

# Solid Lubricant Formulations Containing Starch–Soybean Oil Composites

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**ABSTRACT:** Starch–oil composites comprising purified food-grade corn starch (PFGS) and soybean oil (SBO) were investigated as potential ingredients for water-based solid lubricant formulations. Current solid lubricants are almost exclusively petroleum-based and are used for protecting sheet metal and/or as sheet metal forming lubricants. Starch–oil composites are preferred ingredients for formulating solid lubricants because they are based on renewable and abundantly available raw materials and also have superior environmental and health characteristics. Steel sheets coated with the PFGS–SBO containing solid lubricant were evaluated for boundary coefficient of friction (COF) and wear properties using ball-on-flat test geometry. The COF was highly dependent on the SBO to PFGS ratio in the composite. In the absence of SBO, the COF was high (~0.8) and decreased sharply with increasing SBO content to a minimum value of 0.07. Wear evaluation showed no scratches or lubricant transfer on the ball. There were also no wear tracks observed on the flat sheet before or after the solid lubricant was washed off. It was concluded that water-based solid lubricants formulated with the PFGS–SBO starch–oil composite have acceptable friction and wear properties and should be evaluated further for use in sheet metal forming.

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**KEY WORDS:** Ball-on-flat, coefficient of friction, Fantesk™, friction force, purified food-grade starch, sheet metal forming, soybean oil, starch–oil composites, sucrose, wear.

Starch is one of the most abundantly available and renewable natural products. It is obtained from various plant-based agricultural products such as corn, wheat, barley, potato, and tapioca (1).

Starch is a linear and/or branched polysaccharide of 1,4-glucose units (1). Its molecular weight depends on its structure as well as the plant source (1–4). Starch comprising a linear chain of glucose units is called amylose and generally has a M.W. in the range  $0.5$  to  $2.0 \times 10^6$  (2). Starch comprising branched polysaccharides is called amylopectin and generally has M.W. that are several orders of magnitude higher than amylose (1,3). Starches from most plants comprise 75–80% amylopectin and 20–25% amylose (1). However, certain corn and rice varieties produce only waxy starch, which is a highly branched amylopectin with very high M.W. (3). Also, a special variety of high-amylose corn called Cerestar® (formerly Amylomaize®) contains more than 70% of the linear chain amylose (4).

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Starch, as is or with some modification, is used in the manufacture of a wide variety of food and nonfood products (1,4,5). This is usually accomplished by blending the starch with a variety of ingredients such as sugars, oils, or FA.

Currently, there is an overabundance of starch relative to the demand for starch-based products. This has depressed the price of starch-producing agricultural products and, hence, the income of farmers growing such crops. One way of overcoming this problem is to introduce new starch-based products. Recently, the USDA developed a starch-based product called Fantesk™ that has opened up new applications for starch-based products in food and nonfood industries (6–11). Fantesk is a composite of starch with other ingredients such as oils and lipids. It is obtained by continuous steam jet cooking of a blend of starch and the lipid of choice (12). The resulting dispersion is dried, milled, and used for the intended application. Ingredients that have been blended with starch to make Fantesk composites include synthetic oils, vegetable oils, and formulated products for use in food, medicine, cosmetics, agriculture, coatings, adhesives, and lubricants (13–16). Some of these Fantesk products are currently being developed in collaboration with industrial partners. Studies on the structure of Fantesk have indicated that the Fantesk composites constitute micrometer-sized lipid droplets surrounded by a shell of starch encapsulating the blend component (11). Details of the Fantesk manufacturing process and the structure of the Fantesk composite are given elsewhere (6–16).

In the area of lubrication, Fantesk has been evaluated for encapsulation and delivery of biocides in metalworking lubricants (15) and for the encapsulation of lubricant formulations for application in oil drilling mud (16). The latter application is currently being pursued in collaboration with an industrial partner. Other than these two examples, there have been no other investigations into the lubrication properties of Fantesk composites.

