

Real-time in-situ Rheological Assessment of Sticky Point Temperature and Humidity of Powdered Products[†]

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Abstract

Unwanted changes in powder flow behavior can unexpectedly occur when a product is exposed to certain conditions of temperature and humidity. This can happen during production, but also during transport or storage. The work reported here demonstrates the novel approach of using an amended powder rheology set-up for measuring and predicting such changes in powder flow behavior. The developed methodology makes it possible to vary in-situ the temperature and the relative humidity of the air to which the product is exposed, thereby mimicking realistic conditions of production or related unit operations. An air flow capable of fluidizing the powder particles is controlled at a specific constant temperature and its relative humidity can be altered while measuring the torque in the fluidized powder bed in real time. The fluidization is necessary for generating a homogeneous introduction of temperature and relative humidity. Results obtained using citric acid and commercial coffee whitener products have proven this methodology to provide both similar and in certain instances dissimilar results compared to the more established methodology such as measuring the vapour adsorption isotherms. These observations are explained. In this way, it can be predicted under which combinations of temperature and humidity a product does or does not become sticky. The main advantages of our approach are that the flow properties are directly assessed, the interpretation of the obtained data is more straightforward and that the measurement times are shortened substantially.

Keywords: powders, stickiness, powder flow, caking, humidity

1. Introduction

A wide array of products is in powder or granular form. Quite often these products are available as free-flowing materials for easy transporting, packaging, dosing and handling by the customer. In this respect, the prediction and investigation of powder flow is an important measure (Schulze D., 2008). Different techniques are available for investigating powder flow, and measurement conditions can be varied from non-consolidated to highly consolidated systems. Leturia M. et al. (2014) have convincingly shown that one or more appropriate measurement techniques should often be applied in order to get an understanding of the product's flow properties under realistic

conditions. Surprisingly, Salehi H. et al. (2017) have recently shown that even similar methodologies working with the conventional Jenike shear and ring shear principle can provide inconsistent results. This all makes a reliable assessment of the powder flow properties a complicated task. To make it even more challenging, besides the intrinsic characteristics of the product expressed as particle size, particle shape, porosity and surface area and density, also extrinsic conditions can be of vital importance.

The exposure of stable and non-stable granular products to certain conditions of temperature and relative humidity can transform free-flowing and thus easy-to-handle products into more cohesive and sometimes even sticky materials, eventually leading to caking and clogging of machinery and packaging. Examples of materials that are sensitive towards stickiness are numerous and can be found throughout all industries, e.g. in the food industry (Juarez-Enriquez E. et al., 2017) but also in the pharmaceuticals (Lumay G. et al., 2016; Sandler N. et al., 2010) and chemicals industries (Stanford M.K. et al., 2002 and Sun C., 2009). This is considered a major issue in the powder handling industry and although these phenomena



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are highly undesirable, they often come unexpectedly. The effect of temperature on the flow properties of materials has already been investigated using a high-temperature annular shear cell (Tomasetta I. et al., 2013). Here the changing flow properties of consolidated powders were compared while varying the temperature, yet the humidity was not regulated.

The prediction of stickiness due to interaction with water at a certain temperature is a complicated task and not easily feasible for newly developed or modified products. Moreover, the fact that some quite recent publications deal with the subject of powder flow in relation to extrinsic conditions such as moisture and temperature confirms the relevance of the topic (Lumay G. et al., 2016; Emery E. et al., 2009). In these studies, however, typically the water content of the material was studied as the main descriptor rather than the impact of the relative humidity in the air, although current literature by Juarez-Enriquez E. et al. (2017) strongly recommends that the latter option is much more appropriate.

