



## Original Research Article

# Effects of beeswax on properties and biodegradation of baked corn starch foam

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### ABSTRACT

In this study, baked starch foam was prepared from corn starch, using water as a non-toxic blowing agent. Some additives e.g., guar gum, magnesium stearate, kaolin, and glycerol, were added to improve the properties of the foam. Chemical structure of the foam was confirmed by Fourier-transform infrared spectroscopy (FT-IR). Scanning electron micrographs (SEM) revealed that the foam showed smooth surface with the addition of the additives. When beeswax was added, the foam surface roughness increased with increasing beeswax concentration. Hardness test (Shore C) showed a slight increase in the foam hardness when beeswax concentration increased. Moreover, it was found that the moisture absorption capacity of the foam at 50 %RH decreased with increasing beeswax concentration. Water resistance test confirmed that beeswax played an important role in the dimensional stability of the foam while soaking in water for 12 hrs at room temperature (29 °C). Convincingly, the baked foam prepared from corn starch mixed with the additives and 20 parts per hundred (phr) of beeswax showed the best properties among the developed foams. Besides, such the foam can be totally degraded in soil mainly via hydrolysis.

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## INTRODUCTION

With increasing environmental problems caused by petroleum-based plastics, biodegradable plastics have been received an increasing attention recently. Among them, starch is one of the most interesting biodegradable materials for single use packaging application owing to its low cost, natural abundance, and renewability (Kaewtatip *et al.*, 2018). Undoubtedly, the most commonly used foam packages in current markets are expanded polystyrene foams (EPS) that require more than hundred years to degrade. The development of starch-based foams is the most commonly used way to substitute the traditional petroleum-based foams. Besides, there are many techniques used to produce starch foam, such as baking or compression, extrusion, injection and microwave heating (Sanhawong *et al.*, 2017). Unfortunately, starch foams exhibit high water absorption that affects their mechanical properties and this drawback needs to be resolved.

Previous studies reported that both hydrophobic and hydrophilic additives can be used to reduce water absorption of starch foam. Those additives included cellulose nanofiber (Ghanbari *et al.*, 2018), sesame cake (Machado *et al.*, 2017), corn husk fiber, kaolin and beeswax (Polat *et al.*, 2013), and natural rubber (Sanhawong *et al.*, 2017).

It is well known that hydrophobic fillers are attractive fillers responsible for the reduced water absorption of the matrix composited with them. Beeswax is a natural wax recognized as a hydrophobic filler. It consists of complex mixtures of hydrocarbons C25–C33, (12–16%), free fatty acids C24–C32 (12–14%), esters of fatty acids C40–C48 (35–45%), complex wax esters (15–27%) and fatty alcohol C28–C35 (1–2%) (Polat *et al.*, 2013). With the addition of beeswax, the hydrophobicity of the starch foam is expected to increase.

In this study, baked biofoam was prepared from corn starch by compression, using water as a non-toxic blowing agent to create pores inside the foam matrix. Chemical structure and morphology of the foam were investigated using Fourier-transform infrared spectroscopy (FT-IR) and scanning electron microscopy (SEM), respectively. Effects of beeswax concentration on the properties of the foam, e.g. density, surface roughness, hardness, moisture absorption capacity and dimensional stability were also studied. Finally, biodegradation behavior of a selected foam was investigated by soil burial test.

## MATERIALS AND METHODS

### Chemicals and reagents

Corn starch was purchased from Bangkok Interfood Co., Ltd. Guar gum, beeswax, kaolin, and magnesium stearate (food-grade materials) were from Chemipan Co., Ltd. Glycerol (analytical grade) was from Orec. Calcium chloride (analytical grade) was from Merck. All chemicals were used as received without any further purification.

