



Microscopical behavior of Selected Starches in Presence of Other Food Ingredients Studied by Scanning Electron Microscopy (SEM)-Review

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Abstract:

This review article highlights the microscopical behaviors of selected starches that were studied using scanning electron microscopy (SEM) with data shown in various research publications. The starches of potato, sago, corn, rice, and arrowroot are included in this overview. Scanning electron microscopy (SEM) proved itself to be a cherished method for the study of granulate microstructure and surface characteristics of starch. It is used for the investigation of enzyme effect on the starch granules. The goal of the present work is to reveal by SEM observation, the size, shape, and surface morphology of starch granules from the native starches with other starches which are under various treatments and also used for the production of biodegradable plastics. Swelling and deformation of the starch granules observed in the scanning electron microscope correlated with the loss of birefringence in the light microscope.

Introduction

Starch is an essential component in most foods and a major energy source in human diets. Starch is a glucose biopolymer, it is the major storage component of most economically important crops: cereal (e.g., wheat, rice, corn, oat, barley), legume (e.g., lentil, bean, pea), tuber (e.g., potato, sweet potato), and yam (*Dioscorea*) (Vasanthan, 2001). Starch is a naturally occurring, biodegradable, inexpensive, and abundantly available polysaccharide molecule. It is a major carbohydrate easily extractable from various native sources, like potato, maize, corn, wheat, etc, which finds widespread application in various food and non-food industries. Starch is the most important polysaccharide as well as storage polymer of plants and is most commonly found in tubers, stem, seed, fruit, leaves, and roots of various plants. Starch consists of glucose units $(C_6H_{10}O_5)_n$ with n ranging from 300 to 1000. Starch is made up of a mixture of two polymers referred to as amylose and amylopectin. Depending on its botanical source, Amylose is a linear polymer having a molecular weight of less than 0.5 million Dalton (degree of polymerization of $15 \times 10^2 - 6 \times 10^3$). Amylopectin molecules are much larger and highly branched having a molecular weight of 50–100 million Dalton and a degree of polymerization of about in the range of $3 \times 10^5 - 3 \times 10^6$ (Neelam et al., 2012). From a nutrition point of view, starch can be divided into three categories based on the digestion rate and digestion extent: rapidly digestible starch (RDS, the starch fractions digested within 20 min after ingestion), slowly digestible starch (SDS, the starch fractions digested between 20 and 120 min after ingestion), and resistant starch (RS, the starch fractions that cannot be digested in the small intestine after ingestion). It is important to understand the rheological properties of food products (Ahmed & Ramaswamy, 2004). Gelatinized starch recrystallizes during storage in a process known as retrogradation. Retrogradation affects the texture, appearance, and shelf-life of food products (King & Kaletunc, 2009).

Importance of microscopical behavior:

By using SEM, these authors observed that the high-pressure-treated starches demonstrated two distinctly differentiated zones. The outer zone of granules remained unchanged and it corresponded to the more organized part of the granule, whereas the interior part of the granules was destroyed and formed gel-like structures. The observation by SEM also confirmed by (CP/MAS) ^{13}C NMR studies on pressurized potato starch conducted by Błaszczak and co-authors seemed to be closely related to the previous suggestion of Rubens and Heremans (2000) that the changes in the internal granule structure evoked by high pressure may be resulted from the hydration and/or melting of the crystalline structures. The phenomenon of birefringence loss by the pressurized starch granules may have resulted from the disruption of radial orientation of the crystallites that, according to Yuryev, Wassermen, Andreev, and Tolstoguzov (2004), are predominantly formed by the amylopectin chains in the double helical form.

keywords: Cross-polarisation/magic angle spinning (CP/MAS),

Potato starch :

The changes in the granule structure of potato starch during gelatinization are shown in Figure 1 a-d. The granules in the control sample (Fig. 1a) are relatively free of destruction and impurities. Varying in the structure from small spherical granules to larger ellipsoidal particles. At 60 ° C (Fig. 1b)

changes in the granule structure become evident. Larger granules appear to be broken down initially leaving granule residues that seem to be adhesive and extensible. As the temperature is increased to 62 C (Fig. 1c) there is still evidence of granules that do not look like leached. Two or three of the granules have collapsed inwards forming cup-shaped structures. By 64 C, all but a few of the small granules are gelatinized (Fig. 1d) but residues of large granules stretched over the surface of the specimen are very much in substantiation.

