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## A novel technique for in situ measurements of stress development within a drying film

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#### **ABSTRACT**

A novel technique capable of monitoring the stress evolving process within a drying film has been developed in this work. The device uses a thin cantilever beam made of stainless steel to hold a thin layer of fluid material. The (bending) force exerted on the beam as a result of the lateral stress developed within the film is measured via a pendant-balance transmitting mechanism. The reliability of the device was tested with two different biopolymer fluids (18 wt% sodium caseinate and 30 wt% waxy maize starch) dried at different temperatures and relative humidity (RH). The results revealed three stages of stress development for these biopolymer films: an initial delay, a sharp stress increase, followed by a steady stress plateau. Further analysis of experimental data showed that stress development and moisture loss can be normalised to form a master curve. However, it appeared that the magnitude of stress increase has no direct link with the rate of moisture loss, indicating possibly different mechanisms of the stress increase and the loss of moisture. Results also showed that the total stress of a dried starch film was much higher than that of a dried caseinate film.

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#### 1. Introduction

Drying or dehydration is an operation commonly used in food industry to remove water (or the solvent) from a solid or slurry matrix for the purposes of either a decreased water activity for a longer shelf life, or a reduced weight for easy transportation, or simply for texture modification. The removal of water molecules involves simultaneous heat and mass transfer, regulating respectively the amount and rate of thermal energy supply and the amount and rate of solvent migration. The kinetics of drying or the rate of mass transfer is determined mainly by two sets of controlling parameters: the environmental conditions (e.g. temperature, relative humidity, air flow speed, air flow direction) and the material properties (e.g. surface properties, surface area-volume ratio, microstructure, and composition). The mechanisms and principles underpinning the drying process have been well studied and described in various textbooks ([Barbosa-Cásanovas, 1996; Toledo,](#page--1-0) [1999\)](#page--1-0).

Even though controlling a drying process (the drying time and/ or the rate) can be achieved by using a particular drying device operated at specific drying conditions, some undesirable effects of drying (such as material deformation, shrinking, surface cracking, loss of glossiness) are rather difficult to control. These issues are often great concern to food manufacturers because of their detrimental effects on the visual appearance of a food product and consumers' preference. The internal stress is believed to be the main cause of the above identified side effects. During a drying process, the moisture loss at the external surface will lead to moisture migration from the interior to the exterior surface and develop a moisture content gradient. Particles/molecules within the drier plane will have an intensified tendency of displacement and therefore a lateral stress develops. In order to release this stress, a material will have to either shrink from the outside or simply to crack (mostly at the surface). For a thin film, bending or curling is often an effective way for stress release. The exact rate and mechanisms for such stress development and release are not yet clear, but is generally believed to be material-dependent. [De Gennes \(2002\)](#page--1-0) studied the drying of polymer films and indicated that a ''crust" glassy surface was the direct result of solvent evaporation. He further argued that the crust is under a mechanical tension and could lead to some cracks. [Thill and Spalla \(2003\)](#page--1-0) and [Lee and Routh](#page--1-0) [\(2004\)](#page--1-0) investigated the cracking of drying colloidal films and indicated more specifically that lateral capillary pressure, induced by packed particle fronts travelling horizontally across film, was responsible for the failure (crack) in these films. The occurrence of surface crack depends of course on the balance on the magnitude of the capillary force and the mechanical strength of the material itself. Using both numerical analysis and NMR technique for moisture diffusion, [Augier et al. \(2002\)](#page--1-0) showed that the highest risk of surface cracking was at the beginning of a drying process,





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when the yield stress of the material was relatively low compared to the internal stress caused by moisture loss.

