

**EFFECT OF POWDER AND TARGET PROPERTIES ON FOOD POWDER
COATING AND COMPARISON OF SOLID-LIQUID SEPARATION (SLS) AND
VACUUM CONCENTRATION OF TOMATO JUICE**

DISSERTATION

**Presented in Partial Fulfillment of the Requirements for the Degree Doctor of
Philosophy in the Graduate School of The Ohio State University**

By

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ABSTRACT

Tapioca starch, NaCl (28, 135, and 378 μm), corn starch, cocoa powder, soy protein isolate, cheese powder, wheat protein, modified starch, nacho cheese, and sugar were coated at 0 kV for nonelectrostatic and at 25 kV for electrostatic coating onto metal, wood, unoiled paper, oiled paper, unoiled plastic, oiled plastic, fresh bread, and dry bread. Powders and targets were allowed to naturally tribocharge, or all charge was removed before coating. Powder particle size, flowability, resistivity, and target resistivity were reported. Electrostatic coating produced the same or better wrap around, or percent side coverage as nonelectrostatic coating for every powder and target. The greatest electrostatic improvement was found when using powders that had the worst nonelectrostatic side coverage: large particle size ($>135 \mu\text{m}$), low resistivity, and low cohesiveness, especially on targets that had high-surface resistivity ($2 \times 10^5 \Omega\text{m}$). Tribocharging had a similar effect as electrostatic coating. In both nonelectrostatic and electrostatic coating, percent side coverage increased as powder particle size decreased, cohesiveness increased, or target resistivity decreased. In electrostatic coating, percent side coverage increased as powder resistivity increased; however, in nonelectrostatic coating, as powder resistivity increased, percent side coverage increased on only oiled plastic and dry bread.

Salts were coated on a variety of thick targets. The best transfer efficiency, adhesion (> 70%), and percent side coverage (100%) was obtained when small (< 200 μm) and cohesive (Hausner ratio > 1.20) salt was used with electrostatic coating on targets with high a_w (> 0.7), low resistivity (< $9 \times 10^8 \Omega\text{m}$), and short charge decay time (< 3.8 sec). The shape of salt particles also affected the coating performance; porous cube provided significantly better transfer efficiency and adhesion than flake salt on some targets. There was no significant effect of KCl content on coating performance.

Hot break, cold break and commercially canned tomato juice was concentrated by vacuum concentration and solid-liquid separation (SLS). Tomato products from vacuum concentration had higher soluble solids than those from SLS because of loss of soluble solids into the filtrate. Most volatile levels in vacuum concentration greatly decreased initially then remained constant during further concentration. In SLS, volatile levels linearly decreased with increasing concentration so SLS had greater retention of volatile compounds than vacuum concentration. Viscosity of the rediluted samples decreased with concentration, except in the hot break and commercial samples from SLS, which maintained the same viscosity. Samples from SLS were close to the original color while samples from vacuum concentration were redder due to heat induced Maillard browning. Vitamin C decreased during concentration with greater loss during SLS than vacuum concentration. SLS consumed 45 times less electric power energy than vacuum concentration, which also needed water for creating vacuum conditions.

DEDICATION

To my parents Suchart and Duangdao Sumonsiri.

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FIELD OF STUDY

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CHAPTER 1: LITERATURE REVIEW

1.1 Electrostatic powder coating

1.1.1 Introduction

Electrostatic powder coating was introduced to the food industry from the painting industry in the 1960s (Bailey 1998). The main purposes of electrostatic coating are to produce evenly coated products, improve powder adhesion, minimize dust, and minimize overuse and waste of expensive food powders. The coating on the food target is more even because charged powder particles repel one another to form a cloud which evenly disperses the coating across the surface (Biehl and Barringer 2003; Ricks and others 2002). According to Coulomb's law, the attraction force increases when the distance decreases. Therefore, the charge on the powders produces better adhesion between powders and the target surface (Hughes 1997). Dust creates worker health issues and an explosion risk. Electrostatic coating reduces dust and powder build up because charged powders seek out and land on the target surface, rather than remaining in the air and eventually settling on other surfaces. The reduction of dust and powder build up can shorten the cleaning process; as a result, it can reduce the operating costs.

Powder coating of food products is most often used in the snack food industry. Examples of powder coated foods include potato chips, cakes, donuts, shredded cheese, crackers, and pretzels. Powder coating is used to produce better taste and appearance, as

well as to create more variety in foods to enhance consumer acceptability. Thus a sufficient amount of food powder or seasoning evenly dispersed on these foods is critical.

One of the advantages of electrostatic over nonelectrostatic coating is more even coating. Evenness of coating is used to indicate how well food powder is dispersed on the target, which affects the appearance of food and consumer acceptability (Hughes 1997).

In a study of the uniformity of salt applied on crackers, the variation in salt content between crackers from nonelectrostatic coating was higher than the variation from electrostatic coating, which indicates that the electrostatic coating provides more even dispersion of salt on crackers because powder particles repel each other due to the negative charge given by the coater (Strietelmeier and Reynolds 1969). There are two coating systems that are usually used in snack food seasoning processes: a tumble drum coating system and a conveyor belt coating system (Hanify 2001). The tumble drum is a rotating stainless steel cylinder with internal baffles to flip the pieces, increasing the ability of the system to coat all sides of the target (Figure 1). The powder is charged and dispersed inside the drum by a gun, auger or vibratory feeder to create a spray or curtain of powder. The metal drum is grounded to attract the powder to the bed of food pieces tumbling underneath the powder dispenser.



Figure 1. A tumble drum coating system used in coating popcorn with salt.

The other coating system is the conveyor belt coating system (Figure 2). The gun, waterfall or pneumatic sprayer is positioned over the conveyor belt. The belt is metal or made of conducting plastic to attract the charged powder. The belt system is designed to coat only one side of the target; however, the other sides of the product can be coated by using equipment to flip the target over. Frequently, though, the consumer doesn't notice that only one side is coated.



Figure 2. Saltine crackers being coated in a conveyor belt coating system.

Most application methods use a charging gun or wire. The powder is blown from a reservoir to a feed pipe and through a powder dispenser with an electrode to create the electrostatic field, to coat the target (Bailey 1998). The electrostatic field can be created by a wire in the coating chamber or electrode at the end of a gun. While there is some use of tribocharging and induction charging in other industries, in the food industry almost all powder charging occurs by corona charging. Corona charging involves the ionization of the air, which carries the charge to the powder particles while passing through the exit region of the powder dispenser, because of the Coulombic force of the intense electric field between the corona gun and the target (Bailey 1998). During electrostatic coating, the air flow provides the aerodynamic force which combines with inertia and Coulombic

forces to carry the charged powder particles from the coater to the target (Bailey 1998). These forces are greatly affected by the physical properties of the powders.

Under appropriate conditions, powder particles can not only cover the surface of the target but also coat down the sides of the target (Bailey 1998), which can lead to efficient side coating of powders on a thick target (Figure 3). This wrap around effect is possible because of particle inertial forces and space charge (Bailey 1998). In contrast, the Faraday Cage effect can decrease coating on a target with an uneven surface. This effect is the phenomenon when charged food powders do not deposit inside crevices on the target where there is a strong electrical field between the charging electrode and target, resulting in poor deposition and evenness on the surface (Figure 3), especially on targets with complicated geometry (Boncza-Tomaszewski and Penczek 2002; Mayr and Barringer 2006). There is also a self-limiting phenomenon occurring during electrostatic coating in a corona system due to the back ionization effect. When the total charge in the powder layer exceeds the dielectric strength of the air, powder is blown off the target, producing an uneven layer of powder (Figure 3) (Horinka 1995).

The physical properties of the powders, including particle size, shape, charge, density, flow characteristics, and resistivity, affect the transfer efficiency, dust, adhesion, evenness and functionality of the electrostatically coated powder layer. These properties generally cannot be changed because the powder was chosen to deliver a certain sensory profile. Thus the design of the equipment must be altered to adapt to the powder used.

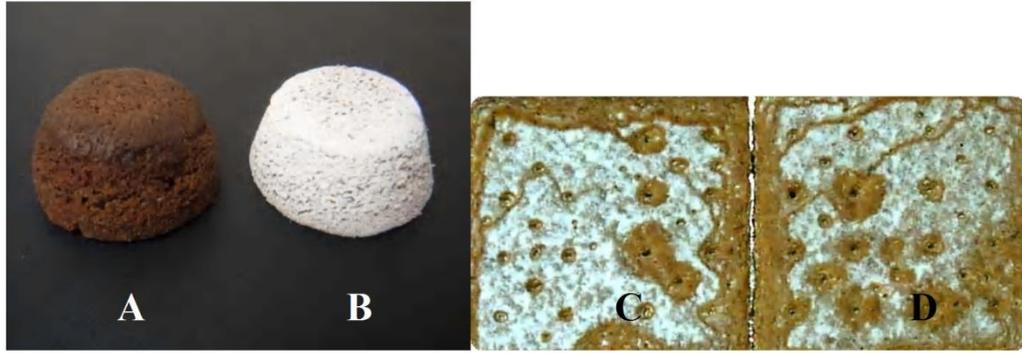


Figure 3. A: Wrap around effect on food targets. Chocolate fudge brownie without coating. B: Electrostatically coated brownie. C, D: Faraday cage and back ionization effects on electrostatically coated graham crackers.

1.1.2 Effect of powder properties on electrostatic coating

1.1.2.1 Particle size

Particle size plays an important role in electrostatic coating just as it does in nonelectrostatic coating. During nonelectrostatic coating, the transfer efficiency may increase or decrease with particle size, depending on whether gravity or aerodynamic forces dominate during coating (Figure 4). The transfer efficiency is a proportion of an amount of powder deposited on the target surface and amount of powder fed into the coating system. An example of a gravity system is in Figure 1, where powder falls out of a tube. Figure 2 is an aerodynamic system, where powder is blown onto the food. With addition of electrostatics, the transfer efficiency increases. With an aerodynamic system, the percent improvement with electrostatic coating increases as the particle size decreases from 330 to 5 μm using salt, corn starch, maltodextrin, cellulose powder, soy flour, cocoa

(Ratanatriwong and Barringer 2007; Ricks and others 2002), sugar (Mayr and Barringer 2006), barbeque, salsa, salt and vinegar, and nacho cheese seasoning (Ratanatriwong and Barringer 2003). Smaller particles under the same charging conditions produce a higher charge to mass ratio than large particles, thus the electrostatic forces have more influence over smaller particles than larger particles. For small particles, electrostatic forces overcome the gravitational and aerodynamic forces so that the powder particles land on the targets rather than stay in the air as dust. When particles are large enough, gravitational forces overcome electrostatic forces resulting in no or little difference in transfer efficiency between nonelectrostatic and electrostatic coating (Ricks and others 2002). With a gravity fed system, the electrostatic transfer efficiency increased as the particle size decreased from 145 to 13 μm using cornstarch, flour, maltodextrin, powdered sugar, salt, and cellulose. However, the percent improvement with electrostatic coating stayed constant at approximately 15% (Biehl and Barringer 2003).

The improvement in adhesion with electrostatic coating is also particle size dependent. The adhesion of electrostatically coated sugar decreased as the particle size increased from 20 to 150 μm (Halim and Barringer 2007). Increasing the particle size greater than 200 μm produced no improvement of adhesion by electrostatics. Larger powders have a lower charge-to-mass ratio, and therefore lower attraction force between particles and the target surface. The adhesion of electrostatically coated salts on potato chips also significantly decreased when large particle sizes, 258 and 388 μm , were used (Buck and Barringer 2007).

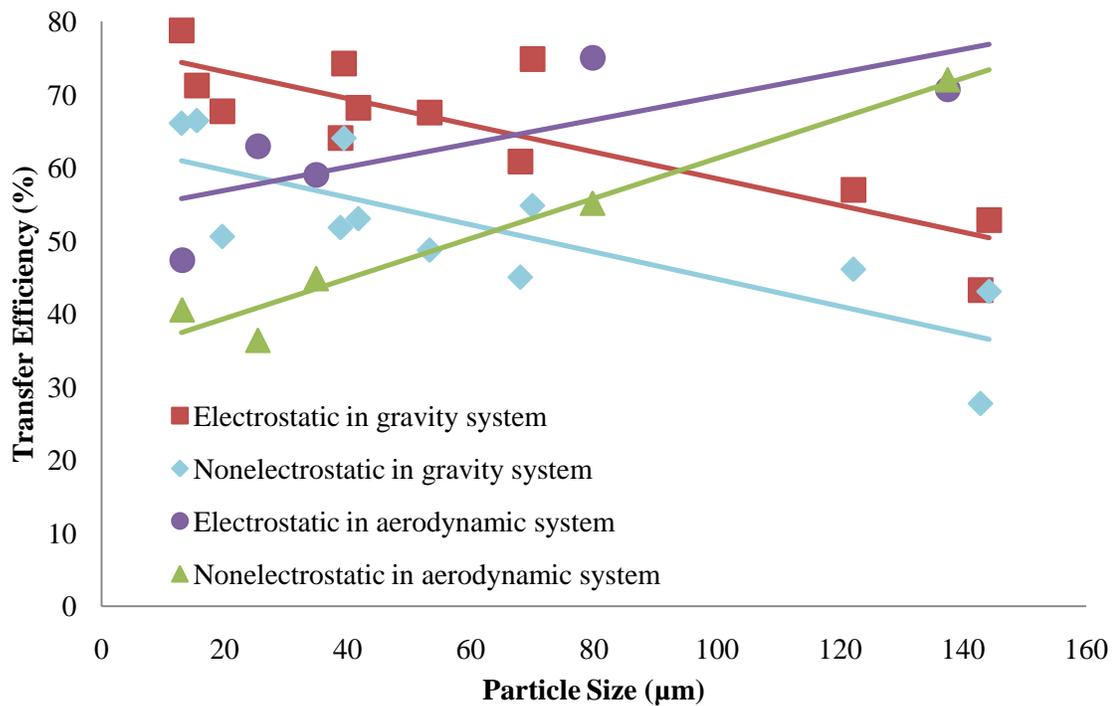


Figure 4. Effect of particle size on transfer efficiency in a gravity and aerodynamic coating system (data from Biehl and Barringer 2003; Mayr and Barringer 2006).

Since electrostatic forces are strongest on small particles, electrostatics is very efficient at decreasing the dustiness of a process. Dust increases as particle size decreases. The use of electrostatic coating has been shown to create a significant dust reduction in most powders, such as corn flour, cellulose powder, salt, soy flour, corn starch, cocoa powder (Ricks and others 2002), sugar (Mayr and Barringer 2006), barbeque, sour cream, salt and vinegar, nacho cheese (Ratanatriwong and others 2003), and a variety of protein powders (Sumawi and Barringer 2005).

1.1.2.2 Powder density

Power density can influence the performance of powder coating; however, it depends on the powders used and the coating system. Among food powders, salt is the most dense, at 2.29 g/cm^3 . Other food powders range from only 1.3 to 1.69 g/cm^3 . Frequently, density is found to have no significant effect on electrostatic coating, whether because of the small range of densities possible or because particle size and other factors are a larger effect (Biehl and Barringer 2004). In a computer simulation, electrostatic transfer efficiency increased when the particle density increased (Figure 5) (Yousuf and Barringer 2007).

