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Study of Surface Morphology of Low-Pressure Plasma Treated Rice by SEM and Contact Angle Measurement

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ABSTRACT

This study focused on using low-pressure plasma processing on polished "white" rice and unpolished "riceberry" brown rice. Plasma treatment reduced the water contact angle on riceberry rice from 120.6°±1.0° to 89.8°±1.5°. Scanning electron microscope (SEM) images showed morphology of an external surface and a cross sectional view of the interior just below the surface of a rice kernel. The SEM image revealed a μm thin layer, 10-μm square-like pockets, and an endosperm of a few-mm across. The underlying pockets contain a lot of starch granules. SEM images also showed many pores on the top surface opening down to the pocket-like structure underneath after low-pressure plasma treatment. This effect caused the rice kernel to be more hydrophilic and agreed well with lower water contact angle. This work has demonstrated the feasibility of applying low-pressure plasma to improve the cooking quality of rice.

INTRODUCTION

Radio-frequency (RF) discharge is one of the most widely used forms of plasma discharge in many applications such as etching, deposition, surface modification, sterilization, etc. It was used to produce a large volume of stable plasma [1]. In principle, RF power is transferred to the working gas through an antenna to generate plasma. One form of RF antenna is an inductive coil, which can be applied either externally from outside or internally inside a plasma reactor. It is possible to use an external inductive coil with, for example, a low-pressure downer reactor and an RF power supply to generate low-pressure plasma for surface treatment of materials [2-3]. The plasma can remove substances from the material's surfaces by chemical reactions and physical etching [4].

Rice (Oryza sativa L.) is the most widely consumed staple food for a large part of the world's human population. The whole grain of rice consists of a hull, rice bran, germ and endosperm. Rice bran is a good source of protein, lipids, fiber and minerals, while the endosperm (the innermost part of the kernel) is mostly carbohydrate. This fact has been widely realized to indicate a higher nutritional value in brown rice, in which the rice bran is still left intact. For normal polished white rice, this bran layer is removed during milling process. It is interesting that the bran and the endosperm layers are affected differently under plasma treatment [5-8].

In this work, we applied a low-pressure plasma downer reactor at 13.56 MHz to two kinds of rice kernel. We would like to investigate the effects of low-pressure plasma treatment on physical properties of rice. For comparison, low-pressure treated and untreated rice was also investigated for each type of rice under study.

METHODOLOGY

Rice sample

Two types of rice were studied: white rice (Kor Khor type 47) and Riceberry rice (brown rice). They were gathered from Suan Dusit rice mill (Prachinburi, Thailand).

Plasma treatment setup

The low-pressure plasma system is shown in **Fig. 1** [9]. It is comprised of the following parts; (i) plasma is generated in a quartz tube with a diameter of 38 mm. (ii) A 10-turn coil is mounted around the tube and is connected to (iii) a RF power supply (13.56 MHz). The applied RF power was 100 W. Argon was fed at the flow-rate of 15 sccm during plasma generation. The pressure in the reactor was kept as low as two to three mbar. About one hundred grams of polished rice kernel (white rice) and non-polished riceberry kernel (brown rice) were placed into the sample storage container. The rice was allowed to pass through a feeding valve into the plasma discharge and eventually to a product-collecting vessel. The treatment was carried out 1 and 5 times for the white rice and the brown rice, respectively. Another treatment for each type of rice kernel was carried out by exposing the rice to only low-pressure with no plasma discharge. Henceforth, they are referred to as vacuum treated rice.

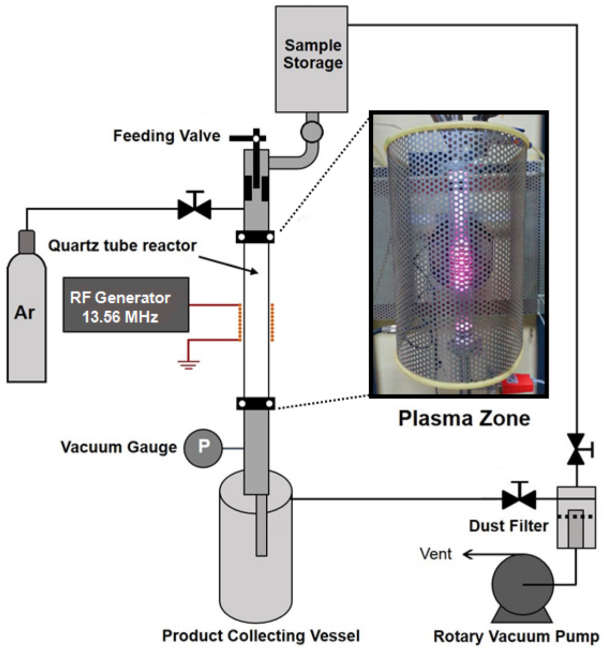

Figure 1 Schematic set up of plasma system

Water contact angle measurement

Contact angle measurement was performed at room temperature by the sessile drop method using a CCD camera. De-ionized water of 0.15 mm^3 was dropped from a microsyringe from the top of the sample surface. The angle of the liquid droplet was analyzed using ImageJ software. The average contact angle values and standard deviations were obtained from at least three different positions on the kernels.

