Use of Silica from Rice Husk Ash as an Antiblocking Agent in Low-Density Polyethylene Film

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ABSTRACT: In packaging applications, blocking is always found in low-density polyethylene (LDPE) films. Practically, such problems can be solved by incorporation of antiblocking agents, for example, silica and talc. The objective of this research was to explore the possibility of using silica from rice husk ash (RHA silica) as an antiblocking agent in LDPE film. Properties of RHA silica were compared with commercial silica, Sylo-1. The appropriate amount of silica to be used as an antiblocking agent in LDPE film was also investigated. The results indicate that RHA silica has a smaller particle size and a higher specific surface area but a higher bulk density than those of Sylo-1 silica. In the plastic film industry, 500–1000 ppm of silica is added in LDPE films as an antiblocking agent. It was also found that LDPE film with 2000–3000 ppm RHA silica showed similar properties to LDPE film filled with commercial silica in terms of its blocking behavior, mechanical strength, and film clarity. © 2003 Wiley Periodicals, Inc. J Appl Polym Sci 88: 848–852, 2003

Key words: additives; polyethylene (PE); silicas

INTRODUCTION

Low-density polyethylene (LDPE) resins have found use in a wide range of applications. Films, blends, or coextrusions produced from LDPE provide considerable variability in properties such as flexibility, clarity, toughness, and processibility, thus allowing products to be designed for particular applications ranging from very thin films (0.3 mil) to very thick sheets (30 mil) and from high clarity to translucent films. Layers of LDPE films often show high cohesion or blocking, a feature that is often a nuisance in both processing and use. Blocking is the tendency of two adjacent layers of films to stick together, especially when they are left under pressure for some time. This situation is often found when films are stacked in cut sheets. Blocking of smooth surfaces hinders destacking and taking off the films, especially for very thin and flexible films. It can also make bags made from films laid flat difficult to open. Other factors that affect blocking behavior are static charges, surface treatment (such as printing pretreatment), and storage conditions. In the industry, this problem can be overcome by incorporation of antiblocking agents such as diatomaceous earth, fine silica, or talc. Antiblocking agents roughen the film surface, thus reducing blocking.^{1,2}

Thailand is an agricultural country that produces a large amount of rice for domestic consumption and

exportation. Rice milling produces millions tons of rice husk annually as agricultural waste. Many researchers have reported on rice husk as an important source of a silica compound.^{3–5} When rice husk is burnt at 600°C for 6 h, the resulting white ash contains about 99.5% silica.⁶

The purpose of this research was to investigate the potential use of silica from rice husk ash (RHA silica) as an alternative antiblocking agent for LDPE blown film. LDPE films filled with silica from rice husk were processed, then compared with LDPE films filled with commercial silica.

EXPERIMENTAL

High-purity silica was prepared from rice husk using the technique described by Leela-adisorn.⁷ The husk was mixed with 0.4M HCl in the ratio of 100 g husk per 1 L acid and heated at 105°C for 3 h until the color of the husk gradually changed from yellow to dark brown. The husk was cleaned to remove acid using tap water. It was then dried in an oven at 110°C. Subsequently, the treated husk was burnt in an electric furnace at 600°C for 6 h. The obtained RHA silica in the form of white ash was ground to reduce its particle size. Some properties of prepared silica were investigated to compare it with the commercial silica normally used as an antiblocking agent. Silica-grade Sylo-1 from the PPG Siam Silica Co. (Bangkok, Thailand) was also used in this experiment for comparison purposes. The particle-size distribution of silica was examined using a laser particle-size analyzer (Malvern

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Properties	RHA silica	Sylo-1 silica	
Average particle size (micron)	4.27	6.61	
Specific surface area (m^2/g)	131.35 ± 2.34	511.97 ± 4.70	
Bulk density (g/cm ³)	0.677	0.319	
pH ^a	5.7	6–8	
Oil absorption $(g/g)^a$	1.92	2	
Silica content (% SiO ₂) ^a	99.6	99.0	

 TABLE I

 Properties of RHA Silica and Sylo-1 Silica

^a Ref. 9.

