Journal of Food Engineering 111 (2012) 186–189

Contents lists available at SciVerse ScienceDirect

Journal of Food Engineering

journal homepage: www.elsevier.com/locate/jfoodeng

Research Note

Influence of the type of gelatinized starch on the soiling of stainless steel

Susann Zahn, Annett Wehner, Harald Rohm *

Institute of Food Technology and Bioprocess Engineering, Technische Universität Dresden, 01069 Dresden, Germany

ARTICLE INFO

Article history:
Received 27 July 2011
Received in revised form 6 January 2012
Accepted 26 January 2012
Available online 6 February 2012

Keywords:
Soiling
Surface cleaning
Starch
Gelatinization

ABSTRACT

Quality assurance of cleaning procedures is important as regards production efficiency, food safety and food quality. Model routines for the assessment of cleaning methods require that materials can be fouled in a reliable manner. The present study was performed to demonstrate that a reproducible soiling of stainless steel can be achieved as long as the flow characteristics of the soiling fluid (i.e., different types of gelatinized starch) and specific aspects of the corresponding procedure for the quantification of soil residues are considered.

1. Introduction

The evaluation of surface cleaning methods plays a key role in quality assurance in the food and pharmaceutical industry. Insufficient procedures may have serious consequences on product safety, product quality and storage properties. It is therefore a prerequisite in responsible processing that the respective hygiene standards are fulfilled, and that cleaning routines receive appropriate attention so that contamination or cross-contamination during production is prevented with a high degree of assurance (Hasting, 2008). Because, on the other hand, cleaning is time-consuming, strategies are required to reduce the corresponding economical and technical effort without impairing product quality. This could be done, for example, by altering the interactions at the interface between machine surface and contamination (Liu et al., 2006). Another possibility is to increase the efficiency of the cleaning routine by optimizing the design of components such as nozzles, or to alter fluid dynamics at the machine/fouling interface to speed-up fouling removal (Augustin et al., 2010).

To benchmark cleaning procedures by considering the amount of residue which is left after cleaning is therefore of tremendous importance. Such tests can be performed with real devices (e.g., pumps, valves, and tableware) and real foods (Benezech et al., 2002; Sigua et al., 2011), or with model substances and procedures for soiling and cleaning (Detry et al., 2011; Wernersson et al., 2004; Whitehead et al., 2010). Model soiling substrates mentioned in literature are predominantly proteins (e.g., whey proteins) and polymeric carbohydrates (e.g., starch), which are known to cause problems during cleaning.

Experimental techniques for soiling food contact surfaces in model systems vary to a great extent, and include spraying organic soils onto surfaces (Boyd et al., 2000; Detry et al., 2009, 2011), soaking specimens in test solutions for a defined period of time (Alameda et al., 2011; Mauermann et al., 2009), soiling surfaces using scraping modules (Augustin et al., 2010), or soiling surfaces using micropipettes and spreaders (Lang et al., 2011; Sigua et al., 2010; Whitehead et al., 2009, 2010). In most studies, the main emphasis has been placed on the detection of residues after applying a cleaning procedure rather than on reproducibility of the preceding method for surface soiling. Potential differences in adhesion behavior, which may result from source, pre-treatment or modification of starches used as model soil (e.g., Emengo et al., 2002; Glenn et al., 2010; Nwokocha, 2011), were scarcely considered.

The aim of the present study was to develop a simple protocol for soiling food-contacting surfaces with respect to reproducibility, and to evaluate differences in adhesion between starch-based model substrates.

2. Materials and methods

2.1. Starch samples

Two native starches from corn (NC; Maisita 21.000) and waxy corn (NWC; Maisita 21.007), three pregelatinized starches from corn (PC; Quemina 21.200), waxy corn (PWC; Quemina 21.214) and potato (PP; Quemina 21.216), and one modified cook-up starch (acetylated waxy corn, MWC; Agenajel 20.320) were obtained from Agrana Stärke GmbH (Aschach, Austria). Starch moisture was determined by drying at 103 ± 1 °C in an oven (triplicate measurements).

* Corresponding author. Address: Institute of Food Technology and Bioprocess Engineering, Technische Universität Dresden, Bergstrasse 120, 01069 Dresden, Germany. Tel.: +49 351 463 32420; fax: +49 351 463 37761.

E-mail address: harald.rohm@tu-dresden.de (H. Rohm).

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doi:10.1016/j.jfoodeng.2012.01.027

2.2. Starch paste preparation

Defined amounts of native and cook-up starch samples were weighed into the outer cylinder (d = 43.4 mm) of a Rheostress 1 rheometer (Haake GmbH, Karlsruhe, Germany), made up with demineralized water (50 °C) to 90.0g, and vigorously mixed with a spatula. After mounting the cylinder on the instrument, a four-bladed vane tool (d = 40 mm, h = 55 mm) was lowered into the suspension. Temperature around the outer cylinder was adjusted to 50 °C by means of a circulator, and vane tool rotation was set to 150 rpm. The temperature/time regime for gelatinization was selected in accordance with ICC standard (ICC, 1992). After 3 min at 50 °C, temperature was linearly increased to 90 °C within 20 min (i.e., 2 K/min) and kept constant for 15 min. After reducing rotational speed to 50 rpm for further 7 min at 90 °C, temperature was reduced to 50 °C at a rate of 1.33 K/min. The measurement cycle was concluded by a 3 min rotation at 50 rpm and 50 °C. Torque was recorded continuously during gelatinization using the RheoWin 3.5 software. To minimize evaporation, the rheometer beaker was covered with parafilm.