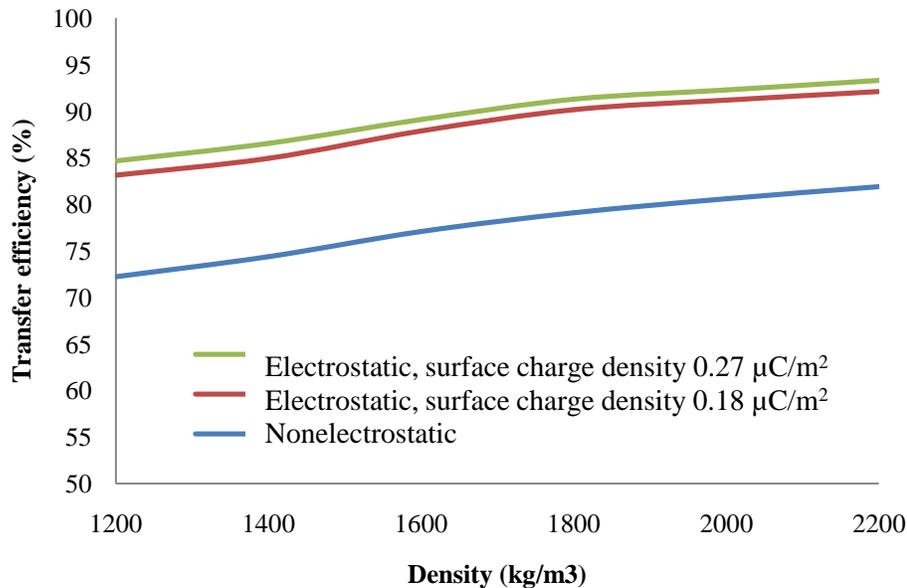


Figure 5. Effect of particle density on nonelectrostatic and electrostatic transfer efficiency in a computer simulation (data from Yousuf and Barringer 2007).

1.1.2.3 Powder charge

The amount of charge on the powder affects particle velocity, transfer efficiency, coating thickness, and adhesion of electrostatic coating (Masui and Murata 1982). How well a powder charges is affected by the powder resistivity, and can be measured by the charge-to-mass ratio. There are three ranges of powder resistivity: greater than 10^{13} Ωm , between 10^{13} and 10^{10} Ωm , and below 10^{10} Ωm (Bailey 1998). Powders with resistivity above 10^{13} Ωm provide good adhesion since they are insulating and have a slow charge decay time (Bailey 1998). Cocoa powder is one of the few examples of a food powder that has resistivity in this range. Powder particles with resistivity from 10^{13} to 10^{10} Ωm have a short charge decay time and poor adhesion (Bailey 1998). The performance of these powders is hard to predict because good adhesion might occur with these powders if other factors are in favor of adhesion. Most food powders have an intermediate resistivity. Powders with resistivity lower than 10^{10} Ωm are conductive so charge easily but lose their charge quickly on the target (Bailey 1998). Salts are the only food powders with resistivity below 10^{10} Ωm . During charging, the lower the resistivity, the more charge is deposited on the powder, and therefore a lower resistivity is desired. On the other hand, after the powders are charged, the higher the resistivity, the slower the charge decay rate and the better the adhesion force between particles and the target therefore a higher resistivity is desired (Hughes 1997). Unlike powders developed for photocopying, the resistivity of food powders cannot be changed during coating.

Resistivity is important in determining the adhesion of food powders. Powders with low resistivity, including salt and maltodextrin, did not produce a significant

difference in adhesion between electrostatic and nonelectrostatic coating (Halim and Barringer 2007). However, most powders with resistivity between 10^{11} and 10^{13} Ωm produced a significant improvement in adhesion with electrostatic coating. Cocoa powder, with a resistivity of 10^{13} Ωm , showed the greatest increase in adhesion with electrostatic coating. Relative humidity also affects resistivity. As the relative humidity increases, the resistivity of cocoa powder decreases because of the water absorbing onto the surface (Halim and Barringer 2007). When the relative humidity increases and the resistivity of powders decreases, electrostatic adhesion is lower (Xu and Barringer 2008).

Charge-to-mass ratio is the most common method to indicate how efficiently the powders are charged during electrostatic coating (Hughes 1997). Charge-to-mass ratio increases as the particle size decreases. A higher charge-to-mass ratio produces a greater radial trajectory; therefore, powders with a larger charge-to-mass ratio can disperse more evenly and produce more uniform deposition (Ali and others 2000). Moreover, powders with large charge-to-mass ratio have better attraction to the target; thus, producing higher electrostatic transfer efficiency (Mazumder and others 1997). Many food powders, including powdered sugar, NaCl, cellulose powder, maltodextrin, whole wheat flour, and soy flour, show an increase in electrostatic transfer efficiency as the charge-to-mass ratio increases (Biehl and Barringer 2003). The charge-to-mass ratio needs to be at least 0.2 $\mu\text{C}/\text{g}$ to have good adhesion on the target (Singh 1978).

1.1.2.4 Flowability

Powder flowability also affects the performance of nonelectrostatic and electrostatic coating. There are numerous methods to measure flow characteristics of

powders, including cohesion, flow index, angle of repose, and Hausner ratio. Cohesion represents the affinity of powder particles for each other and determines how sticky the powders are (Rennie 1999). Cohesion is measured by using either the annular shear cell (Jenike 1964) or the unconfined yield test (Head 1982). A higher value of cohesion indicates that powders are stickier. The flow index is used to indicate how easily powder particles can flow. The angle of repose represents the angle which is formed at the highest peak of a pile of powder (Teunou and others 1999). The Hausner ratio is the ratio between the untapped and tapped bulk density of powder.

The flow properties of powders can be divided into three ranges: non-flowing, cohesive, and free-flowing. Non-flowing powders have a flow index less than 2, angle of repose greater than 50° and Hausner ratio more than 1.4 (de Jong and others 1999; Peleg 1977; Teunou and others 1999). Cohesive powders have a flow index between 2 and 4, angle of repose from 40° to 50°, and Hausner ratio between 1.25 and 1.4. Free-flowing powders have a flow index from 4 to 10, angle of repose less than 40°, and Hausner ratio from 1 to 1.25.

Flowability is an important factor to indicate the cohesiveness of powders and predict how efficiently and evenly powder particles deposit onto a target, which affects the transfer efficiency, adhesion, and dust reduction in coating (Biehl and Barringer 2003). During electrostatic coating, the transfer efficiency increases as powders become more free-flowing because they can be evenly dispersed across the target (Amefia and others 2006; Biehl and Barringer 2004; Ratanatriwong and Barringer 2007). However, since free-flowing powders also perform well in nonelectrostatic coating, the greatest

improvement in electrostatic transfer efficiency occurs when the powders are cohesive (Ratanatriwong and others 2003; Ricks and others 2002). The charged particles repel each other, minimizing clumping, which results in the improvement in transfer efficiency. As expected, adhesion increased when the cohesiveness of powders increased (Sumawi and Barringer 2005). Flowability of powders also significantly affects the amount of dust. Free-flowing powders produce more dust than cohesive powders, but electrostatic coating produces a large reduction in dust for both free-flowing and cohesive powders.

1.1.3 Effect of surface characteristics of the target on electrostatic coating

The characteristics of the target must be considered to achieve the best coating (Bailey 1998). The roughness of the target surface affects the adhesion forces between powders and target (Podczeck 1998). When the roughness of the target surface increases, the adhesion between powder particles and the target surface frequently decreases. This happens because there is less contact area, resulting in less van der Waals force between powders and target (Podczeck 1998).

The oil content on a potato chip's surface influences the adhesion created by electrostatics because a surface with high oil content has high capillary forces which overcome the electrostatic forces. Electrostatic coating on chips with a high surface oil content showed no improvement in adhesion over nonelectrostatic coating, while there was a significant improvement in adhesion when electrostatic coating was used on chips without free surface oil (Buck and Barringer 2007). However, oil also has high resistivity that can lead to charge building up on the surface of targets. This charge repels charged particles when they land on the target and lower the adhesion between particles and the

target surface (Buck and Barringer 2007). Targets with lower resistivity provide better charge flow from the ground and better attraction between the powder and the target surface (Sims and others 2000). Most food targets have a resistivity in the range of 10^8 to 10^{10} Ωm . As food target resistivity increases, the percent electrostatic adhesion improvement increases (Figure 6).

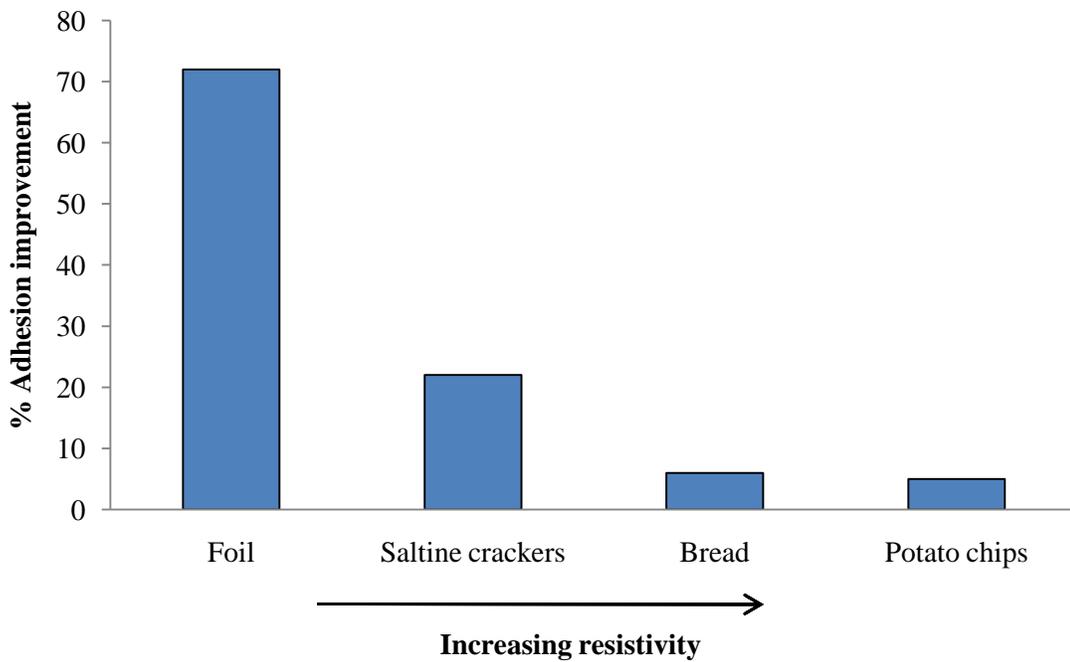


Figure 6. Effect of target resistivity on percent electrostatic adhesion improvement with powdered sugar. * indicates significant improvement in adhesion (data from Halim and Barringer 2007).

1.1.4 Evenness in electrostatic coating

Evenness of coating is important because it affects the appearance and functionality of the powder. In a study of coating evenness of various seasonings on potato chips and on banana chips, electrostatic coating produced a more even color on the surface at the same amount of seasoning (Figure 7) (Ratanatriwong and others 2003; 2009).

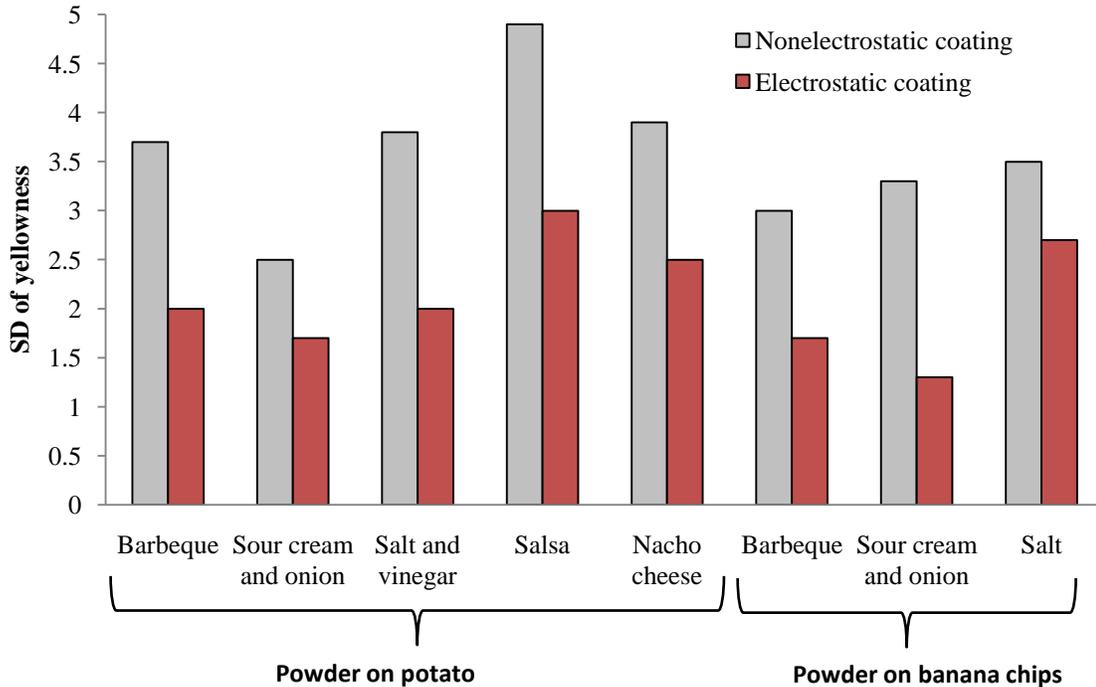


Figure 7. Standard deviation (SD) of yellowness of seasoned potato chips and banana chips. A higher SD indicates more uneven coating (data from Ratanatriwong and others 2003; 2009).

Consumers can see this difference, and significantly preferred electrostatically coated chips over nonelectrostatically coated ones. Meats electrostatically coated with the same amount of sodium erythorbate and/or glucono-delta-lactone were redder than nonelectrostatically coated meats (Barringer and others 2005). Electrostatic coating with glucose or smoke extract produced French fries with a more even color than nonelectrostatic coating (Amefia and others 2006). Electrostatic coating of cheese with a mold inhibitor produced a significantly longer shelf life than nonelectrostatic coating, for the same amount of inhibitor on the cheese (Amefia and others 2006). Similarly, electrostatic coating of meats with antimicrobial agents produced a significantly greater reduction in microbial counts than nonelectrostatic coating.

1.1.5 Conclusions

There are several factors that can influence the efficiency of electrostatic coating during food production, especially the characteristics of the powders used for coating. The greatest improvement with electrostatic coating occurs when using small and cohesive food powders with large charge-to-mass ratio. These parameters play significant roles in the performance of the coating and help the processor to design the optimum system to produce coated food products with the maximum transfer efficiency and minimum operating costs. Moreover, these factors can be used for the prediction of the coating performance of various food powders.

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1.2 Concentration of tomato juice

1.2.1 Introduction

Tomato (*Lycopersicon esculentum*) is one of the most important vegetables for processing with the total value of \$926 million for the U.S. processing in 2010 and the U.S. per capita consumption of processing tomatoes at approximately 67 pounds in 2008 (Boriss and Brunke 2011). The products from tomato processing are typically divided into 4 categories, including tomato pastes, ketchup, tomato sauces, and other products (whole canned tomatoes, puree, and juice) (Boriss and Brunke 2011). After harvesting, tomatoes are usually processed into pastes, which can be used to produce tomato sauces, ketchup, and other products afterwards, because it is more economical that the equipment used in tomato processing can be also used during the off season and the transportation cost for paste is much lower than that for sauce (Barringer 2004).