In order to understand the evolving process of the internal stress (including the creation and the relaxation) within a drying material, various efforts have been made in developing a reliable technique to monitor and quantify such a process. One major progress was an in situ stress measurement apparatus developed a decade ago [\(Payne et al., 1997](#page--1-0)). They used a metal beam as the substrate coated with a thin layer of drying material on its top side. Since the beam was clamped firmly only on one end, it tended to bend slowly upwards as the lateral stress developed. The displacement or the deflection of the beam was monitored using a laser beam reflection technique and was used to quantify the magnitude of the internal stress of the drying film. This cantilever deflection principle was further modified with an improved accuracy and more sophisticated functionalities [\(Martinez and Lewis, 2002; We](#page--1-0)[din et al., 2004\)](#page--1-0). The improved device was not only able to measure in situ the internal stress but also the rate of moisture loss (or the drying kinetics). However, one potential problem of this set up is that the internal stress relaxes continuously as a result of film bending. This continuous relaxation makes it questionable whether the technique is capable to determine the maximum stress of a drying film and to observe surface cracking. This work aims to develop an alternative method to monitor the lateral stress development and to investigate effects of temperature and humidity on drying and stress development. The technique is still based on the cantilever principle, but the beam is restricted for free bending by a pendant acting on the free end. This pendant also functions as a force transmitting mechanism so that the bending force acting on the beam can be sensed and measured. The advantage of this design is that it allows only a minimal film deformation so that the maximum stress can be measured. In this paper, we present results from our initial investigation to demonstrate the feasibility of this novel method.

#### 2. The apparatus

The operation principles of self-developed stress apparatus for film drying is illustrated in Fig. 1. The whole device was housed inside a sealed transparent wooden-framed Perspex chamber with dimensions) of  $\,$  710 mm  $\times$  445 mm  $\times$  705 mm  $\,$  (length  $\times$  width  $\times$ height). The chamber was secured on the top of a solid bench to avoid any vibration and external disturbance. Inside the chamber,



Fig. 1. A schematic illustration of the apparatus capable of measuring the stress development of a drying film.

a thick cast-iron plate topped with a layer of polished stainless still was used as the base, also for the benefit of anti-vibration stability. A stainless metal beam was used as the substrate for film coating and drying. The beam was clamped at one end on to a brass block, which sits on a lubricated sliding trough of the base so that it can be pushed and pulled smoothly to its designated position. The beam was made from a Starrett feeler gauge and has a thickness of 400 lm (Starrett Feeler Stock, Cat No. 667M, The L.S Starrett Company Ltd., Jedburgh, Scotland). The beam has a suspend length of 80 mm and a width of 12.6 mm. Near the free end of the beam, a small indent was made so that the tip of the pendant can rest on it with a precise position. This makes the effective length for film coating 73 mm, from the centre of indent to the clamping wall. In order to enhance the grip or adhesion of coating fluid, the surface of the beam was slightly and uniformly treated with a fine sand paper. A texture analyser (Stable Micro Systems, Surrey, UK) was used to determine the mechanical strength of the beam by measuring the bending force and the deflection displacement of the beam (shown in Fig. 2). The elastic modulus of the beam, E, can then be calculated using the following equation:

$$
E = RM/I,\tag{1}
$$

$$
I = bh^3 / 12
$$
 (2)

where R is the radius of the bended beam, M is the moment of bending,  $I$  is the second moment of cross-section area,  $b$  and  $h$  are the width and the thickness of the beam. The radius of the bended beam can be estimated as an approximation using the following equation:

$$
R = l^2 / 2d'
$$
 (3)

where  $l$  is the effective length of the beam and  $d$  is the deflection distance of the beam. Taking the fact that the beam has an effective length of 73 mm, a width of 12.6 mm and a thickness of 0.4 mm, one can estimate that the beam should have an elastic modulus of 289.0 GPa, larger than the modulus for the one used in previous work (190 GPa) ([Wedin et al., 2004](#page--1-0)).

Beneath the indent, a small disc was attached and dipped into an oil reservoir to form a dashpot. This arrangement was found to be extremely useful in restraining beam vibration for a stable reading. The pendant has its own weight of 97.6423 g. The length of the pendant hook was adjustable so that it can be firmly hooked to the balance but only in a gentle touch with the beam. The net force/weight acting on the beam can be read from the difference of balance reading before and after touching the beam. Normally a net weight of around 1 to 1.5 g was applied to the beam, which makes sure that the tip of pendant is in firm touch with the beam but that the initial bending to the beam is also negligible.



Fig. 2. The deflection curve of the substrate beam used for film drying.

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