# THE PHYSICOCHEMICAL PROPERTIES OF FRUIT POWDERS AND RESIDENCE TIME ON STEEL COUPONS ARE ASSOCIATED WITH THEIR EASE OF REMOVAL FROM STAINLESS STEEL SURFACES USING BRUSHING

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## ABSTRACT

Food powder residual on food contact surfaces can cause a series of problems such as cross-contamination and lowering production efficiency. Dry sanitation practices are commonly used in low moisture food production facilities to ensure that water is not introduced to the food production environment. It is crucial to identify the factors contributing to the ease of removal of fruit powders to improve current dry sanitation programs. In this project, the factors influencing the efficiency of dry sanitation are investigated by using a lab-scaled brushing system. The mechanism of stickiness in fruit powder is discussed. The water activity of fruit powder is found to be the most important factor influencing its stickiness. This work provides insights into designing better dry sanitation programs for low moisture food production facilities.

## BIOGRAPHICAL SKETCH

Quanrun He graduated from the University of California, Davis with a Degree of Bachelor of Science with a Major in Food Science and a Minor in Economics in June of 2020. He started his professional Master's Degree in Food Science at Cornell University in September of 2020 and graduated in December of 2021.

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# 1. Introduction

Fruit powders have a wide range of applications in the food industry including flavoring, nutraceuticals, thickening agents, etc. They are easy to preserve, highly portable, and cost-effective compared to fresh fruits. Most fruit powders are low in moisture content and highly hygroscopic in the ambient environment. Due to their high amorphous sugar content and low water activity, most fruit powders will absorb water from the environment over time, resulting in physical and chemical properties changes.

These changes in fruit powders pose serious food quality concerns to food manufactures. Many low moisture food production facilities use dry sanitation measures to replace typical wet cleaning because the water residual can promote the growth of pathogens. The common dry sanitation measures include brushing, vacuum cleaning, scraping, and sweeping, etc. (Moerman & Mager, 2016). As one of the most common dry sanitation methods, brushing can effectively clean a large area during a short time frame. The mechanism by which brushing is working is to lift the powder by the friction force generated between brush bristles and food contact surfaces (Moerman & Mager, 2016).

The change in stickiness behaviors of fruit powder is mainly attributed to the glass transition. The glass transition is a process in which the amorphous materials experience transitions from glassy states to rubbery states as temperature increases (Dyre, 2006). Once the ambient temperature becomes higher than the glass transition temperature ($T_g$) of fruit powders, the fruit powder will transit from a glassy state to a rubbery state and become sticky. There are several types of inter-particulate interactions causing stickiness during this process: liquid bridge, solid bridge, mechanical interlocking, electrostatic attraction, and Van Der Waals force (Wang & Hartel, 2021). The most common interactions in fruit powders are liquid bridge, solid bridge, and mechanical interlockings. Liquid bridges are formed due to the release of mobile liquid substances from the particles (Rennie et al., 1999). The capillary force and surface tension of the released liquid determined the flow between particles and bridges. Once the relative humidity decrease or temperature of the surrounding environment increase, these liquid bridges tend to form solid bridges (Wang & Hartel, 2021). Once the particle surface becomes wet and irregular after increased/decreased temperature or humidity, mechanical interlocking can further enhance the cohesion/adhesion of food materials (McBain & Hopkins, 1925).

It is important to investigate the factors influencing the efficacy of brushing to establish better dry sanitation protocols. In the experiment, brushing was used to remove the fruit powders from stainless steel coupons to stimulate dry sanitation in food manufacturing facilities. Six types of fruit powder, including apple powder (8/20 mesh), peach powder, apple/pear/plum powder, cherry apple powder, and pumpkin powder, were used to test their difficulties of removal from SS coupon. This paper will provide insights into how the fruit powders change in their ease of removal over time and the mechanism behind these changes.

## 2. Materials and methods

### *2.1 Materials*

Fruit powders (The Tree Top Inc., Selah, WA) were acquired from Tree Top company. Six fruit powders used in the study include apple powder (8 mesh/20 mesh), cherry apple powder, apple/pear/plum powder, peach powder, and pumpkin powder. The detailed compositions of fruit powders are summarized in Table 1. The water activity of fruit powder was measured using a dew point water activity meter (Aqualab Series 4 TE, Decagon Devices Inc., Pullman, WA) at 25°C. Stainless steel (SS) sheets (0.46 mm, 316/316L, cold roll 2B) were cut into coupons with dimensions of 10.16 cm by 10.16 cm by the Machine Shop at Cornell University (Chen et al., 2022). A brush with soft polyester bristle and polypropylene block (Vikan Advancing Hygiene Cleaning, Allentown, PA) was purchased as the treatment tool for the study.

The particle size distribution of fruit powders was determined using a sieve-based particle size analyzer (Gilson Company, Inc., Lewis Center, OH). 10 g of fruit powder was transferred to the top sieve of the shaker with the largest pore size (355 μm) and was shaken for 30 minutes at the highest magnitude setting. To obtain the particle size distribution, the mass of fruit powders on each sieve with different pore sizes was recorded and divided by the total mass of fruit powders.

The bulk density ($\rho_b$) of the fruit powders was calculated by the ratio of their mass (m) to their bulk volume ($V_b$) and was expressed as g/cm$^3$: $\rho_b$=m/$V_b$. The fruit powders were weighed with an analytical balance (Aczet Pvt. Ltd., Mumbai, India) with an accuracy of 0.1 mg, while the bulk volume ($V_b$) was measured with a 25 ± 0.5 mL graduated cylinder. The measurements were done in triplicate.

