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The Application of Scanning Electron Microscope (SEM) to Study the Microstructure Changes in the Field of Agricultural Products Drying

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1. Introduction

The objective of this part: Highlight the significance of microstructure investigation in the field of agricultural product drying or dehydration.

It is a common sense that structure of material determines its function. The change of macroscopic properties of materials is caused by the changes of its microstructure. For example, a porous structure as a honeycomb would facilitate rapid water diffusion or promote a rapid water uptake during drying or cooking. On the contrary, a compact structure or fewer pores at the surface of the product can cause a slower moisture migration during drying or water penetration into the interior during rehydration or cooking. Therefore, microstructure investigation can help quantifying product changes during processing and may also improve the understanding of mechanisms and changes in quality factors, especially the changes in food texture (Aguilera & Stanley, 1999; Xiao et al., 2009). For example, the pore sizes and the number of pores can significantly influence the texture of food. Smaller number of pores and small sizes led to the dense structure. While, larger number of pores and large pore size can cause a decrease of the hardness of the product.

2. The classification of the specific research

The objective of this part: Through concrete examples to introduce the main content and conclusions of the microstructure investigation in the field of agricultural materials drying.

2.1 Observing the microstructure of the material surface before and after the processing to find out the effect of drying on the product microstructure

The scanning electron micrographs on the surface of fresh and dried samples can be used to analyze the microstructure changes during drying or dehydration process. Fresh apple tissue has a well-organized structure consisting of cells and intercellular spaces, however, the breakdown of cell walls, a decreased intercellular contact and collapse of cell structure were found in the dried apple tissues (Deng & Zhao, 2008 a and b).

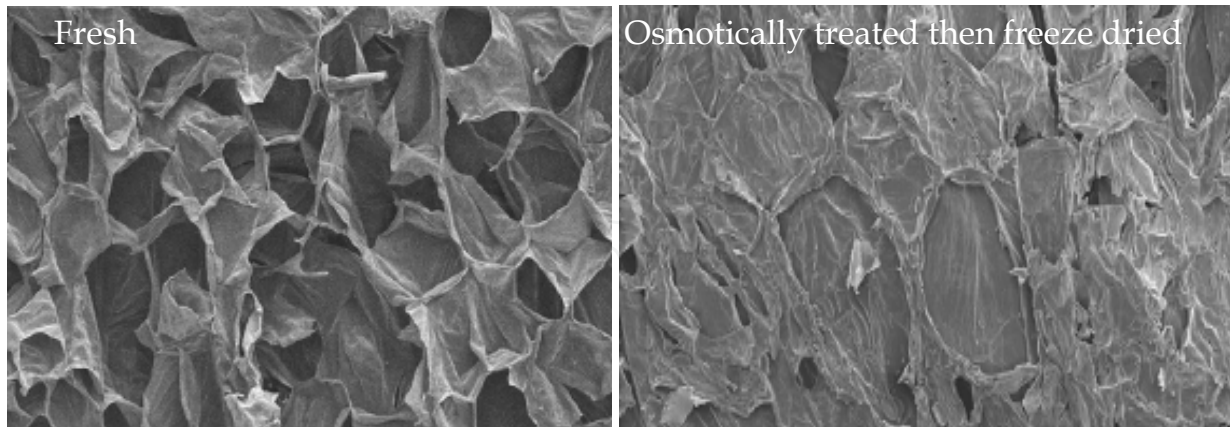


Fig. 1. Scanning electron micrograph of fresh and dried apple (Fuji)
(Deng & Zhao, 2008 a and b)

Take the pressure pulsed osmotic dehydration (PPOD) of salt eggs using NaCl solution for another example. Osmotic dehydration of salt eggs is an ancient method of egg preservation, which can be traced back to several hundreds of years. With special taste and flavour, salt eggs is one of the most popular egg food in Asian countries especially in China. Whereas, presently in China the osmotic dehydration of egg is done manually, the process of which is tedious, time consuming and labor intensive. The traditional osmotic dehydration process involves submerging eggs in NaCl solution in a static situation which last about 30 days at room temperature. During this process the NaCl transfer from salt solution through eggshell into the egg white and yolk. In order to increase the automation and decrease the processing time, the PPOD technology has been applied in salting eggs, which has been proved more efficient reducing the osmotic time from 30 days to 2 or 3 days (Chen and Gao, 2006; Wang and Gao, 2010). However, the mechanisms of PPOD of salt eggs hasn't be explored.

The authors try to find the mechanism using the microstructures of the eggshell before and after PPOD, as shown in Figure 2. From Figure 2, it can be found that the pore sizes and the number of pores in the surface of eggshell was increased after the processed of PPOD, which could significantly facilitate the NaCl immigration from the salt solution to the egg inside. As regards the cross section of eggshell, it can be observed that after PPOD the eggshell become less dense and a few pore channels were formed, which enabled the NaCl to penetrate easily from solution to egg inside and increased the moisture transport from egg to the solution, and thus accelerated the osmotic dehydration process. This phenomenon may be due to the tunneling effect of PPOD, which can create canals in the microstructure of eggshell during PPOD by stretching and enlarging the pore sizes. The membrane of eggshell is the most resistance for mass transfer during osmotic dehydration of salt egg (Chen et al., 1999). From Figure 2, it can be found that the eggshell membrane comprised of multi-layers of "fibers" as a bird's nest built of sticks. It can be also found that after PPOD the microstructure of eggshell membrane become looser. Certainly, such a loose structure can promote mass transport and improve the overall process rate compared to the dense structure.