Mastersizer S). The specific surface area of silica was determined using the BET method (Flowsorb II 2300). Bulk densities were also investigated. The appearance of each silica was examined under a scanning electron microscope (JSM 5410LV). Some predetermined properties of both kinds of silica are presented in Table I.⁸

LDPE resin (blown film grade LD1807G from the Thai Polyethylene Co., Bangkok, Thailand) was mixed with silica using a counterrotating twin-screw extruder equipped with a pelletizer (Collin ZK25). The *L/D* ratio of the screws was 25:1 with a screw speed of 75 cycles/min. The average temperature used was 180°C. The mixture was then processed into film using a Betol 2525 blown film extruder. The screw speed was 75 cycles/min with a 7-m/min film take-up rate. The temperature used was 190°C. The ratio of LDPE and silica in each batch is shown in Table II.

A universal testing machine (Lloyd LR100K) was employed to determine the tensile strength and tear strength, following ASTM D882 and ASTM D1004, respectively. The prepared film was tested in both the machine direction (MD) and the transverse direction (TD). The film blocking force was determined using the universal testing machine equipped with block slip grip (Ceast DY30) complying with ASTM D1893. Film haze and gloss were determined according to ASTM D1003 and ASTM D253 using a haze meter and a haze-gloss tester (BYK Gardner), respectively.

RESULTS AND DISCUSSION

From the investigation using an electron microscope, both kinds of silica have irregular shapes. RHA silica appears to have finer particles than those of Sylo-1, as illustrated in Figure 1.

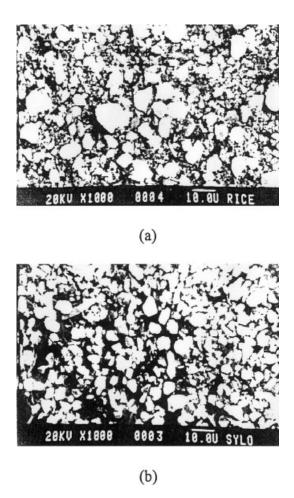


Figure 1 SEM micrographs of (a) RHA silica and (b) Sylo-1 silica.

From Table I, it can be seen that the average particle size of RHA silica was finer than was the average particle size of Sylo-1 silica. Compared with Sylo-1 silica, the size distribution of RHA silica, as shown in Figure 2, has a greater fraction of fine particles. However, it was also observed that the specific surface area of RHA silica was lower than that of Sylo-1 silica. This result suggests that the porosity of RHA silica is less than that of Sylo-1 silica.

In the case of blocking behavior, a drastic decrease in blocking could be observed when the LDPE film was filled with silica. From Figure 3, the film filled with Sylo-1 silica shows less blocking force than that filled with RHA silica. It could be deduced that film

TABLE II Ratio of LDPE and Silica in Each Batch of the Mixture

	Silica content (ppm)							
Material	300	500	1000	1500	2000	3000	4000	5000
Silica (g) LLDPE (g)	0.75 2499.25	1.25 2498.75	2.50 2497.50	3.75 2796.25	5.00 2495.00	7.50 2492.50	10.00 2490.00	12.50 2487.50

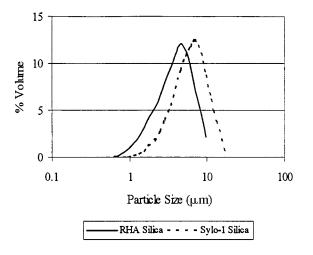


Figure 2 Size distribution of RHA silica and Sylo-1 silica.

surfaces are roughened more with larger particles of Sylo-1, compared with RHA silica.

During the blown film extrusion process, LDPE film is stretched in the machine direction; thus, polymer chains rearrange themselves in an orderly manner. As a result, the tensile strength of the film filled with silica is notably higher in the machine direction than in the transverse direction. The results are shown in Figure 4. It was also found that LDPE film filled with Sylo-1 silica showed a higher tensile strength when compared with LDPE film filled with RHA silica. It was assumed that the higher porosity in Sylo-1 silica contributed to this increasing adhesion between the polymer and the silica. However, when the amount of silica was increased, the tensile strength of the films was decreased, accordingly.

Since the film was stretched in the machine direction during the blowing process, it can be demonstrated in Figure 5 that the elongation at break of the film in the machine direction is less than that of the film in the transverse direction. As well as the tensile strength, it was found that the elongation at break of the LDPE film filled with Sylo-1 silica was higher than that of the film with RHA silica. It was also found that the addition of silica decreased the elongation at break of the film.