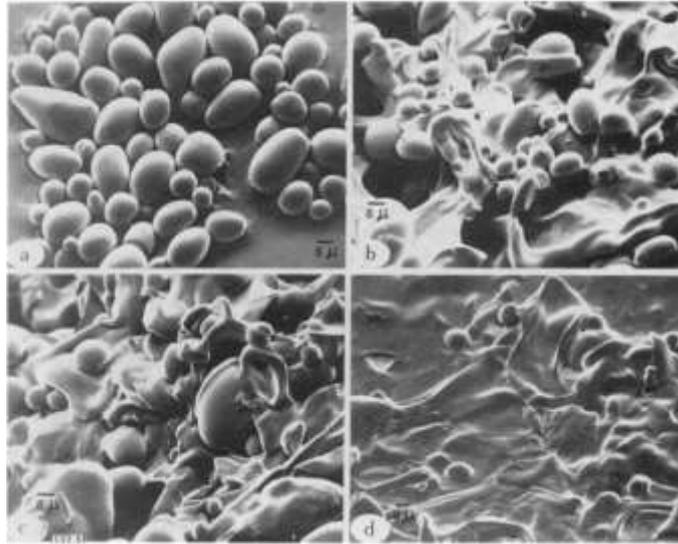


Fig 1. Potato starch granules visualized by SEM [a-control, b- 60°C, c-62 °C, d-64 °C]

Corn starches :

Changes in photographs occurring during the process of gelatinization of corn starch are shown in Figure 2. The control sample (Fig. 2a) contains predominantly large chunky granules that are characteristic of the starch with some small to intermediate spherical granules. The image obtained by scanning electron microscopy (SEM) of native Hylon VII and waxy corn starch showed the typical polygonal shape of corn starch granules with sizes ranging from 5 to 20 and 2 to 30 mm (longer axis), respectively. The detailed analysis under SEM of Hylon VII after treatment for 9 min. (Fig. 2c) appear to explain the reason for the lack of the granule's ability to birefringence under polarised light. While the majority of granules retained granular shape, many of them appeared collapsed, and deformed and demonstrated a hollow appearance. This physical process in starch granules was already observed under polarised light. Although the changes in granules structure were important, the leaching of amylose outside granules which appeared slightly swollen was not observed. The microscopy images of waxy corn starch treated for 9 min (Fig. 2d) showed a complete breakdown of granules. Fig. 2d demonstrates a molecular dispersion of waxy corn starch after treatment for 9 min. That homogenous structure of a gel network was formed by the amylopectin phase. These observations were similar to those made by Stute et al. (1996). These authors showed that the treatment of waxy corn starch over water (5% suspension) at a pressure of 600 MPa for 15 min lead to a complete disintegration of starch granules.

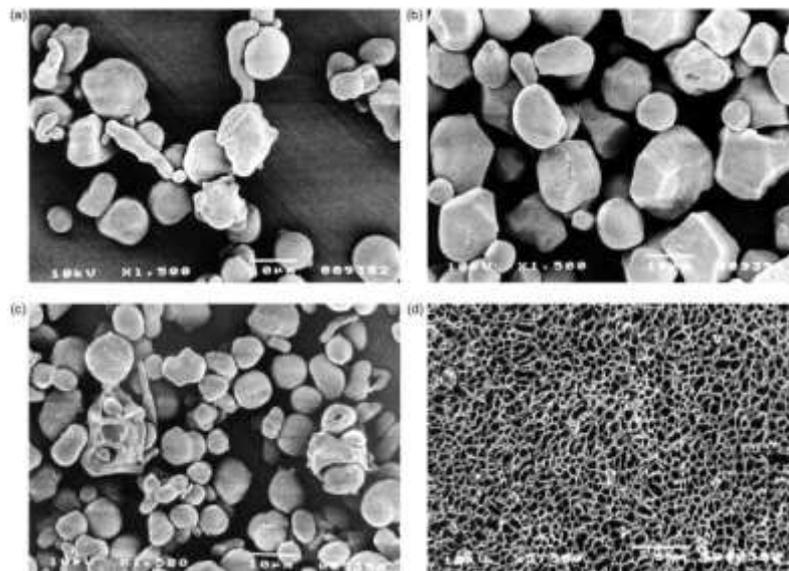


Fig. 2. SEM microstructure of starches: native Hylon VII (a) and waxy corn starch (b), Hylon VII (c) and waxy cornstarch (d) treated with high pressure at 650 MPa for 9 min

Sago starch :

Scanning electron micrographs of native, OPT and HMT sago starch are shown in Fig. 3. Native sago starch granules are in the form of oval granules without hollow areas at the surface (Fig. 3A), as observed in OPT and HMT of sago starch (Figs. 3B, 3C). These SEM results demonstrate the occurrence of the hollow at the central core region only inside the granules as observed by light microscopy.