### *2.2 Cleaning outcome measurement*

Following each pass of brushing, the mass of the remaining powder was measured by an analytical balance with an accuracy of 0.1 mg. The same stable state used in this study was based on the previous study (Chen et al., 2022). The fruit powders deposited on coupons were considered “stable” when the mass change of the remaining powder on the coupon was within 0.5 mg for two consecutive passes of brushing. If the “stable” state cannot be reached, 10 passes of brushing were conducted for consistency.

### *2.3 Treatment application*

A thickness gauge (iGAGING Inc., San Clemente, CA) was used to measure the thickness of the fruit powder layer deposited on SS coupons. The fruit powders were deposited on 10.16 cm by 10.16 cm coupon surfaces using a depositing mode with an inner dimension 7.62 cm by 7.62 cm and outer dimension 10.16 cm by 10.16 cm (Fig. 1B). A custom dry-cleaning system (Fig. 1A) was developed to standardize brushing treatment. The system consists of a motor, a speed controller, a power switch, a rotating column, an adjustable height platform, and a coupon carrier. The speed of the moving stage is 1.92 cm/s. The brush was manually cleaned by hands between each pass of brushing. Each coupon with fruit powder was brushed once a day until it reached a "stable" state to record the percentage of powder remaining for 7 days.

Fruit powders on SS coupons were transferred to closed desiccators containing Lithium chloride (11% RH) and Sodium chloride (76% RH) (Research Product International, Mount Prospect, IL) until they reached equilibrium with the salt solution after 3 days. The fruit powders on the SS coupon were then brushed until the "stable" state was achieved.

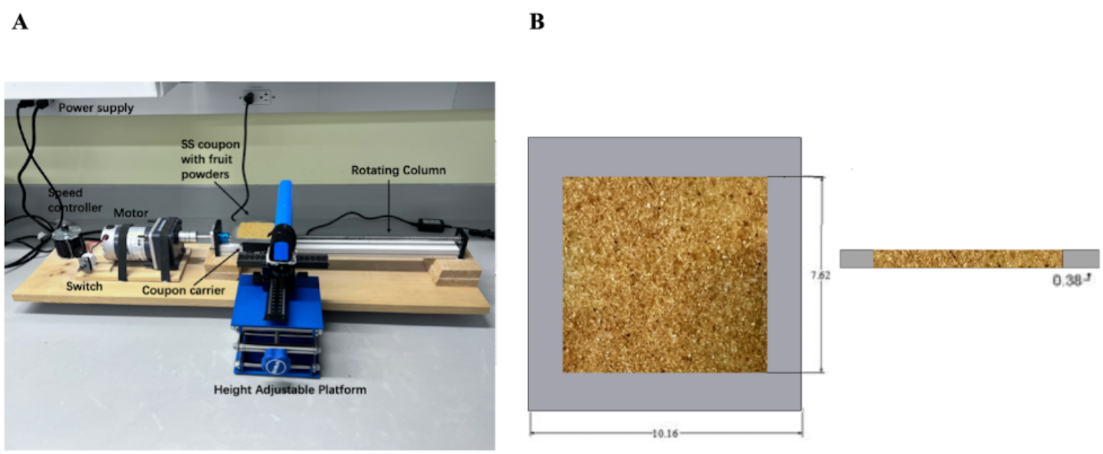



**Figure. 1:** (A) The custom-designed experimental setup for standardized brushing treatments and (B) the dimensions of fruit powders depositing frame (left: top view, right: sectional front view, in cm). (Adapted from Chen et al., 2022).

### *2.4 Adhesion and cohesion measurement*

A shear test method was used to determine the adhesion and cohesion of fruit powders, as initially described by Jenike (Jenike, 1976) and adapted by others (Peleg, 1977) (Li et al., 2014) (Mathlouthi & Rogé, 2003) (Peeters et al., 2011). The working principle for measuring adhesion and cohesion was based on two types of shear tests (Fig. 2), adapted from Peeters et al. (2011) and Li et al. (2014). Two SS circular rings, with (closed) and without (open) a bottom, were manufactured with a height of 1.27 cm and inner and outer diameters of 4.13 cm and 3.81 cm, respectively (Chen et al., 2022). Before cohesion and adhesion tests were performed, the fruit powders and coupons with SS rings were equilibrated in desiccators with 11% RH and 76% RH and equilibrated for 3 days.

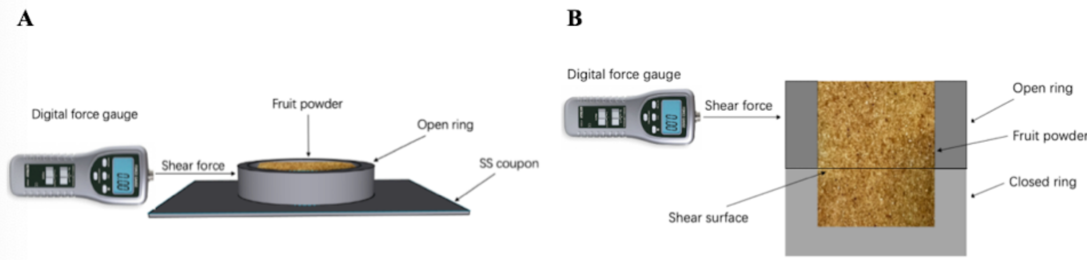



**Figure. 2:** Schematic diagrams (sectional view) of the fruit powder shear tests for (A) adhesion and (B) cohesion determination. (Adapted from Chen et al., 2022).

