# INFLUENCE OF RHEOLOGICAL PROPERTIES AND PULL-OFF FORCES OF NATIVE AND MODIFIED STARCHES ON CLEANING IN PLANE CHANNEL FLOW

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# ABSTRACT

Consumer safety and product quality are of high priority in the food industry. Strongly adhering deposits are formed in processing equipment such as plate heat exchangers, which demand large quantities of water, chemicals, energy and time for cleaning. This study presents an approach to characterize soil properties and to link them to the cleaning behavior to generate a basis for soilspecific cleaning. Six starch soils were rheologically analyzed during swelling, pull-off forces were measured with a micromanipulation device, and swelling progression was determined with a camera. Cleaning experiments were conducted in a plane channel setup. A reptation time without cleaning and a subsequent constant cleaning rate defined the cleaning behavior. The observed cleaning mechanisms were considered as well. Multivariate statistics revealed significant interrelationships between soil properties and cleaning behavior. The complex reasons for the overall cleaning behavior remain unknown, but certain aspects could be explained by the measured soil properties.

# **INTRODUCTION**

Cleaning of strongly adhering deposits is a challenging but crucial task in the food industry as product quality and consumer safety highly depend on safe cleaning processes. Following the latest market trends, food producers strive to individualize their products and reduce batch sizes. This leads to an increasing number of product changes and the necessity of frequent cleaning (Claassen and Hendrix, 2014). However, product-specific cleaning requirements are often not fully known. Since these cleaning processes are not optimally adapted to the respective soil, the use of water, chemicals, energy and time is oversized in many cases. Consequently, knowledge of the individual physicochemical properties of a soil is essential to ensure environmentally friendly cleaning (Fryer and Asteriadou, 2009, Herrera-Márquez et al. 2020, Agüeria et al., 2021).

As regards the cleaning behavior of different soils, four cleaning mechanisms can be distin-

guished. Viscous shifting occurs in viscous deposits. Diffusive dissolution requires the solubility of soil components, while cohesive separation involves breaking up cohesive bonds within the soil layer and removal of larger fragments. Adhesive detachment refers to the debonding of the soil from the substrate (Fryer and Asteriadou, 2009; Joppa et al., 2017; Helbig et al. 2019; Köhler et al. 2021). The composition and structure of the soils, which also depend on drying and ageing, and the cleaning fluid largely define the cleaning mechanism. Furthermore, the mechanical properties of food soils may change during cleaning (Fryer and Asteriadou, 2009). Polymeric networks such as proteins or long-chain carbohydrates swell during contact with the cleaning fluid. Cleaning starts after a short time, called reptation time, when molecular chains disengage into the fluid or when the fluid deforms and displaces the soil layer (Devotta et al., 1994; Xin et al., 2004; Kricke et al., 2021).

Numerous studies investigated the mechanical properties of soils and attempted to relate them to cleaning behavior. Comparatively often, relations between the cleaning behavior and rheological properties of soil layers were found (e.g., Xin et al., 2002; Othman et al., 2009; Goode et al., 2010; Wilson et al., 2014; Palabiyik et al, 2018; Yang et al., 2019). Xin et al. (2002) were the first who linked the cleaning rate of whey protein concentrate deposits to their mechanical properties determined by oscillatory rheometry and indentation tests. The oscillatory shear rheometry method reflects mechanical properties at small strains. In Contrast, rotational rheometry (Wilson et al., 2014, Palabiyik et al., 2018) examines rheological properties at larger deformations. Wilson et al. (2014) highlighted that the effect of temperature on the cleaning rate during impinging jet cleaning of petroleum jelly was directly related to the effect of temperature on critical shear stress. Using rotational and oscillatory rheometry, Palabiyik et al. (2018) identified a linear relationship between a dimensionless cleaning time and the ratio of critical shear stress to surface shear stress for viscoplastic fluids.

However, rheological studies were usually performed on homogenous soil films or pastes placed between roughened parallel plates. In contrast, Helbig et al. (2019) applied and dried egg yolk deposits on the bottom plate of a rheometer. The rheological properties of the soil layers were investigated in different cleaning fluids during swelling.