The interaction of the material of interest with moisture from the environment can be studied by recording water vapor sorption isotherms (Mathlouthi M. et al., 2003). The uptake of water is then measured as a function of the relative humidity at a certain temperature and the isotherm evidences when a certain interaction of water with the material is present. Often kinetic models, such as the two-parameter BET or the three-parameter GAB model (Goula A.M. et al., 2008), are applied to determine the monolayer coverage of the material, the latter model being often used for food products. The models can be used to identify the possible presence of sticky behavior, yet they are not applicable for every material. Since it is not always clear what actually is causing the water uptake, these measurements are often complemented by techniques capable of determining the glass transition temperature (Emery E. et al., 2009). Below the glass transition temperature, an amorphous material behaves as being in a glassy state with a very high viscosity due to the limited molecular movement at low temperatures (Downton G.E. et al., 1982). However, above the glass transition temperature, a glassy material starts to transform into a 'rubbery state' and the viscosity of the material decreases considerably. A relationship between the glass transition temperature and stickiness was already established for a wide range of food powders. Interestingly, however, the glass transition temperature is highly dependent on the presence of water (vapor) in the system, and Bhandari B.R. and Howes T. (1999) as well as Goula A.M. et al. (2008) have convincingly shown that water vapor typically has a strongly depressing influence on the glass transition temperature. This means that techniques capable of measuring phase transitions while working with often a constant (inert) carrier gas flow are restricted to adequately take into account the implication of water.

Furthermore, a phase transition of an amorphous material from a glassy state into a rubbery state does not necessarily have to occur in order to make a powder material sticky. Hygroscopic materials that interact with water can also display sticky-like properties, turning a free-flowing powder into a cake-like compound without being accompanied by a phase transition. Even for materials that are not hygroscopic, an enhancement of inter-particle forces by liquid bridges due to an increased relative humidity has been proven, e.g. by measuring the onset of avalanches of glass beads (Soria-Hoyo et al., 2009). Even for glass beads up to 4 mm, this enhancement of inter-particle forces could be seen. Furthermore, the particular liquid bridging behavior is highly dependent upon surface and solvent polarity as shown in recent work (Jarray et al., 2019). The complex admixture of different polarities in industrially relevant samples (such as the complex admixture of carbohydrates, fatty acids and proteins present in coffee creamer) increases the difficulties. From the adsorption isotherm only, it is often difficult to derive sticky point conditions, even despite the presence of different kinetic models. An additional complicating factor is that stickiness also depends on particle morphology such as the size and the shape of the particles (Shi H. et al., 2018). This impressive set of variables means that assessment of the stickiness of a certain product preferably requires a study of the particular product under realistic conditions of temperature and (relative) humidity.

An attractive tool would thus allow a direct assessment of powder flow under varying conditions of moisture and temperature. First studies by Lazar M.E. et al. (1956) that already appeared in the 1950's report on stickiness in which an impeller stirred by hand was used to identify changes in resistance in the powder material upon increasing the temperature. Though being an effective methodology, the sensitivity of the method was clearly too low to detect the early onset of stickiness.

More recently, Özkan N. et al. (2002) reported on the use of a powder rheometer to accurately measure the sticky point. In this study different variables—temperature, time, and pressure of compaction—were investigated, however, only the moisture content in the product was altered and no study was done on the impact of the relative humidity of the surrounding atmosphere that would be realistic, e.g. during storage and transport. Other works reported so far have mostly used external cabinets to accommodate and equilibrate powder samples externally under certain conditions of temperature and/or relative humidity.

In the work reported here, we discuss the in-situ variation and control of temperature and relative humidity during which changes in the flow of the powder materials are recorded in real time by a novel powder rheometer



set-up in order to determine the range of conditions at which the material can become sticky.

2. Experimental

2.1 Materials

Commercially available coffee creamer with the product name Completa (Friesland Campina, The Netherlands) was purchased from the local grocery store and used without further modification.

All materials were kept in their original containers and stored in a glovebox under nitrogen in order to avoid interaction with moisture.

Citric acid monohydrate with a purity of > 99% was acquired from Boom Chemicals (The Netherlands) and was used with minor modification. The only modification applied was reducing the particle size using a mortar. This was done to obtain a powder with a particle size $< 300 \, \mu m$ that could be fluidized in the rheometer set-up. The citric acid monohydrate sample thus obtained appears to contain 8.2 wt.% water, whereas a true monohydrate should have 8.6 wt.% of water. The implication of the slightly lower water content is discussed in the results and discussion section.

2.2 Methods

Water vapor sorption isotherms were recorded in an IGASorp HT from Hiden Isochema, which employs the dynamic vapor sorption principle. A microbalance with sub-microgram resolution is used to record weight changes due to the adsorption or desorption of water vapor molecules. Isotherms were recorded in a temperature range of 15–75 °C and a relative humidity range of 5–90 %. In a typical experiment, approx. 10–20 milligrams of sample were used.