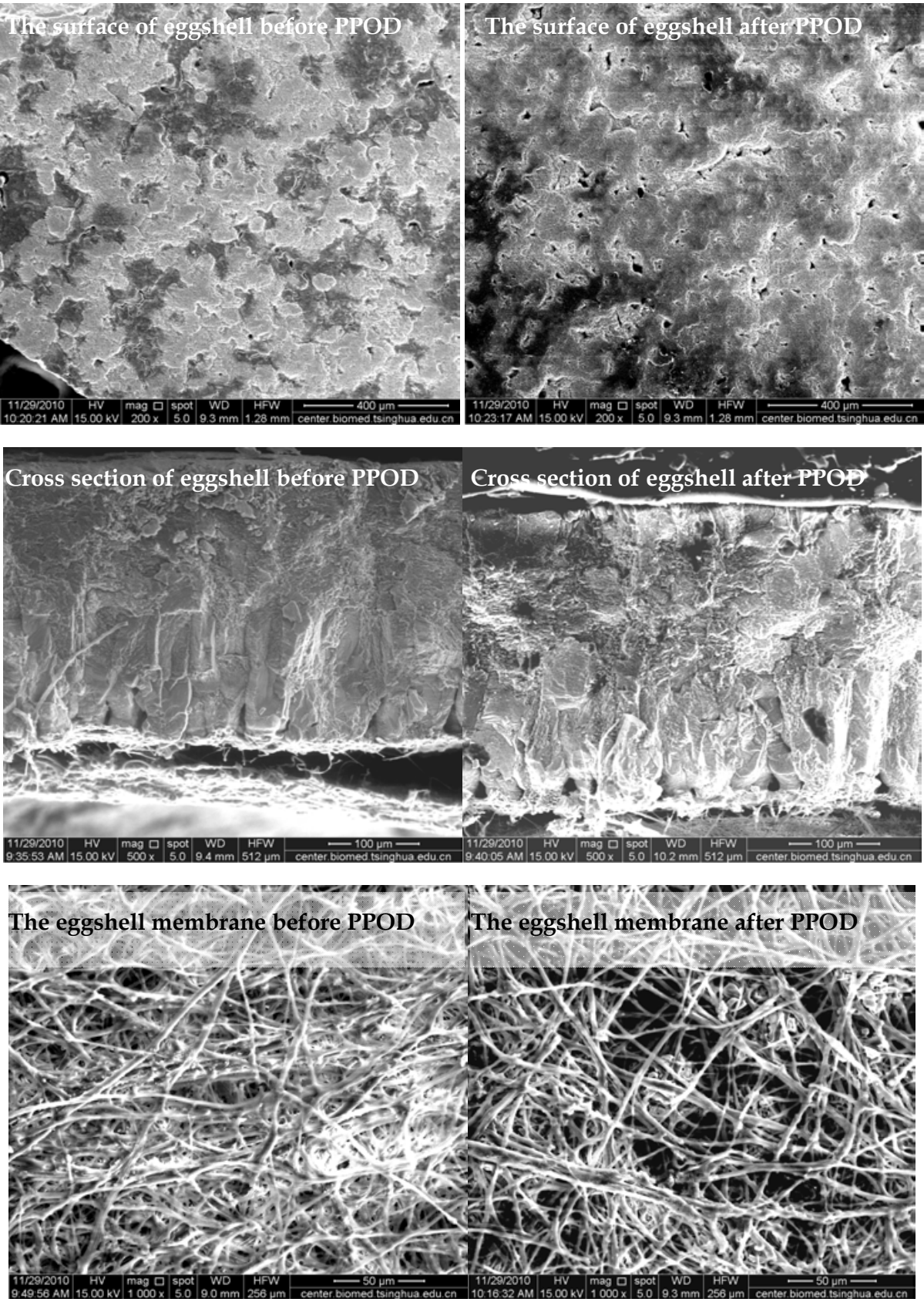


Fig. 2. Scanning electron micrograph of eggshell before and after the pressure pulsed osmotic dehydration (PPOD).

2.2 To investigate the effect of different pretreatment methods and drying conditions on the microstructure of the samples

The information on microstructure changes is essential for enabling better process control and improvement in the appearance by optimizing the pretreatment and drying parameters. The microstructure observation of the product's surface can be carried out to explore the effects of different pretreatment methods and drying temperatures on the microstructure changes of the samples. Recently, many researchers have investigated the microstructure changes of various fruits, vegetables and other food materials during pretreatment and drying process.

Vega-Gálvez et al. (2008) investigated the influence of pretreatment and air drying temperature on the quality and microstructure properties of rehydrated dried red bell pepper. Microscopic evaluation of the rehydrated pepper samples, as shown in **Figure3**, illustrated that the damage to cellular structure was minimized when the samples was pretreated by immersing in a solution containing NaCl, CaCl₂ and Na₂S₂O₅ prior to drying in comparison with the no pretreated samples. They also found that the drying temperature had an significant effect on the microstructure of the dried sample and the damage to cellular structure could be alleviated by decreasing the drying temperature.

Xiao et al. (2009) studied the effect of different pretreatments on drying kinetics and quality of sweet potato bars in terms of textural properties, microstructure, and colour undergoing air impingement drying. Microstructure observation of the surface of dried sweet potato bars, as shown in **Figure4**, was carried out to evaluate the effects of different pretreatments on the microstructure changes of the dried samples. It was found that when the sweet potato bars were subjected to hot water blanching and superheated steam blanching pretreatments, the dried samples had a homogeneous compact structure. In addition, no pores and starch granules were found on the surface of the samples. As a result, the structure would slower water transfer or penetration during drying or rehydration process. However, the samples subjected to citric acid pretreated for 30 min had large with non uniform pores and lots of starch granules on its surface. Absolutely, such porous structure would facilitate rapid water migration during drying. In terms of no pretreated ones, the dried sweet potato tissues showed more numerous starch granules and fewer pores than the citric acid pretreated samples on its surface. Therefore it is interesting to note that different pretreatments cause various changes of microstructure of the samples and lead to product properties varied differently.

