



Zero discharge fluid dynamic gauging for studying the thickness of soft solid layers



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ABSTRACT

Fluid dynamic gauging (FDG) is an experimental technique for measuring the thickness and strength of solid layers on solid or porous substrates by relating the flow rate of liquid through a nozzle positioned near to, but not touching, the layer. This paper shows that such measurements can be made with the liquid both being ejected from and sucked into the nozzle, so that there is no net flow of liquid into or out of the system. This zero discharge (ZFDG) mode has considerable potential for sterile and aseptic operation. Discharge coefficient profiles are reported for a range of flow rates and show very good agreement with computational fluid dynamics simulations, indicating that the shear stress imposed on the surface can be estimated with confidence. The use of ZFDG to study the thickness and removal of soft layers was demonstrated with layers of a commercial petroleum jelly (Vaseline[®]) layer. The Vaseline[®] layers exhibited cohesive breakdown on 316 stainless steel, indium tin oxide coated polyethylene terephthalate (PET), and glass under suction mode testing. The cohesive strength agreed with the yield stress measured in separate rheological tests. When exposed to ejecting liquid on glass, however, the layers switched from cohesive breakdown to an adhesive rupture mechanism which is attributed to an instability in the layer and the observed tendency to slip on smooth substrates.

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1. Introduction

Layers of soft solid material on surfaces immersed in liquid are found throughout the chemical, process and biotechnology industries. Some layers are desired, such as coatings and the biofilms on membrane and solid substrates used in biological waste treatment (Miura et al., 2007). Others are unwelcome, such as biofilms causing biofouling and corrosion, and heat exchanger fouling deposits which reduce heat transfer efficiency, product quality and hydraulic performance (Epstein, 1983). Knowledge of how the layer responds to shear imposed by a flowing liquid is important for ensuring that the layer remains on the surface or, conversely, to promote its removal.

We focus here on applications related to the cleaning of fouling layers but the approach is generic. Food fouling layers exhibit a wide range of behaviours, which Fryer and Asteriadou (2009) classified in terms of their response to chemical and mechanical action. Selection and optimization of cleaning methods requires techniques which can measure the thickness and mechanical properties of such layers measured *in situ*, in real time, under conditions representative of the process environment (Wilson, 2005). Replicating process conditions using scaled-down apparatuses is time- and resource- intensive, resulting in the develop-

ment of bench scale techniques which can be used to study particular aspects of cleaning, such as micromanipulation (Liu et al., 2002; Hooper et al., 2006); cleaning cell (Jurado et al., 2011); radial flow cells (Detry et al., 2009); and atomic force microscopy (Liu et al., 2007).

This paper reports a step change in the fluid dynamic gauging (FDG) technique developed by Tuladhar et al. (2000) for measuring the thickness of soft fouling layers immersed in liquid *in situ* and in real time. This non-contact technique was extended by Chew et al. (2004a), using computational fluid dynamics simulations, to determine the strength of the layer. The FDG technique has subsequently been used to study a range of soft solid layer materials, including biofilms (Salley et al., 2012), with a resolution of thickness measurement currently about $\pm 5 \mu\text{m}$ (Gordon et al., 2012). The technique exploits the relationship between the flow rate through a nozzle such as that shown in Fig. 2, the pressure drop across the nozzle, ΔP , and the clearance between the nozzle and the layer surface, h . The mass flow rate, \dot{m} , is very sensitive to the clearance when the clearance is small, *i.e.* $h/d_t \leq 0.25$, where d_t is the diameter of the nozzle throat. This dependency is used to locate the surface: parallel measurement of the vertical location, z , allows the layer thickness, δ , to be calculated.

In early FDG devices \dot{m} was measured while ΔP was held constant (termed mass flow mode). Later devices measured ΔP at constant \dot{m} , (termed pressure mode), which facilitated measurement at elevated pressure and temperature (Ali et al., 2013). Prior to the work by

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Nomenclature

Latin

a, b, c	fitting parameters, Eq. (2) (-)
C_d	discharge coefficient (-)
d_t	nozzle diameter (m)
d_{tube}	tube diameter (m)
h	clearance (m)
h_c	cohesive failure clearance (m)
\dot{m}	actual mass flow rate (g s^{-1})
\dot{m}_{ideal}	ideal mass flow rate (g s^{-1})
ΔP	pressure drop (Pa)
p_1	nozzle outside pressure (Pa)
p_2	nozzle inside pressure (Pa)
$P_{\text{footprint}}$	pressure of layer surface underneath nozzle (Pa)
Re	Reynolds number (-)
Re_t	Reynolds number in nozzle throat (-)
r	radial co-ordinate (m)
r_i	inner rim of nozzle (m)
r_o	outer rim of nozzle (m)
w_e	nozzle edge width (m)
w_r	nozzle rim width (m)

z distance from clean substrate (m)