Micro- or millimanipulation devices (Ali et al., 2015; Cuckston et al., 2019; Köhler et al., 2021) examine adhesive and cohesive interactions at large deformations. Tsai et al. (2020) and Fernandes et al. (2021) used a micromanipulation device to estimate the critical shear stress of viscoplastic soils in contact with the cleaning fluid and related it to the cleaning behavior. Köhler et al. (2021) used micromanipulation to link the measured adhesive strength to the water mass fraction at the soil-substrate interface and coupled this to the cleaning time of ketchup soil layers.

Fluid dynamic gauging (FDG) is a method to study the thickness of swelling soil layers immersed in a cleaning fluid (Tuladhar et al. 2002; Wang and Wilson, 2015; Pérez-Mohedano et al. 2016). Thereby, the suction force or pressure can be used to measure the strength of soft soil layers (Chew et al. 2004; Hooper et al. 2006).

However, only few studies consider that the rheological properties of soils change during swelling. Regardless of the soil, various authors indicated that cohesive and adhesive strengths decrease with swelling time. (Liu et al. 2006; Helbig et al., 2019; Cuckston et al., 2019; Yang et al., 2019).

Starch is widely used in the food industry as thickener, stabilizer, and gelling agent. Since products containing starch often adhere strongly at the inner surface of pipes and accessories, different cleaning parameters such as temperature, chemistry of the cleaning fluid and methods to analyze the removal of dried starch layers were investigated (Jurado-Alameda et al., 2015; Otto et al., 2016; Vicaria et al., 2017; Sauk et al. 2018; Kricke et al., 2021). The swelling and cleaning behavior of starch soils is affected by the chemical composition and the starch structure, which can result from different botanical origins. Furthermore, varying cleaning mechanisms were observed depending on alkaline concentration and temperature of the cleaning fluid. However, the cleaning behavior of different starches could not be fully explained with the determined swelling properties (Kricke et al., 2021).

This study aims to analyze the influence of strength and elasticity of swelling soil layers on the cleaning behavior of four native and two modified starches. Soil layer thickness measurements were used to obtain information on the swelling progress which supported the interpretation of rheological measurements and pull-off properties obtained from micromanipulation measurements in conjunction with cleaning experiments.

# MATERIALS AND METHODS

#### Investigated soils and cleaning fluids

Four native starches from different botanical sources (waxy maize (WMS), Cargill Deutschland GmbH, Hamburg-Rothenburgsort, Germany; potato (PS), wheat (WS), AGRANA Beteiligungs-AG, Tulln, Austria; rice (RS), BENEO GmbH, Mannheim, Germany), a modified waxy maize starch (acetylated distarch adipate (ADA-S), Cargill Deutschland GmbH, Hamburg-Rothenburgsort, Germany) and a modified potato starch (hydroxypropyl distarch phosphate (HDP-S), Unilever Deutschland GmbH, Auerbach, Germany) were investigated. Soil layers were prepared as described by Kricke et al. (2021). The concentration of each starch was adjusted in a way that, after gelatinization at 95 °C for 45 min with stirring at 1000 rpm, the viscosity of the starch pastes enabled homogenous application using a pipette: WMS and WS, 3.5 g/100 g; PS, 2.5 g/100 g; RS, ADA-S and HDP-S, 3.0 g/100 g. Luminescent, stabilized strontium aluminate crystals (0.0267 g/g dry starch) were added to ensure UV detectability of soil layers during the cleaning tests. Stainless steel substrates (AISI 304; 2B finish;  $R_z \le 1 \mu m$ ) measuring 40 x 20 mm<sup>2</sup> were soiled for micromanipulation and swelling experiments, and 150 x 80 mm<sup>2</sup> substrates were used for cleaning experiments. The dried soil mass coverage  $m_{s,0}^{\prime\prime}$  of the samples was set to  $50 \pm 5$  g/m<sup>2</sup> by applying an appropriate mass of gelatinized starch paste and drying at standard climate (23 °C, 50% relative humidity) for 18 h. For the rheological measurements, the concentration of the starch pastes was adjusted to obtain thicker, plane soil layers: WMS, 10.0 g/100 g; WS and RS, 7.5 g/100 g; PS, 4.5 g/100 g; ADA-S, 3.5 g/100 g; HDP-S, 6.0 g/100 g.

All experiments were carried out at five test conditions: with deionized water at 25 °C or 55 °C, using sodium hydroxide with a concentration of 1.0 g/100 g at 40 °C, as well as with 2.0 g/100 g sodium hydroxide at 25 °C or 55 °C (abbrev. T25H2O, T55H2O, T40N10, T25N20, T55N20).