Bondaruk, Markowski, Blaszcak (2007) investigated the effect of drying conditions on the quality of vacuum-microwave dried potato cubes in terms of colour, starch content, sugar content, mechanical properties and microstructure. Concerning the microstructure, it was observed that compared with the forced convection air drying the application of microwave energy led to different physical changes in the sample microstructure. It was also found that in the case of hot-air drying the intensity of structural changes depended on the drying temperature. In addition, a higher temperature causes greater damage to the microstructure of potato cubes.

Pimpaporn et al. (2007) reported that potato chips dried at 80 and 90°C had more uniform pore size and pore distribution compared with the chips dried at 70°C and more extensive surface shrinkage was found on the samples dried at 70°C. However, Fang et al. (2011)

reported that lower drying temperature led to relatively uniform size and shape with smooth particle surface, whereas higher drying temperature resulted in size variations and wrinkled particle surfaces when they carried out the milk spray drying under different drying temperatures. The SEM micrographs of milk powder under different spray drying temperatures were shown in **Figure5**.

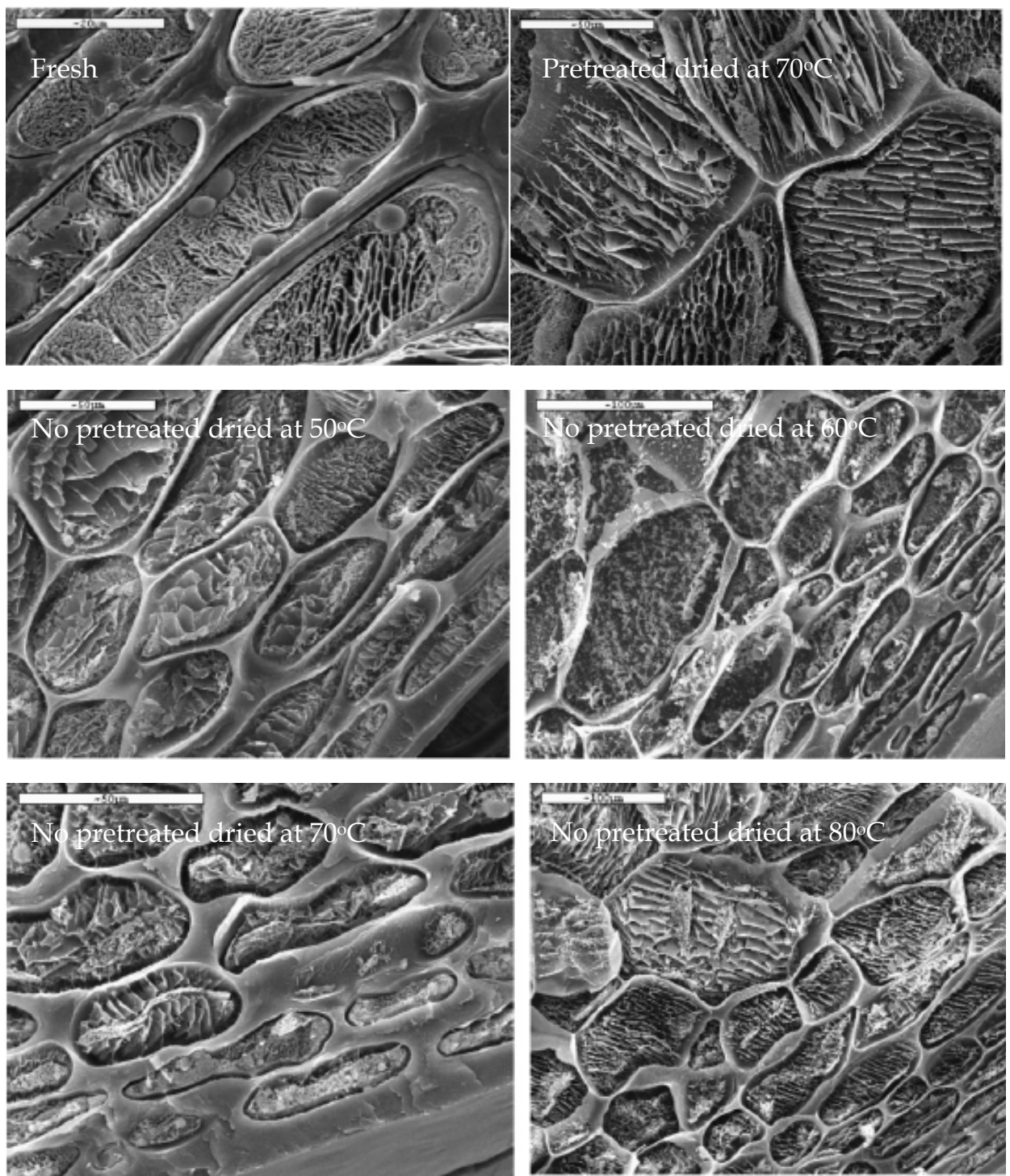


Fig. 3. SEM micrographs of fresh and rehydrated red pepper samples with and without pretreatment dried at different temperatures (Vega-Gálvez et al., 2008).