### Preparation of baked corn starch foam

Several formulations of corn starch foams (Table 1) were prepared using a compression molding machine (Chareon Tut, PR1D-W300L300 HD, Thailand). Corn starch, distilled water, and all additives were mixed using a kitchen aid mixer at room temperature (28–29 °C) for 5 min or until a well dispersion of all the ingredients was obtained. The homogenous batter was poured into a 300 × 300 × 3 mm<sup>3</sup> mold, which was placed in the compression molding machine. The mold temperature was set at 220 °C. The process was carried out at a pressure of 1000 atm for 4.30 min.

### Confirmation of chemical structure

The functional groups of the developed corn starch foam were analyzed by Fourier-transform infrared spectrometer (model system 2000FT-IR, Perkin Elmer, USA) at wavenumbers 400 to 4000 cm<sup>-1</sup>.

### Surface morphology

The cross-sectional morphologies of the corn starch foams were examined by scanning electron microscopy (SEM) (Angstrom Scientific, USA). The dried foam was mounted on a metal stub and coated with gold. SEM micrographs of the foams were recorded on a LEO-1450VP SEM instrument at an accelerating voltage of 15 kV.

### Hardness test

Hardness of the starch foam was measured using Shore C durometer. The test measured the penetration of a specified indenter into the foam surface. The foam sample was cut into a 12 cm × 3 cm × 0.3 cm specimen and conditioned at 29 °C and 50%RH for 14 days prior to the test. The specimen was first placed on a hard flat surface. The indenter for the instrument was then pressed into the specimen ensuring that it was parallel to the surface. The maximum hardness number was read from the scale within one second of firm contact with the specimen.

### Determination of moisture absorption capacity

The moisture adsorption of a foam specimen with the dimension of 2.5 cm × 2.5 cm × 0.3 cm at 50% RH was examined by a gravimetric method. Prior to the test, five specimens of each sample were dried in an oven at 105 °C for 24 h. The specimen was weighed ( $W_0$ ) before placing it into a desiccator in which the RH was controlled at 50% (saturated CaCl<sub>2</sub> solution). Then, the weight of the specimen ( $W_t$ ) was recorded every hour for 12 h. The moisture content was calculated by the following equation:

$$\text{Moisture content (\%)} = ((W_t - W_0) / W_0) \times 100 \quad (1)$$

### Dimensional stability after water soaking

Water resistance of corn starch foams was investigated by witnessing the dimensional stability of a foam after soaking in water. The dimensional stability of the specimen was tested by soaking a specimen with dimensions of 2.5 cm × 5.0 cm × 0.3 cm into 100 mL of distilled water for 24 h. The dimensional change was observed by visualization. Digital photographs of the test specimen were taken intermittently.

### Biodegradation behavior by soil burial test

The soil burial test was carried out on a laboratory scale. The foam specimens with the dimension of 3 cm × 3 cm × 0.3 cm were buried in plastic cups containing gardening soil, which were placed approximately 8 cm from the soil surface. The soil water content was controlled at 40–50%. The pH and temperatures of the soil were 6.5 and 21.0–26.0 °C, respectively. The digital photographs of the tested specimens were recorded every week.