Greek

δ	layer thickness (m)
θ	nozzle angle ($^\circ$)
μ	dynamic viscosity (Pa s)
ρ	density (kg m^{-3})
τ	shear stress (Pa)
$\bar{\tau}$	average shear stress, Eq. (4) (Pa)
φ	spherical co-ordinate

Acronyms

CFD	computational fluid dynamics
FDG	fluid dynamic gauging
ITO	indium tin oxide
LED	light emitting diode
PET	polyethylene terephthalate
SS	stainless steel
ZFDG	zero flow fluid dynamic gauging

Salley et al. (2012), FDG devices had employed suction flows into the nozzle but they showed that the technique worked equally well when the liquid was ejected from the nozzle. Salley et al. (2012) employed a syringe pump to deliver liquid at a controlled, low, flow rate and the use of such units suggests that ejection and suction could be used, in turn, to make measurements over an extended period with no change in the total volume. This concept, of zero discharge FDG, or ZFDG, is demonstrated here. ZFDG has particular advantages for applications requiring (i) aseptic conditions; (ii) minimizing the inventory of (potentially valuable or hazardous) liquid; and (iii) devices with a small footprint. ZFDG is therefore ideal for studying biofilms or soft tissues that are very sensitive to interruption, infection or contamination. The apparatus employed here fits on an optical microscope stage but could equally well be installed on a laser scanning confocal microscope, allowing microstructure mapping similar to that reported by Wagner et al. (2009).

In this paper an FDG apparatus based on that reported by Salley et al. (2012) is operated in both ejection and suction modes. The discharge coefficient for the nozzle is calculated and compared with CFD simulations. Good agreement is obtained, indicating that the shear stress exerted on the layer surface can be estimated with reasonable accuracy. Application of the technique to soft solids is demonstrated using layers of a model material, Vaseline[®], which is a hydrophobic gel consisting of low melting point waxes which exhibits viscoplasticity at room temperature. The onset of deformation of the Vaseline[®] layers observed during FDG testing in water is compared with estimates of its yield stress obtained from parallel rheological tests. In addition to serving as a model material, the mechanical properties of the Vaseline[®] layers are similar to those of fouling layers formed by food fats in process pipelines (Huang et al., 2012) and semi-solid food products such as spreads (e.g. Marmite, White et al., 2008) and molten chocolate (Taylor et al., 2009).

2. Materials and methods

2.1. Fluid dynamic gauging

Fig. 1 shows a schematic and a photograph of the FDG apparatus. A detailed description of the nozzle assembly is given in Salley et al. (2012). A syringe pump (Cole Parmer 210-CE, flow rate accu-

racy for infuse and withdraw modes 0.35%) forces liquid into or out of a convergent nozzle of throat diameter 0.97 mm. The nozzle is located above the test plate by a rigid frame: the position of the nozzle relative to the test plate, z , is adjusted by a vernier micrometer (accuracy $\pm 1 \mu\text{m}$). The plate is located at the centre of a cylindrical perspex tank of internal diameter 105 mm. Flow near the plate was evened out by a cylindrical ring wall (55 mm inner diameter) containing many 2 mm holes. Four white LED lights were mounted in the ring wall (Farnell[™] Electronics, typical value 18,000 mcd) to illuminate transparent test plates being observed during testing by an inverted optical microscope (GX Microscope XJL-17AT).

The nozzle geometry and dimensions are reported in Fig. 1. A pair of absolute pressure transducers (Omega Engineering, PX26) were used to measure the pressure difference across the nozzle. The uncertainty in the differential pressure value due to individual sensor error was significant for $\Delta P < 30 \text{ Pa}$ so an inclined manometer (accuracy $\pm 1 \text{ Pa}$) was used to measure small ΔP values. Low ΔP readings correspond to the nozzle being far from the layer surface, so the FDG technique does not require high accuracy and sensitivity sensors for measuring layer thickness. A Labview[™] (version 7.1) application was used to control the automated parts and collect data.

Measurements of the flow rate and pressure drop yield the nozzle discharge coefficient, C_d , via

$$C_d = \frac{\dot{m}}{\dot{m}_{\text{ideal}}} = \frac{4\dot{m}}{\pi d_t^2 \sqrt{2\rho\Delta P}} \quad (1)$$

where ρ is the liquid density and \dot{m}_{ideal} is the ideal mass flow rate through the nozzle, in the absence of flow losses, for a given value of ΔP . Calibration tests were performed with a clean substrate in place to generate plots of C_d versus h/d_t for a given mass flow rate. These calibration curves were compared with the general functional form identified by Gordon et al. (2010):

$$C_d = a \left\{ 1 - \exp \left[-b \left(\frac{h}{d_t} + c \right) \right] \right\} \quad (2)$$

where a , b and c are fitted parameters. Eq. (2) was then used to determine the clearance, h , during tests on Vaseline[®] layers and the thickness of the layer, δ , calculated from $\delta = z - h$.

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