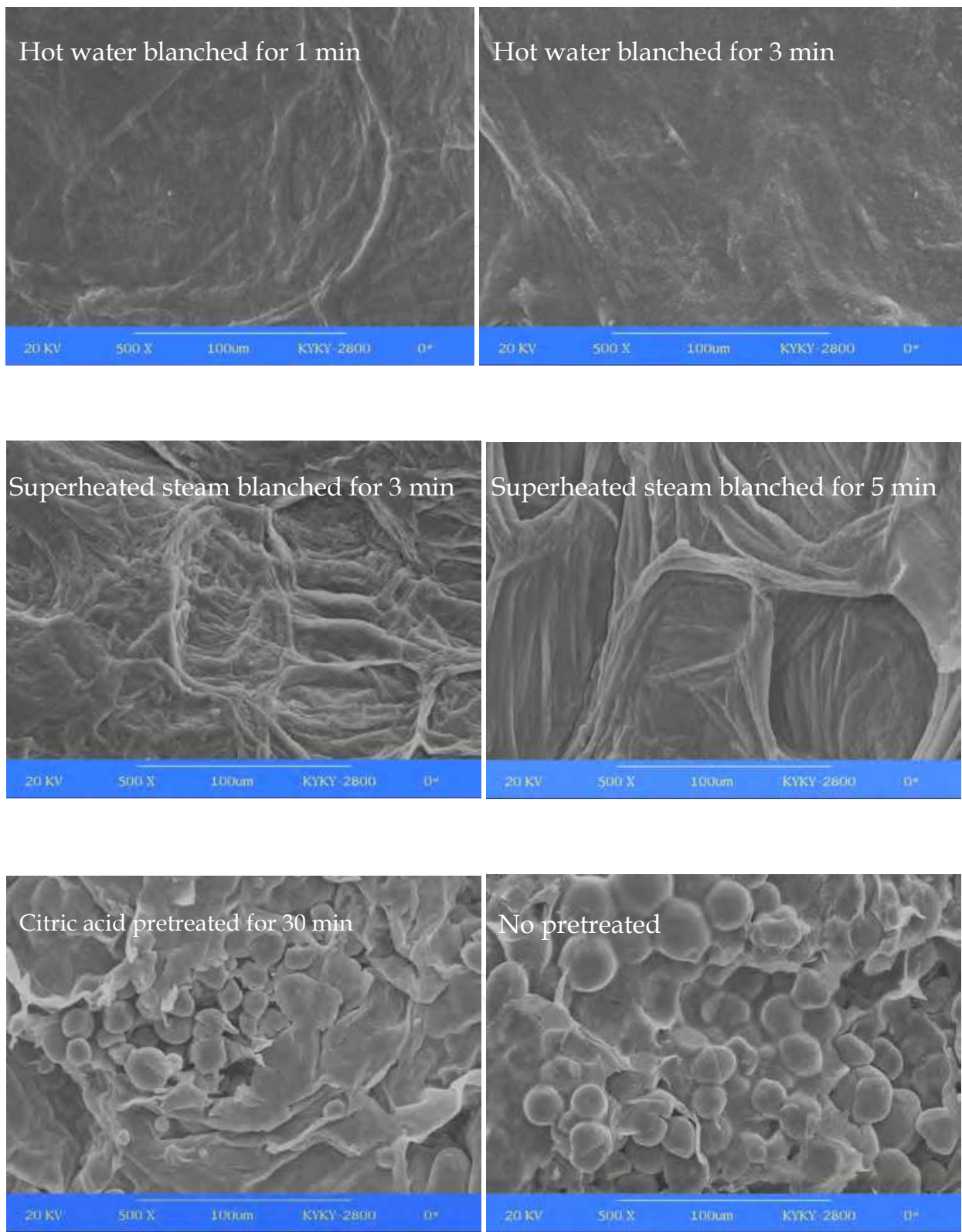


Fig. 4. SEM micrographs of the surface of dried sweet potato bars underwent different pretreatments (Xiao et al., 2009).

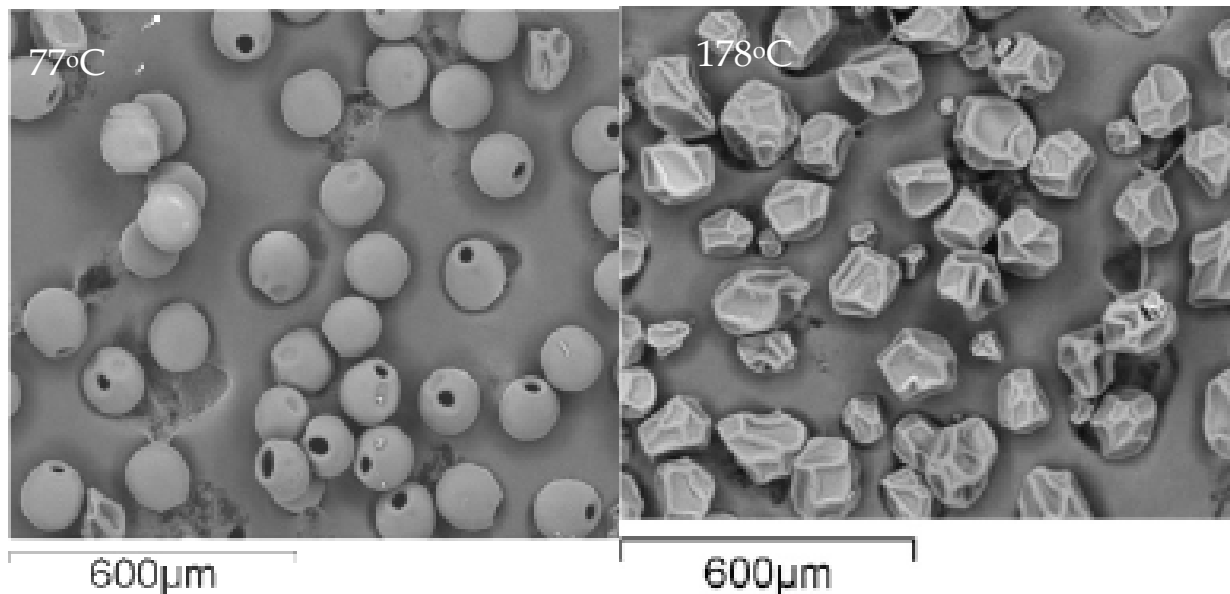


Fig. 5. SEM micrographs of milk powder under different spray drying temperatures (Fang, e t al., 2011).