## RESULTS AND DISCUSSION

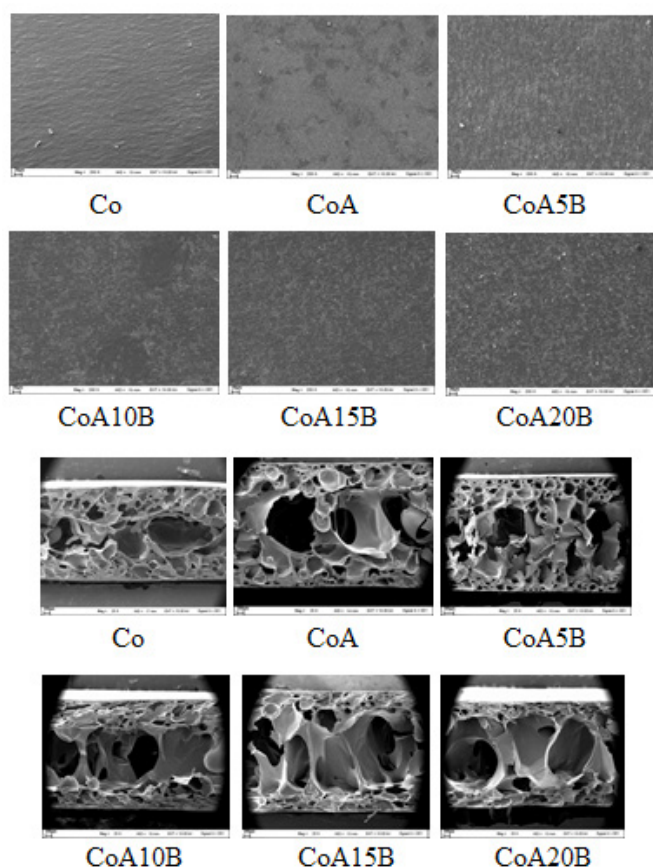
### Processing and morphology

During the processing of the corn starch foams, the starch must gelatinize and dry, while water evaporates. The amount of water used in each foam formulation is shown in Table 1. Except for control (Co), the amount of water was fixed at 115 mL. This amount was suitable to produce the starch batter with proper viscosity. The mold temperature and processing pressure were set at 220 °C, and 1000 atm, respectively, while the processing time was 4.30 min. With suitable conditions and appropriate batter density the water can evaporate properly and the foam structure can be produced (Uslu and Polat, 2012).

**Table 1. List of foam formulations and compositions.**

Sample name	Corn starch (g)	Water (mL)	Fixed-amount additives				Beeswax (g)
			Guar gum (g)	Glycerol (mL)	Magnesium stearate (g)	Kaolin (g)	
Co	100	100	0	0	0	0	0
CoA	100	115	1	4	2	15	0
CoA-5B	100	115	1	4	2	15	5
CoA-10B	100	115	1	4	2	15	10
CoA-15B	100	115	1	4	2	15	15
CoA-20B	100	115	1	4	2	15	20

The morphologies of the developed corn starch foams were displayed in Figure 1. It's clearly seen from the figure that all the developed foams exhibited a sandwich type structure with small, dense closed cells at the exterior and large, expanded open cells in the interior. This structure is typical for starch foam prepared by compression. (Machado et al., 2017). Without any additives, Co exhibited smooth surface. With the addition of fixed-amount additives (guar gum, magnesium stearate, kaolin, and glycerol), the density of the batter increased resulting in the difficulty of water to evaporate.

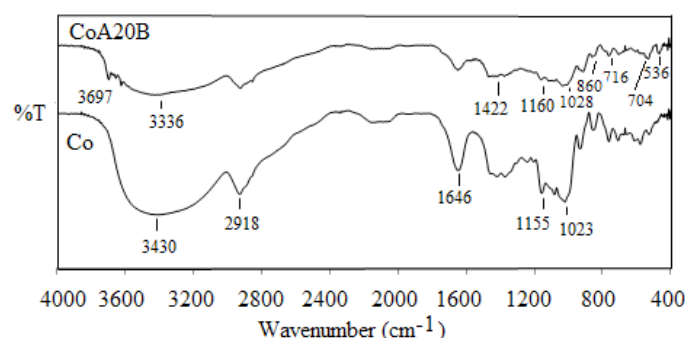


**Figure 1.** Scanning electron micrographs showing surface and cross-sectional morphologies of the developed foams with the magnifications of 200X (surface) and 20X (cross-sectional). (Please specify magnification)

This leads to the increased size of the open cells. Although the phase separation seems to be noticeable owing to poor mixing, CoA shows smoother surface than that of Co. This may be due to the water soluble ability and/or the compatibility with starch of all the added additives. When beeswax was added, the foam surface roughness increased with increasing beeswax concentration, while the size of the open cells firstly decreased at 5 phr of beeswax, but increased with increasing beeswax concentration afterwards. This was because with small amount of beeswax, the incompatibility between aqueous and wax reduced the molecular interactions between wax and starch batter, allowing water to evaporate easier. This resulted in smaller size of the cells. With increased amount of beeswax, although some degree of phase separation occurred (as evidenced by rough surface), the density of the batter became higher. Batter water could evaporate through the mold after getting enough amount and evaporation energy, resulting in large cells with thin cell walls and some ruptured cells.