In this work, Fantesk composites comprising purified food-grade corn starch (PFGS) and soybean oil (SBO) were investigated as ingredients in water-based solid-lubricant formulations for a one-time sheet metal forming application. Factors affecting the blending, application, friction, and wear properties of these solid lubricant formulations were investigated.

Solid or dry film lubricants are used to protect sheet metal surfaces from scratches and/or the environment during transportation and storage (17,18). Solid lubricants are also used to lubricate the desired part during the fabrication of sheet metal. In such applications, the solid lubricant may be the only source of lubricant, or it may be used in conjunction with

a compatible lubricant applied during the forming process. Prior to commercial implementation, the solid lubricant must meet a number of stringent requirements imposed by the type of sheet metal (steel, aluminum, copper, etc.), product application (automotive, appliance, food, beverage, etc.), and other factors (e.g., waste treatment/waste disposal). Currently, solid/dry film lubricants are almost exclusively petroleum based. Successful development of water-based Fantesk solid lubricants provides a number of advantages over petroleum-based solid lubricants including being (i) an unlimited, renewable, and cheap source of raw material, (ii) a completely biodegradable and, hence, environmentally friendly product, and (iii) a nontoxic and, hence, operator-friendly product.

## MATERIALS AND METHODS

**Materials.** SBO was obtained from Procter & Gamble (Cincinnati, OH). PFGS was obtained from A.E. Staley Mfg. Co. (Decatur, IL). Sucrose was obtained from Fleming Companies, Inc. (Oklahoma City, OK). Isopropanol and hexane (both 99.9%) were obtained from Fisher Scientific (Fair Lawn, NJ) and used as supplied. Deionized water was used to prepare solid lubricant formulations. Type 304,  $0.076 \times 30.48 \times 30.48$  cm steel plates were obtained from McMaster Carr Supply Co. (Elmhurst, IL) and cut into  $7.6 \times 15.2$  cm specimens for use in friction experiments. Grade 100, 440-C stainless steel balls were obtained from Altek Co. (Torrington, CT) and had the following specifications: diameter,  $15.88 \pm 0.02$  mm; sphericity, 0.0254 mm; hardness, 57–67 c. The steel balls and flat sheets were cleaned by consecutive 5-min sonications in isopropanol and hexane prior to use in friction experiments.

**Preparation of starch–oil composite.** PFGS was mixed with 2 L of water and blended at high speed in a Waring blender until a homogeneous mixture was obtained. SBO was then added to the mixture gradually while blending at high speed for 5 min. The mixture was then poured into the hopper of the jet cooker and stirred continuously with a mechanical stirrer to prevent rapid separation of the phases. The mixture was then pumped through a Penick & Ford Laboratory Model steam jet cooker (Penford Products Co., Cedar Rapids, IA) operating at  $140^\circ\text{C}$ . Pumping rate through the cooker was about 1 L/min. The hot, jet-cooked dispersion came out at  $100^\circ\text{C}$ . The mixture was then dried on a 45 cm long  $\times$  30 cm diameter double-drum drier heated with steam to about  $140^\circ\text{C}$ . The flakes were passed through a Retsch mill (Model ZM-1; Brinkman Instrument Inc., Des Plaines, IL), equipped with a 1-mm screen, to yield a fine powder. The concentration of SBO in the composite is expressed in parts per hundred (pph) relative to dry PFGS. Since PFGS is composed of about 10% moisture by weight, 110 parts of PFGS is required to obtain 100 parts of dry PFGS. Thus, for example, a composite of 20 g of SBO and 110 g of PFGS will have an oil concentration of 20 pph.