#### **Rheological analyses**

To measure the rheological properties of the starch layers during swelling, a MCR 300 rheometer (Anton Paar GmbH, Ostfildern, Germany) with plate-plate geometry (diameter 50 mm) and Peltier temperature control was used. Dynamic experiments were carried out based on Helbig et al. (2019) with the following adjustments. The soil layer was applied using an acrylic ring with an inner diameter of 60 mm and a thickness of 2 mm. To dry the starch paste, the lower plate was heated to 55 °C for 60 min, followed by an adjustment to the test condition temperature within 5 min. The tempered test fluid was spread by a pipette, and

soaking was stopped after 1 min by lowering the upper plate until a normal force of 15 N was reached. An anti-evaporation cover was fitted. A dynamic time sweep (1 % strain, 1 rad/s) was applied for 15 min to determine the absolute value of the complex modulus  $|G^*|$  as well as the loss factor tan  $\delta$ . The total soaking time was 200 s until the first measured data point. The measurements were repeated for at least three times.

#### **Micromanipulation experiments**

A micromanipulation device described by Helbig et al. (2019) was used to determine the strength of the soil layers, which includes adhesive and cohesive binding forces that need to break during deformation and displacement of the soil layer (Ali et al., 2014; Tsai et al., 2020). After defined soaking times  $t_{\text{soak}}$  of 60 s or 300 s, the soiled substrate was positioned at a gap distance of  $100 \pm 10 \,\mu\text{m}$  between the substrate surface and the lower edge of the blade. The force required to pull off the soil at a constant scraper blade speed of 2.6 mm/s was measured at a frequency of 100 Hz with a force sensor (KD40s, 2 N, ME-Meßsysteme GmbH, Hennigsdorf, Germany).

The pull-off forces were normalized with respect to substrate width *B* of 20 mm to obtain the normalized pull-off force (*F*/*B*). Averaging these pull-off forces ( $\overline{F}/B$ ) over substrate length *L* of 40 mm, as described by Köhler et al. (2021), made it possible to compare the results of the different soils and fluids by one characteristic scalar value.

## Soil layer thickness measurements during swelling

The growing thickness of the starch layers was investigated in a swelling test setup (Köhler et al., 2021). The probe was placed horizontally in a heatable and transparent tank and monitored with a camera and zoom lens (Zoom 70 XL, TV-Tube 1.0X, Opto GmbH, Gräfelfing, Germany) during soaking for 1800 s. Snap shots of a representative swelling experiment are shown in Figure 1. The thickness increase  $\Delta h(t) = h(t) - h(t = 0)$  was captured time-resolved for the whole swelling process using a contrast based evaluation script.



Fig. 1. Swelling progress of a waxy maize starch layer in T55H2O in the swelling test setup.

#### **Cleaning experiments**

The cleaning behavior of the starch soils was investigated in a plane channel flow test setup as depicted and thoroughly described by Joppa et al. (2017). The 150 x 80 mm<sup>2</sup> samples were inserted in a  $78 \times 5 \text{ mm}^2$  cross-sectional plane flow area.

Cleaning was carried out for 1800 s at a bulk velocity of 1 m/s, resulting in turbulent flow with Reynolds numbers ranging from 10,300 to 18,500 depending on the temperature of the fluid. The fluorescent tracer embedded in the soil layer was excited by UV lamps through a transparent PMMA cover and allowed the cleaning observation by a monochrome camera. The gray values were measured over time and averaged within a centered 40 x 40 mm<sup>2</sup> range of interest. The intensity  $I_{raw}(t)$ was normalized with respect to initial intensity  $I_{raw}(t = 0)$  to ensure comparability of the different cleaning measurements.

Joppa et al. (2020) already introduced a method to distinguish the swelling induced intensity increase from the actual cleaning progress. Relevant swelling experiments were carried out within the cleaning test setup. The reptation time  $t_{rep}$ , during which no cleaning occurs, and mean cleaning rate  $\overline{m}''_{s}$  were determined from the intensity plots (Kricke et al., 2021). The cleaning mechanism was additionally documented from visual inspection for all cleaning experiments as introduced by Köhler et al. (2019). Diffusive dissolution and cohesive separation could not be differentiated from macroscopic observations. Therefore, cohesive separation, viscous shifting and adhesive detachment were used to describe the observations.