Powder rheometry investigations were executed in a modified Anton Paar MCR 302 rheometer equipped with the optional powder cell. The cell is cylindrical with a diameter of 50 mm and has a porous material (borosilicate glass Frit; Por. 4 (Schott Duran)). The stirrer used was a two-blade stirrer of rectangular shape measuring 36 mm × 10 mm, and continuously held at a 10 mm gap. Modifications to the stock cell consist mostly of an exchange of the bottom plate of the cell holder from stainless steel to a custom-made PEEK model to avoid extensive heat loss through conductive transfer. Fluidization of the powder sample was used in the actual analysis in order to achieve a homogeneous introduction of the water vapor during exposure to different relative humidity values. A fluidization velocity from 2.5-12.7 cm/s was set for the experiments. Although fluidization of the sample induces substantially lower torque values compared to a packed bed, the sensitive torque sensor and the low residual friction of the air bearing in the rheometer makes accurate and repeatable measurements possible under these conditions. The two-bladed spindle that was used for a continuous recording of the torque in the sample was rotated constantly at fairly high rpm values (50 rpm) to further improve the homogeneous interaction of the water vapor within the sample. The air used for fluidization, controlled by a mass flow controller, was heated by means of an evaporation mechanism powered by an external heater kept constantly above 105 °C and heat tracing of the connection lines. The relative humidity was regulated by means of a Harvard PHD-2000 infusion syringe pump device integrated into the heated gas line for instant evaporation and subsequent dosing of the required amount of water, thereby attaining the desired relative humidity value.

3. Results and discussion

The first experiments performed were using the coffee creamer. Both the water vapor sorption isotherms and the torque curves are recorded at a certain fixed temperature as a function of a stepwise change in relative humidity over time. The original data obtained were therefore plotted as a function of time and can be replotted as a function of relative humidity. For the conventional DVS method, the original data are displayed in **Fig. 1**, and show the uptake of water vapor due to the increase of the relative humidity over time. Initially, the uptake is relatively low and equilibrium is reached fairly quickly.

At a certain RH value, the uptake becomes more significant, and reaching equilibrium requires more time. Therefore the measurement time is different for each

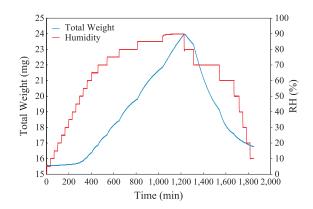


Fig. 1 Typical dynamic vapor sorption curve of the coffee creamer sample at 25 °C, with increase and decrease of the relative humidity at this temperature and the weight change of the material due to this relative humidity change.



point, as it is only when equilibrium is obtained that the relative humidity is increased in order to record a new data point. Upon reaching the maximum RH value of 90 %, the relative humidity is decreased, to identify whether the desorption of water vapor is reversible to the adsorption of water vapor. Usually, for materials that undergo a phase change or materials that are able to strongly adsorb water vapor, the desorption isotherm is not reversible to the adsorption isotherm, which is a direct indication that the material has changed during the measurement. Visual inspection of the material under investigation after the measurement can confirm whether or not this change has occurred. Using the weight change of the material at each equilibrium point, the isotherm can be constructed.

The torque curve obtained with the Anton Paar Rheometer set-up is displayed in Fig. 2 and shows the torque changes due to the increase of relative humidity over time at a certain constant temperature. The humidity change is a manual operation and the stepwise increase is executed with a fixed time interval. At the end of each relative humidity point, the torque is measured and the friction isotherm can be plotted. The main advantage of the technique is that the actual flow behavior of the material is measured, and thus that the increase of the torque at a certain relative humidity value actually describes that the flow ability of the material changes. With the DVS technique, the uptake of water vapor is not necessarily an indication that the flow properties of the material have changed and that the material has become sticky. It is therefore also not necessary to record a decrease in humidity with the rheometer set-up, as the change in flow behavior is directly monitored. This also translates to a much faster determination of the sticky points of materials, as with the Anton Paar Rheometer a measurement can be performed within 6 h, while with the DVS technique, a measurement could last several days.