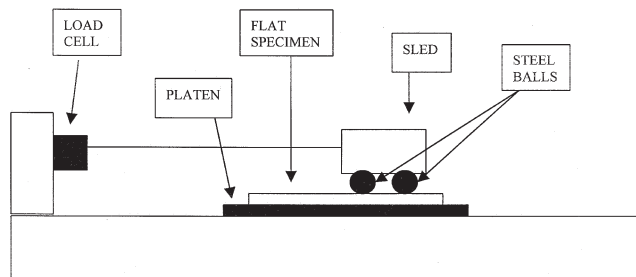
**Formulation and application of solid lubricant.** An aqueous sucrose solution was prepared by dissolving the required weight of sucrose in 130 g of water at  $95^\circ\text{C}$ . The solution was

then transferred to a Waring blender to which the appropriate amount of the Fantesk composite was added. The mixture was then blended at high speed for 5 min and the resulting dispersion applied as a thick film onto the flat steel specimen using a doctor blade with a 0.76-mm gap. The film was then allowed to dry at ambient conditions during the course of several hours. During drying, the appearance of the film changed from opaque to transparent.

**Friction measurement instrument.** Friction was measured under a ball-on-flat configuration. In this configuration, a flat sheet metal coated with the appropriate lubricant and secured onto a platen is pulled from under a sled of specified weight and connected to a load cell. The sled contacts the flat sheet with three balls installed at its bottom. The load cell measures the friction force resisting the movement of the sled. The coefficient of friction (COF) is obtained by dividing the friction force measured with the load cell by the weight of the sled. A schematic of a ball-on-flat friction tester is shown in Scheme 1.

The ball-on-flat instrument used in this study was constructed by combining the SP-2000 slip/peel tester from Imass, Inc. (Accord, MA) with Model 9793A test weight sled from Altek Co. The test weight sled provides the normal load and includes slots for securing the three steel balls. The SP-2000 has the following main features: a 2-kg load cell for measuring the friction force; a platen for securing the flat sheet metal sample and the capability to travel at a range of speeds; and an inbuilt microprocessor system for setting up test parameters, acquiring data, statistically analyzing and displaying results on an onboard and on an external monitor, printing results on an external printer, and transferring data to a personal computer for storage and/or further data manipulations.

**Friction measurement procedure.** Latex gloves were used to handle test specimens, and care was taken to prevent contamination of the test specimen surface. A typical test procedure was as follows: The sheet metal coated with solid lubricant was secured on the platen. The sled with the selected weight was then placed on top, in contact with the sheet metal with its three clean steel balls. The string from the load cell was then connected to the sled and the platen moved to remove the slack from the string. The start button was then pushed causing the platen to move at the selected speed and the load cell to measure the resulting friction force. The measured friction force was automatically recorded by the microprocessor at a maximum rate of 3906 samples/s. At the end



SCHEME 1

of the test period, the platen stopped automatically and a summary of the results was displayed on the onboard screen while a time vs. friction force plot was simultaneously displayed on the external monitor. At the end of each measurement, the instrument automatically displayed the average of the friction force values, in grams. The platen was then brought to the start position and the sled removed for inspection. After inspection, the three balls were either cleaned for reuse or replaced with a new set of clean balls. The sled was then put back onto a fresh surface of the same sheet metal for a repeat test. At the end of the second test, the friction force statistics for each test and for both tests were automatically printed on the external printer.

All friction experiments were conducted at room temperature, using a 1500-g load sled at a speed of 2.54 mm/s for a total test time of 24 s. Two sheet metal specimens were prepared for each solid lubricant sample, and duplicate tests were conducted on each specimen. The COF for a solid lubricant sample was obtained by dividing the average friction force of the four measurements by the sled weight.

## RESULTS AND DISCUSSION

As already mentioned, the Fantesk compositions studied in this work are composed of PFGS and SBO. Fantesk composites with 0 to 45% SBO were prepared and used to blend water-based solid lubricants. The properties of these solid lubricants were investigated and are discussed below. In this paper, Fantesk compositions will be designated as PFGS-xx-SBO, where xx is the concentration of SBO as a percentage of dry PFGS.

