Jurnal Sains Teknologi & Lingkungan
Available online http:// jstl.unram.ac.id
ISSN: 2477-0329, e-ISSN: 2477-0310
Terakreditasi Kemenristek-DIKTI SINTA 4
Nomor: 225/E/KPT/2022

Vol. 11 No. 3 pp: 395-402
September 2025
DOI https://doi.org/10.29303/jstl.v11i3.818

Research Articles

Crystalline Properties of Jambu (*Syzygium aqueum* (Burm. f.) Alston) and Jackfruit (*Artocarpus heterophyllus*) Seed Starches: An XRD Spectroscopic Study

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Manuscript received: 28-04-2025. Accepted: 26-09-2025

ABSTRACT

Starch, a complex carbohydrate found in plants, has become an alternative raw material to make plastics (bioplastics). Bioplastics have been intensively used to replace conventional plastics, which cannot be degraded and thus cause environmental problems. Therefore, starch has been the focus of many studies, where researchers study its properties, such as crystalline properties. These properties influence the properties of starch-based bioplastics: their mechanical strength, water resistance, and degradation rate are affected by the arrangement of starch molecules into crystalline structures. This study aims to characterize crystalline properties of starches of jambu fruit (Syzygium aqueum (Burm. f.) Alston) and jackfruit seeds (Artocarpus heterophyllus). For this purpose, X-ray diffraction (XRD) spectroscopy was used. The results show that the starches of jambu and jackfruit fruit seeds belong to different types: B-type starch for jambu and A-type starch for jackfruit. The index of crystallinity of starch of jackfruit seed, 54.0%, is higher than that of starch of jambu fruit seed, 38.1%. Starch of jackfruit seed has higher crystalline properties compared to starch of jambu seed.

Key words: Starch; Crystalline properties; XRD spectroscopy; Jambu fruit seed; Jackfruit seed.

INTRODUCTION

Starch is a complex carbohydrate found in plants, where it acts as a storage form of energy. It is produced as discrete granules varying in shapes from a sphere to an oval and in sizes from a few µm to more than a hundred µm (Thieme et al., 2023). Granules consist of two major polysaccharides: amylose and amylopectin. Amylose is a linear polymer of α-(1→4)-linked D-glucose units containing a very small number of α-(1→6) linked branches, while amylopectin is a high-branch molecule with α-(1→4)-linked D-glucose backbone and α-(1→6) linked branches (Bertoft, 2017).

Recently, starch has been the focus of many researchers since it can be an alternative raw material to make plastics. Conventionally, plastics are made from crude oil, and the products are not degradable, which causes an environmental problem due to the waste they produce. Plastics from starch, bioplastics, can be a solution to this environmental problem faced nowadays. Consequently, there are many studies of bioplastics such as biodegradable foam (biofoam) and biodegradable film (biofilm) made from starches. For instance, researchers have used starches such as potato (Zang et al., 2020), sago (Gani and Kusumyanti, 2022), and cassava (Fatrozi et al., 2020) to make biofoam and study its properties. Likewise, starches such as corn (Aisyah et al., 2018), cassava (Wanita and Purwaningsih, 2017), and sweet potato (Indrianti et al., 2018) have been used to make biofilm and to study its properties.

The properties of starch itself have been of interest to many researchers. One of the properties studied is its crystallinity. It has been shown that granules consist of rings of alternate crystalline and amorphous parts, where crystalline parts consist of branches of amylopectin forming double helices, and amorphous parts contain junction of amylopectin interacting with long amylose molecules (Bertoft, 2017). Based on the way double helices arrange themselves in crystalline parts, and measured by XRD, starches can be classified as type A, B, and C. Type A starch is indicated by XRD peaks of 2θ at 15^o, 17^o, 18^o, and 23^o, type B starch at 5.6^o, 17^o, 22^o, and 24^o, and type C starch, a combination of type A and type B, at 5.6^o, 15^o, 17^o, and 23^o (Xie et al., 2017). In an A-type starch, the structure of the double helices packing is monoclinic with eight water molecules per unit cell, while in a B-type starch, it is hexagonal with 36 water molecules per unit cell (Bertoft, 2017). It has been shown that the crystalline properties of starch, thus starch types, affect the properties of bioplastics. The arrangement of starch molecules into a crystalline structure influences bioplastic properties such as its strength (Palai et al., 2019) and its water absorption (Copeland et al., 2009).