Scanning electron microscopy (SEM)

The effects of plasma on rice surface was investigated by SEM (Model S-2500 Hitachi, Japan & JEOL Model 6610 LV, Tokyo, Japan). The samples were sputter-coated with gold prior to the analysis. We have made a comparison between plasma-treated and untreated rice kernels. The images were taken at 1000× magnification.

RESULTS AND DISCUSSION

Water contact angle

Water contact angle or surface wettability are summarized in **Table 1**. White rice has a lower water contact angle than riceberry rice because it has no hydrophobic substance (from the bran layer) left anymore. In the case of white rice, the rice kernel became chalky right after exposure to vacuum; hence no further experiment with plasma exposure was performed. We believe that a rapid loss of humidity from rice under vacuum was the cause. There was no such effect in the case of riceberry rice. Plasma treatment dramatically lowered the water contact angle of riceberry rice from 121° to 90° The intact of bran layer, which contains lipid or protein, prevented the riceberry rice from having an even lower contact angle. **Fig. 2** shows photographs of contact angle for two types of rice kernel under different treatment conditions. All kernel surfaces became more hydrophilic, as can be seen from the lower water contact angle, after plasma treatment. This allows water to be absorbed more efficiently.

Table 1 Contact angle of rice kernel

Sample	contact angle (θ)
WRU	69.3±1.1
WRV	65.9±3.4
WRP	na
RBU	120.6±1.0
RBV	129.1±1.2
RBP	89.8±1.5

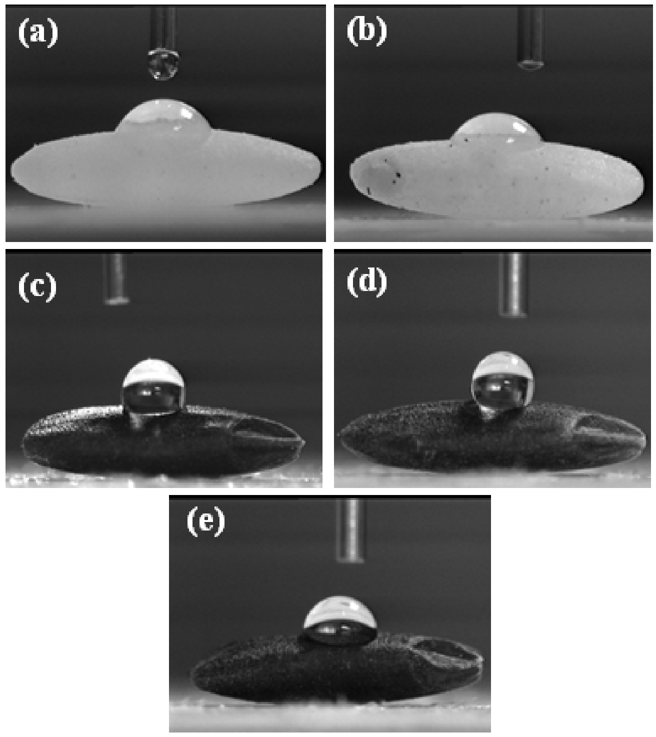

Figure 2 Contact angle of rice kernel; (a) untreated white rice, (b) vacuum treated white rice, (c) untreated riceberry, (d) vacuum treated riceberry, (e) plasma treated riceberry

Microstructure of rice kernel

Rice kernels are not homogeneous on their surface. Most areas of the surface are rather thick or "full grain" **(Fig. 3(a))**. One specific end of the brown rice is much slimmer; the so-called rice germ. This part of the kernel is lost for the polished white rice. The morphology of the two areas is very different as shown in **Fig. 3(b)** and **3(c)**. It is advantageous if plasma treatment can be confined to only the full

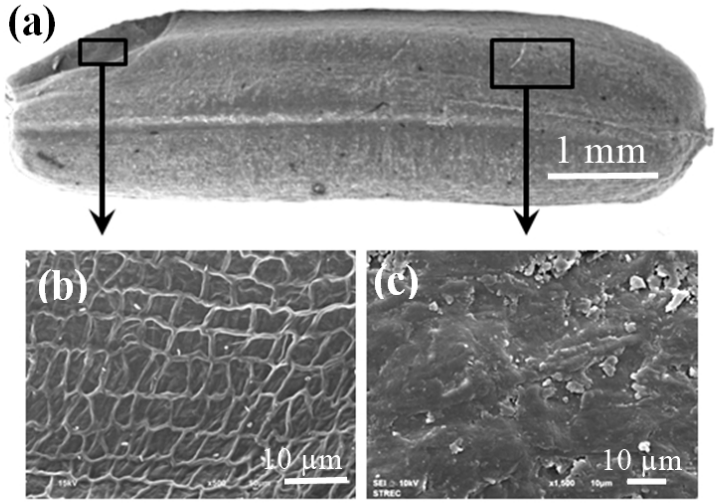

Figure 3 Structure of rice kernel; (b) zoomed in view of a section of rice germ and (c) kernel surface