In the fruit powder adhesion test (Fig. 2A), an open SS ring was placed at the center of a SS coupon. Fruit powder was filled into the open ring, ensuring that the same fruit powder bulk density was in contact with the SS coupon by measuring the same weight of fruit powders for testing. In the test, a digital force gauge (Extech Instruments, Melrose, MA) was used to apply and measure the shear force (N) required to slide the open ring containing fruit powders across the stationary SS coupon surface.

An open ring was layered on top of a closed ring in the cohesion test (Fig. 2B), and 10 g of fruit powder was put into the empty space inside the stacked rings. The shear force required to slide the top open ring of fruit powder over the bottom ring of fruit powder was determined using a digital force gauge. During the cohesion tests, the bottom ring stayed stationary. As demonstrated in Fig. 2B, the shear surface is the interfacial fruit powder layer. Blank tests (no fruit powder) were performed for both adhesion and cohesion setups to quantify the frictional force between the open ring and the SS coupon surface (adhesion test) or between the open ring and the closed ring (cohesion test) so that it could be excluded from shear force calculations. The average shear forces required to move the open ring from the SS coupon surface or the closed ring were 0.03 N and 0.07 N, respectively.

The Adhesion and Cohesion were calculated as:

$\tau$ =Adhesion/Cohesion=$(F-F_0)/A$

F represents the shear force required to overcome the cohesion of fruit powders or the adhesion between fruit powders and SS coupon. $F_0$ was acquired from blank adhesion and cohesion tests described above. A represents the cross-sectional area of the circular ring. $\tau$ represents adhesion/cohesion stress in the unit of $N/m^2$.

### *2.5 Statistical analysis*

The figures were made using Prism 9 (Version 9.3.0) and the statistical analysis was conducted in R studio (Version 1.4.1717). Water activity, powder type, and residence time were treated as discrete variables. The two-way analysis of variance (ANOVA) was used to calculate the interactions between water activity, powder type, and residual weight. Pairwise comparison was performed using Tukey post hoc analysis with emmeans package for any significant interactions identified. The significance level of all statistical tests was 5% ($\alpha = 0.05$).

## 3. Results and discussion

### *3.1 The water activity of fruit powders changes over time is associated with the ease of powder removal from SS coupon surfaces.*

The change in percentage of powder remaining on stainless steel (SS) coupon to achieve a "stable” state under ambient conditions was strongly related to the change of water activities over 7 days period (Fig. 4). Except for apple powder (8 mesh) and pumpkin powder, most fruit powders required 10 times brushing to achieve a "stable" state. The average number of passes needed to achieve a "stable" state for apple powder (8 mesh) and pumpkin powder were 7 times and 9 times, respectively (Fig. 5). Water activity has been used as one of the most important indicators for predicting the behavior of food materials (Moreyra & Peleg, 1981). The initial water activity of cherry apple powder, apple/pear/plum powder, peach powder, pumpkin powder, and apple powder were 0.18±0.006, 0.15±0.0039, 0.19±0.020, 0.27±0.0069, and 0.18 ±0.016, respectively (Table. 1). When the water activity was below 0.2, water molecules existed in fruit powders as monolayer structures. As the water activity of fruit powders exceeded 0.35~0.45, which is the critical water activity for many food materials, the fruit powders underwent many physicochemical changes such as the formation of liquid bridges, interlocking, and recrystallization of sugars (Sherwin & Labuza, 2006). During the first 4 days of residence, the water activities of all fruit powders were relatively stable, ranging from 0.5~0.6. The water activity of fruit powders then experienced a sharp decrease on day 5 and recovered during day 6 and day 7 (Fig. 4). Water as a good plasticizer can significantly depress the glass transition temperature of fruit powders (Drake et al., 2018). When the ambient temperature (25 ºC) exceeded the glass transition temperature of fruit powder, the fruit powder became sticky and harder to be removed from the SS coupon surface (Jaya & Das, 2009). Generally, increased water activity led to a higher percentage remaining of fruit powders. Except for apple powder (8 mesh) and pumpkin powder, the percentage of powder remaining gradually increased to the highest level on day 5 and began to decrease since day 6. For apple powder (20 mesh), the percentage of powder remaining on the SS coupon increased from 1.83±1.17% to 46.66±22.50% during the first 5 days and decreased to 15.82±10.32% on day 7. For apple powder (8 mesh), the percentage of powder remaining on the SS coupon increased from 0.043±0.011% to 4.28±3.48% during the first 5 days and decreased to 1.28±1.96% on day 7. For peach powder, the percentage of powder remaining on the SS coupon increased from 86.13±1.73% to 99.09±0.33% during the first 5 days and decreased to 93.26±2.12% on day 7. For apple/pear/plum powder, the percentage of powder remaining on the SS coupon increased from 48.29±7.55% to 79.16±3.68% during the first 5 days and decreased to 52.88±9.83% on day 7. For cherry apple powder, the percentage of powder remaining on the SS coupon increased from 0.49±0.14% to 12.96±7.49% during the first 5 days and decreased to 1.86±0.44% on day 7.

Interestingly, the percentage of powder remaining experienced sharp increases on day 5 when water activity decreased for all fruit powders except for pumpkin powder and apple powder (8 mesh) (Fig. 4). A phenomenon called "humidity cycling" happens when the food powder is first exposed to high relative humidity and then experienced lower relative humidity (Fitzpatrick, 2013). It will cause the formation of solid bridges (Dopfer et al., 2013). The subsequent increase of water activity might weaken the solid bridges and solid-liquid bridges resulting in a drop in the difficulty of removing fruit powders from the SS coupon.