*Formulation studies. (i) Effect of Fantesk/water ratio.* Initially, solid lubricant formulations were obtained by blending the PFGS–SBO composite with hot deionized water. The resulting solid lubricant blend had a viscosity that was a function of the aqueous composite concentration and the temperature. Viscosity increased with increasing Fantesk concentration and decreasing temperature. Viscosity is a critical factor that affects the applicability of the blend to the flat metal surface. High-viscosity blends are too difficult to apply, whereas low-viscosity blends are hard to control resulting in inconsistent film thickness. Viscosity also had an effect on the subsequent drying process of the lubricant film on the flat sheet metal. Experiments with different Fantesk/water ratios and application temperatures showed that optimal results were obtained when the concentration of Fantesk in water or aqueous sucrose is 17% (w/w) and when such a formulation is applied at 85–90°C.

*(ii) Effect of sucrose/water ratio.* Solid lubricant blends of PFGS–SBO composites in pure deionized water were found to have poor adhesion properties to the flat stainless steel plates. After drying, such blends peeled off from the surface and were very brittle. To overcome this problem, an aqueous sucrose solution was used for dispersing the PFGS–SBO composites. In the presence of sucrose the lubricant film became flexible (eliminated brittleness) and also had improved

adhesion to the surface of the flat stainless steel sheet. Investigation into the effect of sucrose concentration on film properties showed that adhesion properties improved with increasing sucrose concentration to about 8% (w/w) of sucrose in water. Further increase in sucrose concentration did not affect adhesion but resulted in a more flexible film.

*Friction studies.* A typical time vs. friction force profile obtained during the friction measurement on a PFGS–SBO composite-based solid lubricant formulation is shown in Figure 1. In all cases, the friction force showed a rapid increase to a maximal value followed by a rapid decrease to a steady-state value. The maximal friction force corresponds to the static friction force, which is a measure of the friction force when both surfaces are initially at rest. The steady-state friction values correspond to the kinetic friction force, which is a measure of the friction force when there is relative motion between the surfaces. After each measurement, the kinetic friction force values are averaged to calculate the average kinetic friction force for the run. For each solid lubricant formulation, four measurements are conducted on two flat metal specimens. The average kinetic friction force values from these four measurements are then divided by the load (weight of the sled) to obtain the coefficient of friction (COF) of the solid lubricant formulation.

The effects of various formulation and process parameters on COF were studied. The results of these studies will be discussed below.

*Effect of sucrose concentration on COF.* As discussed above, addition of sucrose to the deionized water used for dispersing the starch–oil composite was essential to ensure that the solid lubricant film was not brittle and also properly adhered to the flat sheet metal surface. Studies were conducted to determine the effect of sucrose concentration on the COF of solid lubricant films. Sucrose concentration is expressed as percentage of total (sucrose + water) weight. The compositions studied were PFGS–0-SBO and PFGS–10-SBO. These compositions were used to prepare solid lubricant formulations using aqueous sucrose solutions of concentrations