Keywords: OPT – Osmotic Pressure Treatment, HMT – Heat Moisture Treatment

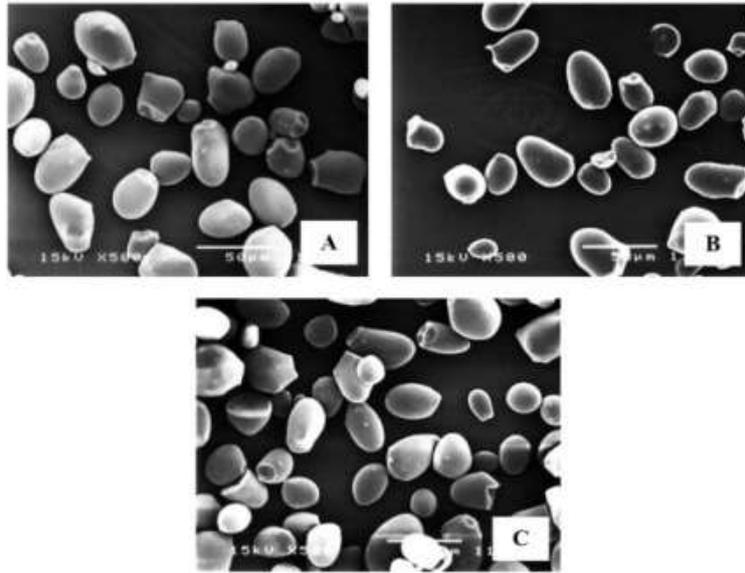


Fig. 3. Scanning electron micrographs of native (A), OPT (B), and HMT sago starch treated at 120°C, 60 min (C) (magnification 500 ×)

Rice Starch :

The granules of this starch are among the tiny cereal starches which were studied with Scanning Electron Microscopy (SEM) (Fig. 4) Because of its small size (3-8 microns), the SEM provides the preferable method of studying the shape and surface details of this starch. Most of the granules are emphatically polygonal, although a few rounded sides can be observed in the micrographs. Most small starches which have been scanned with the SEM have this general shape. Rice starch tends to aggregate into the flock. It has been thought that the aggregation is the result of & “bilobated” granules which were not broken up in the mechanical preparation of the starch or that the granules are cemented by gluten. Schoch suggests that the assemblage of rice granules may also be caused by surface gelatinization induced by the base-forming steep and/or poor control during drying in the commercial manufacture of rice. Poor steeping may not liberate the starch granules from the protein matrix in which they grow, thereby leading to the rice starch agglomerates cemented by gluten as seen by Sjoström Since most of the granules seen in the micrographs are the result of compound granules breaking open, some of the granule faces should conceivably show details of the -internal structure of the rice starch in the native granule.



Fig.4a. SEM micrograph of rice starch (3360 X); some wedge-shaped layering appears on some granules.

