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## Progress in Organic Coatings

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# Sag in drying coatings: Prediction and real time measurement with particle tracking



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## A B S T R A C T

Sag is a coating phenomenon characterized by gravity-driven flow after deposition; excessive amounts of sag can lead to coating defects. In this work, a new method for evaluating and quantifying sag is investigated. The motion of micron-sized Lycopodium spores on an inclined coating surface is tracked during drying, and the resulting surface velocity data is used to determine sag length. This in situ particle tracking method is minimally invasive and permits real time measurements. Measured sag lengths and real time surface velocities in aqueous polyvinyl alcohol solution coatings compare well with a theoretical model. The model is also used to develop a predictive sag regime map, which anticipates the extent of sag given coating properties and process-specific parameters. This map also identifies viable processing windows and aids in intelligent coating design given specific process constraints. The predictions of the sag regime map are compared against experimental sag results from polyvinyl alcohol solution coatings as well as four commercial latex paints, revealing good agreement for coatings with Newtonian or 'Newtonian-like' rheologies.

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### **1. Introduction**

Controlling the development of coating appearance during processing is critically important. Rheology, substrate geometry, and process parameters play complex and highly interdependent roles in this development, and failure to appropriately control these factors can have dramatic consequences on the ultimate coating appearance [\[1\].](#page--1-0) Defects such as orange peel, cratering, and sag are all examples of the possible consequences of poor coating control [\[2\].](#page--1-0)

Sag is a coating phenomenon characterized by gravity-driven flow after deposition and excessive amounts of sag can lead to coating defects, such as drips, in the final dried film [\[2\].](#page--1-0) When inclined at a nonzero angle  $\theta$  (from horizontal), a liquid coating experiences a local shear stress,  $\tau$ :

$$
\tau = \rho g(h - z) \sin \theta,\tag{1}
$$

where  $\rho$  is coating density, g is acceleration due to gravity, z is position relative to the substrate, and  $h$  is the total film thickness [\[3\].](#page--1-0) At the substrate,  $z = 0$  and shear stress is maximized. This stress results in a shear rate, which depends on the specific rheology ofthe coating and ultimately dictates the sag velocity profile throughout the film [\[1,4\].](#page--1-0) For steady, one-dimensional flow down an inclined plane, the velocity of a Newtonian fluid with viscosity  $\eta$  at any time is given by:

$$
v = \frac{1}{2} \frac{\rho g z^2 \sin \theta}{\eta},
$$
 (2)

assuming no slip at the substrate surface and zero shear stress at the coating/air interface  $\left[3\right]$ . Sag velocity profiles for non-Newtonian coating rheologies have been derived  $[4]$  and the inverse dependence on viscosity in Eq.  $(2)$  is a common feature. This dependence is the primary mechanism by which sag is arrested. Upon deposition, viscosity is low to facilitate spreading and wetting, and through either evaporation or curing, viscosity rises (often steeply). This sharp rise in viscosity drives velocity to zero, even while thickness and density remain finite.

The total distance the coating surface flows between deposition and the point when sag becomes arrested is defined here as the sag length,  $l_{\text{sag}}$ . Equivalently,  $l_{\text{sag}}$  can be defined as: [\[5,6\]](#page--1-0)

$$
l_{\text{sag}} = \int_0^\infty v_s dt,\tag{3}
$$

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where *t* is time and  $v_s$  is surface velocity, or  $v$  in Eq. [\(2\)](#page-0-0) at  $z = h$ . Thus,

$$
l_{\text{sag}} = \frac{1}{2} \rho g \sin \theta \int_0^\infty \frac{h^2(t)}{\eta(t)} dt, \tag{4}
$$

for a Newtonian solution with a constant density throughout the course of drying.

Sag has been classified into two categories: uniform and nonuniform [\[6\].](#page--1-0) For uniform sag, also referred to as draining or "smooth" sag  $[6]$ , the coating flows evenly over the width of the substrate. This sag mechanism will dominate when edge effects are negligible, coating thickness is uniform, and the substrate is flat. However, non-uniform sag, referred to as curtaining or dripping  $[6]$ , can occur if the above conditions are not met. Non-uniform sag can lead to large, isolated drips or fingering instabilities [\[7\],](#page--1-0) which become increasingly likely in thicker coatings [\[6\].](#page--1-0) For uniform sag, sag lengths as small as 1 mm have also been shown to have adverse effects on coating appearance by accentuating defects and/or roughness in the substrate  $[8]$  or base coat  $[5,9]$ . Excessive sag can also reveal and magnify microscopic contaminants, such as dust, that have fallen onto the coating and would otherwise be invisible  $[4]$ . Even if local contaminants or defects are absent, excess sag can lead to unsightly accumulation of material at the substrate edges [\[10\].](#page--1-0)

In practice, all processes that utilize liquid applied coatings on non-horizontal surfaces are susceptible to sag and require keen process control. Examples include spray coating [\[11\],](#page--1-0) dip coating, and roll-to-roll or web-based processes operated at nonzero angles  $[12]$ . Processes with baking steps are also susceptible, as the sharp temperature rise at the onset of baking significantly reduces viscosity and increases sag velocity  $[10]$ , per Eq.  $(2)$ .