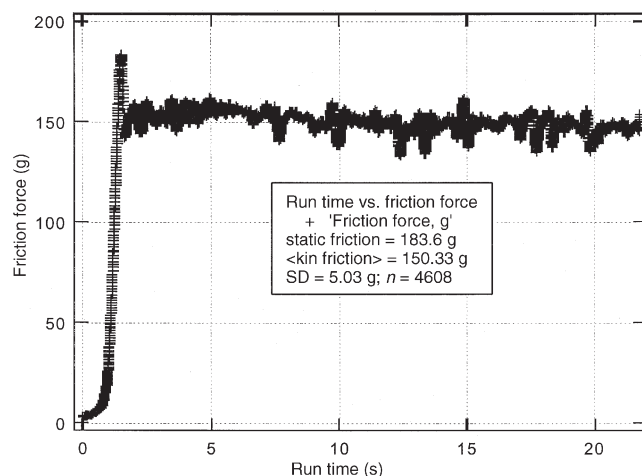
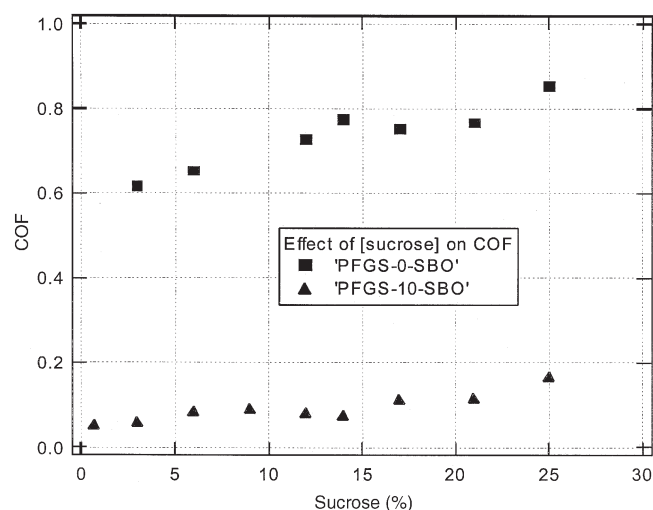


FIG. 1. Typical run time vs. friction force data of a solid lubricant obtained on a ball-on-flat friction tester.

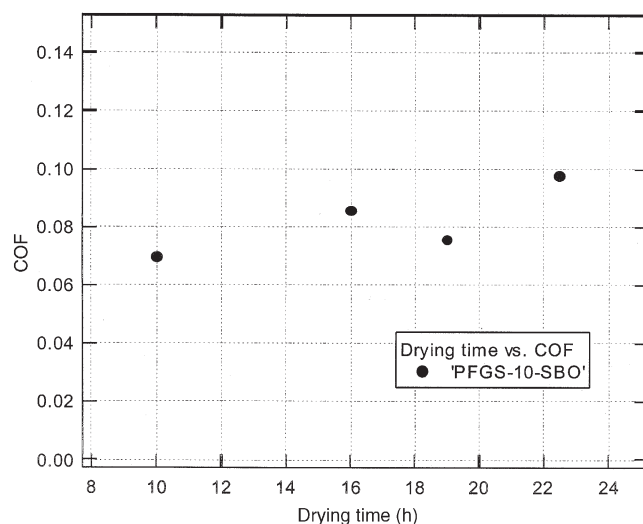
ranging from 3 to 25%. The resulting COF values of these solid lubricants are summarized in Figure 2. Two important observations from Figure 2 were made: (i) For both compositions, the COF increased gradually with increasing sucrose concentration, and (ii) for all sucrose concentrations, the incorporation of 10% SBO into Fantesk resulted in a dramatic drop in COF (detailed discussion on the effect of SBO–PFGS ratio on COF is given later). It is clear from this study that the effect of sucrose concentration on COF is rather mild and will not contribute to COF variability as long as a fixed sucrose concentration is used in all solid lubricant formulations. Thus, a 10% aqueous sucrose solution was selected for preparing the solid lubricants used in the rest of this study.

**Effect of lubricant film drying time on COF.** Another factor that could potentially influence the COF of dry film lubricants is the moisture content (MC) of the film. MC is a function of the drying conditions such as temperature, time, and ventilation. In this work, the sheet metals coated with the dry film lubricant formulations were kept on top of a lab bench and allowed to dry at ambient temperature and relative humidity. In such a setup, the only variable that can influence MC is the drying time. Experiments were conducted to investigate the effects of drying time on COF. Solid lubricant formulations based on the PFGS–10-SBO composite were used in this study. The result of this study is summarized in Figure 3. As shown in Figure 3, the COF showed a slight increase with increasing drying time. It is clear from this observation that drying time will not present large variability in COF values as long as it is kept constant. Based on these results and also for the sake of convenience, we selected 16 h of drying time for all subsequent solid lubricant samples.