Many starches have been classified based on XRD data; for example, wheat and corn have been classified as type A starch (Todica et al., 2016; Xie et al., 2017), avocado and kluwih seeds type B starch (Indarti et al., 2022), and pea type C starch (Xie et al., 2017). Jambu (Syzygium aqueum (Burm. f.) Alston) and jackfruit fruit seeds have also been identified as starches (Santos et al., 2024; He et al., 2025). However, from crystalline studies of starches, jambu and jackfruit (Artocarpus heterophyllus) fruit seeds have not received much attention.

In this study, crystalline properties of starches of jambu and jackfruit fruit seeds are characterized by using XRD spectroscopy to determine the type of starches and the index of crystallinity of both starches.

MATERIALS AND METHODS

Preparation of Fruit Seed Starches of Jambu and Jackfruit

The fruit seed starches of jambu and jackfruit were prepared from the seeds of the fruits taken from a local garden in Ambon Island, Indonesia.

The seeds were washed using tap water and left to dry. The outer layer of the dried seeds was removed using a knife, and the seeds were ground using a blender. After grinding, the paste was mixed with distilled water at a ratio of 1:3, and the slurry was filtered through a cloth bag. The filtered slurry was then allowed to rest for two hours for the process of sedimentation. The liquid at the top was decanted and discarded. The process of filtration was repeated three times, and the sediment was dried in the oven at 45ºC for 48 hours and stored in a container for further analysis.

XRD Analysis

The crystalline property of starch was studied using an X-ray diffractometer, XRD Rigaku MiniFlex 2. The monochromator used was Cu-Kα radiation with λ=1.5405 Å at a power of 30 kV, 15 mA under an angle of 2θ ranging from 3 - 145º with an interval of 0.02º. The data of 2θ was truncated between 14º to 26º and was smoothed by applying a Savitzky-Golay filter with the polynomial degree of 3 and 40 points for clarity.

The index of crystallinity, I_C, of the starch was determined following protocol of the previous study (Patty and Wattimena, 2023). It was calculated using

$$I_C = \frac{A_C}{A_C + A_A} \quad \text{.......................................(1)}$$

where A_C and A_A are the areas of crystalline and amorphous parts, respectively. The A_C and $A_C + A_A$ were determined by integrating the area under the peaks and the whole area of the XRD data, respectively.

RESULTS AND DISCUSSION

Type of Starch

Figure 1 shows XRD data of fruit seed starch of jambu: raw data (grey line) and smoothed data (black line), where the data was smoothed as explained in the method section. There are three main peaks of 2θ: 17º, 22º, and 24º, and two minor peaks: 15º, and 20º. Based on the main peaks of the data, jambu seed starch can be categorized as a B-type starch.

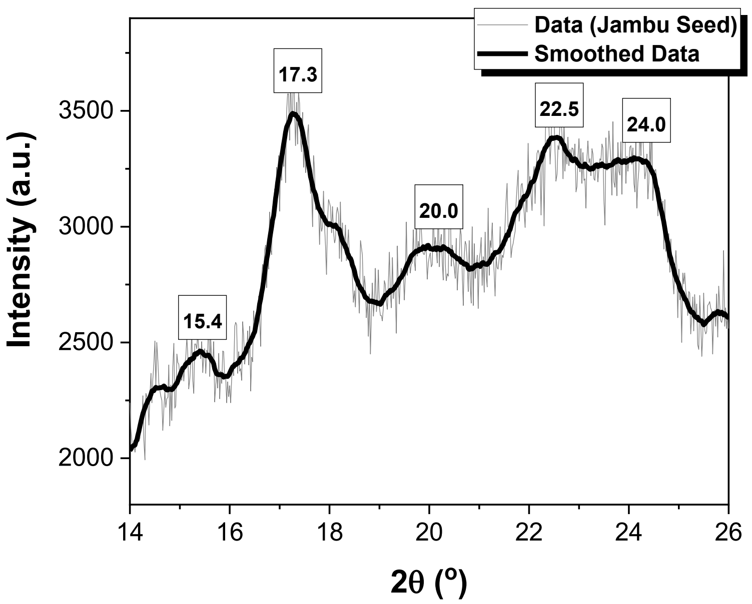

Figure 1. XRD data of jambu seed starch, where the raw data has been smoothed as explained in the method.