The behaviors of pumpkin powder and apple powder (8 mesh) were different from other fruit powders, and they exhibited very little adhesion and cohesion. The physical property and composition of pumpkin powder and apple powder (8 mesh) may play important roles in their lower overall stickiness.

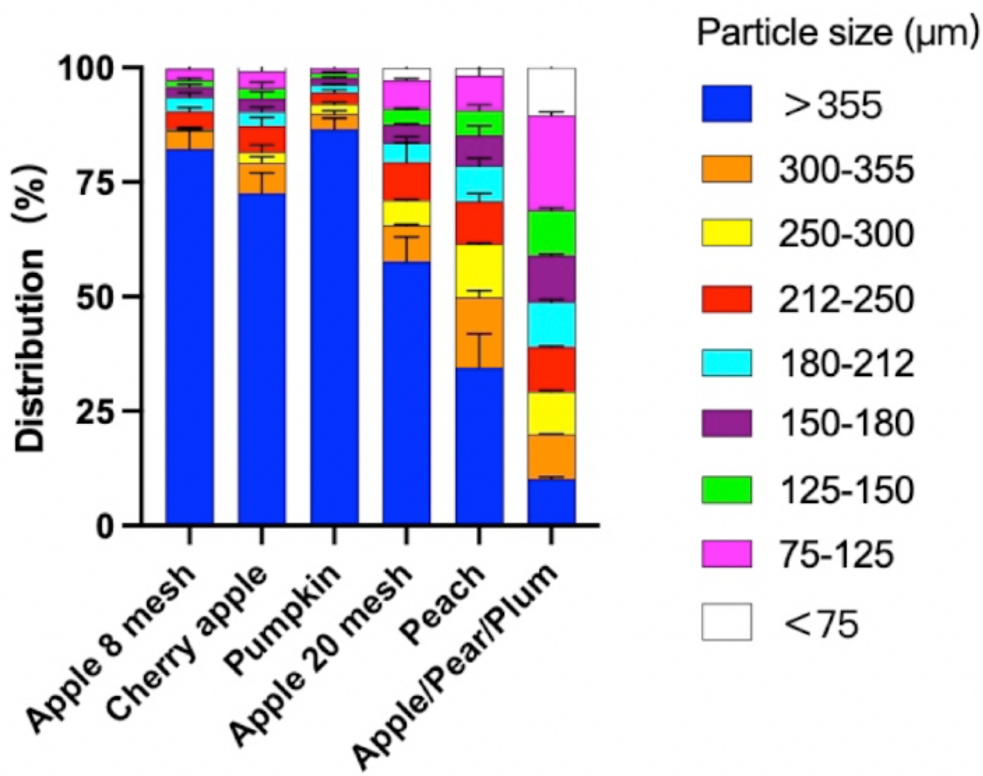



**Figure. 3:** Particle size distribution of different types of fruit powders.

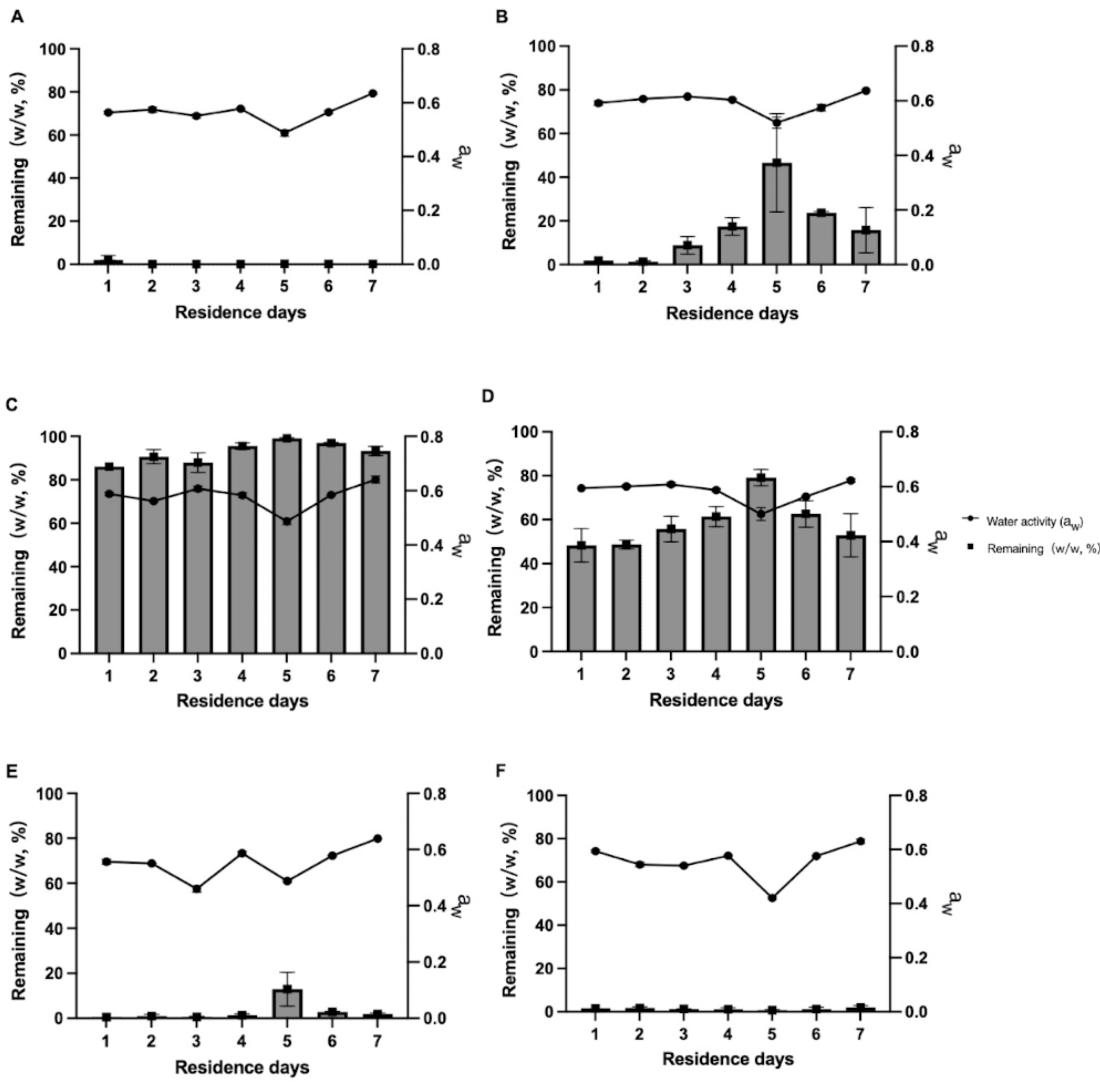



**Figure. 