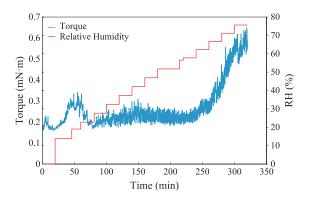


Fig. 2 Torque curve of the coffee creamer sample at 23 °C obtained with the Anton Paar Rheometer, with an increase of the relative humidity at the set temperature and the torque change of the material due to this relative humidity change.

3.1 Powder Rheometry of Coffee Creamer

The isotherms obtained from the torque profiles of the coffee creamer sample at different temperatures are plotted in Fig. 3. The isothermal plots show a stable torque value at lower relative humidity values and an increased torque value when the relative humidity exceeds a certain value. The increase in torque is a direct indication that the flow properties of the material are decreasing and thus that the material becomes sticky. Two distinct regions can be discerned and a linear fit has been plotted in these regions. The cross-section of both fits determines the point where excess water is present and the material becomes sticky. The resulting sticky point values for the coffee creamer at different temperatures are quantitatively summarized in Table 1.

Since this approach is solely based on the flow properties of the material, the sticky point does not have to be derived from a water vapor sorption isotherm using a certain method or model, as is the case for DVS. The sticky point can be directly determined and is a direct indication of when the flow properties change and thus when the material becomes sticky.

3.2 Water Vapor Sorption of Coffee Creamer

The water vapor sorption isotherms are given in Fig. 4, where the sample weight is plotted as a function of relative humidity at different temperatures. The dynamic vapor adsorption measurements with water vapor over the coffee creamer sample reveal that for each temperature, at lower relative humidity values, an almost stable weight is obtained. For the measurements performed at all four temperatures, three distinct regions can be discerned, namely a region where the material shows hardly any uptake, a quite linear second region where the sample weight of the material steadily increases and, at higher humidity values, a third region can be seen where after the steady increase of the sample weight, a sort of exponential increase occurs. The uptake seen can be caused by adsorption in pores of the material, adsorption on the external surface area or by the transition of a certain component (from amorphous to crystalline). Each of these regions describes a different state the material is in, and in order to know when the materials become sticky, to derive the sticky point, is not an easy task. Since the sticky point has to be derived from the obtained isotherms, it can be debated what is the best method to do so.

The GAB (Guggenheim-Anderson-de Boer) model is widely used on food materials and could provide the best fit. The model has been applied on the acquired data sets, and the monolayer uptake is obtained, from which subsequently the sticky point can be determined. The obtained sticky points are listed in **Table 1**.

A similar procedure as used when obtaining the Anton Paar values was applied that determines the intersection



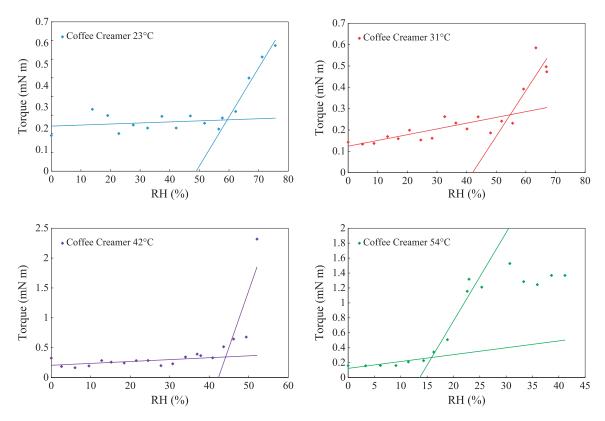


Fig. 3 Torque isotherms of the coffee creamer sample at 4 different temperatures, 23 °C, 31 °C, 42 °C and 54 °C, with the fitted regions in the isotherms obtained with the Anton Paar Rheometer technique.

Table 1 Sticky point values for coffee creamer recorded via the powder rheometer methodology and derived from the water vapor sorption isotherms using the various methods, in the temperature range from 23–54 °C.