# Statistical analyses

The statistical analyses were carried out with SPSS 27.0 (SPSS Inc., Chicago, USA). Significant differences between mean values were identified by one-way analysis of variance followed by a Tukey-B post-hoc test at  $P \le 0.05$ . Relationships between rheological, pull-off, swelling, and cleaning properties were identified using nominal or, in case of  $|G^*|$ , logarithmic data by applying principal component analysis (PCA).

#### **RESULTS AND DISCUSSION**

#### **Rheological properties**

Dynamic shear rheometry is able to provide information about the viscoelastic behavior of materials. The complex modulus  $G^*$  describes the entire deformation resistance of a material and includes the storage modulus G' and the loss modulus G'' as real and imaginary part of a complex variable. The loss factor tan  $\delta$  represents the ratio of viscous to elastic contributions.

For most starches, the complex modulus and loss factor slightly changed within the first three minutes of measurement and thereafter reached a stable level (data not shown). The complex modulus mostly showed an initial increase in T25H2O and an initial decrease in T55N20. ADA-S showed a steady decrease of the complex modulus and increase of the loss factor in all cleaning fluids throughout the whole measurement period. The branched network of solid bonds in the acetylated distarch adipate is less susceptible to swelling than the network of native starches (Leach et al., 1959). Unfortunately, the rapid change in rheological properties of native starch soil layers were only recorded to a limited extend caused by the methodrelated 200 s time delay after soaking started.



Fig. 2. Rheological properties a) complex modulus  $|G^*|$  and b) loss factor tan  $\delta$  of all investigated starches and fluid properties after soaking for 200 s. Error bars represent standard deviation of three replicate measurements. Mean values with different letters within a starch type show significant difference at  $P \leq 0.05$ .

The complex modulus and loss factor depended on the type of starch as well as on temperature and sodium hydroxide concentration of the fluid (Figure 2). WS had the highest complex modulus and lowest loss factor of all analyzed starches, therefore it had a higher deformation resistance with a more elastic behavior. On the other hand, the soil layers of WMS were more viscous with the highest loss factor and less deformation resistant. The high loss factor of WMS might result from a high water solubility of WMS in comparison with other starches (Schmidt et al., 2022). During swelling in heated water, the WMS layer became more viscous, while the ADA-S soil layer was most elastic. In contrast, NaOH in both concentrations weakened the soil network of ADA-S but increased the loss factor during swelling at all temperatures. The loss factor of PS was low in water or T40N10, but twice as high when in contact with high concentrated sodium hydroxide (2.0 g/100 g). Nor Nadiha et al. (2010) assumed that the high polymerized amylose of potato starch is less vulnerable to low concentrations of sodium hydroxide but tends to alkaline hydrolysis when a critical concentration is exceeded. During alkaline hydrolysis, monomers of PS split off, resulting in a high water solubility (Schmidt et al., 2022) and therefore a loss of elastic bindings.

# Pull-off properties from micromanipulation experiments



Fig. 3. Representative force plots F/B during pulloff of potato starch soil layers after soaking in T25H2O for 60 s, T40N10 and T55N20 for 300 s.

Three representative types of force plots were determined in the micromanipulation experiments (Figure 3). Caused by a larger surface area, fluid penetration was obviously pronounced at the edges of the soil layer, resulting in reduced pull-off forces at the beginning and the end of the measurement. Depending on the fluid used, the rate of penetration varied. The cohesive binding forces in the center of the soil layer remained resistant to deformation for a longer time, e.g., after soaking of PS in T25H2O for 60 s. In contrast to the continuously increasing and decreasing pull-off force after soaking of PS in T25H2O, the forces after soaking of PS in T40N10 for 300 s were comparably low at the beginning and at the end of the force measurement. Higher forces were only measured in a small central part of the soil layer. Once the fluid completely penetrated the soil, the pull-off forces decreased over the entire length, as demonstrated by the graph of F/Bafter soaking of PS in T55N20 for 300 s.

The averaged pull-off forces are shown in Figure 4 for all starches and fluid conditions after soaking for 60 s. The same tendencies, but at lower magnitudes, were observed after soaking for 300 s (data not shown). All starches showed the highest averaged pull-off forces after soaking in T25H2O for 60 s. The averaged pull-off forces were reduced when the temperature was increased to 55 °C. Comparable to this, lower pull-off forces were observed with increasing temperature after soaking in sodium hydroxide (2.0 g/100 g) with exception of WS. For all investigated starches, soaking of the layers in T40N10 led to pull-off forces similar to that in T55N20.