4:** The final percentage of (A) Apple powder (8 mesh) (B) Apple powder (20 mesh) (C) Peach powder (D) Apple/pear/plum powder (E) Cherry apple powder (F) Pumpkin powder remaining on SS coupon after multiple passes of brush once a day for 7 days and the water activities change of each powder during 7 days in ambient environment.

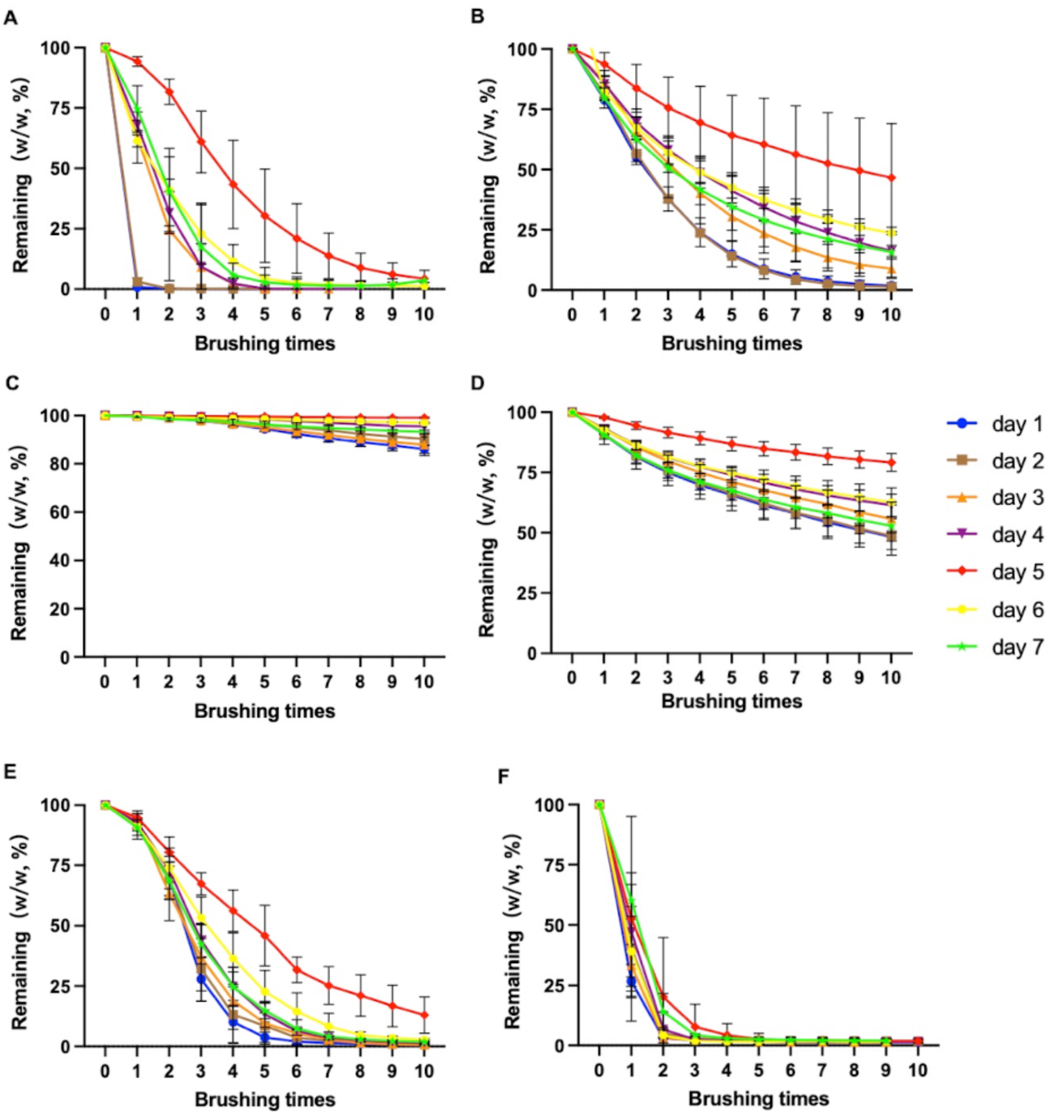



**Figure. 5:** The (A) Apple powder (8 mesh) (B) Apple powder (20 mesh) (C) Peach powder (D) Apple/pear/plum powder (E) Cherry apple powder (F) Pumpkin powder remaining percentage change on SS coupon after each brushing rested in ambient environment for 7 days.