Figure 2 shows XRD data of fruit seed starch of jackfruit: raw data (grey line) and smoothed data (black line), where the data were smoothed as explained in the method section.

There are four main peaks of 2θ: 15º, 17º, 18º, and 23º, and one minor peak: 20º. Based on the main peaks of the data, jackfruit seed starch can be categorized as an A-type starch. The minor peaks at 20º shown in Figures 1 and 2 for both samples indicate the presence of V-type complexes containing amylose and endogenous lipids (Chen et al., 2015).

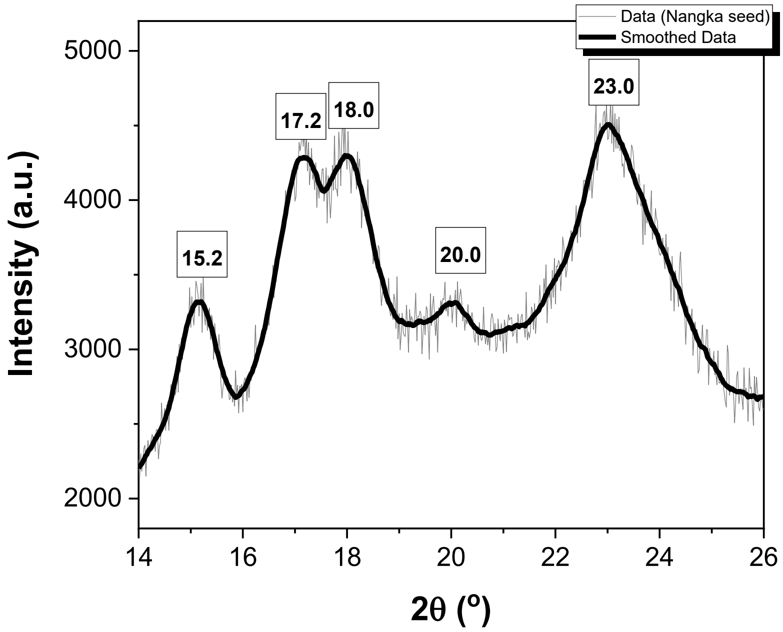

Figure 2. XRD data of nangka fruit seed starch, where the raw data has been smoothed as explained in the method.

The data of Figures 1 and 2 show that two seeds of different fruits show two different starch crystalline structures, types A and B. Previous studies on other fruit seeds also show two different crystals. For example, it has been shown that mango seed is an A-type starch (Shahrim et al., 2018), while avocado seed (Macena et al., 2020; Indarti et al., 2022), kluwih seed (Indarti et al., 2022) are B-type starches. For other starches than the fruit seeds, wheat and corn are type A starches (Todica et al., 2016; Xie et al., 2017)

Index of Crystallinity

Index of crystallinity was determined using Equation 1. The crystalline area was determined by integrating the area under the curve of the peaks, as shown as a shaded area in Figure 3 for jambu seed starch. The area of each peak is summarized in Table 1, and the total crystalline area was found to be 13,106.9 a.u.