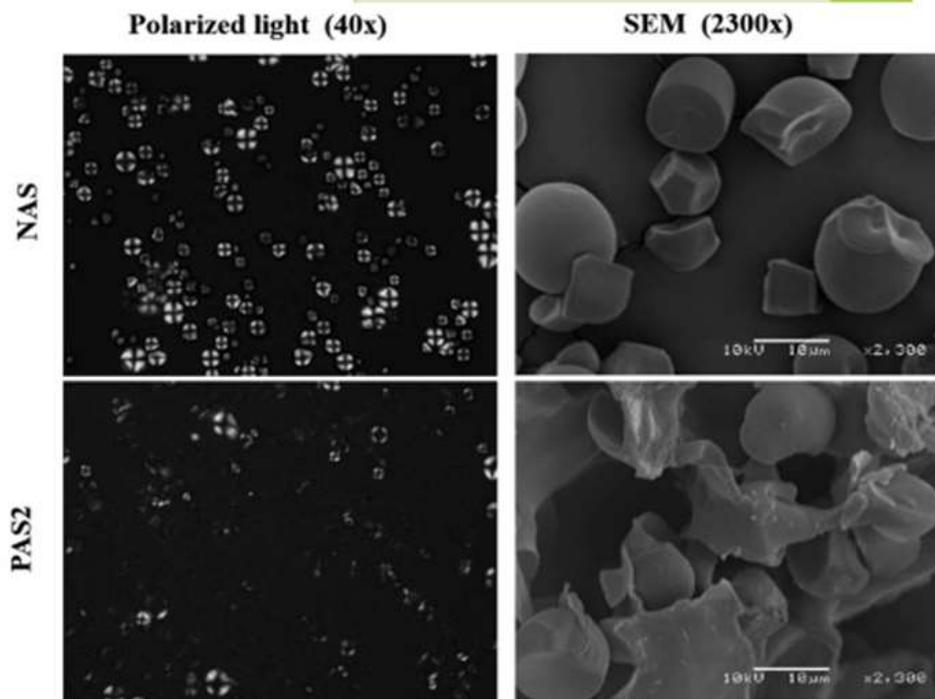


Fig. 4b. SEM micrograph of rice starch (6400 X); some wedge-shaped layering appears on some granules

Arrowroot starch :

Polarized light and scanning electron micrographs (SEM) of NAS and selected HHP-processed arrowroot starches (PAS) granules are shown in Figure 5. NAS granules were irregularly shaped, showing an oval or shortened form, as has also been reported by Madineni et al. (2012). The granular morphological characteristics (size and shape) were retained after processing at 400 MPa/25°C suggesting that there was no pressure effect seen on the material under these processing conditions. This was further evidenced by the presence of Maltese crosses in both native and 400 MPa/25°C (PAS1 and PAS4) processed starches under polarized light, which indicates the degree of molecular order in the starch granules was unchanged (Honey, 1998) At 500 MPa/50°C only a few Maltese crosses remained in the material, while mostly broken granules were observed. These events of disorder within the granules indicate hydration and swelling of the starches The images in Figure 5 suggest that at 650 MPa (sample PAS12; data not shown for 25°C), complete granule disintegration occurs, and the starch forms a continuous and weak gel-like network. These findings, together with a no Maltese cross observation, suggest that the HHP completely disrupted the ordered structure of the starch granule (Liu et al., 2008)