The composition of the tomato depends on the variety, state of ripeness, conditions during growing, year, irrigation, and fertilization. Ripe tomatoes have average of 94.5 % water (U.S. Department of Agriculture (USDA), Agricultural Research Service 2011), which is a disadvantage during concentration into paste. In total solids of tomatoes (vary between 5 and 10 %, with average at 6 %) half of them are reducing sugars while the other are citric, malic, and dicarboxylic amino acids, minerals, lipids, and alcohol-insoluble solids, such as pectic substances, cellulose, protein, and hemicelluloses.

Tomatoes are also a good source of vitamin C and A at 13.7 mg and 833 IU per 100 g of ripe tomatoes respectively (U.S. Department of Agriculture (USDA), Agricultural Research Service 2011).

1.2.2 Processing of tomato juice, paste, and sauce

Most tomatoes for processing are made into juice, which is then concentrated into paste. The paste is remanufactured into a wide variety of sauce products. After harvesting, tomatoes are graded, washed, sorted, chopped, put through a break system, extracted into juice (Barringer 2004).

1.2.2.1 Grading

One of the initial steps tomatoes go through is grading, to determine the price paid to the farmer. This is done at the processing facility or at a centralized station before going to the processing facility. Individual companies may set their own grading standards, use the voluntary USDA grading standards, or use locally determined standards, such as those of the Processing Tomato Advisory Board in California. The farmer is paid based on the percentage of tomatoes in each category. Typically, companies hire USDA graders or hold an annual grading school to train their graders.

The USDA divides tomatoes for processing into categories, the highest being A, and followed by B, C, and culls (U.S. Department of Agriculture (USDA) 1983). Grading is done on the basis of color and percentage of defects. Color can be determined visually by estimation of what percentage of the surface is red, with an electronic colorimeter on a composite raw juice sample, or with a portable colorimeter on a whole tomato. Defects include worms, worm damage, freeze damage, stems, mechanical damage, anthracnose,

mold, and decay. The allowable percentage of extraneous matter may also be specified. Extraneous matter includes stems, vines, dirt, stones, and trash.

Tomatoes for canning whole, sliced, or diced are graded on the basis of color, firmness, defects, and size. Solids content is unimportant, unlike in tomatoes for juice or paste. Graders must be trained to evaluate and score color and firmness. Color should be a uniform red across the entire surface of the tomato. Color is graded using USDA issued plastic color comparators, the Munsell colorimeter or the Agtron colorimeter, or the tomato is ground into juice and used in a colorimeter with a correlation equation to convert it to the Munsell scale. Firmness, or character, is important to be sure the tomato will persist canning conditions. Soft, watery tomatoes or tomatoes possessing large seed cavities give an unattractive appearance and therefore receive a lower grade. Size is not a grading characteristic per se, but all tomatoes must be above a minimum agreed upon size.

The Processing Tomato Advisory Board inspects all tomatoes for processing in California. Their standards are similar to those of the USDA, but more geared for the paste industry. They inspect fruit for color, soluble solids, and damage. A load of tomatoes may be rejected for any of the following reasons: > 2% of fruit is affected by worm or insect damage, > 8% is affected by mold, > 4% is green, or > 3% contains material other than tomatoes, such as extraneous material, dirt, and detached stems (California Department of Food and Agriculture 2001).

1.2.2.2 Washing

Washing is a critical control step in producing tomato products with a low microbial count. After harvesting, fruits and vegetables are washed to eliminate soil, dirt, surface microorganisms, mold, insects, *Drosophila* eggs, fungicide, insecticide, and other pesticide residues (FAO 1995). The efficiency of the washing process will determine microbial counts in the final product. Spoiled fruits and vegetables should be removed before washing to minimize the contamination of washing tools, equipment, and produce during washing. The washing process should reduce the surface microorganisms by a six fold, or one log, reduction and there should not be any molds and yeast in the water from the final wash. Lye or surfactants may be added to the water to improve the efficiency of dirt removal; however, surfactants have been shown to promote infiltration of some bacteria into fruits and vegetables by reducing the surface tension at the pores (Bartz 1999), which jeopardizes food safety. The washing step also serves to cool fruits and vegetables. Since some of them are harvested on hot summer days, washing removes the field heat, slowing respiration and therefore quality loss.

Several methods can be used to increase the efficiency of the washing step. Agitation increases the efficiency of soil removal. The warmer the water spray or dip, up to 90 °C, the lower the microbial count (Adsule and others 1982; Trandin and others 1982), although warm water is not typically used because of economic concerns. Immersion or spraying is usually used with the application of detergents, 1.5% HCl solution, warm water (approximately 50 °C), or high water pressure (for spray or shower washing).

Tomatoes are typically transported in a water flume to minimize damage to the fruit. Therefore, tomato washing can be a separate step in a water tank or it can be built into the flume system (Figure 8). The final rinse step uses pressurized spray nozzles at the end of the soaking process. Flume water may be used in a counter flow system, so that the final rinse is with fresh water, while the initial wash is done with used water. In either system, the first flume frequently inoculates rather than washes the tomatoes because all of the dirt in the truck is washed into the flume water (Heil and others 1984). When the water is reused, high microbial counts on the fruit may result if careful controls are not kept.



Figure 8. Tomato washing in a tank before transporting to the lye peeler.

Chlorine is frequently added to the wash water. Chlorine will not significantly reduce microbial counts on tomatoes itself because the residence time is too short. However, chlorine is effective at keeping down the number of microorganisms present in the flume water (Heil and others 1984). When there is a large amount of organic material in the water, such as occurs in dirty water, chlorine is used up rapidly, so it must be continuously monitored.

1.2.2.3 Break

During processing into juice, chopped tomatoes can be put through either a cold break or hot break system in a heat exchanger. Vacuum condition can also be used in some break systems in order to minimize oxidation and ascorbic acid degradation (Trifiro and others 1998).

In hot break system, chopped tomatoes are rapidly heated at least 82 °C, usually at 93 – 99 °C, to inactivate the pectolytic enzymes: polygalacturonase (PG) and pectin methylesterase (PME) in order to produce the product with high viscosity. Most juice is processed by hot break system since most juice is concentrated into paste and high viscosity in tomato paste is desired for using in products such as ketchup and pizza sauce (Barringer 2004).

In the cold break method, lower temperature at 60 – 66 °C is used to enhance the enzymatic activity, increase yield, and preserve color and flavor in tomato juice and juice-based drinks (Madhavi and Salunkhe 1998). Cold break juice has a lower viscosity because of the activity of the enzymes.

1.2.2.4 Extraction

After the break system, the heated tomato pulp is extracted through a screw-type or paddle-type extractor, pulper, or finisher, where seeds and skins are also removed. In screw-type extractors, the tomatoes are pressed between the screw and the screen. The expanding helical screw is used to force the tomatoes through the screen. In the paddle-type extractors, the tomato pulp is beaten against the screen, which incorporates more air into the juice when compared to the screw-type extractors. During extraction, air incorporation is not desired since it accelerates the oxidation of lycopene and ascorbic acid. The texture and viscosity of tomato juice are generally affected by the finisher screen size (Hayes and others 1998; Barringer 2004).

1.2.2.5 Concentration into paste

The traditional concentration of tomato juice into paste occurs in three- or four-effect vacuum evaporators with forced circulation. At each successive effect, the juice is heated to 48 – 82 °C by using vapor collected from later effects to conserve energy. In this concentration, color and flavor are preserved by the reduced pressure, which lowers the concentration temperature (Barringer 2004; Smith 2011). The tomato paste usually has a final solids content of at least 24% NTSS (natural tomato soluble solids) (U.S. Department of Agriculture (USDA) 1977). Commercial paste is available in a range of solids contents, finishes, and Bostwick consistencies. The larger the screen size, the coarser the particles and the larger the finish. Bostwick may range from 2.5 to 8 cm (tested at 12% NTSS).

1.2.2.6 Solid-liquid separation (SLS) in tomato concentration

The solid-liquid separation (SLS) system (Figure 9) is a new innovative technology to dewater products with low energy consumption. This equipment uses different belts, including dewatering and membrane belts, to separate liquids or filtrate (Figure 9D) from solids or concentrate (Figure 9C) by adhesion and cohesion. It is claimed to be capable of separating 18 – 45% solids from dilute liquids without forced drying or heating (Coho and Youngs 2010). The operation of SLS consumes up to 90% less energy than a centrifuge. Therefore, the SLS is expected to concentrate tomato product with better energy conservation and product quality preservation.

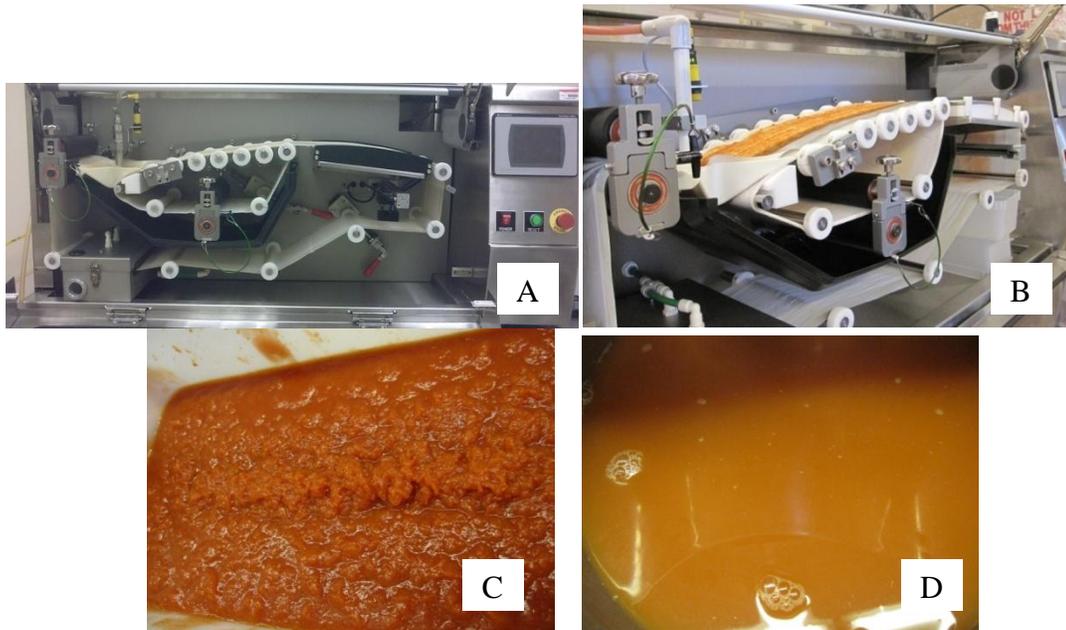


Figure 9. A: solid-liquid system (SLS). B: SLS during concentration of tomato juice. C and D: concentrate and filtrate from SLS concentration respectively.

1.2.3 Measurement and changes of quality during processing

1.2.3.1 Total solids, degrees Brix, NTSS, and sugar content

Tomato solids are important quality attribute since they influence the changes of consistency and yield of the final product. Total solids of the product, usually reported as % total solids, are determined by drying in a vacuum oven or in a microwave oven (AOAC Official Method 985.26). Soluble solids are determined by a refractometer (AOAC Official Method 970.59). Refractometers are calibrated directly in percentage sugar, or degrees Brix, which is the majority of soluble solids in tomatoes. Natural tomato soluble solids (NTSS) are determined by degrees Brix, minus any added salt (Barringer 2004).

Fully ripe tomatoes have the highest sugar content (Hobson and Gierson 1993). In processing, reducing sugar content in tomatoes decreases due to Maillard reaction, caramelization, and the formation of 5-hydroxymethyl furfural during heat treatment (Eichner and others 1996; Barringer 2004).

1.2.3.2 Flavor

The volatile compounds in tomatoes determine the aroma, and together with the titratable acidity and total sugars, the taste of tomato products (Malundo and others 1995). Sensory evaluation is generally used for flavor evaluation. Selected ion flow tube-mass spectrometry (SIFT-MS) and gas chromatography are also used to determine the exact volatile compounds in the products.

Sugar and organic acid content, especially fructose and citric acid, are the main components that contribute sweet-sour taste in tomatoes (Stevens and others 1977). The

character and intensity of the taste are also influenced by free amino acids, salts, and their buffers (Petro-Turza 1987). There are 9 important compounds responsible for fresh tomato flavor: hexanal, (*E*)-2-hexanal, (*Z*)-3-hexanal, (*Z*)-3-hexenol, (*E*)-2-(*E*)-4-decadienal, 2-isobutylthiazole, 6-methyl-5-hepten-2-one, 1-penten-3-one, and β -ionone (Petro-Turza 1986), while 11 compounds are the most important in the aroma of tomato paste: dimethyl sulfide, β -damascenone, β -ionone, 3-methylbutanal, 1-nitro-2-phenylethane, eugenol, methional, 3-methylbutyric acid, 6-methyl-5-hepten-2-one, phenylacetaldehyde, and linalool (Buttery and others 1990b). Some volatile compounds, such as (*Z*)-3-hexenal, hexanal, 1-penten-3-one, and 2-phenylethanol, evaporate during heat processing, such as hot break, concentration, and pasteurization. Other compounds, such as pyrrolidone carboxylic acid and dimethyl sulfide, can be formed by the breakdown of carotenoids, amino acids, and sugars during thermal processing (Buttery and others 1990).

Aroma in processed tomatoes is different from that in fresh tomatoes due to the loss and formation of volatile compounds. Thermal processing causes volatilization of many volatiles while oxidation of carotenoids causes the formation of terpenes and terpene like compounds, and the Maillard reaction forms volatile carbonyl and sulfur compounds. During processing, many volatiles responsible for the fresh tomato flavor are lost, especially (*Z*)-3-hexenal and hexenal (Buttery and others 1990b). (*Z*)-3-hexenal, which is an important compound in fresh tomato, is transformed into (*E*)-2-hexenal, which is more stable, during thermal processing (Kazeniak and Hall 1970). 2-

isobutylthiazole, a compound responsible for a tomato leaf green aroma, is also lost during processing of tomato puree and paste (Chung and others 1983).

Breakdown of sugars and carotenoids forms volatiles responsible for the cooked aroma of heated tomatoes, especially dimethyl sulfide (Buttery and others 1990b, Thakur and others 1996). Dimethyl trisulfide, 1-octen-3-one (Buttery and others 1990a), acetaldehyde, and geranylacetone (Kazeniak and Hall 1970) may also be responsible for the cooked aroma. Some volatile compounds are degraded due to thermal processing. Lipoxygenase, which is responsible for the formation of fresh tomato aroma, are also inactivated during heating (Goodman and others 2002).

1.2.3.3 Viscosity and consistency

Viscosity, the resistance of fluid to the relative shear, is another important attribute in the quality of tomato products. Products with high quality have high viscosity (Thakur and others 1996). Viscosity can be determined by analytical rheometers. Tomato products are non-Newtonian; therefore, consistency is measured rather than viscosity. The Bostwick consistometer is a standard method used for determining the consistency of tomato products. It measures the shear stress under a fixed shear rate while Efflux viscometers, such as the Canon-Fenske (for serum viscosity) and the Libby tube (for tomato juice), measure shear rate under fixed shear stress..