### *3.2 The effect of residence time on ease of removal is dependent on the type of fruit powder*

The percentage of fruit powders remaining on the SS coupon varies among different fruit powders. The peach powder is the most difficult powder to remove with an average percentage remaining 92.75±4.96%. The apple/pear/plum powder is the second with a percentage remaining 58.41±11.41%. The apple powder (20 mesh) is the third with a percentage remaining 16.35±16.82%. The cherry apple powder has a percentage remaining 3.03±4.85%. The pumpkin powder has a percentage remaining of 1.54±0.61%. The apple powder (8 mesh) was the easiest to remove with a percentage remaining 1.05±1.88% (Fig. 5).

The physicochemical properties of fruit powders significantly affect their overall stickiness. Many factors such as low molecular sugars, relative humilities, temperatures, and particle size can influence the difficulty of removing different fruit powders. Water activity and temperature have high contributions, and the level of low molecular sugars and particle size have medium contributions to the overall stickiness of food powders (Adhikari et al., 2007).

**Table 1:** Composition and physical properties of fruit powders used in this study.

| Powder type | Total carbohydrates (g/100g) | Sugars (g/100g) | Total Fat (g/100g) | Saturated Fat (g/100g) | Protein (g/100g) | Bulk density(g/cm$^3$) | Initial water activity |
|---|---|---|---|---|---|---|---|
| Cherry apple | 86.43 | 48.88 | 1.81 | 0.13 | 2.44 | 0.57 | 0.18 |
| Apple/Pear/Plum | 74.65 | 50.42 | 0.3 | 0 | 1.66 | 0.74 | 0.15 |
| Peach | 78.99 | 59.12 | 1.37 | 0.06 | 6.46 | 0.58 | 0.19 |
| Pumpkin | 76.88 | 28.66 | 3.1 | 0.64 | 12.38 | 0.2 | 0.27 |
| Apple powder | 91.2 | 52.3 | 0.9 | 0 | 2.58 | 0.65 | 0.18 |

The type of fruit powder has a significant impact on the percentage remaining on the SS coupon. Fruit powders contain a high amount of low molecular weight sugar which is highly hygroscopic (Oliveira et al., 2014). Sugar levels can significantly influence the stickiness behavior of fruit powders (Tze et al., 2012). The percentages of sugar per 100 g for cherry apple powder, apple/pear/plum powder, peach powder, pumpkin powder, and apple powder were 48.88%, 50.42%, 59.12%, 28.66%, and 52.30%, respectively (Table. 1). The peach powder with the highest sugar level was also the hardest powder to be removed from the coupon surface, and pumpkin powder with the lowest sugar content was the easiest to be removed from coupon surfaces.

The contribution of sugar to the overall stickiness of the system is more complex than water. Firstly, the interaction between sugar and water has an impact on the $T_g$ of fruit powders. The glass transition temperature for fructose is 7~17.8°C (Truong et al., 2004). The addition of sugar with a lower glass transition temperature can significantly lower the overall glass transition temperature of the food system. Secondly, the sugars exist as an amorphous state or crystal forms and they change the surface and volume of the food powder (Wang & Hartel, 2021).

Besides sugar, protein also has a minor influence on the stickiness of fruit powders. The higher protein content of pumpkin powder (12.38%) might explain its lower stickiness because protein usually has a much higher molecular weight than sugars, thus having stronger anti-plasticizing capabilities. The fat content of pumpkin powder (3.1%) is also higher than other fruit powders (Table. 1). Fat has lower surface tension and can reduce the contact surface areas between particles, therefore, reduce cohesion (Wang & Hartel, 2021).

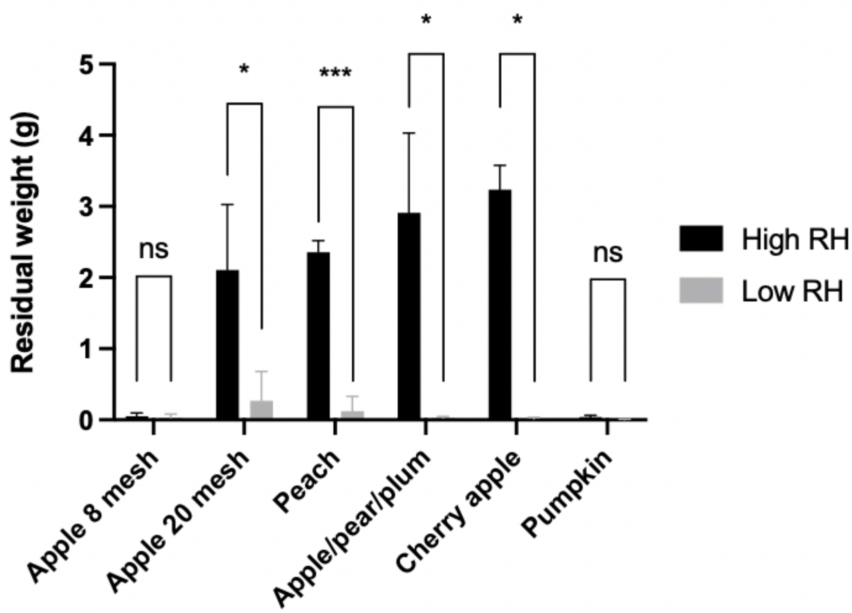
Particle size also plays a critical role in determining the ease of removal for fruit powders. Particle size is inversely proportional to the stickiness of fruit powders