Pregelatinized starches were dissolved in demineralized water (50 °C) using a glass beaker and a magnetic stirrer device. After complete dissolution, the starch pastes were transferred into the rheometer. At 50 °C, torque was monitored at vane tool rotation of 150 rpm for 15 min and, subsequently, at 50 rpm for another 15 min.

2.3. Measurement of starch paste viscosity

The vane tool was calibrated using torque/stress and rotational speed/shear rate conversion factors as described previously (Krulis and Rohm, 2004). Immediately after gelatinization, flow curves of the starch pastes were measured at 50 °C by reducing shear rate from 200/s to 0.01/s in 31 log-spaced increments within 15 min. The shear rate in the system depends on vane and cylinder radius and angular velocity, and a rotational speed of 50 rpm corresponds to a shear rate of 31.4/s.

2.4. Soiling of stainless steel

Prior to use, electropolished stainless steel coupons (40.0 × 20.0 × 1.0 mm; X2CrNiMo17-12-2) which were mounted onto a fixture for handling were cleaned with water and a detergent, rinsed with demineralized water and ethanol, and dried. A 5 mm wide parafilm strip was then wrapped around the coupons to ensure a uniform accessible surface of 25.0 × 20.0 mm on either side (Fig. 1). After starch gelatinization in the rheometer, the stainless steel coupons were dipped into the starch paste for 2 s. Liquid remnants were allowed to drip off the coupon by holding it above the rheometer beaker in a 45° angle for 20 s, and multiple contact with a soft tissue ensured that no bulging remnants occurred at the specimen edge. Finally, the parafilm strip was carefully removed, and the coupons were allowed to dry at room temperature.

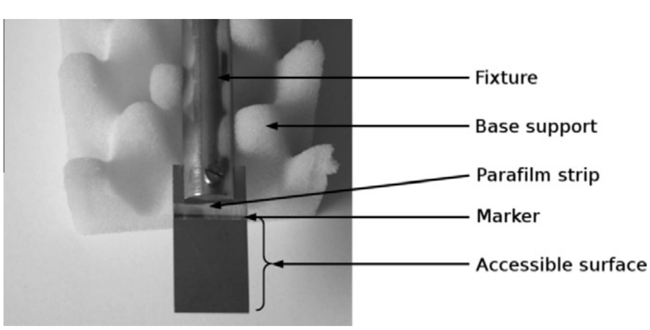

Fig. 1. A test coupon mounted on the fixture.

2.5. Starch removal from stainless steel coupons

The soiled, dry coupons were transferred into plastic tubes filled with approximately 20 mL of 2 mol/L NaOH. The tubes were placed in an ultrasonic cleaning bath and sonicated for 60 min at 60 °C. Each coupon was then rinsed with demineralized water above the tube. pH was adjusted to 4.5 ± 0.1 with 10 mol/L HCl, and the liquid was quantitatively transferred into a 50 mL volumetric flask and made up with demineralized water.

For the evaluation of the starch removal procedure, 50–100 µL drops of starch paste were spread on one side of the steel coupons using a micropipette. The absolute amount of starch transferred onto the specimen was calculated from drop mass determined by an analytical 770-60 balance (Kern & Sohn GmbH, Balingen-Frommern, Germany), and concentration of the starch paste. Finally, the starch film was allowed to dry.

2.6. Starch determination

Starch concentration in the solutions with the removed material was, after appropriate dilution, analyzed in duplicate by means of an enzymatic test assay (R-Biopharm AG, Darmstadt, Germany; Beutler, 1978). In preliminary tests, the specific recovery of the assay was checked using all starches under study. After gelatinization of aqueous suspensions in measuring flasks, recovery values ranged between 97% and 103% (n = 4).

3. Results and discussion

3.1. Preliminary experiments

By considering moisture content of the starch samples, gelatinization experiments were performed at different starch concentrations. Based on the torque at the end of the gelatinization cycle (data not shown), concentrations of each starch were systematically varies until a torque of 1.5 mNm (accuracy ± 5 %) was obtained. It is evident from Table 1 that concentrations necessary to achieve that torque ranged between 35 and 90 g/kg. The development of torque during starch gelatinization (native and cook-up starches), and the dependency of pastes of pregelatinized starch on measurement time is evident from Fig. 2. It was possible to generate systems which exhibited 1.5 mNm after the specific time/temperature/rotation treatment despite differences in torque evolution during gelatinization, and despite torque differences at high rotational speed for pregelatinized starch. As torque is proportional to shear stress, it can also be used as a measure of apparent viscosity as long as rotational speed is kept constant.

Fig. 3 shows averaged (n = 3) flow curves of the gelatinized starch pastes, which were recorded immediately after cessation of the gelatinization experiments at 50 °C. Apparent viscosity at a shear rate of 36.2/s, which is close to the shear rate in the second stage (low rotational speed) of the gelatinization experiments, ranged between 253 ± 12 mPa.s for MWC starch paste, and 273 ± 15 mPa.s for PP starch paste. It is also evident that rheological properties of the pastes differ to some extent. Pastes of NC and MWC showed Herschel–Bulkley behavior with a yield stress of 0.7 and 1.5 Pa and a power law exponent of approximately 0.55. A similar relation between shear stress and shear rate has also been observed by, e.g., Lee and Yoo (2011) and Berski et al. (2011). NWC and PC pastes had a lower yield stress (0.1–0.2 Pa), and the power law exponent was approximately 0.65. For PP and PWC, yield stress was negligible, and the exponent was 0.75 and 0.76, respectively.