**Effect of starch–oil ratio on COF.** PFGS/SBO composites ranging from 0 to 45% SBO were formulated into solid lubricants and the COF measured. These results are summarized in Figure 4. The SBO concentration vs. COF profile of Figure 4 can



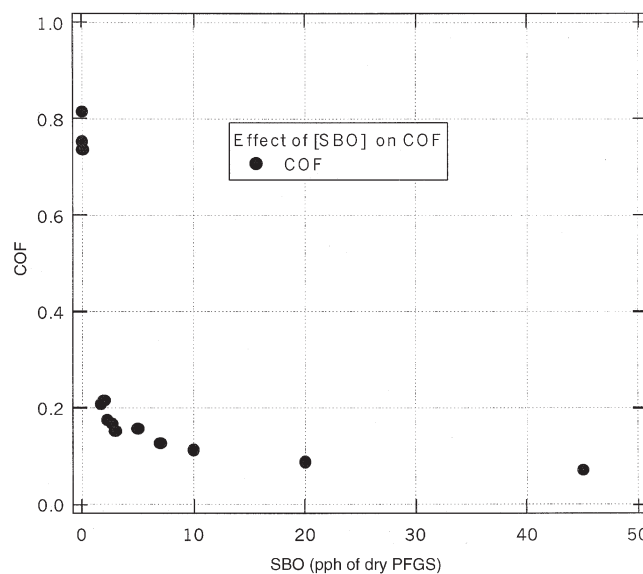
**FIG. 2.** Effect of sucrose concentration on the coefficient of friction (COF) of solid lubricant formulation containing purified food-grade corn starch–soybean oil (PFGS–SBO) composite. In “PFGS–xx-SBO,” xx is the concentration of SBO as a percentage of dry PFGS.



**FIG. 3.** Effect of drying time on the COF of solid lubricant formulation containing PFGS–10-SBO composite. For abbreviations see Figure 2.

be divided into three distinct regions: (i) a region of very high COF corresponding to pure PFGS solid lubricant, (ii) a region of sharp decrease of COF with increasing SBO concentration, encompassing the region from 0 to about 4% SBO (PFGS–0-SBO through PFGS–4-SBO), and (iii) a region of very low COF that is more or less independent of SBO concentration, including the region of SBO concentration of about 4% and higher.

These results are similar to observations on thin lubricant films under boundary friction in metal/metal contact (18–20). In fact, the effect of SBO concentration in PFGS on boundary COF is similar to that of the effect of SBO in hexadecane (21). The only difference is that the friction here is metal/Fantesk instead of metal/metal. However, in spite of this difference, the results from this work can be explained using a similar approach.



**FIG. 4.** Effect of SBO concentrations in PFGS–SBO composite on the COF of solid lubricant. For abbreviations see Figure 2.

The PFGS–SBO composite can be assumed to comprise a SBO dispersed phase in a continuous PFGS matrix. In the absence of SBO, friction is between the PFGS surface and the steel balls and, as a result, the COF is very high (~0.8). Incorporation of SBO into the composite results in SBO covering some fraction of the PFGS matrix surface and causing a reduction in the measured COF. As the concentration of SBO in the PFGS–SBO composite increases, so does the concentration of SBO on the PFGS surface, resulting in a corresponding decrease in measured COF. The COF reaches a minimum at full surface coverage of the PFGS matrix by SBO, which happens at about 4% SBO in the composite. Further increase in the concentration of SBO in the composite could result in multilayers of SBO molecules on the PFGS matrix surface. Since the boundary COF of a multilayer is indistinguishable from that of a monolayer, no change in COF is observed beyond an SBO concentration of ~4% in the PFGS–SBO composite.