High standard deviations appeared for several test conditions, e.g., native and modified potato starch (PS, HDP-S) after soaking in T25H2O. In these cases, continuously increasing and subsequently decreasing pull-off forces were observed.



Fig. 4. Averaged Pull-off force  $\overline{F}/B$  of all investigated starches and fluid properties after soaking for 60 s. Error bars represent standard deviation of three replicate measurements. Mean values with different letters within a starch type show significant difference at  $P \leq 0.05$ .

#### **Swelling properties**

As shown in Figure 5, the soil layer grew steadily over time with a pronounced increase at the beginning. Subsequently, the growth continued at a lower rate until the swelling experiment was stopped after 1800 s. These thickness increases depended on the individual starch and the used fluid. Some starch layers detached from the substrate after swelling in certain fluids probably due to lowered adhesive forces and a strong multidirectional volume increase. The earliest detachment was observed around  $t_{\text{soak}} = 300$  s. Kricke et al. (2021) showed that the initial swelling rate had an important influence on the cleaning behavior of starch soils. The current data of the thickness increase  $\Delta h$  at  $t_{\text{soak}} = 60$  s gave a measure for the initial swelling rate. Therefore, and since defined scalar values were required for further statistical analysis,  $\Delta h$  at 60 s was taken as indicator for describing the swelling behavior.

The thickness increase showed a strong dependency on the fluid after soaking for 60 s (Figure 6). These tendencies were similar for all starches. The thickness increased just slightly in T25H2O. A stronger thickness increase was observed in T55H2O. Within sodium hydroxide, the thickness increase was even more pronounced at the respective temperature compared to deionized water.



Fig. 5. Representative plots of thickness increase  $\Delta h$  over soaking time of waxy maize starch soil layers during soaking in T25H2O, T55H2O and T55N20.



Fig. 6. Thickness increase  $\Delta h$  of all investigated starches and fluid properties after soaking for 60 s. Error bars represent standard deviation of three replicate measurements. Mean values with different letters within a starch type show significant difference at  $P \leq 0.05$ .

#### **Cleaning behavior**

The reptation time and mean cleaning rate obtained from the cleaning experiments are shown in Figure 7. The reptation time was set to 1800 s and the mean cleaning rate equals zero for the test points where no cleaning occurred.

For all starches, the reptation time depended on temperature and sodium hydroxide concentration of the cleaning fluid. None of the starches was cleaned by T25H2O except modified potato starch (HDP-S). A temperature increase to 55 °C initiated the cleaning process for most starches, except WS and RS. Once sodium hydroxide was used as cleaning fluid, all starches were removed from the surface. The cleaning process was initiated after a comparably long reptation time at T25N20 for most starches. Interestingly, the reptation time showed equally low values for T40N10 and T55N20 for most starches, except WS.



Fig. 7. a) Reptation time  $t_{rep}$  and b) mean cleaning rate  $\overline{m}_{s}^{\prime\prime}$  of all investigated starches and fluid properties.  $t_{rep}$  was set to 1800 s and  $\overline{m}_{s}^{\prime\prime}$  to zero when no cleaning occurred. Error bars represent standard deviation of three replicate measurements. Mean values with different letters within a starch type show significant difference at  $P \leq 0.05$ .

The mean cleaning rate showed opposite dependencies on the cleaning fluid, since an effective cleaning process is marked by a low reptation time on the one side, and a high cleaning rate on the other. Noticeably, the mean cleaning rate was highest for PS at T40N10 and T55N20 as well as for RS at T40N10. At these test points, an adhesive detachment of the soil layers was observed as cleaning mechanism, and the soil layer was teared off in large cohering parts as depicted in Figure 8b. Adhesive detachment was also observed as dominant cleaning mechanism for PS at T25N20 as well as for WS at T55N20. Slightly smaller cohering parts were removed and the detachment of the complete soil layer took longer at these test points. WMS was cleaned at a comparably high cleaning rate.