(Buma, 1971). Smaller particles in fruit powders have more contact surface areas than larger particles, thus enabling more interactions between each particle (Sebhatu & Alderborn, 1999). Cohesion will also decrease with the increase of particle size (Rennie et al., 1999). Peach powder and apple/pear/plum powder with the smallest particle sizes exhibited the highest stickiness among all the food powders (Fig. 3). For example, the percentage remaining of apple powder (20 mesh) is significantly higher than that of apple powder (8 mesh) because of its smaller particle size. The residual weights are 0.44±0.93 g and 16.53±16.86 g for apple powder (8 mesh) and apple powder (20 mesh), respectively (Fig. 4).

***3.3 Increased RH results in increased residue weight, adhesion, and cohesion***

Compared with powders in low relative humidity conditions, powders in higher relative humidity conditions required significantly more brushing times to reach the "stable" state. Under low humidity conditions, 4 passes for cherry apple powder, 3 passes for pumpkin powder, 5 passes for apple powder (8 mesh), 6 passes for apple/pear/plum powder, 6 passes for peach powder, and 8 passes for apple powder (20 mesh) were required to reach the "stable" state (Fig. 6). However, all fruit powders reached 10 times brushing limit under high humidity.

Under high relative humidity, not only more brushing times were required to remove the fruit powder, but also the residual weights of fruit powders were significantly higher than the residual weight under low relative humidity for all fruit powders except for apple powder (8 mesh) and pumpkin powder.




**Fiugre. 6:** The residual fruit powder weights on SS coupon after brushing of fruit powders equilibrated at high relative humidity (76%) and low relative humidity (11%) for 3 days.

Under high humidity conditions, the residual weight for peach powder, cherry apple powder, apple powder (20 mesh), and apple/pear/plum powder were 2.36±0.16g, 3.24±0.34g, 2.11±0.92g, and 2.91±1.12g, respectively which were significantly higher than pumpkin powder and apple powder (8 mesh) with residual weights of 0.039±0.02g and 0.05±0.04g, respectively.

Under low humidity conditions, there is no significant difference in residual weight. The residual weight for peach powder, cherry apple powder, apple powder (20 mesh), and apple/pear/plum powder were 0.12±0.21g, 0.016±0.019g, 0.27±0.41g, and 0.025±0.021g, respectively. The residual weight for pumpkin powder and apple powder (8 mesh) were 0.0041±0.0047g and 0.031±0.051g, respectively (Fig. 6).