#### Chemical structure

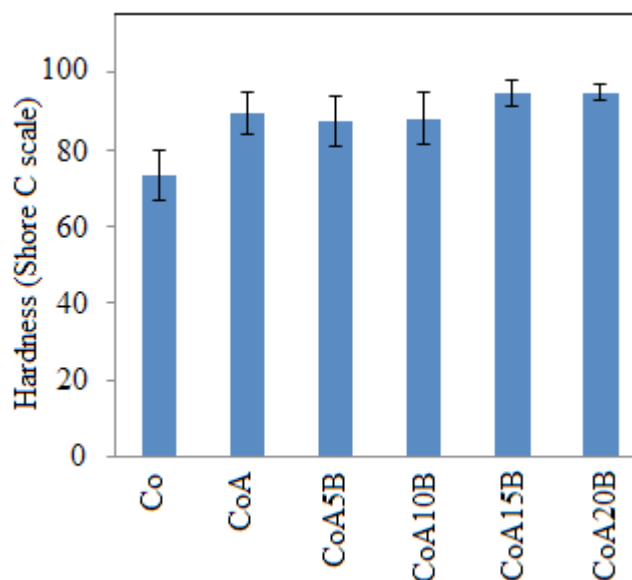
The functional groups of the developed corn starch foams, namely Co and CoA20B, were analyzed and the FTIR spectra of the selected samples were shown in Figure 2.



**Figure 2.** FTIR spectra of two selected developed foams, namely Co and CoA-20B.

Co displayed typical peaks of starch: O-H stretching at 3430 and 1646  $\text{cm}^{-1}$ , C-H stretching at 2918  $\text{cm}^{-1}$  of aldehyde group (CHO), and C-O-C stretching of glycosidic bond at 1155 and 1023  $\text{cm}^{-1}$  (Cremer and Kaletunc, 2013). Those starch characteristic peaks found in Co were also found in CoA20B with relatively lower intensity. Besides, the characteristic peaks of the additives added were found in CoA20B. O-H stretching of glycerol was found at 3336  $\text{cm}^{-1}$  (Liu et al., 2011). C=O stretching of magnesium stearate was found at 1422  $\text{cm}^{-1}$  (Javadzadeh et al., 2013), while that of beeswax was found at 1160  $\text{cm}^{-1}$  (Muscat et al., 2014). C-O-C stretching of guar gum was found at 1028  $\text{cm}^{-1}$  (Shahid et al., 2013). The

characteristic peaks of kaolin were found at 3697 cm<sup>-1</sup>, and 1157 cm<sup>-1</sup>, corresponding with O–H stretching and Si–O stretching, respectively. The peaks at 860, 761 and 704 cm<sup>-1</sup> are due to Al–O bond in kaolin. Moreover, a peak at 536 cm<sup>-1</sup> is from Si–O–Al bond (Ghorbel and Samet, 2013). These findings implied that all the additives were not chemically bonded to starch molecules.



**Figure 3.** Durometer hardness (Shore C) scale of the developed foams.

### Hardness

Generally, shore durometer is one of several methods to measure the hardness of a material. Higher numbers indicate harder materials and lower numbers indicate softer materials. By shore durometer, hardness may be defined as a material's resistance to indentation. There are several scales of durometer used for materials with different properties. In this study, the durometer hardness of the foam samples was measured using shore C scale. Figure 3 displays durometer hardness (shore C) of all the developed corn starch foams.