Factors affecting the viscosity of tomato products include tomato variety, solids content, particle size, and processing (Hayes and others 1998). During processing into juice, tomatoes can be put through either a cold break or hot break system, which greatly affect the viscosity of the final products. In the cold break method, a lower temperature is

used to enhance the activity of pectolytic enzymes, which can degrade pectin in tomatoes into smaller molecules of galacturonic acids and decrease viscosity of the juice, as well as to increase yield, and create flavor in the tomato juice (Madhavi and Salunkhe 1998). In hot break, high temperature is used to inactivate the pectolytic enzymes in order to produce a product with high viscosity. Most juice is processed by hot break systems since most juice is concentrated into paste and high viscosity in tomato paste is desired. However, at very high break temperatures, such as 100 °C, the structure of pectin can collapse and the viscosity decreases again (Trifiro and others 1998). During concentration, the higher the evaporation temperature is, the greater the loss of viscosity (Trifiro and others 1998).

1.2.3.4 Color

Color is important to the quality of tomato products since it is the first quality parameter observed by consumers and is presumed to predict other qualities such as flavor (Thakur and others 1996). There are several methods for measuring color. In voluntary USDA grading USDA grading standards for processing tomatoes, Munsell disk colorimeter can be used to determine the color (U.S. Department of Agriculture (USDA) 1983). Agtron colorimeter is commonly used to determine the color in fresh tomatoes, especially for tomato juice and halves. In processed tomato products, the Hunter colorimeter is generally used to measure the L , a , and b values, which can be converted to hue angle.

Carotenoids, especially lycopene, create the color in tomatoes. Lycopene constitutes 80–90 % of the carotenoids present. They are more stable than other

pigments; however, they can be degraded and lead to the loss of color (von Elbe and Schwartz 1996). The main cause of carotenoid degradation during processing is oxidation in the presence of light, oxygen, and lipoxygenase. During hot break, the hotter the break temperature, the greater the loss of color, even under a vacuum condition (Trifiro and others 1998). Concentrating tomato juice to paste in the presence of oxygen also causes the degradation of lycopene. Heat concentration of tomato pulp can cause up to 57 % loss of lycopene (Noble 1975). With current evaporators, it is likely very little destruction of lycopene occurs.

During processing, a change in color is also caused by brown pigments in the products (Thakur and others 1996). Browning is caused by several reactions, such as caramelization, Maillard reaction, and degradation of ascorbic acid (Mudahar and others 1986). These reactions can occur during all steps of processing, including breaking, concentrating, and canning (Eichner and others 1996).

1.2.3.5 Vitamin C

Tomatoes are a good source of vitamin C, which provide 20% of the recommended daily allowance of vitamin C per serving (90 g of fresh tomatoes) (U.S. Department of Agriculture (USDA) 2009). The amount of vitamin C in tomatoes can be determined by 2,6-dichloroindophenol titration (AOAC Method 967.21, 45.1.14), which measures ascorbic acid by direct titration with 2,6-dichloroindophenol dye. Vitamin C is very sensitive to changes in air, light, pH, and heat. During processing to paste, vitamin C content in tomatoes decreases to approximately 50% of its original value due to the

degradation of ascorbic acid. The main processing steps responsible for the loss of vitamin C are hot break and pasteurization (Capanoglu and others 2008).

1.2.4 Conclusions

Tomato paste is one of the important products, which can be used in the production of tomato sauce, ketchup, and salsa. The processing steps of tomato juice, sauce, and paste include grading, washing, sorting, size reduction, a break system, extraction, and concentration. Tomato paste is usually concentrated from tomato juice using multiple effect evaporators, which can cause the loss of some important quality of the final products, such as vitamin C and volatile compounds. A new technology, the solid-liquid separation (SLS), which does not require any heat to dewater the product, was therefore expected to preserve the quality of tomato concentrate, as well as the lower consumption of energy. After processing, the quality attributes of the products, including total solids, soluble solids, volatile compounds, viscosity, color, and nutritional value, such as vitamin C, are determined to maintain the quality of the products since most of them change during processing.

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CHAPTER 2: EFFECT OF POWDER AND TARGET PROPERTIES ON WRAP AROUND EFFECT DURING COATING

2.1 Abstract

Tapioca starch, NaCl (28, 135, and 378 μm), corn starch, cocoa powder, soy protein isolate, cheese powder, wheat protein, modified starch, nacho cheese, and sugar were coated at 0 kV for nonelectrostatic and at 25 kV for electrostatic coating onto metal, wood, unoiled paper, oiled paper, unoiled plastic, oiled plastic, fresh bread, and dry bread. Powders and targets were allowed to naturally tribocharge, or all charge was removed before coating. Powder particle size, flowability, resistivity, and target resistivity were reported. Electrostatic coating produced the same or better wrap around, or percent side coverage as nonelectrostatic coating for every powder and target. The greatest electrostatic improvement was found when using powders that had the worst nonelectrostatic side coverage: large particle size ($>135 \mu\text{m}$), low resistivity, and low cohesiveness, especially on targets that had high-surface resistivity ($2 \times 10^5 \Omega\text{m}$). Tribocharging had a similar effect as electrostatic coating. In both nonelectrostatic and electrostatic coating, percent side coverage increased as powder particle size decreased, cohesiveness increased, or target resistivity decreased. In electrostatic coating, percent side coverage increased as powder resistivity increased; however, in nonelectrostatic

coating, as powder resistivity increased, percent side coverage increased on only oiled plastic and dry bread.

Key words: powder, electrostatic, coating, size, resistivity

2.2 Practical application

The evenness of powder coating on food is very important for consumer acceptability, since consumers judge food from its appearance before they have a chance to taste it. If thick food targets, such as cakes, donuts, and marshmallows need to be coated, the side coverage, due to the wrap around effect, is important. Choosing powders with small particle size, high cohesiveness and high-powder resistivity, and using electrostatic coating can produce food targets coated on all sides.

2.3 Introduction

The wrap around effect is the ability of a powder to coat down the sides of a thick target during coating; therefore, powder particles can not only cover the top surface of the target but also down the sides (Bailey 1998), which can lead to efficient side coating of powders on a thick target. This wrap around effect is possible because of particle inertial forces, gravitational forces, and aerodynamic forces. In electrostatic coating, there are also electrostatic forces and a space charge effect (Bailey 1998).

Electrostatic coating is the use of positively or negatively charged powders during coating and is used to produce evenly coated products and improve powder adhesion. Electrostatic coating is often more even than nonelectrostatic coating because charged

powder particles repel one another to form a cloud that evenly disperses the coating across the surface (Ricks and others 2002; Biehl and Barringer 2003). The charge on the powders also produces better adhesion between powders and the target surface than powders without charging (Hughes 1997).

During coating, charge can also be produced by tribocharging, which occurs when there is a charge exchange between surfaces, such as powder particles or powders and surfaces, as they contact each other (Bailey 1998). Tribocharging produces more even powder deposition on a target that has complicated geometry (Boncza-Tomaszewski and Penczek 2002); however, it depends heavily on the properties of the powders. Some targets or powders readily tribocharge since they exchange electrons easily while others do not (Sumawi and Barringer 2005).

The performance of powder coating is affected by food powder properties, such as particle size, flowability, and resistivity. Nonelectrostatic transfer efficiency increases as the particle size increases (Ricks and others 2002; Ratanatriwong and others 2003; Biehl and Barringer 2004; Mayr and Barringer 2006). The larger particles fall on the target because of their high inertia while the smaller particles stay in the air as dust due to the effect of aerodynamic forces (Ricks and others 2002; Yousuf and Barringer 2007). With electrostatics, the opposite occurs: transfer efficiency increases when powders with smaller particle size are used (Ricks and others 2002; Ratanatriwong and others 2003; Ratanatriwong and Barringer 2007; Yousuf and Barringer 2007). The electrostatic forces have greater influence over smaller particles than larger particles. These forces can overcome gravitational and aerodynamic forces to land on the targets

rather than stay in the air as dust (Mayr and Barringer 2006). The improvement in adhesion is also particle size dependent. The adhesion of nonelectrostatically and electrostatically coated sugar decreases as the particle size increases (Halim and Barringer 2007).

Flowability of powders can be measured by angle of repose. It is used to indicate the cohesiveness of food powders, which affects the evenness of coating and the adhesion of powders on the target. When more free-flowing powders are used, the transfer efficiency increases since the powder evenly disperses on the target (Biehl and Barringer 2004; Amefia and others 2006; Ratanatriwong and Barringer 2007). However, powders that are more cohesive provide better adhesion after coating on the target (Sumawi and Barringer 2005).

Powder resistivity indicates the amount of charge a powder can acquire during charging, which will affect the attraction to the target and adhesion of powder during electrostatic coating (Masui and Murata 1986). Powders with high resistivity, more than $10^{13} \Omega\text{m}$, have a strong attraction and produce good adhesion because they are insulators with slow charge decay. Powders with lower resistivity have faster charge decay; therefore, they produce lower attraction and less adhesion on the target (Bailey 1998). Because of weak adhesion, powders with low resistivity do not stay on targets after landing (Sims and others 2000), so they produce low-transfer efficiency.

The characteristics of the target must also be considered to achieve the best coating (Bailey 1998). Targets with lower resistivity provide better charge flow from the ground and better attraction between the powder and the target surface during

electrostatic coating (Sims and others 2000). As food target resistivity increases, the percent electrostatic adhesion improvement increases (Halim and Barringer 2007).

The physical properties of the powders and targets, including particle size, flowability, powder resistivity, and target resistivity, affect the wrap around effect during coating. However, there is no research done on this effect on a thick target yet. The objective of this research is to determine how electrostatic coating, tribocharging, powder particle size, powder flowability, powder resistivity, and target resistivity affect the percent side coverage.

2.4 Materials and methods

The 12 different powders analyzed in this study were modified starch (Modified National 377 Starch, Amphur Muang, Thailand), 28 μm NaCl (Extra-fine 325 Salt, Morton Intl., Inc., Chicago, Ill., U.S.A.), 135 μm NaCl (Snack flour salt, Morton Intl., Inc.), 378 μm NaCl (Alberger flake salt, Cargill Inc., Minneapolis, Minn., U.S.A.), corn starch (Pure-Dent B700 Food Corn Starch, Grain Processing Corp., Muscatine, Iowa, U.S.A.), cocoa powder (The Great American Spice Co., Fort Wayne, Ind., U.S.A.), cheese powder (Kraft Cheese Powder, Kraft Food Ingredients, Memphis, Tenn., U.S.A.), nacho cheese (Givaudan, Cincinnati, Ohio, U.S.A.), wheat protein (Manildra Isolated Wheat Protein, Manildra Group, Mission, Kans., U.S.A.), sugar (Baker's Special Superfine Castor Sugar King Arthur Flour, Norwich, Vt., U.S.A.), tapioca starch (AVEBE America Inc., Princeton, N.J., U.S.A.), and soy protein isolate (Cargill Inc., Wayzata). All experiments were done at 25 ± 5 °C and 35 ± 5 % relative humidity.

The powders were stored in desiccators with drierite desiccant for at least 3 d prior to all tests. The particle size of the powders was determined by a Malvern particle size analyzer (Mastersizer X standard bench, Malvern Instruments Ltd., Worcestershire, UK). The volume mean diameter $D[4,3]$ of each powder was reported.

The angle of repose was determined by slowly passing the powder through a funnel onto a powder-collecting cup (cup radius = 3.75 cm). The peak height of powder was measured by a caliper and the angle of repose was calculated by the following equation: Angle of repose = \tan^{-1} [peak height/cup radius].

The powder resistivity of each powder was measured using a powder test cell (Electrostatic Solutions, Bassett, UK), electrometer (Model 6514, Keithley Instruments Inc., Cleveland, Ohio, U.S.A.), voltmeter (LCD Auto Range Digital Multimeter, Model 22-163, Radio Shack, Fort Worth, Tex., U.S.A.), and a highvoltage power supply (ABC Model 125-1DM, Kepco, General Electric Co., Flushing, N.Y., U.S.A.). Powder (5 cm³) was measured by volumetric cylinder and put into the powder test cell. The powder was then compressed to remove air pockets. The applied voltage read from the voltmeter was adjusted to 125 V. When there was no change in the current value from the electrometer for more than 15 s, the current value was recorded and the resistivity was calculated by the following equation: $\Delta = (KV)/I$ where Δ = resistivity (Ω m), K = the cell constant = 0.014, V = the voltage applied (V), and I = the measured current (A).

The targets used in this study were blocks of wood, wood covered with aluminum foil, wood covered with freezer paper (Reynolds, Reynolds Consumer Products, Richmond, Va., U.S.A.), and wood covered with polyethylene (Glad Cling Wrap,

TheGlad Products Co., Oakland, Calif., U.S.A.), all 7.62 x 7.62 x 20 cm. The paper and polyethylene were coated with 1 g canola oil (Kroger pure canola oil, The Kroger Co., Cincinnati, Ohio, U.S.A.) before coating and compared with the same targets without oil coating. The food target was bread (Kroger's Texas Style Toast, The Kroger Co.). The bread was used as fresh bread (9.9 x 9.0 x 22 cm; $A_w = 0.928$) and dry bread (9.8 x 8.9 x 21 cm; $A_w = 0.356$), which was left outside a container for 3 d prior to the study in order to produce bread with 2 different surface resistivities.

The resistivity of the target surface was measured using an electrometer and a high-voltage power supply. The targets used in this measurement were 7.6 x 7.6 x 2.4 cm. Two electrodes, one connected to the power supply and one connected to the electrometer, were attached in the center of the top surface and bottom surface of the target, respectively. The applied voltage read from the voltmeter was adjusted to 125 V. When there was no change in the current value from the electrometer for more than 15 s, the current value was recorded and the resistivity was calculated by the following equation: $\Delta = (VA)/(Il)$ where Δ = resistivity (Ω m), V = the voltage applied (V), A = area of electrodes attached to the target surface (m^2), I = the measured current (A), and l = the linear distance between the two electrodes (m).

The coating was done manually by coating 3 g of food powder on each target using a powder applicator (Terronics Development Corp., Elwood, Ind., U.S.A.) at 0 kV and 25 kV for nonelectrostatic and electrostatic coating, respectively (Figure 10). Powder was fed through a pneumatic sprayer into a chamber with 2 electrode wires to create an electrostatic field and charge the powder. The charged powder was attracted to the target

in the chamber. Each powder was coated on the different targets by placing the target on a cardboard tray wrapped with aluminum foil. After coating, the distance each powder extends down from the top at one-fourth, one-half, and three-fourth of the length along each side of the target was recorded. The percent side coverage was calculated by the following equation: $P = (B/C) \times 100$, where P = percent side coverage, B = average distance of powder extending along side of the target, and C = height of the target. This calculation was used to compare the wrap around effect on targets with different heights. The charge on both powders and targets was also removed using a Nucleospot static eliminator (Local Air Ionizer Model P-2042, NRD LLC., Grand Island, N.Y., U.S.A.) to study the wrap around effect without tribocharging.

Two-way analysis of variance with Tukey's HSD (Honestly Significant Difference) test and t-test were used for statistical analysis to determine if there was a significant difference between the coverage of powder on the target and the difference between the nonelectrostatic and electrostatic coating, respectively. Two-stage least squares analysis was used for the regression analysis to determine if the powder properties correlated to the percent side coverage. A P-value of 0.05 or lower indicates a significant difference between variables.