2.3 Using SEM micrographs of materials to analyze the moisture transfer mechanisms during drying process and interpret the rehydration characteristics or the texture properties of the dried products

In general, most of the drying occurs in the falling rate period and moisture migration controls the whole process. Due to the limited information on the mechanism of moisture movement during drying and the complexity of the process that may involve molecular diffusion, capillary flow, Knudsen flow, hydrodynamic flow, surface diffusion and all other factors which affect drying characteristics, the moisture transfer mechanism hasn't been described completely (Madamba et al., 1996). Knowledge of the microstructure in which moisture and heat transfer take place may assist in finding the mechanisms and their relative contributions to the transport phenomena. Yang et al. (2010) made a try when they carried out an experiment to investigate the influence of glutinous components in plant tissue on the drying characteristics of plant materials taking Chinese angelica and Astragalus slices as the samples.

From the SEM micrographs of the sample (as shown in **Figure6**), they found that the trachea with relative large pore diameter was surrounded by massive parenchyma cells. It implied that during the drying process there was two parallel ways for moisture transfer from parenchyma cells to surrounding drying media: the direct moisture diffusion through some pores or open structures on the surface layer and the moisture emigrate from the parenchyma cells inside matrix to the surrounding drying media. Further more, they also reported that the process of moisture transfer from the parenchyma cells inside matrix to the surrounding drying media included three steps: firstly from the parenchyma cell to the adjacent cell via plasmodesma; secondly from the parenchyma cell to the adhered trachea via aperture; thirdly from trachea to the surrounding drying media.

Rehydration ratio and texture of the samples, which is the macro performance of the material microstructure, is strongly dependent on the product microstructure. Therefore, the SEM micrographs can be used to analyze, interpret or even predict the rehydration characteristics or the texture properties of the dried products.

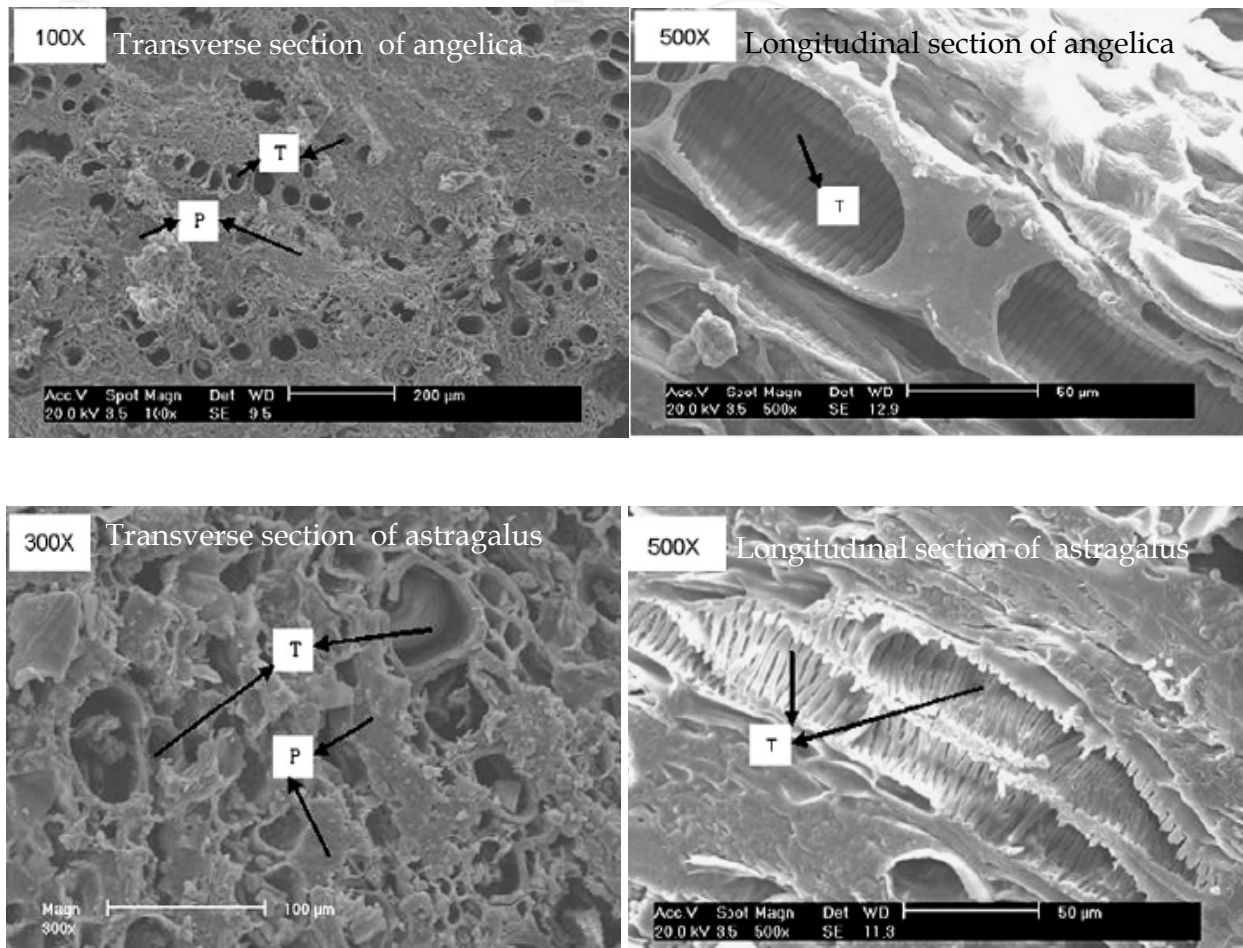


Fig. 6. The SEM micrographs of transverse section and longitudinal section of Chinese angelica slices and Astragalus sices (T part: trachea; P part: parenchyma cells). (Yang et al., 2010)

Thuwapanichayanan et al. (2011) studied the influence of drying temperatures on the moisture diffusivity and quality attributes of the dried banana slices in terms of volatile compound, shrinkage, colour, texture and microstructure. On the subject of microstructure, as shown in Figure7, they found that the drying temperature strongly affected the microstructure of dried banana and on the surface the pore sizes and the number of pores increased with increasing drying temperature, which significantly influenced the product texture in terms of hardness. The hardness of the samples dried at 90°C was lower than that dried at 70°C but was not significantly different from that dried at 80°C. This might be due to the effect of puffing that occurred more at higher temperatures and probably increased the porosity and resulted in a decrease of hardness and less shrinkage of the samples.