Keywords: NAS – Native Arrowroot starch, PAS - Processed Arrowroot starch



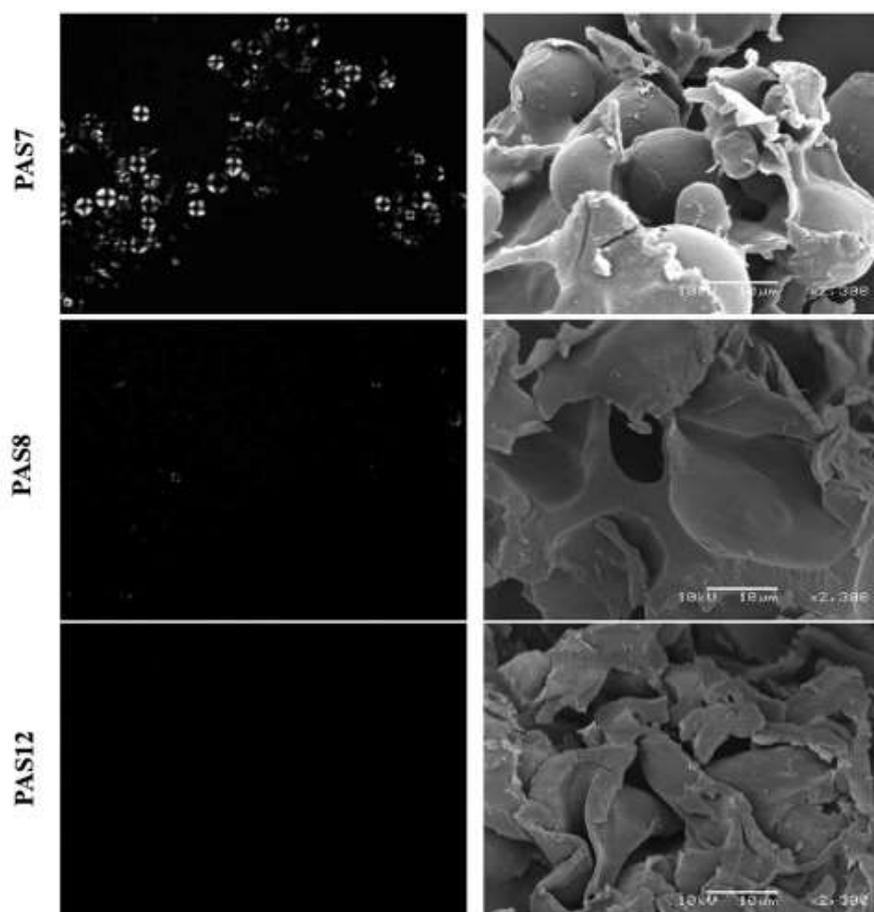


Fig.5. Polarized light and scanning electron micrographs of native and selected processed arrowroot starches

Conclusion :

From the SEM images, the size distribution of the granules was inferred and compared for the different samples. These investigations will contribute to the characterization of native starches as raw materials for the production of biodegradable plastics. Microscopy analysis (LM, SEM) showed that pressurization of waxy corn starch resulted in a complete breakdown of the granules, whereas Hylon VII retained a granular structure after 9 min of treatment.

References :

- Neelam, K., Vijay, S. and Lalit, S., 2012. Various techniques for the modification of starch and the applications of its derivatives. *International research journal of pharmacy*, 3(5), pp.25-31.
- Vasanthan, T., 2001. Overview of laboratory isolation of starch from plant materials. *Current protocols in food analytical chemistry*, (1), pp.E2-1.
- Błaszczak, W., Fornal, J., Valverde, S. and Garrido, L., 2005. Pressure-induced changes in the structure of corn starches with different amylose content. *Carbohydrate Polymers*, 61(2), pp.132-140.
- Zhou, F., Liu, Q., Zhang, H., Chen, Q., and Kong, B., 2016. Potato starch oxidation induced by sodium hypochlorite and its effect on functional properties and digestibility. *International journal of biological macromolecules*, 84, pp.410-417.
- Stolt, M., Oinonen, S. and Autio, K., 2000. Effect of high pressure on the physical properties of barley starch. *Innovative Food Science & Emerging Technologies*, 1(3), pp.167-175.
- Guo, Z., Zeng, S., Zhang, Y., Lu, X., Tian, Y. and Zheng, B., 2015. The effects of ultra-high pressure on the structural, rheological, and retrogradation properties of lotus seed starch. *Food Hydrocolloids*, 44, pp.285-291.
- Ural, N., 2021. The significance of scanning electron microscopy (SEM) analysis on the microstructure of improved clay: An overview. *Open Geosciences*, 13(1), pp.197-218.
- Hall, D.M., and Sayre, J.G., 1970. A scanning electron-microscope study of starches: Part II: Cereal starches. *Textile Research Journal*, 40(3), pp.256-266.

Oliveira, L.C., Macnaughtan, B., House, O., Villas-Boas, F., Clerici, M.T., Bakalis, S., Muttakin, S. and Cristianini, M., 2021. Extending the functionality of arrowroot starch by thermally assisted high hydrostatic pressure. *Journal of Food Processing and Preservation*, 45(9), p.e15756.

Pukkahuta, C. and Varavinit, S., 2007. Structural transformation of sago starch by heat-moisture and osmotic-pressure treatment. *Starch-Stärke*, 59(12), pp.624-631.

Hill, R.D. and Dronzek, B.L., 1973. Scanning electron microscopy studies of wheat, potato, and corn starch during gelatinization. *Starch-Stärke*, 25(11), pp.367-372.

Elgadir, M.A., Bakar, J., Zaidul, I.S.M., Rahman, R.A., Abbas, K.A., Hashim, D.M. and Karim, R., 2009. Thermal behavior of selected starches in presence of other food ingredients studied by differential scanning calorimetry (DSC)–Review. *Comprehensive Reviews in Food Science and Food Safety*, 8(3), pp.195-201.

Błaszczak, W., Valverde, S. and Fornal, J., 2005. Effect of high pressure on the structure of potato starch. *Carbohydrate Polymers*, 59(3), pp.377-383.