<i>T</i> rheometer °C	T DVS °C	Sticky point rheometer % RH	Sticky point DVS, GAB model % RH	Sticky point DVS, linear fit (1 and 2) % RH	Sticky point DVS, linear fit (1 and 3) % RH	Sticky point uptake point DVS % RH
23	25	59.4	62.2	51.9	70.5	50
31	30	54.7	39.8	46.0	68.2	45
42	40	44.1	43.3	40.5	70.4	40
54	54	15.9	27.8	32.4	71.7	35

of two linear fits through the first two regions in the isotherms. The suitability of this procedure has been verified for materials and isotherms where the GAB model was applied and this has proven to produce consistent—insignificantly different—results. However, the isotherms display three different regions and it is unclear in which region the material starts to become sticky. Linear region 1 (the region of low relative humidity values of the isotherm) is fitted and regions 2 and 3 have been fitted and the results of both fittings are listed in **Table 1**.

Another method that can be used to derive the sticky point of a material from the water vapor adsorption isotherms is by taking the point where the material starts to take up water, the uptake point method, and these points have also been added to **Table 1**.

Table 1 clearly illustrates for the data obtained with the Anton Paar rheometer that with an increasing temperature the sample becomes sticky at a lower relative humidity value. The data display a linear trend, with the exception of the sticky point determined at 54 °C, which deviates from this fit. During this measurement, it could be observed that water vapor insertion into the gas stream occurred unequally. This led to spikes where the RH values were increased, which caused the material to become sticky at an earlier stage than expected, not corresponding to the set RH values. Therefore, this point is omitted in further calculations and comparisons.

The sticky points obtained with the GAB model show some similarities but also some large deviations when compared with the sticky points of the rheometer.



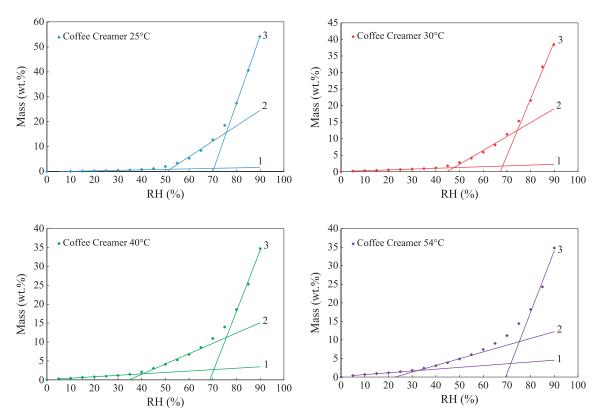


Fig. 4 Adsorption isotherms of the coffee creamer sample at 4 different temperatures, 25 °C, 30 °C, 40 °C and 54 °C, with the three fitted regions in the isotherms obtained with the Dynamic Vapor Sorption technique.

Furthermore, a linear trend between the 4 sticky points obtained with the GAB model cannot be obtained. During fitting of the isotherms with the GAB model it could already be seen that the data could not be fitted adequately. Lewicki P.P. (1997) and Maroulis Z.B. (1988) have shown that the regression method used to fit the model can result in inaccurate results, but also that not all isotherms can be accurately fitted using the model. In our case, the coffee creamer material is not completely dry when adsorption commences and this results in a minor mass decrease, which in turn does not allow an accurate fit of the GAB model. This procedure is used in order not to alter the material prior to the measurement which could occur, and is described vide infra for the citric acid material. The model has been applied on the isotherm obtained over dried coffee creamer material and this resulted in an adequate fit and good correlation with the rheometer values. It is thus very important how the vapor sorption data is obtained whether or not the GAB model can be applied.

Fitting of the linear regions of the isotherms results in sticky points that have a rather good correlation with the data obtained with the rheometer, and a nice linear trend is found when regions 1 and 2 are used, as using the high relative humidity region (region 3) results in sticky points that do not correlate with the rheometer results and also do not provide a nice linear trend. The latter statement

can be made regarding the results obtained when the sticky point is determined using the uptake point method, at the onset of excess water adsorption.

In a Mollier diagram the temperature is plotted versus the absolute quantity of water, which is expressed as kg water per kg air. The obtained sticky points at a certain temperature are converted from RH values to absolute water quantities and can be plotted in such a diagram. Using the best fit, a curve can be constructed through the obtained data points and two regions are obtained, a sticky region and a non-sticky region. The Mollier curves derived from the sticky points obtained with the Anton Paar Rheometer and the DVS system are shown in Fig. 5. The Mollier diagrams of only the GAB model and the linear fit of the regions 1 and 2 have been calculated, since only these methods provide linear results.