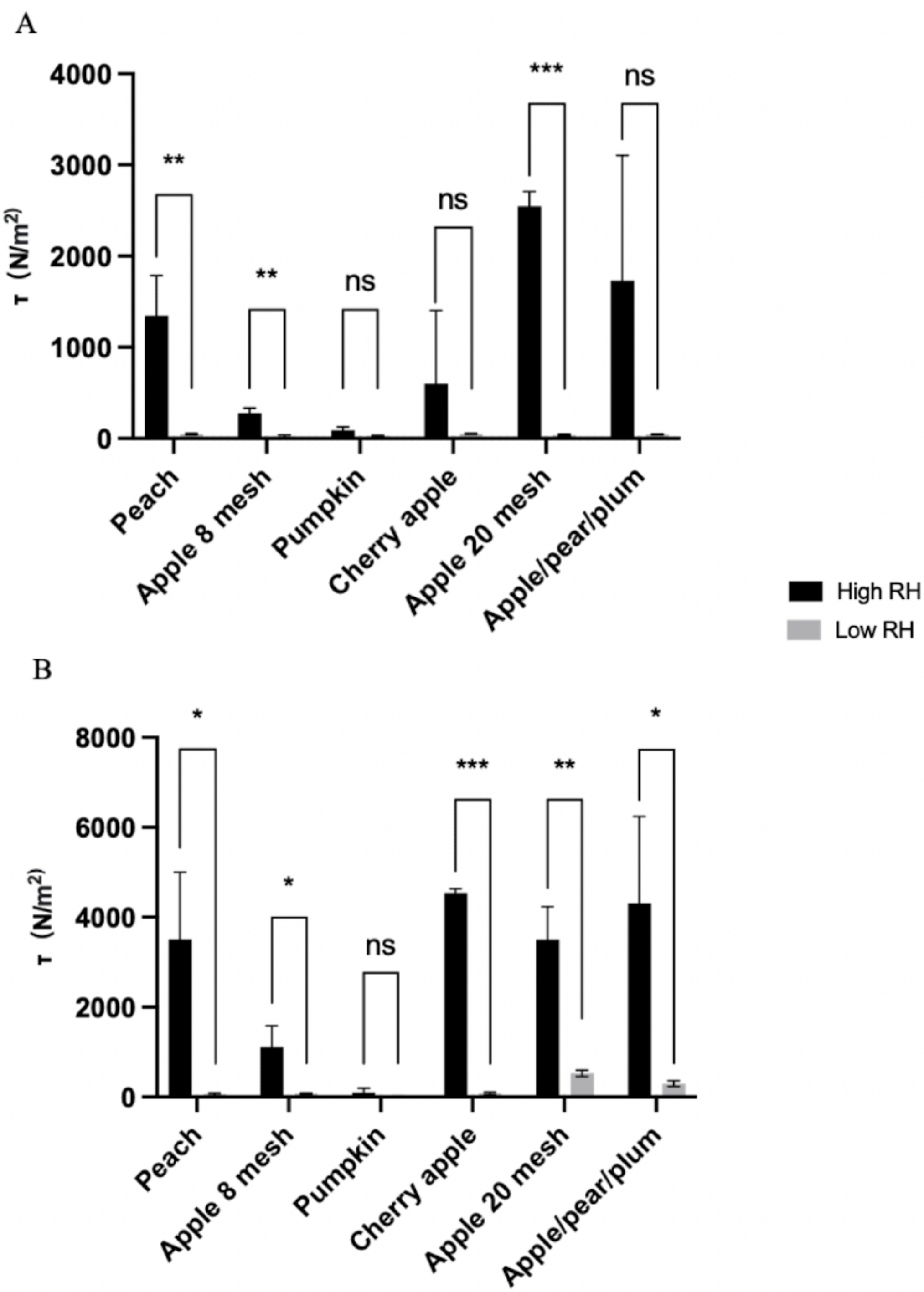



**Figure. 7:** (A) Adhesion of fruit powders to SS coupon surface and (B) cohesion between fruit powder particles equilibrated at high relative humidity (76%) and low relative humidity (11%) for 3 days.

Residual weight of fruit powders on SS coupon is strongly correlated with adhesion and cohesion. Cohesion and adhesion combined determined the stickiness of fruit powder to the food contact surfaces (Adhikari et al., 2001). While the cohesion is dependent on the types of interparticle interactions, the adhesion of fruit powders to stainless steel coupons mainly depends on the adhesive energy balance between the fruit powder and food contact surface (Frabetti et al., 2021). When fruit powders are deposited on the stainless-steel coupon, gravitational force and compression force from powders on the top cause the powder in contact with the coupon to adhere to the coupon surface.

Relative humidity in the environment had a major impact on the cohesion and adhesion of fruit powders. For amorphous fruit powders, adhesion and cohesion exist when the viscosity of particle surface is less than $10^8$ Pa s (Downton et al., 1982). The study has shown that the flowability of powder will decrease with increasing relative humidity levels (Teunou & Fitzpatrick, 1999).

Adhesion and cohesion tests were performed under high relative humidity (76% RH) and low relative humidity (11% RH). The level of relative humidity has a significant impact on the adhesion of peach powder, apple powder (8 mesh), apple powder (20 mesh) (Fig.7). For peach powder, the adhesion stress increased from 52.63±8.77 N/$m^2$ to 1347.95±439.15 N/$m^2$ under 11% RH and 76% RH, respectively. For apple powder (8 mesh), the adhesion stress increased from 29.24±13.40 N/$m^2$ to 277.78±56.40 N/$m^2$ under 11% RH and 76% RH, respectively. For apple powder (20 mesh), the adhesion stress increased from 40.94±10.13 N/$m^2$ to 2549.71±159.43 N/$m^2$ under 11% RH and 76% RH, respectively.

The level of relative humidity has a significant impact on the cohesion of peach powder, apple powder (8 mesh), apple powder (20 mesh), cherry apple powder, and apple/pear/plum powder (Fig. 7). For peach powder, cohesion stress increased from 55.56±25.32 $N/m^2$ to 3511.70±1490.75 $N/m^2$ under 11% RH and 76% RH, respectively. For apple powder (8 mesh), cohesion stress increased from 67.25±13.40 $N/m^2$ to 1119.88±466.67 $N/m^2$ under 11% RH and 76% RH, respectively. For apple powder (20 mesh), the cohesion stress increased from 520.47±70.36 $N/m^2$ to 3502.92±732.88 $N/m^2$ under 11% RH and 76% RH, respectively. For cherry apple powder, the cohesion stress increased from 76.02±25.32 $N/m^2$ to 4538.01±101.67 $N/m^2$ under 11% RH and 76% RH, respectively. For apple/pear/plum powder, the cohesion stress increased from 292.40±62.23 $N/m^2$ to 4312.87±1931.27 $N/m^2$ under 11% RH and 76% RH, respectively.

The influence of relative humidity on adhesion/cohesion was not significant for pumpkin powder. The adhesion stress increased from 32.16±5.06 $N/m^2$ to 90.64±36.52 $N/m^2$, and cohesion stress increased from 11.70±5.06 $N/m^2$ to 102.34±93.80 $N/m^2$ (Fig. 7).

The peach powder, apple powder (20 mesh), cherry apple powder, and apple/pear/plum powder have higher adhesion and cohesion stress than apple powder (8 mesh) and pumpkin powder which is consistent with the trend of their higher residual weights after brushing (Fig. 6).

## 4. Conclusion

Overall, the removal difficulty of fruit powders from food contact surfaces is determined by various factors such as water activity, temperatures, and food compositions, etc. The water activity of fruit powders has the most significant impact on stickiness, which is also reflected in the adhesion/cohesion tests. Besides water activity, compositions such as sugar content and particle size can also influence the stickiness of fruit powders, and these factors together determine the stickiness behaviors of fruit powders.

More studies are needed to determine the specific glass transition temperatures and critical water activities for these fruit powders to provide more detailed guidelines for food manufactures.

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