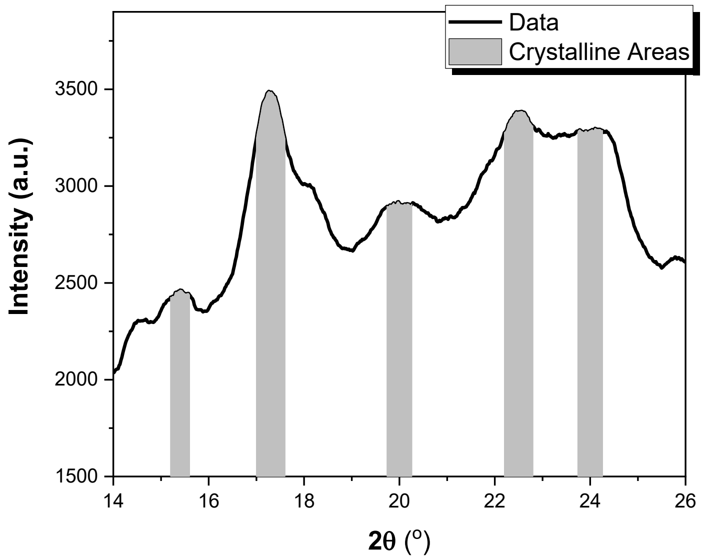

Figure 3. Crystalline areas (Shaded area), the area under the curve of the peaks of jambu starch XRD data

Tabel 1. Area under the curve of the peaks in jambu seed starch XRD data

No	Peaks (^{0})	Area (a.u)
1	15.4	1936.2
2	17.3	4716.8
3	20.0	2892.1
4	22.5	1918.2
5	24.0	1643.6
	Total	13,106.9

The whole area, crystalline plus amorphous areas, was determined by integrating the area under the curve of the whole data, as shown as a shaded area in Figure 4. The total of the whole area was found to be 34,379.2 a.u. The index of crystallinity of jambu seed starch was then found to be 38.1%.

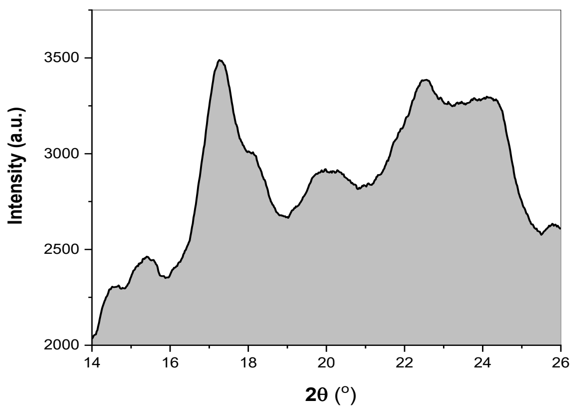

Figure 4. The area under the curve of jambu starch XRD data

The same calculation was conducted for jackfruit seed starch. Table 2 summarises the results of integrating crystalline area, areas under the curves of XRD peaks, where the total crystalline area was found to be 21,958.6 a.u. The total of the whole area was found to be 40,691.8 a.u, so that the index of crystallinity of jackfruit seed starch was found to be 54.0%.

Tabel 2. Area under the curve of the peaks in jackfruit seed starch XRD data

No	**Peaks (^{0})**	**Area (a.u)**
1	15.4	3099.2
2	17 - 18	8121.9
3	20.0	2607.4
4	23	8130.1
	Total	21,958.6

The results show that the index of crystallinity of jackfruit seed starch, 54%, is greater than that of jambu seed starch, 38.1%. Index of crystallinity of jackfruit starch is close to that of avocado seed 56,09% (Macena et al., 2020), while that of jambu seed starch is close to that of cassava 41% (Patty and Wattimena, 2023), corn starch 36% and tapioca 42% (Dome et al., 2020). Both values are larger than that of potato 29% (Dome et al., 2020) and banana 23% (Chavez-Salazar et al., 2017).

CONCLUSION

This study found that fruit seed starches of jambu and jackfruit show two different crystalline structures as revealed by XRD spectroscopy. The seed of the jambu fruit is a B-type starch, while the seed of the jackfruit is an A-type starch. Moreover, the study showed that the index of crystallinity of starch of jackfruit seed, 54.0 %, is higher than that of starch of jambu fruit seed, 38.1 %. The starch of jackfruit seed has higher crystalline properties compared to the starch of jambu seed. In terms of application to bioplastics, starches with a relatively high crystallinity index tend to produce bioplastics with relatively high tensile strength, stiffness, and water resistance.

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