The relative humidity values of the sticky points obtained with the rheometer method and the linear fitting methods are rather similar when compared with each other, and the Mollier diagrams derived from these humidity values are also very similar, indicating that consistent results can be obtained with both methods. Both diagrams show that the top of the curve is at 80 °C, with 0.043 kg water/kg air obtained with the rheometer and 0.044 kg water/kg air with the DVS system using the linear fit methodology. Both diagrams also have a maximum



temperature of approx. 100 °C, which matches nicely with the maximum degradation temperature obtained with a TGA experiment performed on the material. The relative humidity values of the sticky point obtained with the GAB model were already quite different compared to those of the rheometer, and distinct differences can be seen when the Mollier diagram is constructed from these results. The top of the curve is at 65 °C with 0.026 kg water/kg air, and the maximum temperature has shifted from 100 °C to 80 °C.

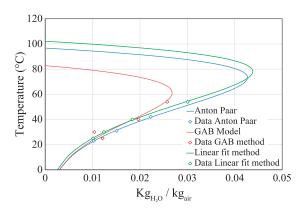


Fig. 5 Mollier diagram of the coffee creamer material derived from the sticky points obtained via the Anton Paar Rheometer and the DVS system (GAB model and linear fit).

If one would simply apply the GAB model and use the data to construct the Mollier diagram, an underestimation would be made, which will negatively impact on the flexibility in handling the material during production and storage.

3.3 Powder Rheometry of Citric Acid Monohydrate

The isotherms obtained from the torque profiles of the citric acid monohydrate sample at different temperatures are plotted in **Fig. 6**. The isothermal plots show a stable torque value at lower relative humidity values and an increase when the relative humidity exceeds a certain value. Two distinct regions can be discerned and a linear fit has been plotted in these regions. The cross-section of both fits determines the point where excess water is present and the material becomes sticky. The resulting sticky point values for the citric acid monohydrate at different temperatures are quantitatively summarized in **Table 2**.

3.4 Water Vapor Sorption of Citric Acid Monohydrate

The dynamic vapor adsorption isotherms over the citric acid monohydrate material are plotted in Fig. 7 and reveal that initially, at lower relative humidity values, the material displays a loss in mass rather than an uptake. This initial loss in mass can be ascribed to the (partial) transition of citric acid monohydrate to citric acid anhydrate. Then a

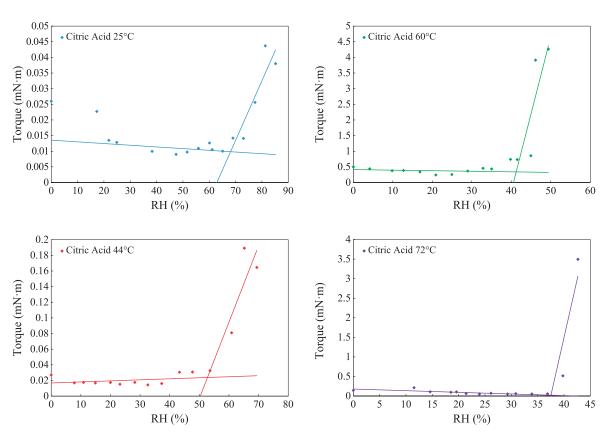


Fig. 6 Torque isotherms of the citric acid monohydrate sample at 4 different temperatures, 25 °C, 44 °C, 60 °C and 72 °C, with the fitted regions in the isotherms obtained with the Anton Paar Rheometer technique.



nice plateau is obtained and at a certain relative humidity value, a substantial linear uptake can be discerned. This linear uptake could be caused by several phenomena, namely due to the transition of the anhydrate to the monohydrate form or by uptake of water in the pores and/or external surface area of the material or by multilayer adsorption. A third phenomenon was also observed, namely that at these higher relative humidity values coupled to the temperatures, the material started to liquify. So besides the fact that it is not always evident where to derive the sticky point from a water vapor isotherm, the fact that all these phenomena occur simultaneously makes it almost impossible to make a good distinction and prop-

Table 2 Sticky point values for citric acid monohydrate recorded via the DVS using the curve fitting method and powder rheometer methodology in the temperature range of 25–72 °C.