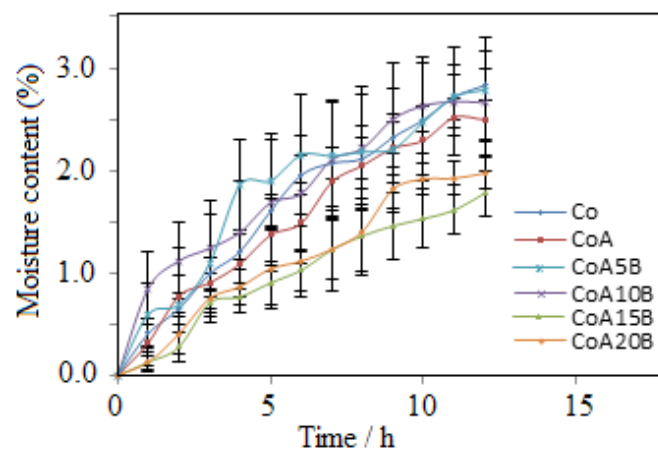
Co exhibits shore C hardness of 73 ± 12. With the addition of the fixed-amount additives, the hardness of CoA increased by 16% compared with Co. This may be due to the strong molecular interactions (e.g. hydrogen bondings) between starch molecules and their compatible additives. Although the hardness of the foams did not significantly change with the addition of beeswax, the shore C hardness showed a slight increase when beeswax concentration increased.

### Moisture absorption

Due to the hydrophilic nature of starch, typically, the starch foams exhibit high moisture absorption leading to poor moisture barrier properties, deterioration of its mechanical properties and reduction of its bacterial resistivity (Sanhawong et al., 2017). In this study, the water resistance of the developed corn starch foams was expected to improve by the addition of beeswax.

Figure 4 reveals the effect of beeswax concentration on the moisture absorption behavior of the developed foams as a function of time at 50%RH. All the foam samples demonstrated the increase in moisture content with the moisture exposure time. With the

addition of beeswax up to 10 phr, there was no significantly variation in moisture content among the 4 samples, namely Co, CoA, CoA5B, and CoA10B. This was due to the high hydrophilicity of native starch. On the other hand, the decrease in moisture content of the foams was observed for the developed foams with high beeswax concentration e.g. CoA15B and CoA20B. This has proven that the water resistance of the foams can be improved by the addition of appropriate amount of beeswax. This is due to the hydrophobicity of beeswax, which is responsible for the decrease in the hydrophilic nature of the foams.