Here, partial viscous shifting was observed as dominant cleaning mechanism when T40N10 and T55N20 were used as fluid (Figure 8c). The soil layer formed a wavy surface at T55H2O and T25N20 (Figure 8d). Cohesive separation was identified as dominant cleaning mechanism for all other starches and test points (Figure 8a).

a) cohesive separation ADA-S in T55H2O		
<i>t</i> = 150 s	<i>t</i> = 300 s	<i>t</i> = 450 s
b) adhesive detachment	c) viscous shifting	d) wavy soil surface
PS in T55N20	WMS in T55N20	WMS in T55H2O

Fig. 8. Images from representative cleaning experiments with the observed cleaning mechanisms a) cohesive separation at three time steps, b) adhesive detachment, and c) viscous shifting as well as the observed formation of a d) wavy soil surface.

#### Statistical correlations and interdependencies



Fig. 9. Factor plot of the principal components analysis of all determined rheological, pull-off, swelling and cleaning properties of the investigated starches at the different test conditions (KMO = 0.570).

Figure 9 presents the result of the principal component analysis considering the rheological, pull-off, and swelling properties as well as the cleaning behavior. Overall, 30 objects, representing the six different starches at five test conditions, were included. It is clearly visible that most of the variables loaded on the first principal component (PC 1) which explained 50.8 % of the overall variance. The second (PC 2) and third (PC 3) principal component explained 17.1 % and 13.7 % of the overall variance and should be considered to un-

cover the underlying interdependencies. Table 1 illustrates bicorrelations between rheological, pull-off, swelling, and cleaning properties.

The complex modulus  $|G^*|$  and the averaged pull-off force  $\overline{F}/B$  as well as the reptation time  $t_{rep}$ loaded strongly positive on PC 1, and slightly positive on PC 2. In contrast, the loss factor tan  $\delta$  loaded negative on PC 1 and PC 2. This indicates correlations between rheological and pull-off properties. The factors representing cleaning behavior ( $\overline{m}_{s}''$ ,  $t_{rep}$ ) loaded in the opposite direction of each other, which is consistent with previous findings (Kricke et al., 2021). The thickness increase  $\Delta h$  loaded in the same direction as the cleaning rate  $\overline{m}_{s}''$ .

Table 1: Bicorrelations of rheological, pull-off, swelling and cleaning properties of the investigated starches at the different test conditions. Marked value (\*) are significant at  $P \le 0.05$ .

	$\log_{10} G^* $	$\tan \delta$	$\overline{\dot{m}}_{ m s}^{\prime\prime}$	trep	$\Delta h$	$\overline{F}/B$
$\log_{10} G^* $	1					
tan $\delta$	-0.71*	1				
$\overline{\dot{m}}_{ m s}^{\prime\prime}$	-0.28	0.20	1			
trep	0.43*	-0.33*	-0.31*	1		
$\Delta h$	-0.39*	0.27	0.25	-0.67*	1	
$\overline{F}/B$	0.45*	-0.60*	-0.28	0.27	-0.57*	1

As presented in Table 1, bicorrelations were revealed between the rheological properties and the averaged pull-off force. This substantiates the assumption that the averaged pull-off force highly depended on the rheological properties of the swollen soil layer. On the one hand, a high complex modulus increased the pull-off resistance of the soil layer. On the other hand, an increasing loss factor represents a rising viscous condition of the soil layer, which led to a decreasing pull-off resistance in return. Furthermore, the complex modulus and especially the averaged pull-off force were negatively correlated to the thickness increase during swelling, substantiating that the inner strength of the soil layer was strongly influenced by the rapid swelling process.

Turning to the cleaning properties presented in Table 1, the reptation time correlated positive with the complex modulus and negative with the loss factor and the thickness increase. The results also showed that none of the investigated properties alone was responsible for a longer or a shorter reptation times. Only slight relations, but nonsignificant correlations between the cleaning rate and the rheological and swelling properties were revealed. The correlation between the thickness increase during swelling and the cleaning rate, as indicated by PCA, was not confirmed in bicorrelation, since both were oppositely loaded on PC 3 (data not shown) indicating another influencing factor. Different cleaning mechanisms observed during the cleaning experiments might explain these findings.



Fig. 10. Radar plot of all determined rheological, pull-off, swelling and cleaning properties at the different test conditions for a) waxy maize starch and b) waxy maize acetylated distarch adipate. For illustrative purpose, reptation time  $t_{rep}$  equals 600 s when no cleaning occurred.