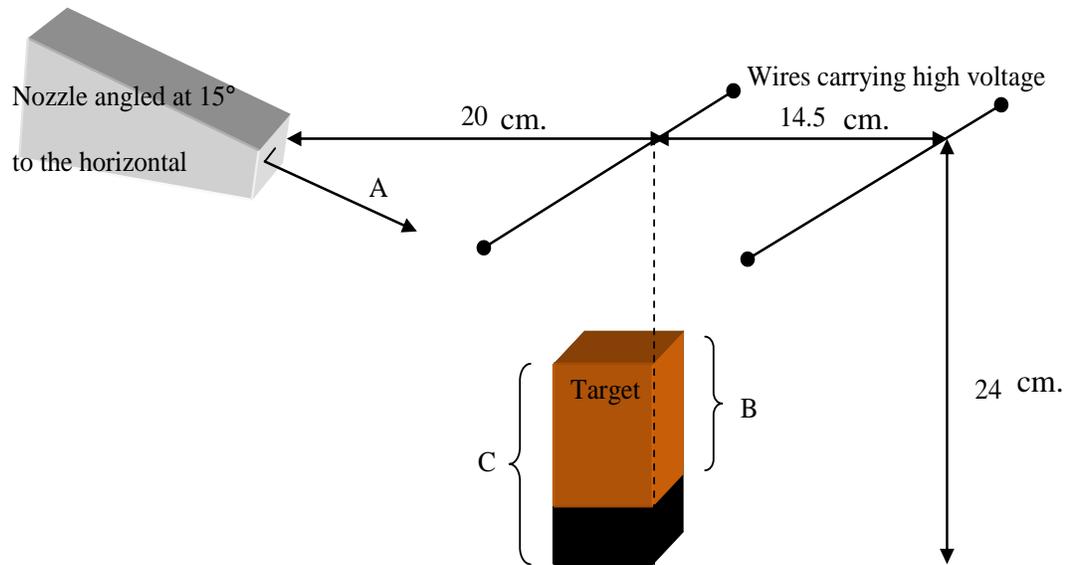


Figure 10. Schematic of powder coating. A: powder entering the chamber. B: coating height on the target. C: target height. B at three locations on each side of the target divided by C is the percent side coverage.

2.5. Results and discussion

The food powder physical properties measured in this study were particle size, resistivity, and flowability (Table 1). Food powders were chosen to provide a wide range of each physical property. Some of the powders were similar in composition but different in properties, such as NaCl, which has 3 different sizes. A range of targets, including several nonfood targets, were chosen to provide a wide range of target characteristics such as targets with different resistivity and adhesion.

Table 1. Physical properties of food powders.

Powder	Particle size (µm)	Angle of Repose (°)	Flowability category (Peleg 1977)	Resistivity (Ωm)	Resistivity category (Bailey 1998)
Native Tapioca Starch	22	37.4	Free-flowing	5.98×10^9	Low resistivity
Small NaCl	28	31.9		1.30×10^7	
Corn Starch	28	31.9		4.66×10^{11}	Medium resistivity
Cocoa Powder	30	40.9	Cohesive	4.80×10^{11}	
Soy Protein Isolate	68	42.7		8.47×10^9	Low resistivity
Cheese Powder	97	40.6		9.05×10^{10}	Medium resistivity
Wheat Protein	109	35.9	Free-flowing	5.67×10^8	Low resistivity
Medium NaCl	135	31.0		9.50×10^6	
Modified Starch	189	28.3		1.03×10^{10}	Medium resistivity
Nacho Cheese	200	35.7		8.10×10^{11}	
Sugar	216	27.5		5.67×10^8	Low resistivity
Large NaCl	378	24.6		5.64×10^5	

2.5.1 Effect of electrostatic coating on percent side coverage

For every powder and target, electrostatic coating produced the same or significantly better percent side coverage as nonelectrostatic coating (Figure 11). The space charge and electrostatic forces present in electrostatics produces better side coverage (Bailey 1998). According to Coulomb's law, the attraction force increases when the amount of charge increases. Therefore, the charge produced on the powders by electrostatics produces both better attraction to the target, and better adhesion between powders and the target surface (Hughes 1997), resulting in more attraction to the sides of target during electrostatic coating. Powders with large particle size, low resistivity, and that were free-flowing, showed the greatest improvement in percent side coverage with

electrostatic coating, especially on targets with high resistivity. These powders and targets showed poor nonelectrostatic coverage; therefore, they produced the most improvement when electrostatic coating was applied. The effect of the different particle characteristics will be discussed in the following sections.

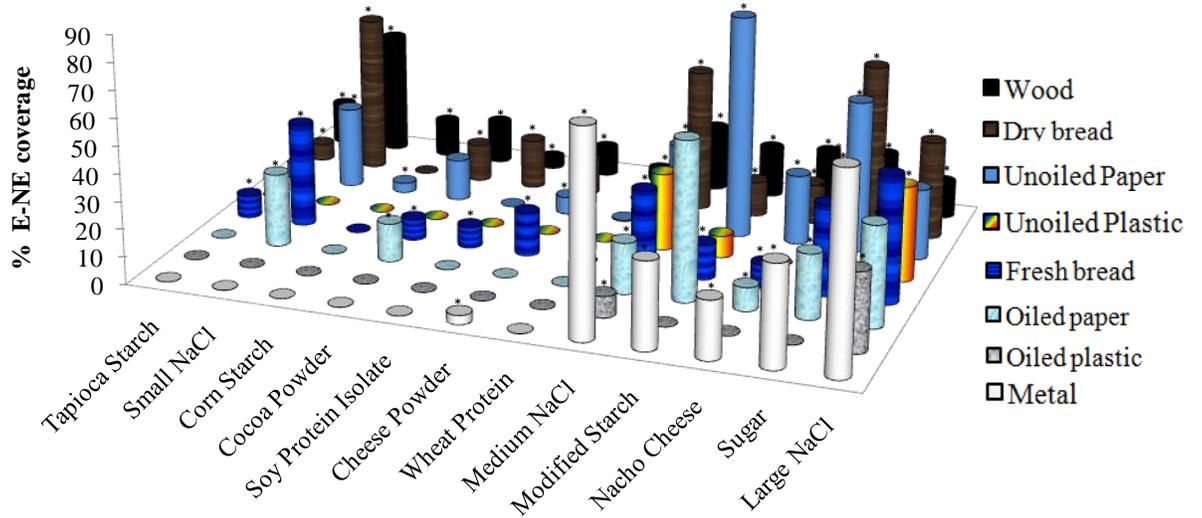


Figure 11. Improvement in percent side coverage with Electrostatic (E) instead of Nonelectrostatic (NE) coating (%E – NE coverage) of food powders on each target.

* indicates a significant difference between electrostatic and nonelectrostatic percent side coverage.

2.5.2 Effect of tribocharging on percent side coverage

Powders and targets were allowed to naturally tribocharge, or all charge was removed with an ionizer to study the effect of tribocharging on percent side coverage. Tribocharging is the charge on powders produced by frictional contact among powder particles or between powders and a target (Bailey 1998). The difference between coating with and without tribocharging was larger in nonelectrostatic coating than in electrostatic coating. This was expected since the charge produced by electrostatics is typically greater than by tribocharging so tribocharging had little effect when electrostatic coating was used. Similar to electrostatics, tribocharging produced the greatest improvement in samples with the worst nonelectrostatic percent side coverage: large, free flowing, low resistivity powders on high resistivity targets.

2.5.3 Effect of particle size

Particle size was the most important factor, with the strongest correlation to percent side coverage. Both nonelectrostatic and electrostatic percent side coverage significantly increased as particle size decreased for all targets (Table 2 and 3; Figure 11 and 12). The percent side coverage on bread targets is shown as an example (Figure 12). In powder coating, size affects aerodynamic force, electrostatic force, and adhesion. The wrap around effect increases when the aerodynamic forces, including turbulence, dominate over the gravitational force; therefore, powders deposit on the sides of the target instead of on the conveyor belt. Small powders are affected more strongly by aerodynamic forces while large powders are more affected by gravitational force (Bailey 1998). With electrostatic force, as the particle size decreases, electrostatic force increases

since the charge to mass of small particles increases more than large powders (Mazumder and others 1997), resulting in an increase of attraction of powders to the target surface (Ricks and others 2002). For larger particles, gravitational forces dominate over the electrostatic forces (Ricks and others 2002) resulting in less powder on the sides of the target. Once the powders land on the target, the adhesion between the powder and target surface prevents the powders from falling off the target. There is a decrease in adhesion as the particle size increases (Buck and Barringer 2007; Halim and Barringer 2007), so even if large powders deposit on the side of the target, they are more likely to fall off of the target, resulting in lower percent side coverage. In addition, the charge from electrostatics creates electrostatic adhesion, which is stronger for smaller particles (Mayr and Barringer 2006).

The targets coated with oil had higher percent side coverage than the corresponding targets without oil, except when both samples had 100% side coverage (Table 2 and 3). On oily surfaces, capillary forces become dominant over van derWaals forces, gravitational forces, and in the case of electrostatic coating, electrostatic adhesion forces. As capillary forces increase, adhesion increases, resulting in better coating performance (Buck and Barringer 2007). Thus, adhesion is a major factor in improving side coverage. However, when electrostatic coating was applied, some targets coated with oil had significantly higher percent side coverage than those with nonelectrostatic coating, because of the improved transfer efficiency of powders onto the target, with electrostatic coating.

Table 2. Average nonelectrostatic percent side coverage on each target.

Powder	Nonelectrostatic percent side coverage (%)							
	Metal	Wood	Unoiled Paper	Oiled paper	Unoiled Plastic	Oiled plastic	Fresh bread	Dry bread
Tapioca Starch	^a 100 ^A	^a 78 ^B	^a 91 ^C	^a 100 ^A	^a 100 ^A	^a 100 ^A	^a 92 ^C	^a 94 ^C
Small NaCl	^a 100 ^A	^b 15 ^B	^b 21 ^C	^b 62 ^D	^a 100 ^A	^a 100 ^A	^b 62 ^D	^b 29 ^E
Corn Starch	^a 100 ^A	^c 75 ^B	^c 96 ^C	^a 100 ^A	^a 100 ^A	^a 100 ^A	^c 100 ^A	^c 100 ^A
Cocoa Powder	^a 100 ^A	^d 8 ^B	^d 77 ^C	^c 86 ^D	^a 100 ^A	^a 100 ^A	^a 92 ^E	^d 86 ^D
Soy Protein Isolate	^a 100 ^A	^e 96 ^B	^a 100 ^A	^a 100 ^A	^a 100 ^A	^a 100 ^A	^a 92 ^C	^e 82 ^D
Cheese Powder	^b 97 ^A	^f 5 ^B	^f 94 ^C	^a 100 ^D	^a 100 ^D	^a 100 ^D	^d 84 ^E	^d 87 ^F
Wheat Protein	^a 100 ^A	^e 95 ^B	^e 100 ^C	^a 100 ^A	^a 100 ^A	^a 100 ^A	^c 100 ^A	^c 100 ^A
Medium NaCl	^c 25 ^A	^g 37 ^B	^g 12 ^C	^d 81 ^D	^b 72 ^E	^b 92 ^F	^e 70 ^G	^f 48 ^F
Modified Starch	^d 68 ^A	^b 16 ^B	^h 42 ^C	^e 92 ^D	^a 100 ^E	^a 100 ^E	^f 87 ^F	^g 80 ^G
Nacho Cheese	^e 79 ^A	^h 1 ^B	ⁱ 63 ^C	^e 91 ^D	^a 100 ^E	^a 100 ^E	^a 91 ^D	^e 82 ^E
Sugar	^f 63 ^A	ⁱ 0 ^B	^j 17 ^C	^f 76 ^D	^c 67 ^E	^a 100 ^F	^g 56 ^G	^h 19 ^F
Large NaCl	^c 25 ^A	ⁱ 0 ^B	^k 0 ^B	^g 54 ^C	^d 22 ^D	^c 71 ^E	^h 0 ^B	ⁱ 0 ^B
Target resistivity (x 10 ⁻³ Ωm)	0.903	2.22	2.61	5.04	163	216	265	300

a-k indicate significant difference between the powders. A-G indicate significant difference across the targets.

Table 3. Average electrostatic percent side coverage on each target.

Powder	Electrostatic percent side coverage (%)							
	Metal	Wood	Unoiled Paper	Oiled paper	Unoiled Plastic	Oiled plastic	Fresh bread	Dry bread
Tapioca Starch	^a 100 ^A	^a 93 ^B	^a 96 ^C	^a 100 ^A				
Small NaCl	^a 100 ^A	^b 60 ^B	^b 51 ^C	^b 89 ^D	^a 100 ^A	^a 100 ^A	^a 100 ^A	^b 87 ^E
Corn Starch	^a 100 ^A	^c 89 ^B	^c 100 ^A	^a 100 ^A				
Cocoa Powder	^a 100 ^A	^d 23 ^B	^d 92 ^C	^a 100 ^A				
Soy Protein Isolate	^a 100 ^A	^e 100 ^A	^c 100 ^A	^a 100 ^A				
Cheese Powder	^a 100 ^A	^f 16 ^B	^c 100 ^A	^a 100 ^A				
Wheat Protein	^a 100 ^A	^e 100 ^A	^c 100 ^A	^a 100 ^A				
Medium NaCl	^a 100 ^A	^b 60 ^B	^e 43 ^C	^a 100 ^A				
Modified Starch	^a 100 ^A	^g 87 ^B	^a 98 ^C	^a 100 ^A				
Nacho Cheese	^a 100 ^A	^h 21 ^B	^f 88 ^C	^a 100 ^A	^a 100 ^A	^a 100 ^A	^a 100 ^A	^c 97 ^D
Sugar	^a 100 ^A	^h 21 ^B	^g 72 ^C	^a 100 ^A	^a 100 ^A	^a 100 ^A	^b 89 ^D	^d 81 ^E
Large NaCl	^b 97 ^A	ⁱ 13 ^B	^h 26 ^C	^c 91 ^D	^b 56 ^E	^a 100 ^F	^c 46 ^G	^e 36 ^F

a-i indicate significant difference between the powders. A-G indicate significant difference across the targets.

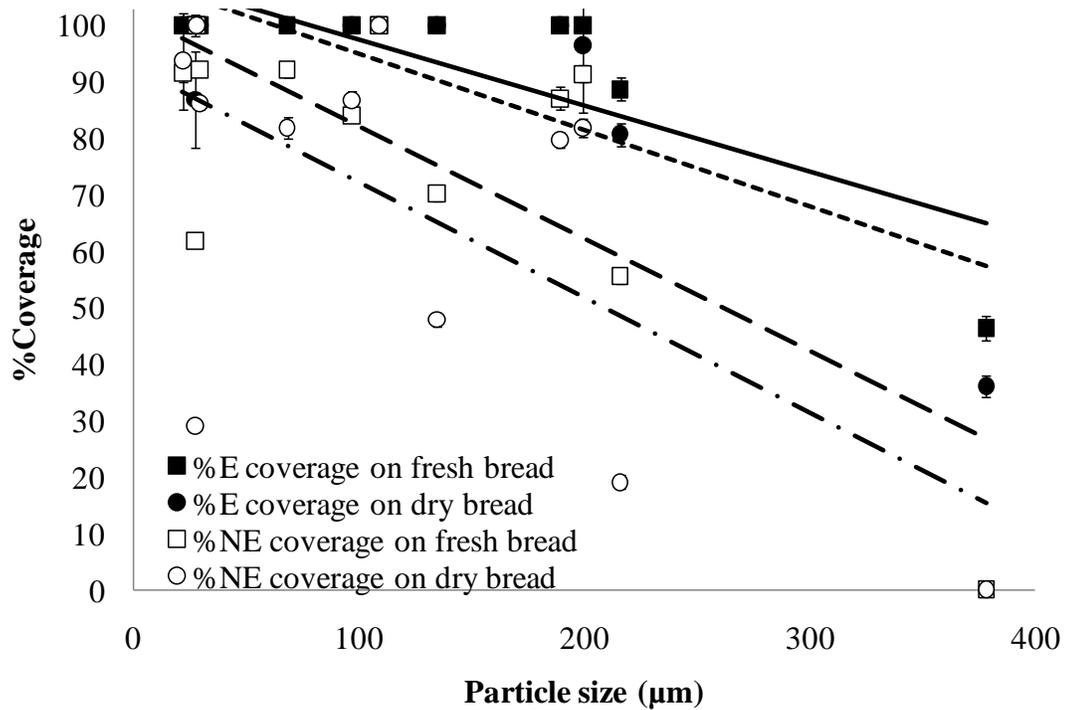


Figure 12. Effect of powder particle size on percent side coverage of nonelectrostatic (NE) and electrostatic (E) coating on fresh and dry bread.