T rheometer °C	T DVS °C	Sticky point rheometer % RH	Sticky point DVS % RH
25	25	66.4	69.0
44	40	52.7	66.7
60	65.7	41.2	61.7
72	71.3	37.5	57.3

erly determine the sticky point of the material, even though it was visually observed that the material became sticky during the measurements.

The GAB model could not be applied for this material, and again the procedure using linear fitting over the two regions has proven to be the most adequate.

Drying the material prior to the measurement eliminates the weight loss observed at low relative humidity values. However, the water vapor uptake occurs at the same relative humidity values as the undried material.

For the data obtained with the Anton Paar rheometer, **Table 2** also clearly illustrates that with an increasing temperature the sample becomes sticky at a lower relative humidity value. A linear trend can be fitted through all four data points.

Fitting of the linear regions of the water vapor isotherms results in sticky points that can be fitted with a linear trend, yet clearly deviate from the results obtained with the rheometer, as the relative humidity values of the obtained sticky points via the linear trend method are much higher. This is due to the fact that during the uptake of the citric acid monohydrate at higher relative humidity values, different phenomena occur (change from anhydrous to monohydrate, water uptake at the external surface area and liquefaction), and the DVS system is not able to distinguish between these several phenomena. This therefore

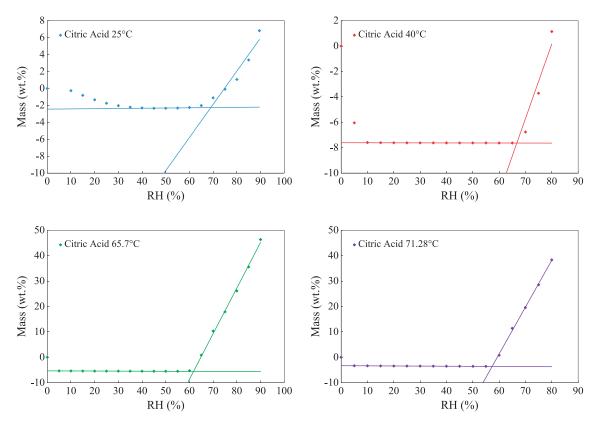


Fig. 7 Water vapor isotherms of the citric acid monohydrate sample at 4 different temperatures, 25 °C, 40 °C, 65.7 °C and 71.3 °C, with the fitted regions in the isotherms obtained with the Dynamic Vapor Sorption technique.



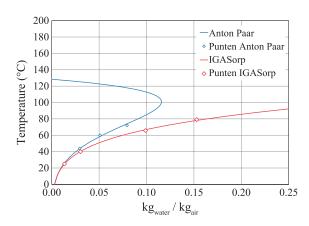


Fig. 8 Mollier diagram of the citric acid monohydrate material derived from the sticky points obtained via the Anton Paar Rheometer and the DVS system (linear fit).

leads to an incorrect determination of the sticky point.

When the Mollier diagrams are constructed, the large differences between the two techniques become even more apparent. The Mollier diagram constructed with the rheometer data shows a nice top around 100 °C with 0.12 kg water/kg air and a maximum temperature around 130 °C. This maximum temperature nicely approaches the melting point of citric acid, which is at 153 °C.

The Mollier diagram constructed with the DVS system has a top at a very high temperature of 290 °C with 7.5 kg water/kg air. The maximum temperature is around 360 °C. These values suggest that for almost all temperatures and moisture contents present, the material will not become sticky, while it was clearly observed that the materials do become sticky, and the maximum temperature exceeds the melting point of citric acid where the material will surely become sticky. This thus indicates that for more complex materials, the DVS technique is not really capable of accurately determining the sticky points and constructing the Mollier diagram.

4. Conclusions

From this study it can be concluded that with the rheometer set-up and linear fit approach, results can be obtained that correlate very nicely with results obtained from the DVS method. In some cases, e.g. the citric acid material, the rheometer set-up is capable of producing more reliable results.

This is due to the fact that the rheometer set-up directly measures the flow properties of a material and is thus capable of directly assessing the sticky point. The DVS technique measures water vapor uptake and is not capable of making a distinction between the several phenomena that may occur during the measurement.