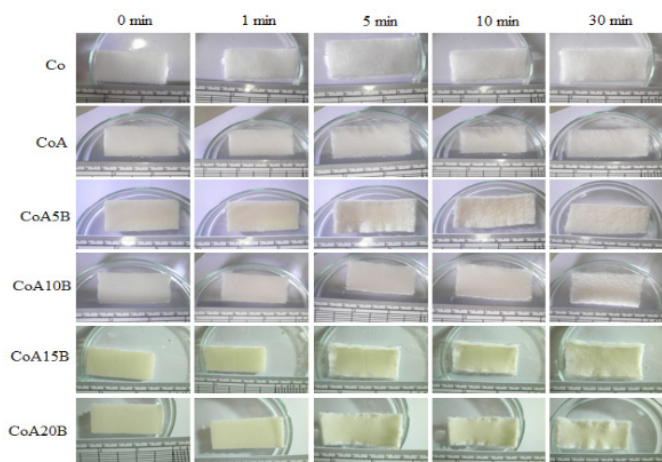


**Figure 4.** Moisture content as a function of time for the developed foams.

### Dimensional stability after water soaking

As shown in the results obtained from moisture absorption test that water resistance of the foams can be improved by the addition of the appropriate amount of beeswax. Therefore, the dimensional stability of the foams is expected to increase accordingly. Dimensional stability is a measure of the extent of swelling or contraction, resulting from the movement of moisture in materials (Dündar et al., 2016). Figure 5 shows the effect of beeswax content on the dimensional change of the foams, caused by water sorption, as a function of time. When taking into contact with water, Co rapidly absorbed water and became slimy within 1 min. Moreover, it did not retain its inherent dimension and immediately swelled after soaking in water. This was due to the inherently high hydrophilicity of starch molecules. With the addition of additives, CoA showed the obvious dimensional change within 5 min. When beeswax was incorporated, the dimensional change of CoA5B and CoA10B was found only at the edges of the foams after 5-min soaking in water. The hydrophobicity of beeswax is responsible for the decrease in water uptake of the foam, leading to the decrease in the moisture content. Hence, the foams possess better ability to withstand the dimensional change by the addition of beeswax. With increasing beeswax's concentration, CoA15B and CoA20B can resist the dimensional change for 10 min. This is related to the results obtained from moisture absorption test, showing that the decrease in moisture content of the foams was observed for the developed foams with high beeswax concentration e.g. CoA15B and CoA20. Further investigation for longer period evidenced that that beeswax played an important role in the dimensional stability of the foam while soaking in water for 12 hrs. This has proven that the dimensional stability of the foams can be improved by the addition of beeswax. The higher the content of beeswax in the foam, the better its dimensional stability.

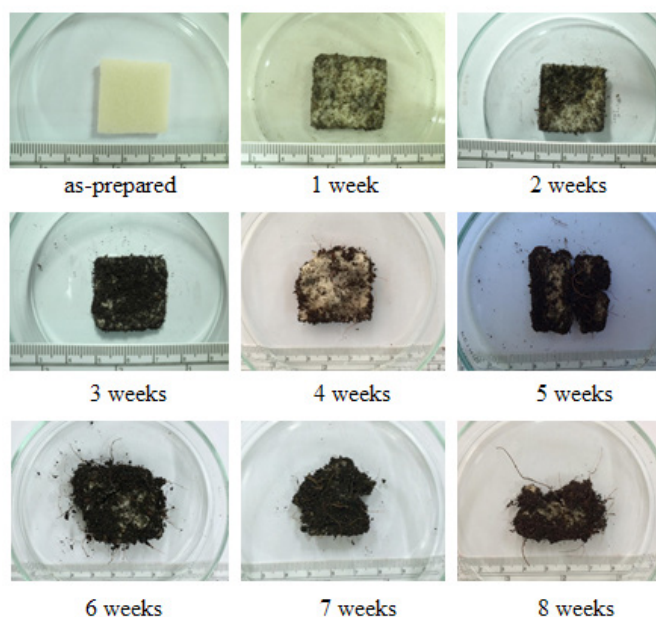




**Figure 5.** Digital photographs showing the dimensional change of the developed foams as a function of time.

### Soil burial test

According to the test results, it is convincing that the baked corn starch foam prepared from corn starch mixed with the fixed-amount additives and 20 phr of beeswax showed the best properties among the developed foams. Therefore, CoA20B was selected as a model foam for biodegradation study via the burial of the sample in soil. The digital photographs showing the degradation behavior of CoA20B as a function of time are demonstrated in Figure 6.



**Figure 6.** Biodegradation behavior by soil burial test for CoA20B biofoam.

Commonly, in soil burial test of starch foam, the weight change of the baked starch foam was not be able to measure because of experimental difficulties, such as the loss of sections of the foam and the association of the biomass with soil (Sanhawong *et al.*, 2017). In this study the biodegradation behavior of the foam was monitored by visual change, while the digital photographs were taken. It can be seen in the figure that CoA20B noticeably swelled after a week of the test by the absorbed soil water. This is recognized as one of

the phenomena leading to the macroscopically observable result of polymer degradation (Albertson, 2000). Conclusively, the foam can be degraded in soil mainly via hydrolysis.

At the 4th weeks of the test, the fragmentation of the foam was clearly evidenced and the fragmented pieces of the foam were prominently observed within 5 weeks. The longer period of time, the smaller pieces of the foam were witnessed. Not surprisingly, the entire piece of the foam disappeared from the soil environment within 9 weeks. Therefore, the waste of this foam could be easily disposed in the environment without causing any environmental problems.

### CONCLUSIONS

Spray-dried rice bran extract powder (SD-RBE) showed the ability to reduce browning in potato puree and potato PPO activity inhibition. It suggested that SD-RBE could be a potentially natural antibrowning agent for potato puree.

### ACKNOWLEDGEMENTS

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