2.5.4 Effect of flowability

The flowability was measured by angle of repose. Powders with angle of repose more than 50 degrees are nonflowing, 40–50 degrees are cohesive and less than 40 degrees are free-flowing (Peleg 1977; de Jong and others 1999; Teunou and others 1999). As the angle of repose increased (powders were more cohesive), the nonelectrostatic and electrostatic percent side coverage significantly increased for all targets (Table 1–3,

Figure 13). When the cohesiveness of powder increases, the adhesion increases (Sumawi and Barringer 2005), so cohesive food powders adhere well without falling off of the target, which produces higher percent side coverage. The effect of flowability on percent side coverage is opposite to its effect on transfer efficiency, which increases when free-flowing powders are used (Biehl and Barringer 2004; Amefia and others 2006; Ratanatriwong and Barringer 2007). This difference likely occurs because transfer efficiency depends on even dispersion of the powder so that it lands on the target. This is also required for a high percent side coverage, but adhesion after landing on the target is a more important factor, as already shown in the section comparing oiled targets with high adhesion, to targets with lower adhesion.

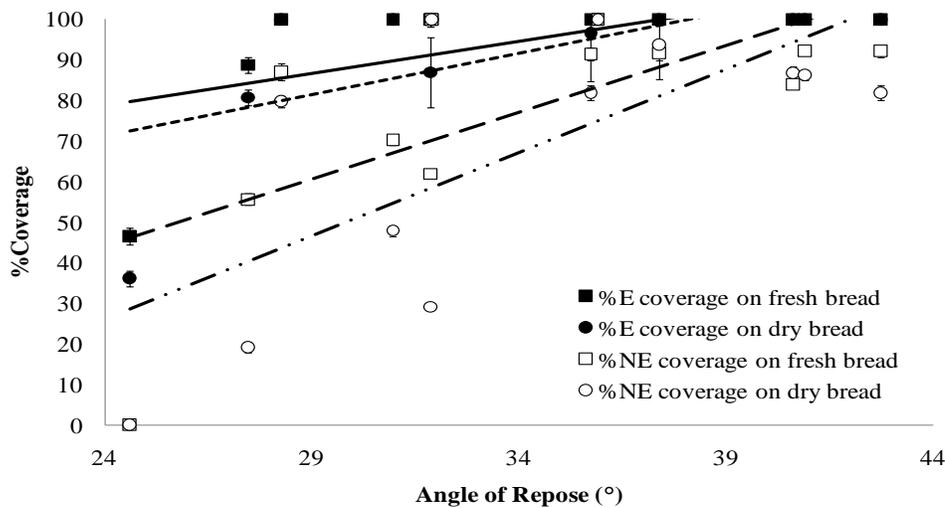


Figure 13. Effect of powder angle of repose on percent side coverage of nonelectrostatic (NE) and electrostatic (E) coating on fresh and dry bread.

2.5.5 Effect of powder resistivity

Powder resistivity also played a role in the percent side coverage. In nonelectrostatic coating, as the resistivity increased, percent side coverage significantly increased on oiled plastic and dry bread; however, other targets did not show any effect, as expected (Table 1–3, Figure 14). In electrostatic coating, as the powder resistivity increased, the percent side coverage significantly increased on all targets (Table 1–3, Figure 14). During electrostatic coating, resistivity affects how quickly the charge on powder decays (Bailey 1998). The rate of charge decay affects both how long the charge remains on the powder when landing on the target surface, and whether electrostatic adhesion is created. During electrostatic coating, the charged powders with higher resistivity have a slower charge decay rate. Thus, they hold the charge long enough to be directed to the target by electrostatic forces and produce electrostatic adhesion between the particles and target surface (Hughes 1997). However, some powders with medium resistivity also showed high improvement, such as nacho cheese and modified starch. This is because these powders had a large particle size, which had more influence on side coverage than the resistivity.

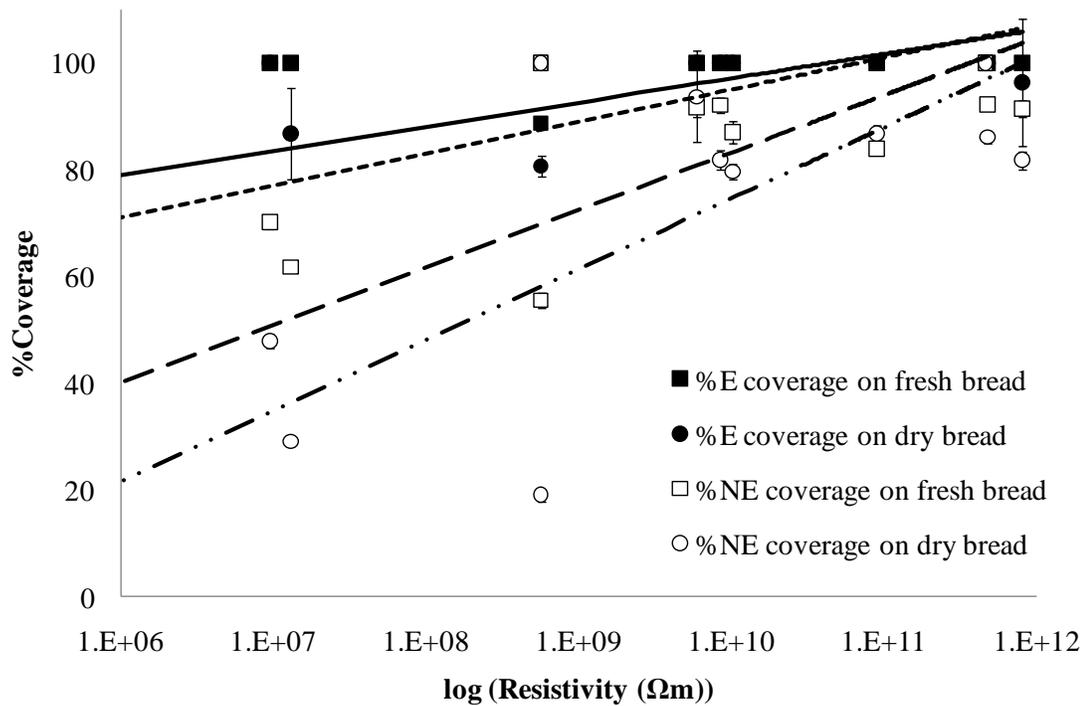


Figure 14. Effect of powder resistivity on percent side coverage of nonelectrostatic (NE) and electrostatic (E) coating on fresh and dry bread, excluding NE fresh bread which had no significant effect.

2.5.6 Effect of target resistivity

As target resistivity decreased, nonelectrostatic and electrostatic percent side coverage significantly increased for all powders (Table 2 and 3, Figure 15). For the 5 targets with a resistivity less than $2 \times 10^{-5} \Omega\text{m}$, only the 2 largest powders did not always produce 100% side coverage during electrostatic coating. In electrostatic coating, targets

with lower resistivity provide better charge flow from the ground and thus better attraction between the powder and the target surface (Sims and others 2000). As charged powders land on a low resistivity target, charge flows to the ground quickly, allowing more charged powder to land on the target. Therefore, charged powders have more adhesion on low resistivity targets than on targets with high resistivity. On the other hand, targets with high resistivity build up a charge from powders that have already landed and repel additional powder.

The best resistivity for target and powder is opposite. High resistivity powders hold a charge, allowing electrostatic force to direct them to the target while low resistivity targets remove that charge so that additional powder can be deposited. Unfortunately, most food targets have low resistivity. Thus differences in food powders have a greater effect than differences in food targets.

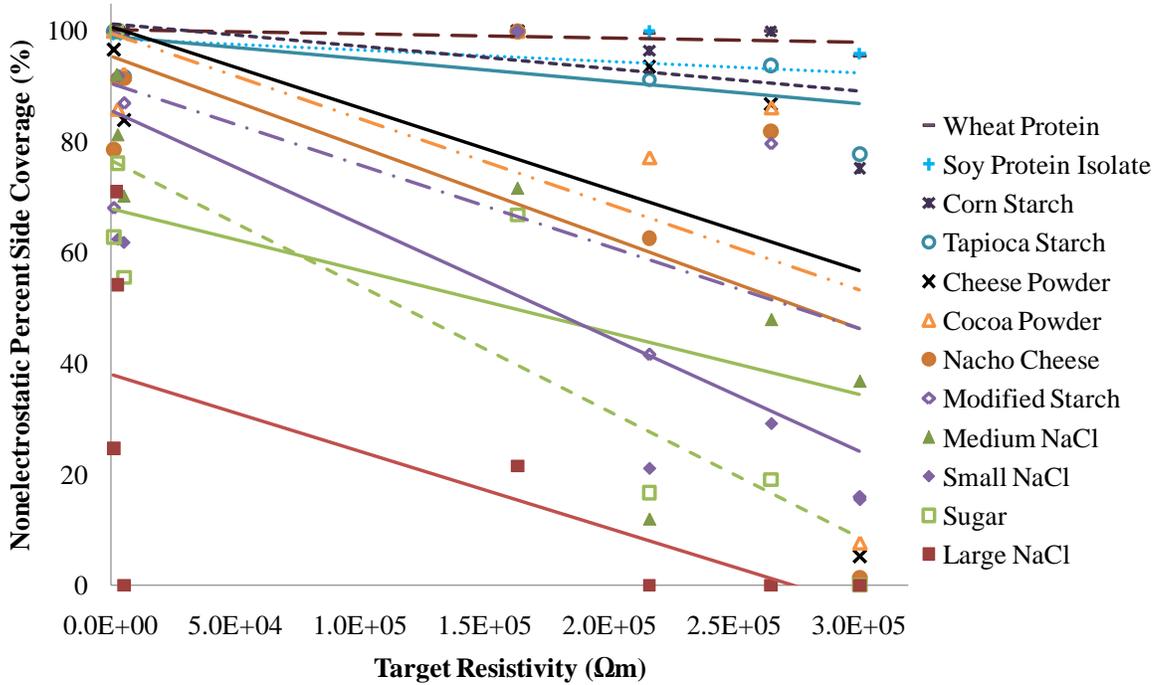


Figure 15. Effect of target resistivity on nonelectrostatic percent side coverage.

2.6 Conclusions

Electrostatic coating produced the same or better percent side coverage as nonelectrostatic coating for every powder and target. The greatest electrostatic improvement can be found when using powders with large particle size, lower cohesiveness, and lower resistivity, especially on wood, uncoiled paper, and dry bread, which have high resistivity. Similar results were found with tribocharging. In both nonelectrostatic and electrostatic coating, the percent side coverage increased as particle size of the powders decreased, cohesiveness increased, and target resistivity decreased.

Nonelectrostatic percent side coverage increased as the resistivity of powder increased on oiled plastic and dry bread but other targets did not show any effect. In electrostatic coating, as powder resistivity increased, percent side coverage increased on all targets.

2.7 References

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CHAPTER 3: EFFECT OF SODIUM CHLORIDE AND FOOD TARGET PROPERTIES ON NONELECTROSTATIC AND ELECTROSTATIC COATING

3.1 Abstract

Salts were coated on a variety of thick food targets. The best transfer efficiency, adhesion (> 70%), and percent side coverage (100%) were obtained when small (< 200 μm) and cohesive (Hausner ratio > 1.20) salt was used with electrostatic coating on targets with high a_w (> 0.7), low resistivity (< $9 \times 10^8 \Omega\text{m}$), and short charge decay time (< 3.8 s). The shape of salt particles also affected the coating performance; porous cube provided significantly better transfer efficiency and adhesion than flake salt on some targets. There was no significant effect of KCl content on coating performance.

Key words: powder, corona discharge, electrical resistivity, adhesion, electrostatic charge

3.2 Practical applications

Coating performance has a significant effect on the flavor of the food, and waste produced. Use of small, cohesive, porous cube salt with electrostatic coating on targets with high a_w , low resistivity, and short charge decay time can maximize the coating performance.

3.3 Introduction

Electrostatic powder coating is used in the snack food industry to produce even coating and improve powder adhesion. There are several snack foods that are coated with salt, such as potato chips, popcorn, pretzels, peanuts, tortilla chips, and crackers. With electrostatics, the powder particles repel one another to form a cloud and disperse evenly across the food targets (Biehl and Barringer 2003; Ricks and others 2002). Charges from electrostatic forces also produce stronger attraction between charged food powders and the target surface (Hughes 1997). Therefore, electrostatic coating usually produces better coating performance.

The physical properties of food powders, such as particle size and flowability, have a significant effect on the coating performance. In nonelectrostatic coating, the transfer efficiency increases as the particle size of food powders and seasonings increases (Ricks and others 2002; Biehl and Barringer 2004; Mayr and Barringer 2006). The particle size also affects the adhesion and wrap around effect (percent side coverage) during coating. As particle size increases, adhesion decreases because large powders have a lower charge to mass ratio, which causes less attraction between powder particles and the target surface (Buck and Barringer 2007; Halim and Barringer 2007). The percent side coverage decreases as particle size increases since large particles have less adhesion, resulting in falling off of the target, instead of depositing on the side of the target (Sumonsiri and Barringer 2010).

Flowability of powders indicates the cohesiveness, which affects adhesion of powders on the target surface and the evenness of powder coating. Flowability can be

measured by Hausner ratio (Peleg 1977). Transfer efficiency increases when powders with more free-flowing characteristic are used because they disperse evenly on the target surface (Amefia and others 2006; Ratanatriwong and Barringer 2007). However, more cohesive powders are likely to produce better adhesion on the target surface after coating (Sumawi and Barringer 2005). Cohesive powders also produce higher percent side coverage since they adhere well on the target surface, instead of falling off of the target (Sumonsiri and Barringer 2010).

Shape and composition of food powders also affect the coating performance. The adhesion between salt particles and the target surface is affected by the geometric shape of salts and surface area, which affects the charge to mass ratio (Kuntz 1994). Cubic crystals provide better adhesion over flake and dendritic crystals when coating potato chips with salts (Buck and Barringer 2007). In coating popcorn with salts using a tumble drum coating system, the highest coating efficiency was produced by flake, followed by porous cube salts (Miller and Barringer 2002). Potassium (K) can be used to replace sodium (Na) in salts to reduce the sodium content in food, and so KCl should be studied to determine how it affects food powder coating performance.