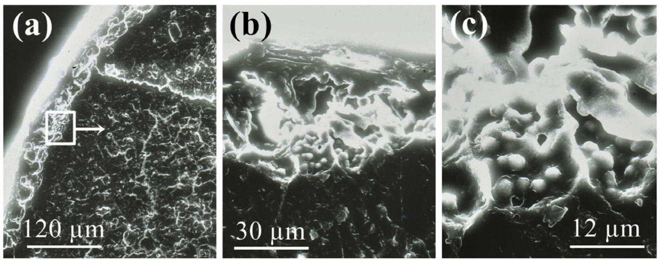

Figure 4 SEM images for the cross-sections of rice kernel; (a) the rice edge at low magnification. (b) a few pockets at higher magnification; (c) inside a pocket at further magnification.

grain area, which leads to better water absorption of the endosperm, hence linking directly to the cooking property of rice. Fig. 4 shows a cross-sectional micrograph of the brown rice, the bran layer and its details. Under the bran layer, there are many pocket-like structures (as shown in Fig. 4(b)). In each pocket, there are a lot of starch granules. These granules, also found in the inner part, play very important roles in determining the cooking properties of the kernel. For any modifications to work, any opening or pores reaching to these pockets, or further in, are crucial.

Microstructure of rice surface

Rice kernel surfaces before and after plasma treatment are shown in Fig. 5 at 1000× magnification. The kernel surfaces of the untreated group (Figure 5(a)) look smoother than those of the vacuum treated group (Fig. 5(b)). Under vacuum, white rice became chalky as mentioned earlier. There were also a few openings exposing the underlying pockets. The degree of modification by vacuum is much lower on the riceberry rice (see Fig. 5 (d) and (e)) because of the presence of the bran layer. The degree of damage has significantly increased with plasma treatment as can be seen from Fig. 5 (b) to (c) and Fig. 5 (e) to (f), in the case of white rice and brown riceberry rice, respectively. This leads to higher exposure of the underlying "starch" part of rice to water during the cooking process, for example. This tends to lower the degree of contact angle as shown in the previous topic, and leads to higher water absorption and reduced cooking time to a few ten percents (in a separate study and not shown in contribution).

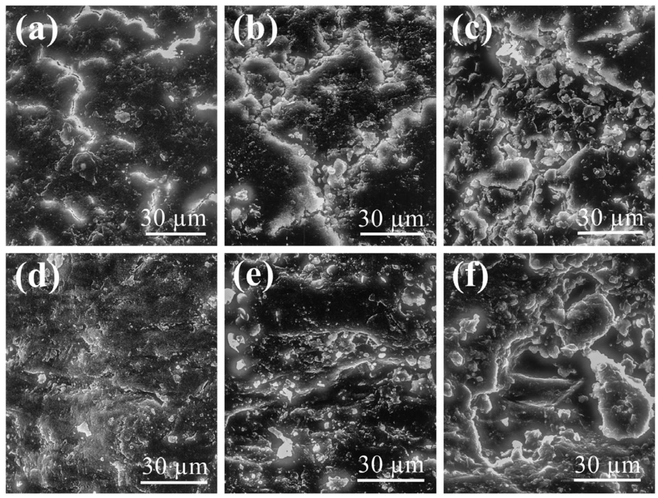

Figure 5 SEM micrographs (at 1000x magnification) of rice kernel; (a-c) white rice and (d-f) brown riceberry rice. (a) untreated white rice, (b) vacuum treated white rice, (c) plasma treated white rice, (d) untreated riceberry, (e) vacuum treated riceberry, (f) plasma treated riceberry.

CONCLUSION

This work has shown that low-pressure plasma treatment changes rice surface morphology significantly. The degree of changes varies with prior processing of the rice, between polished (white) or non-polished (brown) rice. The interactions of plasma treatment caused the rice surface to crack. After plasma treatment, the plasma treated rice had decreased water contact angle and absorbed water easier than the untreated rice. Changes in the structure of the outer layer down into the underlying layer demonstrated the effect of plasma treatment and were clearly observed under SEM micrographs. The potential for applying low-pressure plasma as a non-chemical means for rice quality improvement can be realized. The downer reactor under this study also demonstrates the possibility to scale the treatment to larger scale.

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