Brown et al. (2008) evaluated the microstructure characteristics of carrot pieces that had been dried using different techniques. They found that samples dried in ethano-modified supercritical carbon dioxide possessed many pores which could facilitate the movement of water into the internal structure and decrease the rehydrated and cooked time compared with the air-dried samples. Recently, similar results has been reported by Yang et al. (2010), who revealed that the larger porosity and total volume of the sample, the higher rehydration ratio of the sample.

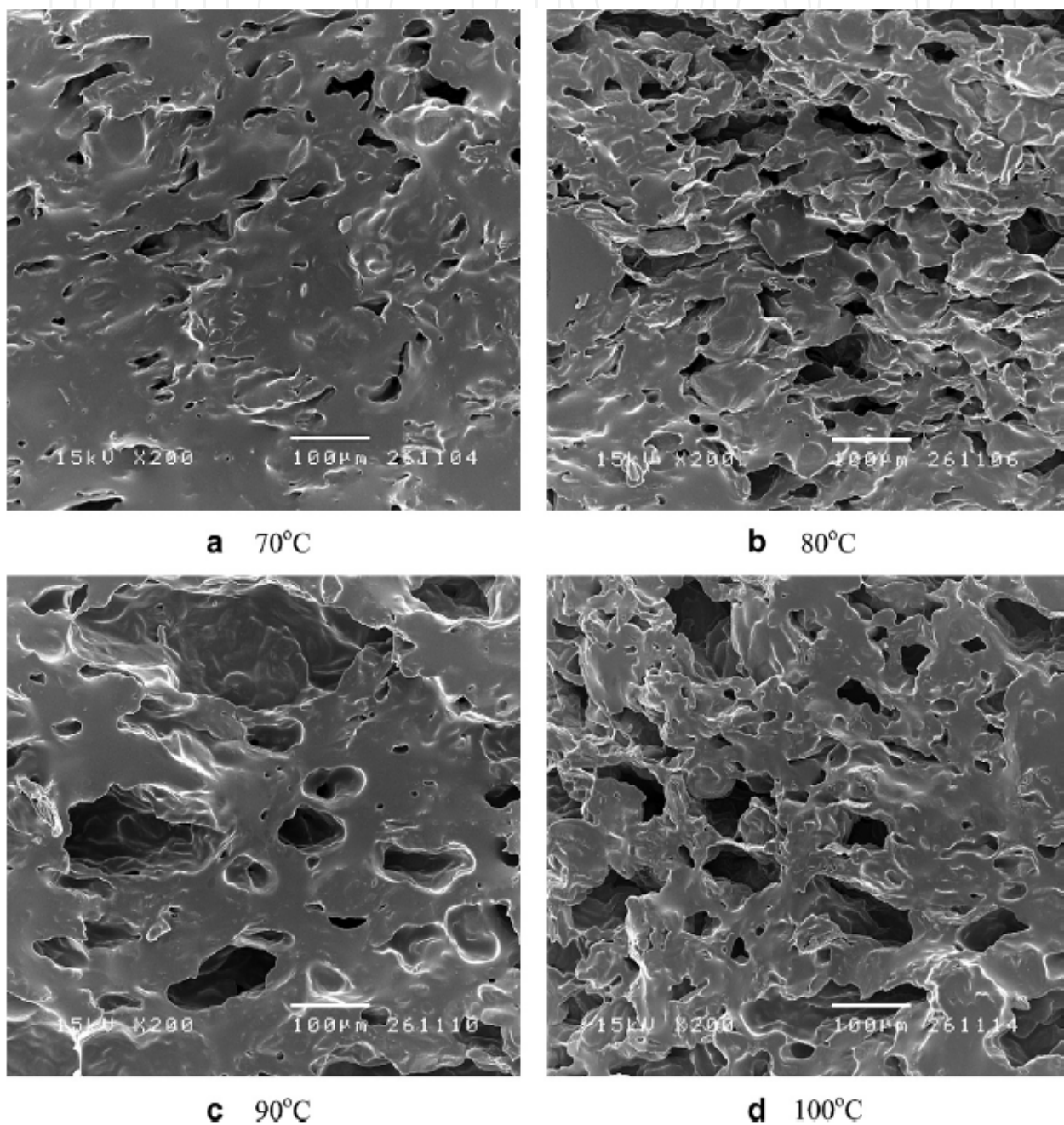


Fig. 7. SEM micrographs of banana slices dried at different temperatures (Thuwapanichayanan et al., 2011).

3. The causes leading to microstructure changes

The objective of this part: Analysis of the reasons behind the microstructure change phenomenon through specific case

3.1 Thermal and moisture gradients can cause cell wall disruption, deformation and folding during drying process

Deng & Zhao (2008b) explored the effect of different osmoconcentration pretreatment on glass transition temperature, texture, microstructure and calcium penetration of dried apples. In terms of microstructure, which was observed using SEM, it was found that it impossible to distinguish between cells and other spaces because of the disruption of cell walls, a decreased intercellular contact and the collapse of cell structure. Furthermore, they pointed that this phenomenon might be due to the fact that during drying process the transient thermal and moisture gradients causing cell wall breakdown, deformation and folding. Additionally, they also reported that the structural deformation, folding and collapses of cell structure might also associated with surface tension, environment pressure, moisture transport mechanism, and generation of internal pressure in samples during drying.