The influence of temperature und sodium hydroxide concentration of the fluid on the different properties and cleaning behavior varied starchspecifically. Therefore, two radar plots for a) waxy maize starch and b) acetylated waxy maize starch are given in Figure 10, presenting the rheological, pull-off, swelling and cleaning properties at the different test conditions.

For WMS, no cleaning occurred in T25H2O. The use of deionized water at 55 °C or sodium hydroxide evoked the cleaning process and a decline of the reptation time. Higher temperature of the cleaning fluid led to a strong increase of the cleaning rate along with a rising thickness increase during swelling as well as a decreasing elasticity and strength of the starch soil layer, indicated by the increasing loss factor and decreasing averaged pull-off force. Wavy soil surfaces were formed during cleaning with T55H2O and T25N20. With T40N10 and T55N20, viscous shifting was observed as dominant cleaning mechanism, which provoked a high cleaning rate. It is assumed that the formation of these wavy soil surfaces and viscous shifting were caused by the viscous behavior of the soil layer.

No cleaning occurred for ADA-S in T25H2O either. A decrease of the reptation time was ob-

served when the fluid temperature was raised, or when sodium hydroxide was used. Contrary to WMS, the highest cleaning rate was achieved using T55H2O. The loss factor was lowest at this test point, and complex modulus and averaged pull-off force showed high values. Therefore, the soil layer was characterized by a high strength and elasticity, and cohesive separation was observed as cleaning mechanism at this test point. Once sodium hydroxide solution was used as soaking fluid, the loss factor increased strongly, indicating a more viscous behavior of the soil layer. The swelling thickness increased as well, but furthermore, low values of the complex modulus and averaged pull-off force indicated a decreased inner strength.

#### CONCLUSION

This study identified starch-specific interdependencies between rheological, pull-off and swelling properties of food soils and their cleaning behavior. The presented set of methods is a useful basis to characterize soils with regard to their cleaning properties. It should be emphasized that relations between the loss factor and the viscous behavior during cleaning were identified by principal component analysis. However, the soil properties change strongly time-dependent during cleaning. It is crucial but challenging to identify and extract representative scalar values for statistical analysis. Distinct dependencies can be expected when the cleaning mechanism is included in the analysis. Further research should be undertaken to explain the complex reasons for soil-specific cleaning behavior. The focus should be on intra- and intermolecular attractive forces that strengthen or weaken the soil layers.

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# NOMENCLATURE

# Abbreviation

acetylated distarch adipate
hydroxypropyl distarch phosphate
principal component
principal components analysis
rice starch
potato starch

T25H2O	T = 25  °C, deionized water
T55H2O	T = 55 °C, deionized water
T40N10	$T = 40 \text{ °C}, c_{\text{NaOH}} = 1.0 \text{ g}/100 \text{ g}$
T25N20	$T = 25 \text{ °C}, c_{\text{NaOH}} = 2.0 \text{ g}/100 \text{ g}$
T55N20	$T = 55$ °C, $c_{\text{NaOH}} = 2.0$ g/100 g
WMS	waxy maize starch
WS	wheat starch

# Latin symbols

A	area, m <sup>2</sup>
В	width, m
С	concentration, g/100 g
Gʻ	storage modulus, Pa
$G^{\prime\prime}$	loss modulus, Pa
$G^*$	complex modulus, Pa
$D_{ m hyd}$	hydraulic diameter, $D_{\text{hyd}} = 4A/p$ , m
F	force, N
h	height, m
Ι	intensity, -
KMO	Kaiser-Meyer-Olkin criterion, -
L	length, m
т	mass, g
$m_{\rm s}^{\prime\prime}$	soil mass per area, g/m <sup>2</sup>
$\overline{\dot{m}}_{ m s}^{\prime\prime}$	mean cleaning rate per area, g/(s·m <sup>2</sup> )
p	wetted perimeter, m
P	P-value, -
Rz	average surface roughness, µm
Re	Reynolds number, $\text{Re} = u_b D_{hyd} / v$ , -
t	time, s
Т	temperature, °C
и	velocity, m/s

#### Greek symbols

tan $\delta$	loss factor, -
v	kinematic viscosity, m <sup>2</sup> /s

# Subscript

0	initial
b	bulk
hyd	hydraulic
raw	original
rep	reptation
s	soil
soak	soaking

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