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# อนุภาคทรงกลมของแป้งข้าวเหนียวที่ผลิตโดยใช้เทคนิคการพ่นแห้งแบบละอองฝอย

# The spherical particles of glutinous rice starch produced by spray drying technique

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## Abstract

The spray drying technique has been widely used for drying heat-sensitive materials, pharmaceuticals, and other substances. Because it leads to rapid solvent evaporation from droplets. In addition, this process is also useful as a method for agglomeration and changing in some of the physicochemical properties of powdered materials. Therefore, this work proposes the use of a spray dryer system to produce spherical particles for improving the fluidity of solid. The slurries of glutinous rice starch containing with several kinds of additives were spray dried using a pneumatic atomizer. The adsorbents such as Aerosil, Avicel PH102, plasticizer such as polyethylene glycol 400 (PEG400), stabilizers such as polyvinylpyrrolidone (PVP K30), polyvinyl alcohol (PVA), and fillers such as lactose, maltodextrin were used as the additives in this study. The particle size and size distribution were determined by Mastersizer. The morphology were examined by scanning electron microscopy (SEM). The results showed that the particle size and the morphology of modified glutinous rice starch which produced by spray drying was bigger and the shape was more sphere than the native glutinous rice starch. Although, the 20% maltodextrin addition could produce the biggest particle size when compared with the other additives but it didn't produced more spherical shape of product. Thus, it is possible that the glutinous rice starch can be modified and produced to the spherical particle using spray drying technique by its own natural properties.

**Key words:** spray drying, glutinous rice starch, scanning electron microscopy

## บทคัดย่อ

เทคนิคการพ่นแห้งแบบละอองฝอยถูกใช้อย่างกว้างขวางสำหรับการทำแห้งสารวัตถุดิบที่ไวต่อความร้อน, สารทางเภสัชกรรม, และสารอื่นๆ เนื่องจากกระบวนการดังกล่าวจะนำไปสู่การทำให้เกิดการระเหยของตัวทำละลายอย่างรวดเร็วจากหยดน้ำ กระบวนการนี้เป็นวิธีการที่ทำให้เกิดการจับตัวกันเป็นกลุ่มก้อนและยังส่งผลถึงการเปลี่ยนแปลงคุณสมบัติทางเคมีกายภาพบางประการของสารที่ได้ ดังนั้นวัตถุประสงค์ของงานวิจัยนี้เพื่อใช้ระบบการพ่นแห้งแบบละอองฝอยในการผลิตอนุภาคทรงกลมสำหรับการพัฒนาสภาพของไหลของสารแห้ง โดยสารขั้นเหนียวของแป้งข้าวเหนียวดัดแปรซึ่งเติมด้วยสารช่วยประเภทต่างๆจะถูกพ่นเป็นละอองฝอยและทำให้แห้งโดยใช้หัวพ่นแบบใช้กำลังอัดของอากาศ โดยสารช่วยดูดซับ เช่น Aerosil, Avicel PH102, สารเพิ่มความยืดหยุ่น เช่น polyethylene glycol 400 (PEG400), สารเพิ่มความคงตัว เช่น polyvinylpyrrolidone (PVP K30), polyvinyl alcohol (PVA) และสารเพิ่มปริมาณ เช่น lactose, maltodextrin จะถูกใช้เป็นสารช่วยในการศึกษาครั้งนี้ ขนาดอนุภาคและการกระจายตัวของอนุภาคจะถูกวัดค่าโดยเครื่อง Mastersizer สัณฐานวิทยาของผลิตภัณฑ์ถูกตรวจสอบโดยกล้องจุลทรรศน์อิเล็กตรอนแบบส่องกราด (เอสอีเอ็ม) ผลการทดลองแสดงว่าขนาดอนุภาคและสัณฐานวิทยาของอนุภาคแป้งข้าวเหนียวดัดแปรซึ่งผลิตโดยใช้เทคนิคการพ่นแห้งแบบละอองฝอยมีขนาดอนุภาคใหญ่ และเป็นทรงกลมมากกว่าแป้งข้าวเหนียวดิบ แม้ว่าการเติม 20% maltodextrin จะสามารถผลิตขนาดอนุภาคที่ใหญ่ที่สุดเมื่อเปรียบเทียบกับสารช่วยชนิดอื่น แต่ก็ไม่สามารถผลิตผลิตภัณฑ์ให้มีอนุภาคที่มีความเป็นทรงกลมได้มากขึ้น ดังนั้นอาจเป็นไปได้ว่าแป้งข้าวเหนียวสามารถถูกดัดแปรและถูกผลิตให้เป็นอนุภาคทรงกลมได้ โดยการใช้เทคนิคการพ่นแห้งแบบละอองฝอยด้วยคุณสมบัติธรรมชาติของแป้งข้าวเหนียวเองได้