In recognition of the importance of minimizing sag, several researchers have developed various methods to measure it. Wu  $[4,13]$  was one of the first to extensively analyze sag and, using an 'indicator' method, was able to measure sag lengths by recording the distance traveled by a blue indicator band in his coatings. In this case, only uniform sag was studied. Croll and Kisha [\[6\]](#page--1-0) later adopted this technique and also extended it to include nonuniform sag. However, in employing this method with "uniform" sag, it was noted that the average of several measurements over the width of the substrate was necessary as the location of the indicator band front was slightly nonuniform. In both of the abovementioned studies, only final sag lengths, measured after drying was complete, were reported. Overdiep  $[14]$  measured sag with a 'sagging balance,' which employs a sensitive force transducer to monitor flow. Colclough et al. [\[15\]](#page--1-0) used a viscometer to simulate coating conditions and predict sag behavior based on the subsequently measured viscosity increase. Several optical techniques have also been devised. For example, Frisch and Scopazzi [\[5\]](#page--1-0) correlated measured distinctness of image to sag length and Bosma et al. [\[16,17\]](#page--1-0) used a 'falling wave' technique to track moving surface ripples down the coating, where the observed wave shift was used to calculate a sag length.

Much effort has also been devoted to developing a simplified, but less quantitative, method for evaluating sag. Several [\[18–20\]](#page--1-0) early attempts were made to develop and standardize such a technique and today the most popular tool for accomplishing this is a multinotched applicator [\[21\],](#page--1-0) used in ASTM standard test D4400 [\[22\].](#page--1-0) In this test, the multinotched applicator is used to coat a series of increasingly thick, parallel lines; information on the sag resistance of the coating can be extracted from the sag behavior of these lines. While this method is simple and reproducible, direct comparisons between test performance and expected sag behavior in situ can prove difficult [\[23\].](#page--1-0)

Despite the individual merits of all the existing techniques, there still remains a need for a quantitative sag evaluation technique that is simple, versatile, and provides measurement in real time on local length scales. Additionally, this technique should be minimally invasive and adapt to the intended coating process, not the other way around as is the case with all of the above-mentioned methods. Song [\[24\]](#page--1-0) recently employed Lycopodium spores to monitor sag in drying coatings. While success using these spores was reported, the method has yet to be fully explored and validated. In this paper, the merits of using Lycopodium spores to monitor sag in real time and locally (micron length scales) are explored. Because the spores remain at the surface of the coating, surface velocity can be monitored throughout the entire process. To validate this method, a model is formulated which predicts and corroborates these measured surface velocities. Using Eq.  $(3)$ , these measured velocities are used to calculate sag lengths. While this work focuses on uniform sag, the general techniques described herein can be extended to the measurement of non-uniform sag.

In the second part of this paper, the concept of a sagging regime map is introduced and developed using the above-mentioned model. This map predicts how much a coating will sag (viz. the sag length) based on its solution properties such as viscosity, density, and initial solids weight fraction, as well as other coating parameters like evaporation rate, film thickness, and angle of inclination. Such a map is capable of providing users with an intuitive, fundamentals-based approach to predicting and controlling sag. To illustrate the utility of such a map, sag in polyvinyl alcohol (PVA) solutions and several commercial latex paints is measured and then compared against the predictions of the map, revealing good agreement for coatings with Newtonian or 'Newtonian-like' rheologies.

## **2. Experimental**

### 2.1. Materials

#### 2.1.1. Polyvinyl alcohol and latex paints

Aqueous PVA (99+% hydrolyzed,  $M_w$  = 130 kg/mol, Sigma– Aldrich Co., St. Louis,MO) solutions were prepared in distilled water by heating ingredients at 97 ◦C overnight and then stirring until cool. Prolonged, elevated temperature was necessary to quicken PVA dissolution. All solutions were prepared on the same day they were used to avoid any effects of gelling, which can become significant in PVA solutions that are several days old [\[25\].](#page--1-0) PVA concentrations of 7.2 wt.% were used for all particle tracking experiments and concentrations ranging from 5.0 to 12.1 wt.% were used for viscosity measurements.

Four commercial water-based, latex paints were tested: Behr Marquee Interior [A], Behr Premium Plus: Paint & Primer in One [B], Behr Premium Plus: Interior Ceiling [C] (Behr Process Corp., Santa Ana, CA), and Glidden Interior Premium [D] (Glidden Co., Strongsville, OH). Paints will henceforth be referred to by their single letter abbreviations, as denoted in brackets above. Each paint was white in color and had a flat sheen. Samples were thoroughly stirred prior to coating.

#### 2.1.2. Lycopodium spores

Lycopodium is one genus of clubmoss [\[26\]](#page--1-0) whose spores form a fine, yellow powder. A scanning electron microscopy (SEM) micrograph of Lycopodium spores (Duke Scientific, Palo Alto, CA) is shown in [Fig.](#page--1-0) 1., revealing that the spores are roughly spherical and very similar in size. Their size ( $\sim$ 25  $\mu$ m) makes them large enough to be easily visualized by an optical microscope, yet small enough to probe local velocities. Their surfaces are covered with an extremely hydrophobic, waxy coating [\[27\],](#page--1-0) allowing them to sit on the coating surface during drying.

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