When the amount of silica in the film was increased, the results indicate that the tear strength was insignificantly different. LDPE film filled with Sylo-1 silica has a higher tear strength as opposed to the film filled with RHA silica, as shown in Figure 6.

The haze of the LDPE film filled with Sylo-1 silica is greater than that of the film filled with RHA silica, as shown in Figure 7. This is probably because Sylo-1 has a lower bulk density and larger average particle size. Light is scattered more when encountering large particles. As the amount of silica increases, the haze increases accordingly due to increased light scattering.

As shown in Figure 8, LDPE film filled with Sylo-1 silica shows slightly lower gloss characteristics than those of LDPE film filled with RHA silica. This finding confirms that particles of Sylo-1 silica are larger than are those of RHA silica. Generally, rough surfaces exhibit less gloss than do smooth surfaces. Large particles accounted for rougher surfaces. This is probably the reason for lower gloss in the LDPE film filled with Sylo-1 silica.

CONCLUSIONS

Silica prepared from RHA, although composed of finer particles, shows less porosity than that of commercial silica (Sylo-1), resulting in a lower specific surface area. Nevertheless, it is possible to use the obtained RHA silica as an antiblocking agent for LDPE film. The addition of RHA silica to LDPE film modifies the film blocking behavior by inducing film roughness. However, the blocking is reduced not as much as in the case of Sylo-1 silica. To the same antiblocking capability, 2000–3000 ppm of RHA silica is needed as opposed to 500–1500 ppm of commercial silica nor-

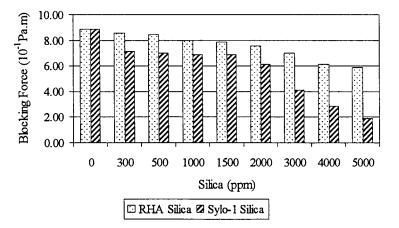


Figure 3 Blocking force of LDPE film filled with silica at various ratios.

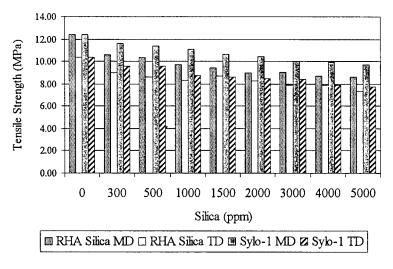


Figure 4 Tensile strength of LDPE films filled with silica at various ratios.

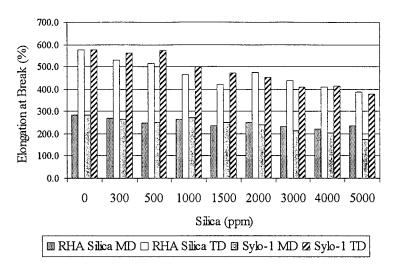


Figure 5 Elongation at break of LDPE film filled with silica at various ratios.

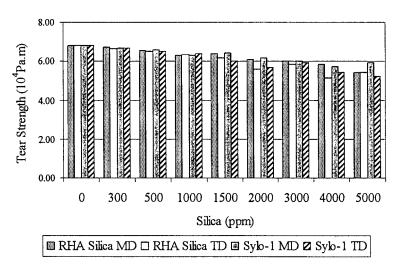


Figure 6 Tear strength of LDPE films filled with silica at various ratios.

70.00 60.00 50.00 40.00 20.00 10.00 0 300 500 1000 1500 2000 3000 4000 5000 Silica (ppm) C RHA Silica Z Sylo-1 Silica

Figure 8 Gloss of LDPE film filled with silica.

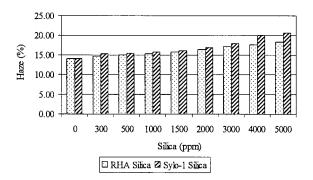


Figure 7 Haze of LDPE films filled with silica.

mally used in the plastic film industry. It was found that, as the amount of silica increased, certain mechanical properties, that is, tensile strength, elongation at break, and tear strength, were decreased markedly. Haze and gloss characteristics of the film were altered as the particle size and amount of silica changed.

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