**คำสำคัญ**: การพ่นแห้งแบบละอองฝอย, แป้งข้าวเหนียว, กล้องจุลทรรศน์อิเล็กตรอนแบบส่องกราด (เอสอีเอ็ม)

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## Introduction

Glutinous rice or sticky rice (*Oryza sativa* L.) (Eliasson, 2004) is a kind of rice commonly cultivated in Thailand, especially in the northeast area. The glutinous rice starch is a natural hydrophilic polymer. The glutinous rice starch is contained almost 100% amylopectin. The native glutinous rice starch is insoluble in cold water (Sodhi and Singh, 2003). Many different methods of modifying starch are widely used as means of changing native starch functionality. The physical modification of glutinous rice starch that can dissolve and swelling in cold water is an interesting modification for novel excipients. Spray drying is by definition the transformation of a pumpable feed from a fluid state (solution, dispersion, or emulsion) into a dried particulate form by spraying the feed into a hot drying medium. This technique involves evaporation from an atomized feed by mixing the spray and the drying medium. Due to rapid solvent evaporation, the temperature of the droplets can be kept far below the drying air temperature; for this reason, spray drying can be applied to both heat-resistant and heat-sensitive materials. This may result in more desirable characteristics in the dried product. There have been no studies on the physical properties of starch powders agglomerated by spray drying or the effect of the composition of the formulation prior to spray drying on these properties. Thus, the purposes of the present study were produce spherical particles of glutinous rice starch with various additives by spray drying technique.

## Materials and methods

### Preparing the slurry of glutinous rice starch

6% starch slurry were prepared in the reactor. The degree of starch gelatinization condition was controlled at the temperature of 75ºC for 12 minutes. The additive suspension were added into the starch slurry. The additives were 0.5% Aerosil, 5% Avicel PH102 as adsorbent, 3% PEG400 as plasticizer, 10% PVP K30, 5% PVA as stabilizer, and 10% lactose , 20% maltodextrin as fillers. This slurry was spray dried.

### Particle size and size distribution

The modified glutinous rice starch and modified glutinous rice starch with additives were determined the particle size and size distribution by Mastersizer which used a dry cell to detect the average mean diameter (n=9).

### Morphology

The appearance, size and shape of the powder samples were examined with scanning electron microscope (SEM). The samples were mounted on an aluminum stub using a double sided adhesive tape and thereafter making it electrically conductive by coating with a thin layer of gold in vacuum. The scanning electron microscope was operated at an acceleration voltage of 15 kV.

## Results

Table 1 The particle size and size distribution of modified glutinous rice starch with various additives

| Sample | | $D_{10}$ (μm) | $D_{50}$ (μm) | $D_{90}$ (μm) |
|---|---|---|---|---|
| Pregel | Additives | | | |
| - | 100% Native GRS | 5.042±0.3 | 14.80±0.14 | 38.60±0.72 |
| 100%Pregel | - | 7.03±0.45 | 19.92±0.24 | 46.41±0.86 |
| 100%Pregel | 0.5%Aerosil | 6.8±0.2 | 18.43±0.42 | 39.2±0.92 |
| 100%Pregel | 50%Avicel PH102 | 6.39±0.18 | 19.83±0.24 | 46.85±0.78 |
| 100%Pregel | 3%PEG400 | 7.86±0.08 | 20.53±0.12 | 43.64±0.55 |
| 100%Pregel | 10%PVP K30 | 8.56±0.3 | 20.9±0.21 | 42.48±0.05 |
| 100%Pregel | 5%PVA | 7.48±0.45 | 20.7±0.67 | 46.75±0.95 |
| 100%Pregel | 10%Lactose | 7.57±0.2 | 19.6±0.46 | 43.45±1.54 |
| 100%Pregel | 20%Maltodextrin | 9.41±0.19 | 26.14±04 | 53.73±0.17 |

The results from Table 1 showed that the particle size of modified glutinous rice starch were 19.92±0.24μm which is bigger than the native glutinous rice starch. The particle size of modified glutinous rice starch with various additives were in the range of 18-26 μm. The 20% maltodextrin addition could produce the biggest particle size at 26.14±04 μm when compared with the other additives.

