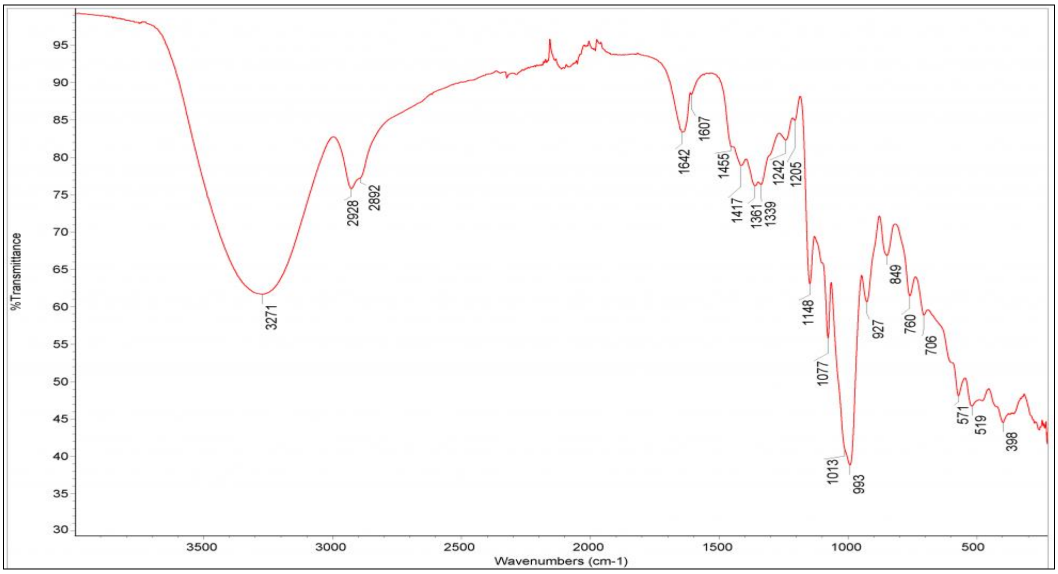

Fig 2: IR spectrum of starch

In Fig. 2 the broad band at 3271 cm^{-1}, for starch, is due to the stretching mode of O-H groups. The adsorption band at 1642 cm^{-1} is attributed to an intermolecular H-bond involving the carboxyl group. The bands at 1148 cm^{-1}, 2926 cm^{-1} are assigned to C-O and C-H stretching, respectively [12-17].

Conclusion

Infrared (IR) spectroscopy proved to be an effective and reliable technique for evaluating starch composition and molecular structure. The characteristic absorption bands observed in all samples confirmed the polysaccharide nature of starch and enabled differentiation between ordered and amorphous regions associated with amylopectin and amylose, respectively. In particular, the fingerprint region (1200–900 cm^{-1}) provided valuable information on starch organization, with the absorbance ratio at 1047/1022 cm^{-1} serving as a useful indicator of relative molecular order and compositional variation.

Differences in O–H stretching vibrations further reflected variations in hydrogen bonding patterns, which are closely linked to starch chain structure and composition. These spectral features highlighted the influence of amylose and amylopectin content on starch molecular arrangement. Although IR spectroscopy does not allow absolute quantification of starch components, it offers a rapid, non-destructive, and reproducible approach for comparative analysis.

Overall, IR spectroscopy represents a practical complementary method to conventional analytical techniques for starch characterization. Its ability to provide insight into starch composition and structural organization makes it particularly valuable for applications in food science, agriculture, and material research, where starch functionality is strongly dependent on molecular structure.

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