The best coating performance can also be achieved by choosing the appropriate target characteristics (Bailey 1998). Targets with higher water activity have lower electrical resistivity, providing faster charge flow from the ground and better electrostatic adhesion (Cross 1987; Sims and others 2000, Halim and Barringer 2007). Targets with lower electrical resistivity have slower charge decay time, or slower charge relaxation time, resulting in better electrostatic adhesion (Cross 1987). The electrostatic percent side

coverage also increases as the target resistivity decreases (Sumonsiri and Barringer 2010).

The performance of nonelectrostatic and electrostatic coating is affected by physical properties of food powders and targets. Previous studies were done on thin metal sheets, but thick targets with a wide range of resistivity, representative of typical food items, have not been studied. The objective of this research is to determine how salt particle size, salt flowability, salt shape and % KCl, target water activity, target electrical resistivity, and target charge decay time affect the transfer efficiency, adhesion, and side coverage during nonelectrostatic and electrostatic coating.

3.4 Materials and methods

The eight different salts analyzed in this study were NaCl of 119, 179, 201, 242, and 332 μm porous cube, 274 μm porous cube with 33% KCl, 277 μm porous cube with 25% KCl (Morton Intl., Inc., Chicago, Ill., U.S.A.), and 182 μm flake (Alberger flake salt, Cargill Inc., Minneapolis, Minn., U.S.A.). 179 μm porous cube was a mixture of 119 and 201 μm porous cube (Morton Intl., Inc.) in a proportion of 1:3.

The salt samples were stored in desiccators with desiccant for at least three days prior to all tests. The particle size was determined by a Malvern particle size analyzer (Mastersizer X standard bench, Malvern Instruments Ltd., Worcestershire, UK). The volume mean diameter $D [4,3]$ of each sample was reported. The Hausner ratio was determined by tapping 100 ml of salt using a Vanderkamp tap density tester (Van-Kel

Industries, Inc., Chatham, N.J., U.S.A.) for 1250 times. The tapped volume was recorded and the Hausner ratio was calculated by the following equation: Hausner ratio=untapped volume (ml)/tapped volume (ml).

The targets used in this study were blocks of wood (ratio of the vapor pressure of water in the target to the vapor pressure of pure water at the same temperature ($a_w=0.46$) (Balsawood, McMaster-Carr, Aurora, Ohio, U.S.A.), and wood covered with freezer paper ($a_w=0.50$) (Reynolds, Reynolds Consumer Products, Richmond, Va., U.S.A.). The food target was crackers ($a_w=0.46$) (Kroger's Thin & Crispy Saltines Unsalted Top Crackers, The Kroger Co., Cincinnati, Ohio, U.S.A.), bread ($a_w=0.93$) (Kroger's Texas Style Toast, The Kroger Co., Cincinnati, Ohio, U.S.A.), carrot ($a_w=0.98$), and romaine green leaf lettuce ($a_w=0.98$). Bread was kept in three different desiccators to create different water activity of samples (0.7, 0.5, and 0.3). In addition, the fresh bread was coated with 2 g canola oil (Kroger pure canola oil, The Kroger Co., Cincinnati, Ohio, U.S.A.) before powder coating to study the effect of adhesion on transfer efficiency. Crackers were glued together, and slices of bread were cut and combined into a cubic shape. Lettuce and slices of carrots covered a block of foam to produce the same cubic shape as the other food targets. All targets were 5.0 x 5.0 x 5.0 cm.

The electrical resistivity of the target was measured using an electrometer (Model 6514, Keithley Instruments Inc., Cleveland, Ohio, U.S.A.) and a power supply (ABC Model 125-1DM, Kepco, General Electric Co., Flushing, N.Y., U.S.A.). Two probe point electrodes, one connected to the power supply and one connected to the electrometer, were attached in the center of the top surface and bottom surface of the target

respectively. The applied voltage read from the power supply was adjusted to 125 V. When there was no change in the current value from the electrometer for more than 15 s, the current value was recorded and the resistivity was calculated by the following equation: $\rho = (VA)/(Il)$ where ρ = resistivity (Ω m), V = the voltage applied (V), A = area of electrodes attached to the target surface (m^2), I = the measured current (A), and l = the linear distance between the two electrodes (m).

To measure the charge decay time of the target surface, each target was placed on a 20 cm x 30 cm aluminum ground plane and charged to 20 μ A with 20 kV by a linear array of stainless steel pins. The space between pins and target surface was 0.5 cm. The samples were charged for a minute before the decay of charge with time was measured by a Monroe Model 236 noncontacting electrostatic voltmeter (Monroe Electronics Inc., Lyndonville, N.Y., U.S.A.). The charge decay time was the time until charge remained 10% of the original charge.

The coating was done using 8.00 g of salt for each target applied with a powder applicator (Terronics Development Corp., Elwood, Ind., U.S.A.) charged at 0 kV and 25 kV for nonelectrostatic and electrostatic coating respectively (Figure 16). During coating, powder was fed and sprayed through a pneumatic sprayer. In electrostatic coating, a wire was used to create an electrostatic field and the powder was charged before landing on a target. Each powder was coated on the different targets by placing the target on a cardboard tray wrapped with aluminum foil. The weight of the targets before and after coating was recorded to calculate the transfer efficiency (TE) using the following equation: $\%TE = \frac{(\text{weight of coated target} - \text{weight of target before coating})}{(\text{weight of NaCl applied})} \times 100$. The distance

each salt extends down from the top at $\frac{1}{4}$, $\frac{1}{2}$, and $\frac{3}{4}$ of the length along each side of the target was recorded. The percent side coverage was calculated by the following equation: $P=(B/C) \times 100$ where P=percent side coverage, B=average distance of salt extending down the side of the target, and C=height of the target. This calculation was used to compare the wrap around effect on different targets. After recording weight of salt and side coverage, the targets were manually shaken 20 times before weighing to determine the adhesion loss. The percent adhesion was calculated by the following equation:

$$\% \text{adhesion} = \frac{(\text{weight of coated target after shaking} - \text{weight of target before coating})}{(\text{weight of coated target} - \text{weight of target before coating})} \times 100.$$

All experiments were done at 25 ± 5 °C and 35 ± 5 % relative humidity.

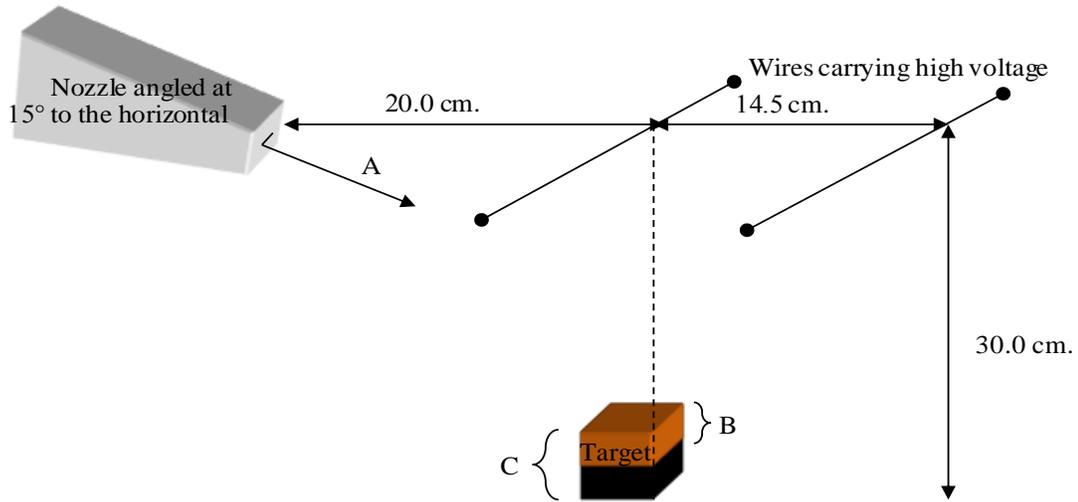


Figure 16. Schematic of powder coating. A: powder entering the plastic chamber (80.0 x 53.0 x 30.0 cm.). B: coating height on the target. C: target height. B at three locations on each side of the target divided by C is the percent side coverage.

Two-way analysis of variance with Tukey's HSD (Honestly Significant Difference) test and *t*-test were used for statistical analysis to determine if there was a significant difference between samples in transfer efficiency, percent coverage of powder on the target, and adhesion loss and the difference between the nonelectrostatic and electrostatic coating respectively. A p-value of 0.05 or lower indicates a significant difference between variables.

3.5 Results and discussion

The particle size of salts used in this study is in the commercial range used on snack foods. The effects of shape and % KCl mixed with NaCl in salts with the same particle size were studied. Various food and nonfood targets were chosen to provide a wide range of target properties, including water activity (a_w), electrical resistivity, and charge decay time. Previous studies have been limited to a narrow range of targets, most of which are not food. These targets were chosen to represent a much larger range of resistivity, covering the range of resistivity represented by food, to allow for prediction of trends during food coating. In addition, large targets were used to quantify side coating, since most foods are large, as opposed to the flat targets used in previous studies.

3.5.1 Effect of salt particle size

Adhesion between salt particles and the target surface strongly correlated with salt particle size. As particle size increased, both nonelectrostatic and electrostatic adhesion significantly decreased for all targets. Figure 17 presents the results for electrostatic coating. The same trend occurred during nonelectrostatic coating. Gravitational force has

more effect than electrostatic forces and dominates for larger particles. In addition, the force of attraction between particles and the target surface decreases as particle size increases because of a lower charge to mass ratio (Bailey 1998); therefore, larger particles easily fall off of the targets after coating and produce lower adhesion.

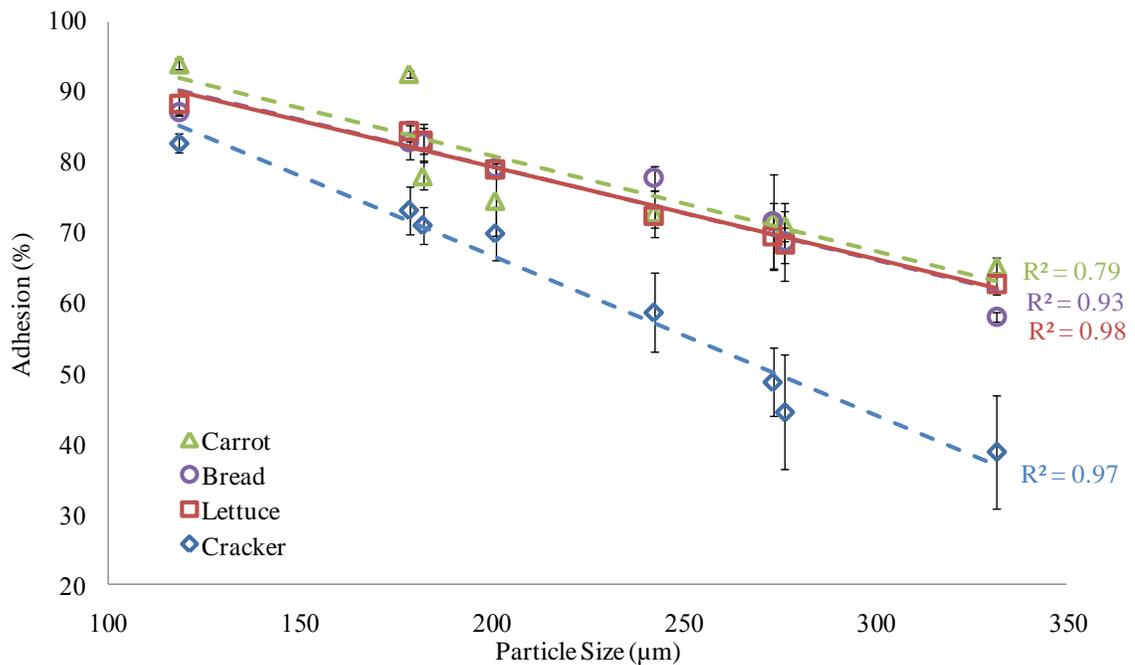


Figure 17. Adhesion (%) significantly decreased with increasing salt particle size for electrostatic coating on all food targets (n = 3, R^2 = correlation coefficient, error bars: standard deviation).

Transfer efficiency had the strongest correlation to particle size of all the powder and target properties studied. Others have also found that particle size has a dominant effect on nonelectrostatic and electrostatic transfer efficiency (Bailey 1998; Mazumder and others 1997; Ratanatriwong and Barringer 2007). Electrostatic transfer efficiency significantly decreased as particle size increased for all targets (Figure 18) and nonelectrostatic coating showed the same results. Because of aerodynamic forces, smaller food particles remain in the air as dust while larger particles fall on the target surface due to their high momentum and gravitational forces. Adhesion between salts and the target surface has a significant effect on the direction of the interaction between particle size and transfer efficiency. When bread was covered with oil to create strong adhesion, transfer efficiency increased with increasing salt particle size, as expected. When adhesion is strong or does not affect transfer efficiency, transfer efficiency increases with increasing particle size (Biehl and Barringer 2004; Mayr and Barringer 2006). However, in studies where adhesion is weak, transfer efficiency decreases with increasing particle size (Miller and Barringer 2002). Since adhesion decreases with particle size in this study (Figure 17), larger particles more easily fall off the target, resulting in lower transfer efficiency.

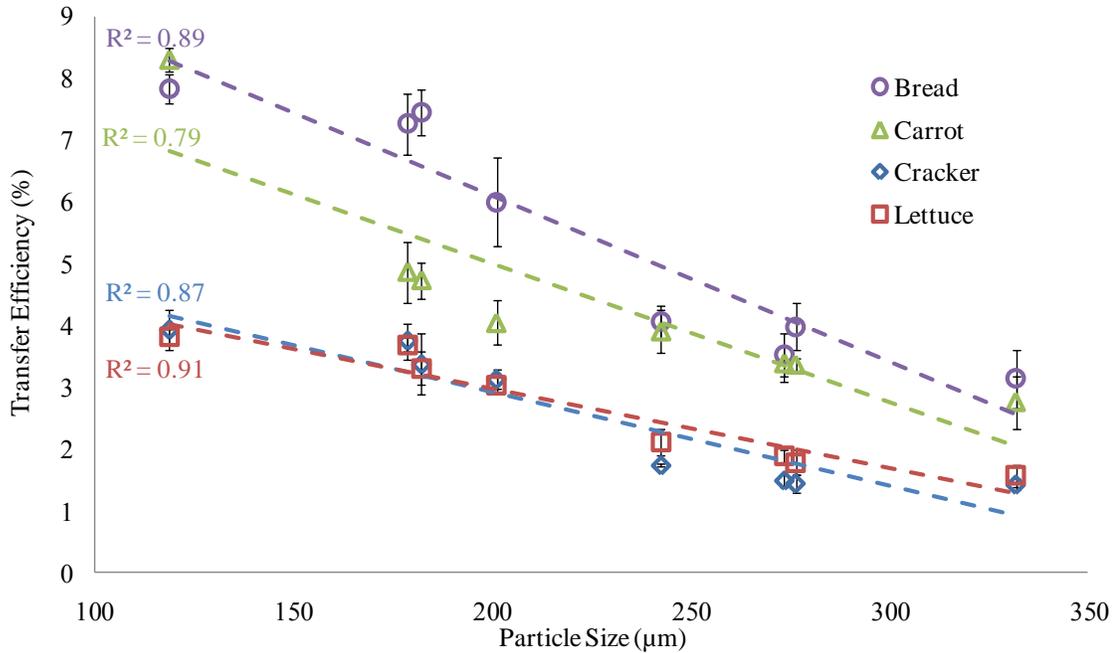


Figure 18. Transfer efficiency (%) significantly decreased with increasing salt particle size for electrostatic coating on all food targets ($n = 3$, $R^2 =$ correlation coefficient, error bars: standard deviation).