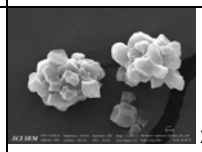
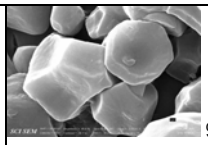
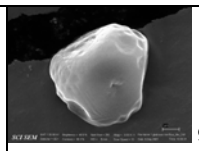
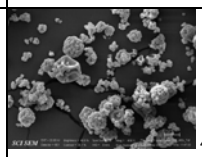
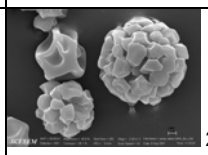
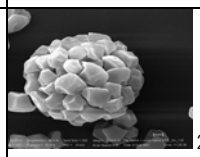
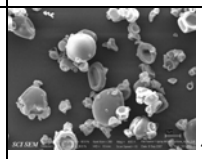
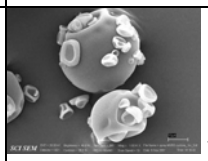
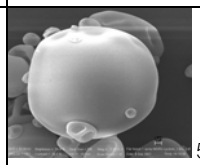
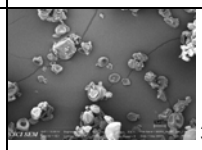
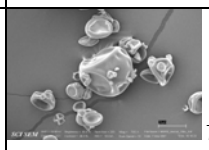
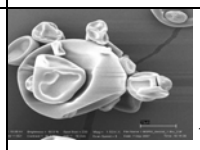
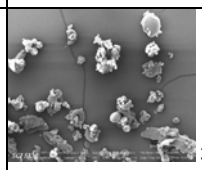
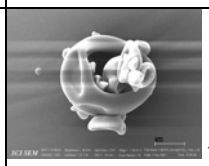
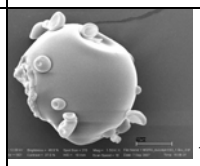
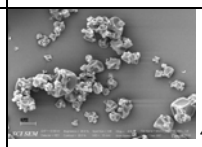
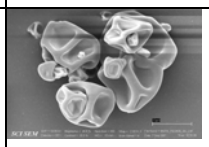
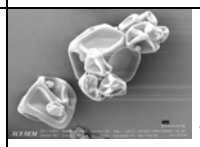
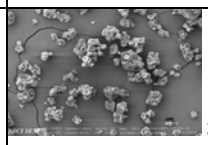
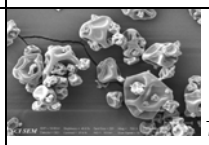
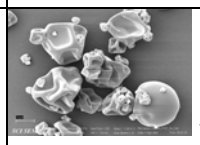
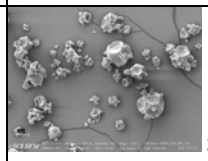
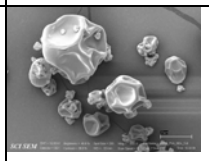
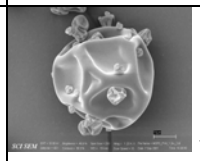
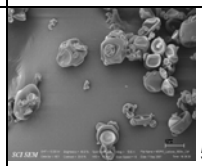
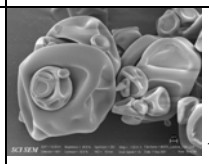
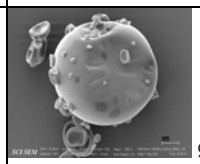
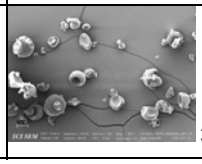
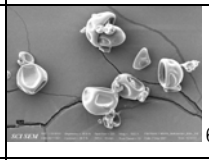
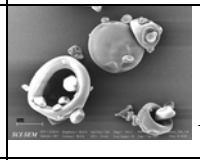
| Sample | Morphology | | |
|---|---|---|---|
| Native GRS |  2000X |  9000X |  9000X |
| Spray native GRS |  400X |  2000X |  2000X |
| Spray MGRS |  400X |  1000X |  500X |
| Pregel + Aerosil |  300X |  700X |  1500X |
| Pregel +Avicel PH102 |  300X |  1500X |  1500X |
| Pregel+ PEG400 |  400X |  2000X |  1200X |
| Pregel + PVP |  300X |  700X |  1000X |
| Pregel + PVA |  300X |  800X |  1200X |
| Pregel+ Lactose |  500X |  1800X |  900X |
| Pregel + Maltodextrin |  300X |  600X |  700X |

