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A multi-method approach to quality control illustrated on the industrial powder coating process

Denis Schütz*, **Elke Riedl**, **Richard Romirer**, **Katja Hartmann**, **Oliver Sack**

Anton Paar GmbH, Anton Paar Strasse 20, 8054 Graz, Austria

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ABSTRACT

Industrial powder coating processes offer a unique object of application for rheometers.

Compared to classic coating procedures with liquid materials, powder coating is still a relatively young procedure. First industrial applications date back to the mid-1960s. Since then these applications have considerably increased, which can be mainly attributed to the desire for more environment-friendly coating systems. The powder coating process is dependent on one end on pneumatic conveyance behavior, namely the transport as a dense fluidized phase of powder and the associated bulk solid values, on the other end on bulk polymer behavior to determine and keep constant the curing point at which the applied powder bonds and produces an (ideally) smooth surface. This multivariant process is an ideal object of quality control for the MCR rheometers and two accessories: the Powder Cell and a disposable Plate–Plate Geometry with Peltier heated hood (P-PTD200, H-PTD200).

In this work we will show the influence of both artificial aging behavior as well as flow aids on the powder flow properties (collapsed and fluidized) as well as the melting and polymerization steps and show a way to optimize processes utilizing this complex system.

1. Introduction

Powder coatings are a growing technology with numerous applications. Initially designed to provide greater resilience than comparable liquid applied coatings the desire for an environment-friendly solvent free process nowadays drives the development and application of this technique (Huang et al., 2010).

It consists of several processing steps. Initially the powder coating is kept fluidized before being introduced into the pneumatic transport stage leading up to the electrostatically assisted application (via spraying) onto a prepared surface. After application the applied coat is then introduced into a heat treatment stage where the polymerization reaction takes place and the coat both bonds to the surface as well as needs to form a continuous and smooth surface.

This necessitates rather complex mixtures of powders including but not limited to e.g. systems that use two co-polymers along with the catalysts (so called hybrid coatings) or (in the simplest cases) such as polyethylene coatings where the polymer mixture consist of the monomers along with the necessary catalysts (Samimi and Soroush, 2011).

To ensure the smooth operation of this process several material variables need to be present within the powder mixture, which serves as a raw material (Mazumder et al., 1997).

It needs to be fluidizable to ensure both a good transport as well as application to the substrate. The fluidization constants (both minimum as well as full) need to be known in order to ensure optimal calibration of the equipment. To further ensure optimal usage of the equipment it would be of use to measure a value of apparent viscosity of the system. The industry standard here is to use DIN/ISO 3081:1–12, which is currently being updated.

This is a less than ideal situation since the transport process but especially the spraying process subjects the fluidized powder to vastly different shear rates and fluidized beds have complex shear rate dependent viscosity akin to some complex fluids. This will be discussed along with the measurements below. In brief we use a cylinder setup (profiled) and calculate the apparent viscosity through the use of the Bingham equations (DIN 53019 and ISO 3219 as well as (Mezger, 2006)). This is similar in setup to the work of Tardos et al. (who did not calculate viscosity) Schügerl et al. and more recent Chen et al. (Tardos et al., 2003; Ritzmann et al., 1974; Landi et al., 2012; Chen et al., 2015).

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* *Corresponding author*.
E-mail address: denis.schuetz@anton-paar.com (D. Schütz).

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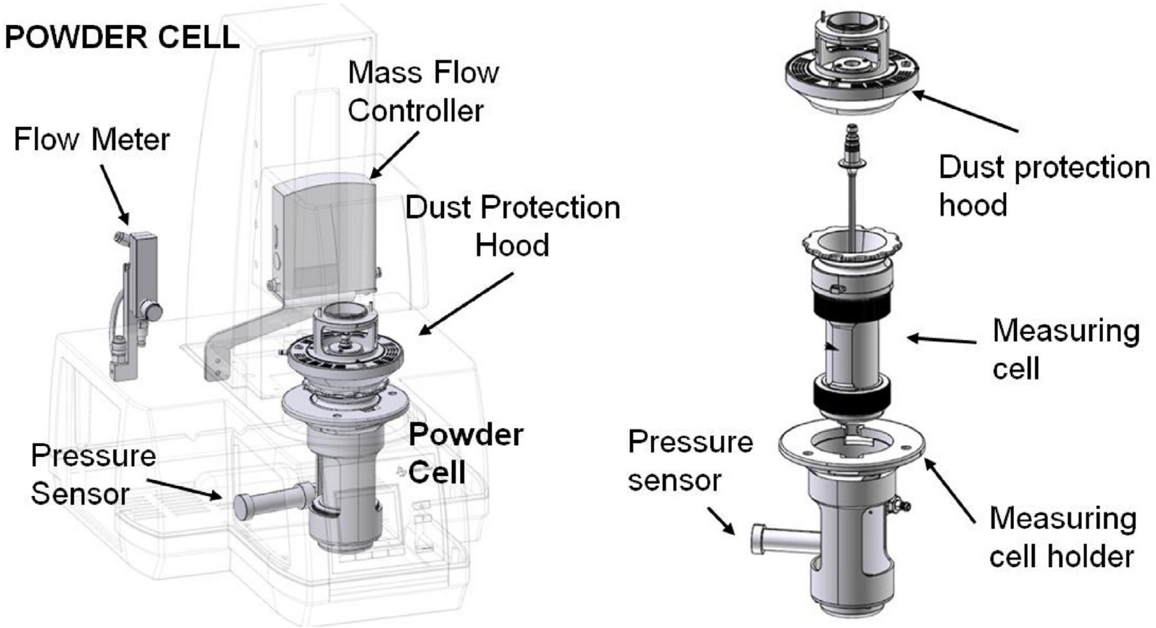