Furthermore, the sticky point has to be derived from the water vapor sorption isotherms using a model or method, and this could easily result in inaccurate results, while with the rheometer, such problems can be avoided.

Another advantage of the rheometer set-up is that the measurement times are drastically reduced.

Acknowledgements

"This scientific work could not have been completed without the practical efforts of Swasti Soekhradj."

Nomenclature

BET Brunauer–Emmett–Teller
DVS Dynamic Vapor Sorption

GAB Guggenheim-Anderson-de Boer

PEEK Polyether ether ketone
RH Relative Humidity (%)
TGA Thermogravimetric Analysis

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Authors' Short Biographies



Johan Groen

Johan Groen studied chemistry in Delft and obtained his PhD. in chemical technology from the Delft University of Technology on the theme of zeolite catalysis. During his stay at the university, Johan published over 40 scientific papers and contributions in books, and he is the inventor of one patent. In 2008, Johan co-founded Delft Solids Solutions, a contract research institute in the Netherlands devoted to particle and powder engineering, of which he is co-owner and science & technology director. His interests lie in the coupling between fundamental properties of particles and powder behaviour typically encountered in the powder handling industry.



Wim Kooijman

Wim Kooijman studied Chemistry in Rotterdam and obtained his BsC. in chemistry from the Hogeschool Rotterdam in September 2017. Wim wrote his graduation thesis about powder stickiness as an intern at Delft Solids Solutions in Wateringen. After he graduated Wim joined Chevron Oronite Technology Netherlands as a Laboratory Technician. In March 2019, Wim switched jobs, becoming a Project Technician for the development of Automotive Engine Oils at Chevron. He is interested in analytical and organic research in Chemistry, which results into the development of new, more environmentally friendly products.



Authors' Short Biographies



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Djamilla van Belzen studied Chemistry at the University of Applied Sciences in Leiden. Djamilla got her B.Sc. in 2018 after her internship at Delft Solids Solutions. After her internship Djamilla started to work for CarbonX, located in Delft, as a QA/QC technician. Her interests lie in the physical part of chemistry and technology.



Gabrie Meesters

Dr Gabrie Meesters has 27 years of industrial formulations experience at Gistbrocades, Genencor International and DSM. He has 23 years of experience as a part-time Professor at TU Delft in particle technology and product design. From January 2019, he became a full-time Assistant Professor at the TU Delft, Faculty of Applied Sciences, Product and Process Engineering. He is a contributor to several books on formulations. He is the editor of three books on product design and solids processing. He published over 70 refereed papers, has more than 20 patents and supervised more than 100 BSc, MSc and PhDs. He is a regular speaker at conferences and workshops. He was the organizer of the 2010 Partec and the 2010 World Congress on Particle Technology in Nuremberg, Germany.



Timothy Aschl

Timothy Aschl holds a PhD in chemistry from the École Polytechnique, France, and a Master's degree in applied physics from the Technical University of Graz, Austria. His research was focused on biosensors and biofuels. Timothy joined Anton Paar as a global product manager for powder rheology, and in this role he is a frequent speaker at seminars and training courses for companies and universities for a wide range of branches and industries around the world.



Denis Schütz

Denis Schütz earned his Master's degree in chemistry at the University of Graz in 2009 and afterwards joined the PhD programme for materials chemistry and chemical engineering at TU-Graz, finishing in 2013. He did postdoc research at Graz University of Technology and postdoc research at Jozef Stefan Institute. At present he is principal scientist for powder rheology and applied scientist for materials at Anton Paar GmbH. His current interests are in granular media and powder flow research as well as high-temperature processing and device building.



Patrick Verolme

Patrick Verolme studied chemistry in Rotterdam and received his Bachelor of Science degree from the Rotterdam Hogeschool, after performing his graduation study at Delft Solids Solutions on the topic of water vapor sorption studies. Since 2012, Patrick has been working at Delft Solids Solutions in the field of solid particle/powder research, with his main area of expertise being specific surface area, porosity and vapor sorption studies. His role has expanded to the research of bulk powder characteristics, on which he is a frequent speaker at seminars and courses.