Figure 1 The morphology of modified glutinous rice starch with various additives

The morphology of the native of glutinous rice starch was irregular shape, but after spray dry produced the agglomerate particle to spherical shape. The spray dry of pregelatization starch was spherical particle. When added with various additives, it produced the particle shape wasn't more spherical.

## Discussion

The particle size and the morphology of modified glutinous rice starch which produced by spray drying was bigger and the shape was more sphere than the native glutinous rice starch. Although, the 20% maltodextrin addition could produce the biggest particle size when compared with the other additives but it didn't produced more spherical shape of product. Thus, it is possible that the glutinous rice starch can be modified and produced to the spherical particle using spray drying technique by its own natural properties.

## Summary

Thus, it is possible that the glutinous rice starch can be modified and produced to the spherical particle using spray drying technique by its own natural properties.

## Acknowledgements

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## Literature cited

Berggren J., G. Frenning and G. Alderborn. 2004. Compression behaviour and tablet-formimg ability of spray-dried amorphous composite particles. **Eur J Pharm Sci**. 22(2-3): 191-200.

Eliasson A.C., editor. 2004. **Starch in Food**. New York, CRC.

Follain, N., C. Joly, P. Dole and C. Bliard. 2005. Properties of starch based blends. Part 2. Influence of poly vinyl alcohol addition and photocrosslinking on starch based materials mechanical properties. **Carbohydr Polym**. 60: 185-92.

Jonas B., F. Goran, A. Goran. 2004. Compression behaviour and tablet-formimg ability of spray-dried amorphous composite particles. **Eur J Pharm Sci**. 22(2-3): 191-200.

Luz P.P, A.M. Pires and O.A. Serra. 2007. A low-cost ultrasonic spray dryer to produce spherical microparticles from polymeric matrices. **Quim Nova**. 30(7).

Maury M., K. Murphy, S. Kumar, L. Shi and G. Lee. 2005. Effects of process variables on the powder yield of spray-dried trehalose on a laboratory spray dryer. **Eur J Pharm Bio**. 59(3): 565-73.

Pringels E., D. Ameye, C. Vervaet, P. Foreman and J.P. Remon. 2005. Starch/Carbopol spray-dried mixtures as excipients for oral sustained drug delivery. **J Controlled Release**. 103(3): 635-41.

Ravenelle F., R.H. Marchessault, A. Legare and M.D. Buschmann. 2002. Machanical properties and structure of swollen crosslinked high amylase starch tablets. **Carbohydr Polym**. 47(3): 259-66.

Shogren R.L., J.W. Lawton and K.F. Tiefenbacher. 2002. Baked starch foams: starch modifications and additives improve process parameters, structure and properties. **Industrial Crops and Products**. 16: 69-79.

Smits A.L.M., P.H. Kruiskamp, J.J.G.V. Soest and J.F.G. Vliegenthart. 2003. Interaction between dry starch and plasticizers glycerol or ethylene glycol, measured by differential scanning calorimetry and solid state NMR spectroscopy. **Carbohydr Polym**. 53: 409-16.

Soares L.A., G.G. Ortega, P.R. Petrovick and P.C. Schmidt. 2005. Dry granulation and compression of spray-dried plant extracts. **AAPS Pharm Sci Tech**. 6(3): E359E366.

Sodhi, N.S. and N. Singh. 2003. Morphological, thermal and rheological properties of starch separated from rice cultivars grown in India. **Food Chem**. 80: 99-108.