3.2 The microstructure changes of samples is closely associated with the stress developed in the tissue, which may be set up by shrinkage

Drying is a simultaneous heat and mass transfer process, shrinkage takes place when the materials are heated and moisture is lost. Since the composition of materials is very difference, take corn as an example which is constituted of soft floury endosperm, hard vitreous endosperm, germ and pericarp as shown in **Figure 8**. In addition, each substance has different shrinkage characteristics. These fact leads to the occurrence of non-uniform shrinkage during drying process. The non-uniform shrinkage within the product results in two types of internal stresses: the thermal stress which is due to temperature gradients within the material and the hydro stress which is due to moisture gradients within the product. When the combination of thermal and hydro stress exceeds the binding force between cells of the material stress crack or burst phenomena occur, which can change the macrostructure and microstructure of the product during drying process. As Wang and Brennan (1995) pointed that during drying internal cracks are formed and shrinkage stresses pull the tissue apart. Lewicki & Pawlak (2003) also demonstrated that physical changes are mostly due to stress and are pronounced by macro and micro alterations of size, shape and internal structure.

3.3 Stress in cell walls, phase changes in membrane lipids and chemical changes can also cause structural modifications of the product

Vega-Gálvez et al. (2011) explored different pretreatments such as high hydrostatic pressure, blanching, enzymatic and microwaves on the microstructure of Aloe vera gel during convective drying at 70°C, as shown in **Figure9**. It was found that the intact cellular structure of aloe was transformed into a more separated and ruptured cellular structure with non-distinct middle lamella. They ascribed this effect to the degradation of

pectinacious material and the damage of most cell wall due to excessive strain in membranes and stress in cell walls during processing.

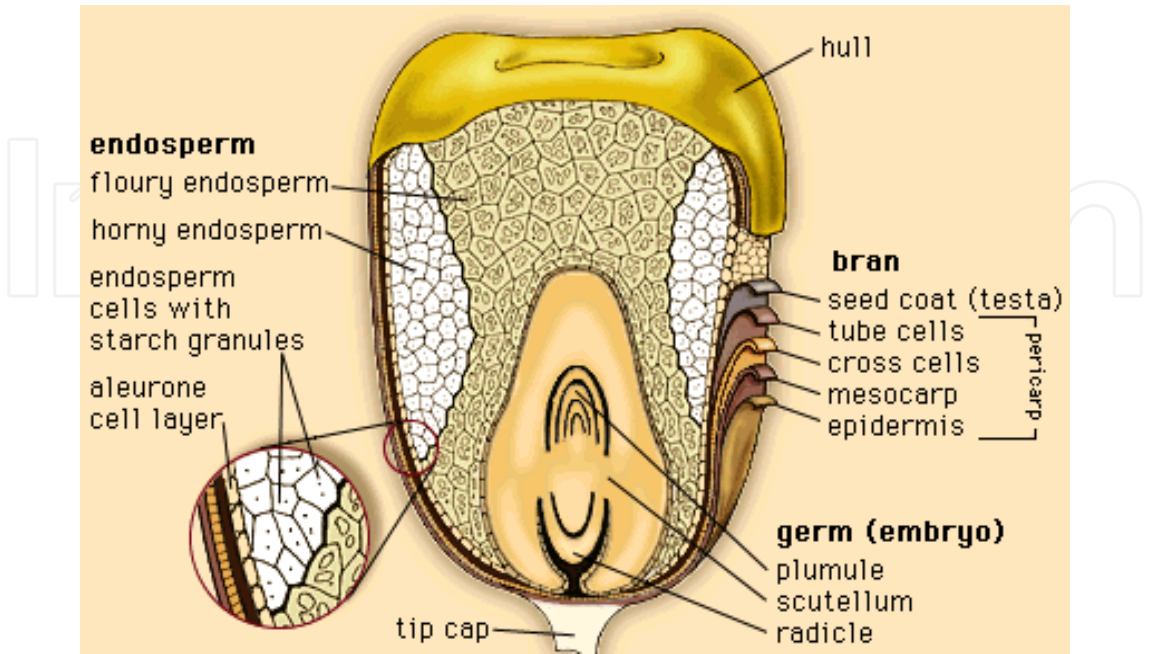
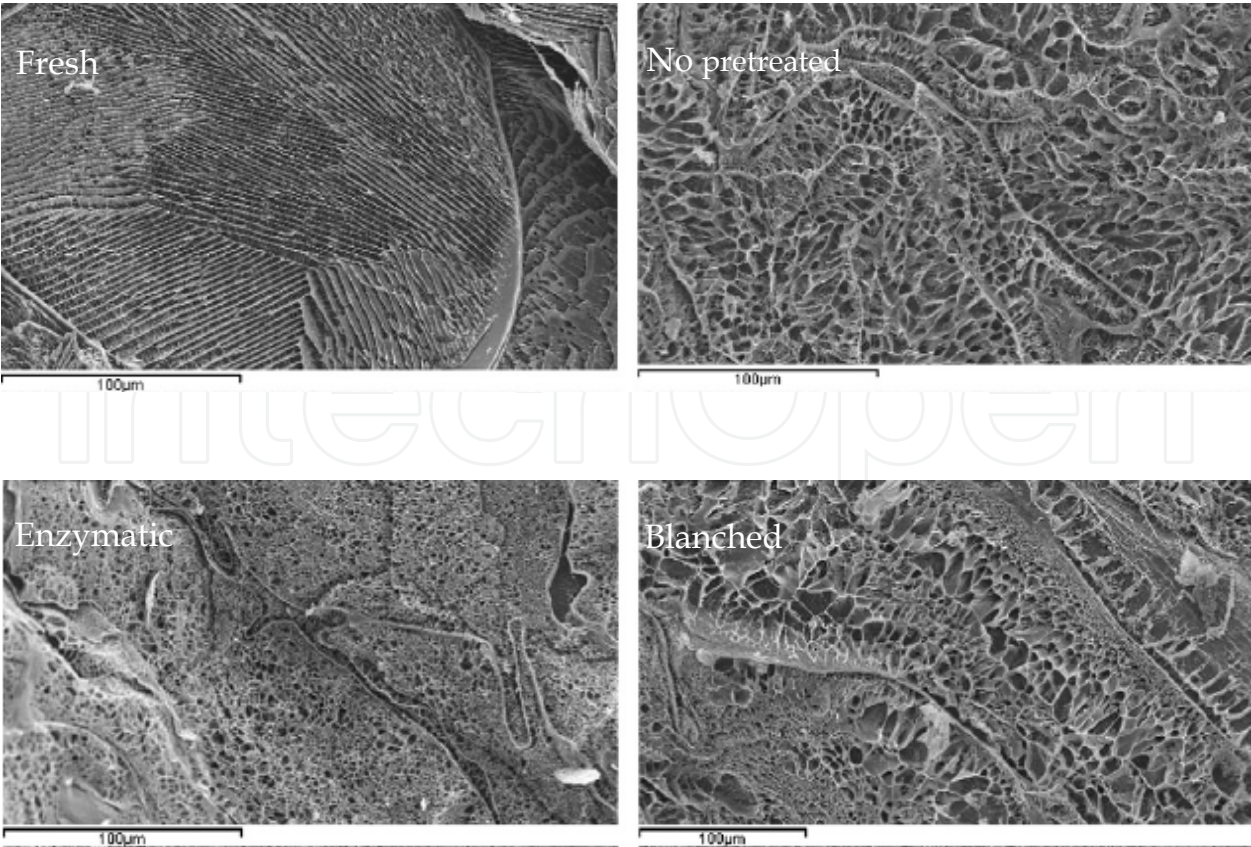