Fig. 1 – Overview of the setup used for the powder rheology and pressure drop measurements this is the Anton Paar Powder Cell within a MCR Rheometer (left) and a detailed explosive view of the Powder Cell insert itself (right). The pressure sensor is below the porous medium (frit) attached to a pressure manifold that ensures laminar flow along the sensor. The frit itself is mounted under the measuring cell.

In the unfluidized state it needs to have high enough cohesion (interparticulate adhesion at zero applied normal force) so the coating does not “crumble off” before or during introduction to the heat treating stage.

It needs to be transportable in the manner of a continuous dense phase fluidized flow (Crowe et al., 2011; Mills et al., 2004; Yang, 2003), meaning it needs to have a high air retention capability (usually checked by bed collapse experiments) as well as (checked in the previous step) the necessity to be fluidizable.

Lastly if there are any steps involving hoppers or even silos of course the powder mixtures need to be able to discharge continuously and without core flow.

In general powder coatings can be assigned into Group C of Geldarts classification (Geldart, 1973), both from their fluidization behavior as well as their particle size. As such the unstirred fluidization behavior is often uneven and channels are expected to form during the fluidization tests for this reason both an unstirred as well as a stirred fluidization point is gathered and compared.

Characteristic for this application is the inclusion of fine particles of fumed (pyrogenic) silica (or alternatively Al_2O_3) as a flow aid as well as helping with fluidization, in principle even degraded powders can be made flow satisfactory, however while improving the powder flow behavior significantly the pyrogenic silica will also affect the melting point, molten viscosity and polymerization of the coating.

To this effect we added 1 permille by weight of pyrogenic silica to demonstrate the effect on both flow behavior as well as melting and polymerization characteristics.

In case of a melting point this may lead to obvious problems in the process. The influence on the viscosity in the molten state is detrimental in regard to the evenness of the coating on the surface and is therefore also of large importance for the process itself. Within reason, the lower the viscosity, the better the surface finish.

Most important however is the influence of the flow aid on the polymerization process, meaning that too much flow aid addition can lead to a delay and therefore an incomplete polymerization and a drastically less resilient coat if it is not addressed downstream of the process. Like the molten viscosity this can be addressed by changing the temperature of the heat treating process.

The melt rheological steps simulate the heat treatment (curing) process by employing parallel plate geometry and a Peltier heating stage in an oscillatory measurement mode, more details below.

Being a reactive mixture that is polymerized in-situ, shelf life plays an important role both for the powder flow measurements as well as the later melt rheology steps to this effect we heat-treated the powder to simulate degradation. Consequently a larger amount (0.5% per weight) of flow aid was added to demonstrate the effects.

The influence of flow aids on powder flow aids have largely been attributed to the large size difference and therefore an easier separation of the grains during flow in the collapsed state (Chen et al., 2008), a similar explanation is given to the increase in fluidizability (Fulchini et al., 2017), the influence of apparent viscosity is as of yet unexplored.

2. Experimental

2.1. *Equipment and raw materials*

The used powder coating system was Tiger Lacke: 009 polyester–epoxide hybrid coating (color RAL 3000).

Particle size is given at: “$\sim$80 μm” by the manufacturer.

46.35 g corresponding to approximately 80 ml of material was used for every measurement.

Since the mixture is still highly reactive, an artificial aging step was added to simulate storage. This was done by heat treating the powder at 45 °C (10 °C below the nominal melting point) for 1 h, significantly changing the flow behavior in similar to the slow reaction taking place in storage at ambient temperatures. Heat treated is used in analogue later in this work.

The used fumed silica (pyrogenic silica) was Evonik Aerosil R974.

For powder measurement a MCR 302 with a Powder Cell scientific (Fig. 1) was used. The Torque accuracy is given at ± 10 nNm for this setup. A mass flow controller capable of airflow from 0.05 to 5 l/min was connected. The measurement tube was 50 mm in diameter and an uncoated borosilicate glass tube was used. As a porous plate material borosilicate glass frit of porosity 4 (Schott) was inserted. The measurement systems were: a profiled cylinder for fluidization (24.4 mm diameter; 40 mm length 10 mm gap to the porous frit) and apparent