At this time, the mechanism of how the SBO molecules end up on the surface of the PFGS matrix is not clear. The current hypothesis on the structure of Fantesk composites is that the oil is fully encapsulated by a starch shell. However, as discussed above, the COF results indicate that (i) the oil also resides on the surface of the starch matrix, and (ii) the concentration of the oil on the surface of the starch matrix is a function of the starch/oil ratio in the Fantesk composite. At this time, the mechanism of the surface coverage of the starch matrix by oil molecules is not clear. Also unknown is the relationship, if any, between surface concentration and encapsulation of oils in Fantesk composites. Work at clarifying issues related to the structure of Fantesk composites is in progress.

**Wear studies.** One of the requirements of a solid lubricant in sheet metal forming is that it allows for the proper formation of the metal part without causing damage to the part and/or the tool used in the forming process. To meet this requirement, the solid lubricant not only must reduce friction between the tool and workpiece (sheet metal) but also must be able to prevent them from contacting each other and causing wear during the forming operation (17).

The wear properties of PFGS–SBO composite-based solid lubricants of various SBO concentrations were evaluated by inspecting the three balls and the flat sheet after each friction measurement. The wear evaluation began with the inspection of the balls under 10× magnification for any sign of wear, scratch, or transferred lubricant. Next, the flat sheet was inspected visually for any sign of wear tracks. Finally, the solid lubricant was washed off the flat sheet, and the surface of the clean sheet was visually inspected for any sign of damages. From the inspections the following observations were made: (i) None of the solid lubricant formulations showed any wear, scratches, or materials transferred onto the balls, (ii) none of the solid lubricants showed any discernible wear tracks on the flat sheets immediately after the friction test, and (iii) no discernible damages, dents, scratches, or lines were observed on the steel sheets after the solid lubricants had been cleaned off. The quality of the steel sheets did not change as a result of applying, testing, and removing the solid lubricant.

*Properties of solid lubricants based on starch–SBO composites.* The tribological properties of the solid lubricants discussed in this work combine the desired properties of SBO and starch. It has been shown that SBO dissolved in liquid hydrocarbon lubricants can adsorb onto friction surfaces and effectively reduce their boundary COF (21). As discussed above, SBO dispersed in a starch matrix also effectively reduced the boundary COF of the starch–oil composite. In fact, the SBO concentration vs. COF profiles in hexadecane and in starch are almost identical. This means that incorporation of SBO into a Fantesk starch matrix did not affect its boundary lubrication properties. However, in spite of its good boundary properties, SBO is incapable of effectively separating the friction surfaces and thereby preventing wear of the friction surfaces.

Jet-cooked starch, on the other hand, can be applied in sufficient thickness to effectively separate the friction surfaces and thereby protect them from any associated wear. However, unlike SBO, jet-cooked starch has a very poor boundary friction property against steel. As discussed above, a COF of ~0.8 was observed between starch film and steel balls. Such a high COF will result in excessive tool wear during metal working and makes jet-cooked starch unsuitable for use as a solid lubricant in sheet metal forming.

The starch–SBO composite-based solid lubricant, however, combines the desired tribological properties of SBO and jet-cooked starch. This solid lubricant combines the COF-lowering property of SBO with the surface protection property of jet-cooked starch. The result is a Fantesk-based solid lubricant that has low COF between the lubricant and the ball, thus preventing tool wear, and that at the same time effectively separates the two surfaces to prevent damage to the sheet metal (workpiece) and the ball (tool).

In addition to these important tribological properties, the Fantesk solid lubricant has several important characteristics that are highly desirable in solid lubricants formulations. It is environmentally friendly; i.e., (i) it is dispersed and applied from water rather than from some other solvent that might require special handling, and (ii) all of its ingredients are biodegradable. These solid lubricants are also nontoxic since all of the ingredients are food based. Finally, all of the ingredients of the Fantesk solid lubricants are abundantly available from renewable agricultural sources. These features make Fantesk-based solid lubricants attractive alternatives to petroleum-based solid lubricants and favor their further evaluation in sheet metal forming.

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