Fig. 8. The structure and compositon of corn kernel.



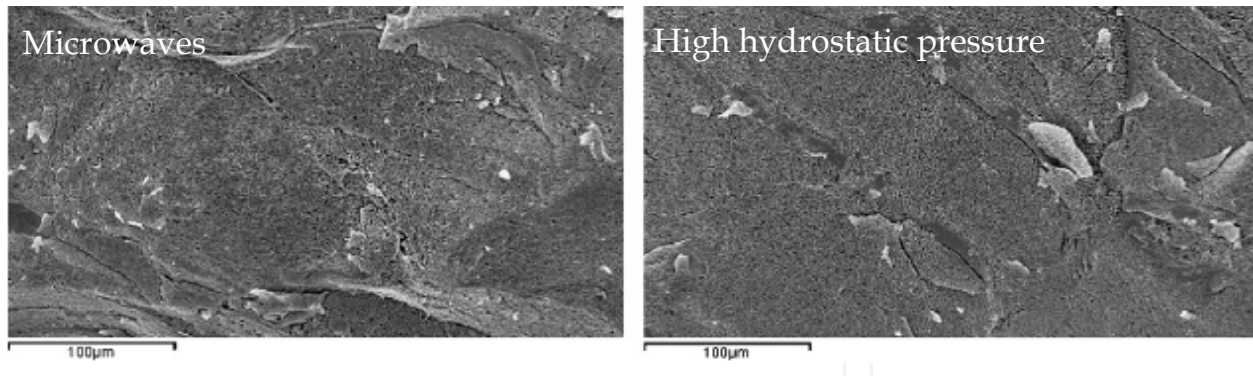


Fig. 9. The SEM micrographs of fresh and dried Aloe vera gel under different pretreatments (Vega-Gálvez et al., 2011).

4. Further research suggestion

The objective of this part: Point out the inadequacy of current research and the future research directions.

The microstructure of fresh and dried samples can provide a powerful tool and strong evidence for analyzing the properties changes of the samples during drying process. In this sense, research about the relationship between microstructure and the properties of the dried samples should be enhanced. In detail, more work needs to be performed on how the microstructure changes of dried food affects the food properties such as texture, rehydration ratio as well as its functionality, or even the availability of bioactive components.

Since information on microstructure change kinetics during products processing would be useful in predicting the quality changes during drying such as the texture and the surface shape of the product, thereby enabling better process control and improvement in the appearance by optimizing the process parameters. In addition, understanding the microstructure changes of the product is very important to clarify the change mechanism of quality during it processing. Therefore, the change kinetics of the microstructure during processing should be carried out in the further research.

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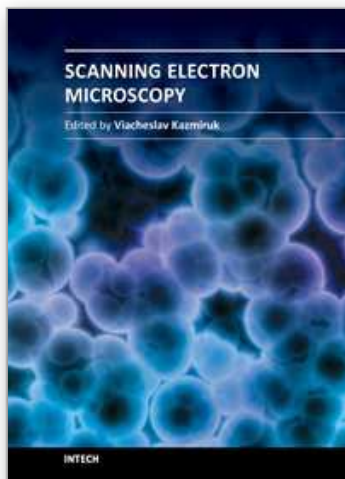
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Today, an individual would be hard-pressed to find any science field that does not employ methods and instruments based on the use of fine focused electron and ion beams. Well instrumented and supplemented with advanced methods and techniques, SEMs provide possibilities not only of surface imaging but quantitative measurement of object topologies, local electrophysical characteristics of semiconductor structures and performing elemental analysis. Moreover, a fine focused e-beam is widely used for the creation of micro and nanostructures. The book's approach covers both theoretical and practical issues related to scanning electron microscopy. The book has 41 chapters, divided into six sections: Instrumentation, Methodology, Biology, Medicine, Material Science, Nanostructured Materials for Electronic Industry, Thin Films, Membranes, Ceramic, Geoscience, and Mineralogy. Each chapter, written by different authors, is a complete work which presupposes that readers have some background knowledge on the subject.

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