Percent side coverage significantly decreased as particle size increased on 3 of the 9 targets: wood, paper, and cracker in both nonelectrostatic and electrostatic coating (Figure 19), while the other targets had 100% side coverage for all but the largest size of salt. When aerodynamic forces dominate over gravitational force, the wrap around effect increases. Therefore, powders deposit on the sides of the target instead of on the conveyor belt. Small powders are affected more strongly by aerodynamic forces while large powders are more affected by gravitational force (Bailey 1998). Percent side coverage decreases when larger food powders are used (Sumonsiri and Barringer 2010).

With an increase in particle size, adhesion between particles and target surface, as well as transfer efficiency, decreases (Figure 17 and 18).

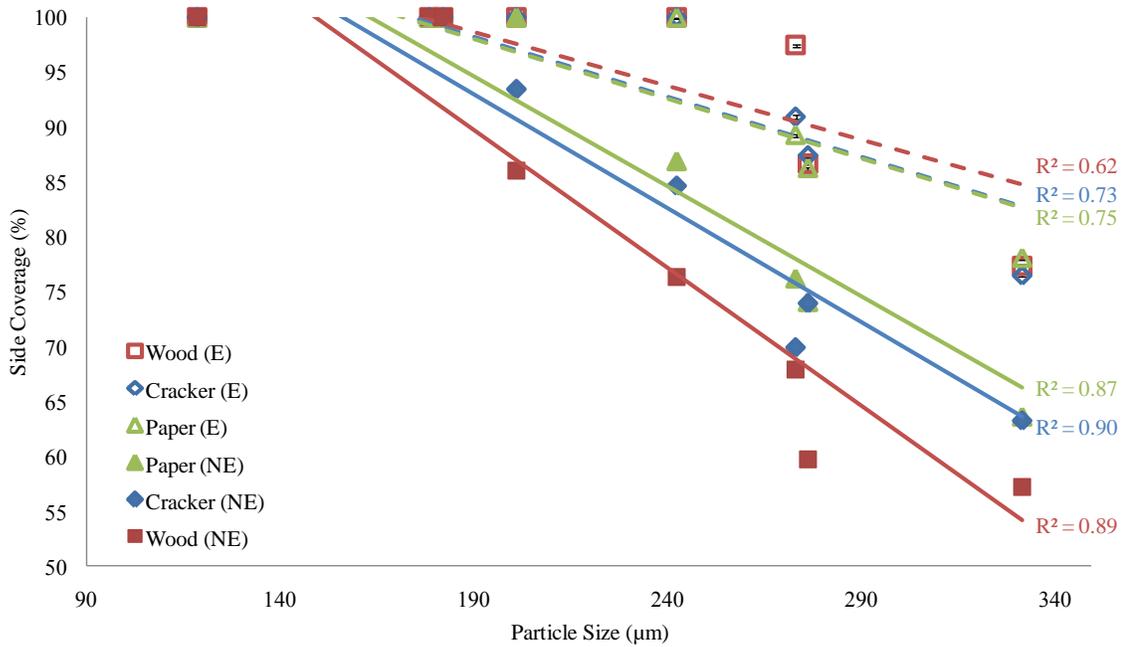


Figure 19. Side coverage (%) significantly decreased with increasing salt particle size for nonelectrostatic (NE) and electrostatic (E) coating on some targets ($n = 3$, $R^2 =$ correlation coefficient, error bars: standard deviation).

3.5.2 Effect of salt flowability

The flowability of powders was measured by Hausner ratio. Powders with Hausner ratios more than 1.4 are non-flowing, 1.25 - 1.4 are cohesive, and 1 - 1.25 are free-flowing (de Jong and others 1999). As cohesiveness of salts increased, electrostatic

adhesion significantly increased for all targets (Figure 20), and nonelectrostatic coating showed the same results. Cohesive powders provide better adhesion between powder particles and the target surface after coating (Sumawi and Barringer 2005). With increased cohesiveness, food powders adhere well on the target surface without falling off of the targets.

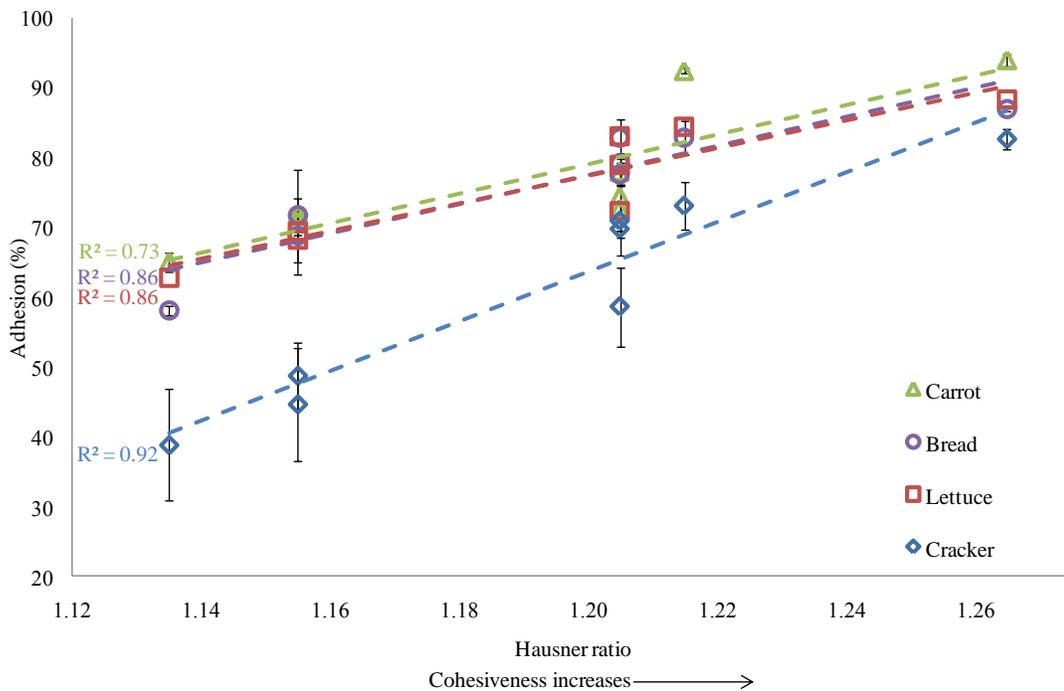


Figure 20. Adhesion (%) significantly increased with increasing salt Hausner ratio for electrostatic coating on all food targets ($n = 3$, $R^2 =$ correlation coefficient, error bars: standard deviation).

As the Hausner ratio increased (salts became more cohesive), transfer efficiency significantly increased for all targets in electrostatic (Figure 21) and nonelectrostatic coating. In contrast, previous studies reported that free-flowing powders disperse well across the target, resulting in better transfer efficiency (Ricks and others 2002; Ratanatriwong and Barringer 2003). The powders in those studies had Hausner ratio from 1.20 to 1.50, which are cohesive to non-flowing powders while all but one of the salts used in this study are free-flowing. Thus, all of these salts could disperse evenly across the target, eliminating that factor, but with a thick target, adhesion between salt particles and the target surface became more important to coating performance. As cohesiveness increases, adhesion increases between powders and the target surface (Figure 20); therefore, salts with higher Hausner ratio produce better transfer efficiency.

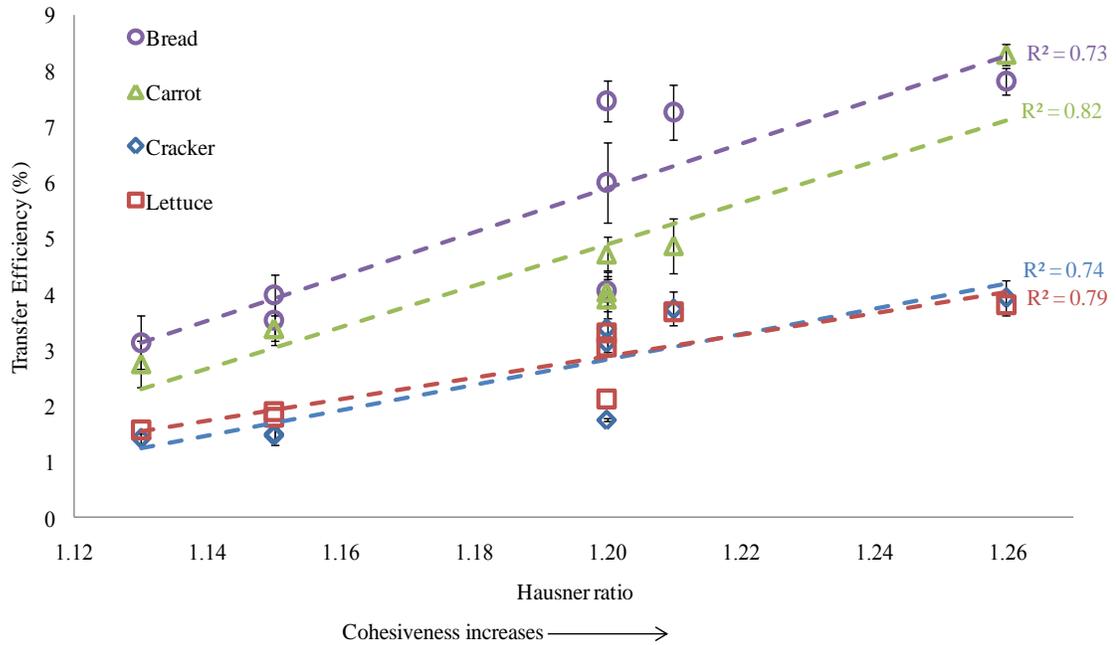


Figure 21. Transfer Efficiency (%) significantly increased with increasing salt Hausner ratio for electrostatic coating on all food targets ($n = 3$, $R^2 =$ correlation coefficient, error bars: standard deviation).

As the Hausner ratio increased, percent side coverage significantly increased on 3 and 2 targets in nonelectrostatic and electrostatic coating respectively while the other targets, except wood, had 100% side coverage for all but the largest salt (Figure 22). Nonelectrostatic and electrostatic percent side coverage increases as more cohesive food powders are used (Sumonsiri and Barringer 2010). More cohesive food powders have more adhesion to the target surface (Figure 20), so they are not likely to fall off of the side of the target easily, producing higher percent side coverage.

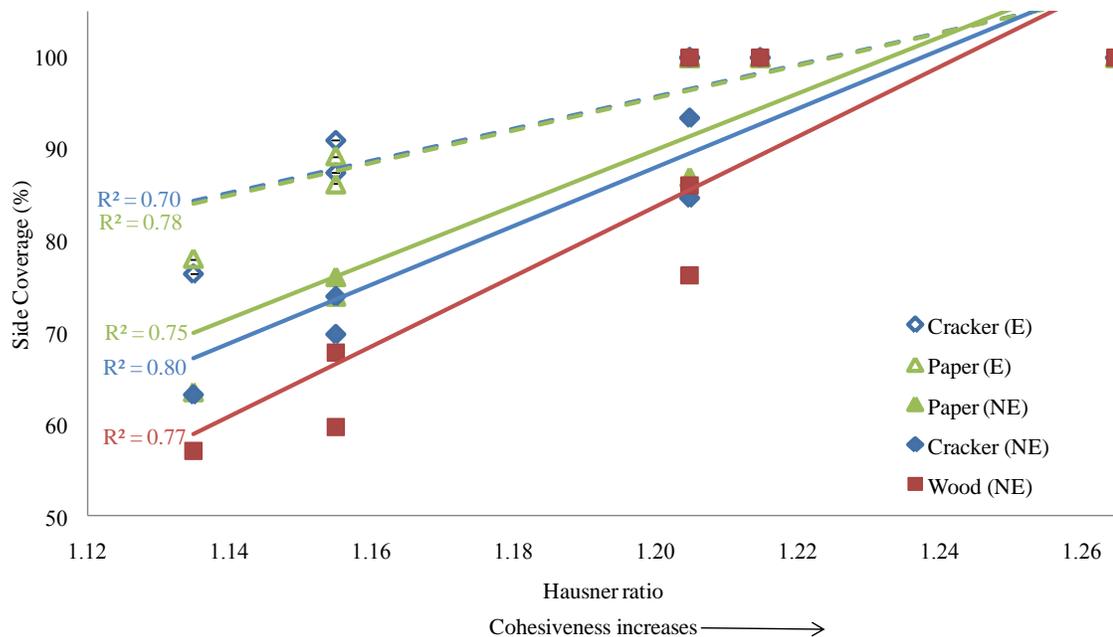


Figure 22. Side coverage (%) significantly increased with increasing salt Hausner ratio for nonelectrostatic (NE) and electrostatic (E) coating on some targets ($n = 3$, $R^2 =$ correlation coefficient, error bars: standard deviation).

3.5.3 Effect of target properties

In this study, many types of targets, including food and nonfood targets, were carefully chosen to provide a wide range of target resistivities (Table 4). Paper and wood targets were used to provide high resistivity targets, similar to dry bakery products. These targets were chosen instead of the specific food products because they could be shaped into the cubic forms to allow comparison between the targets while representing the desired resistivity. Vegetables were chosen to provide low resistivity targets, and have the

ability to be shaped into the cubic forms. The correlation found between targets with these properties and coating performance should be useful to predict coating performance in other food targets with similar properties.

Table 4. Resistivity of targets.

Target	Resistivity (Ωm)
Lettuce	5.97×10^8
Carrot	7.48×10^8
bread (a_w 0.9)	8.24×10^8
bread (a_w 0.7)	9.35×10^8
Cracker	2.98×10^9
bread (a_w 0.5)	5.39×10^9
Paper	1.06×10^{10}
bread (a_w 0.3)	2.34×10^{10}
Wood	3.59×10^{10}

Target properties did not have as great an effect on coating as powder properties did. As target resistivity increased, nonelectrostatic and electrostatic transfer efficiency significantly decreased for only 5 and 1 of the 8 salts respectively. By using only the data from bread at 4 water activities, there was only one more significant correlation for salt in nonelectrostatic coating (Figure 23). Resistivity should affect both adhesion and attraction of the powder to the target. With higher powder resistivity, adhesion between powders and the targets increases due to slow charge flow and slow charge decay during electrostatic coating (Sims and others 2000). A similar effect was expected for target

resistivity as is seen for powder resistivity. However, in this case, adhesion does not explain the effect of resistivity. Adhesion did not significantly correlate to resistivity, and for the different water activities of bread samples, the resistivity of only 2 of the smallest salts, 119 and 201 μm , significantly correlated with electrostatic adhesion. Resistivity has the opposite effect on attraction to the target. After depositing on a low resistivity target, charge on the powders quickly flows to the ground, resulting in more charged powders landing on the target. On targets with higher resistivity, some charge remains on the target surface repelling additional powder; resulting in lower attraction to the target (Sims and others 2000). Thus, decreasing attraction with increasing resistivity appears to be the dominant factor, though overall target resistivity only weakly influences transfer efficiency.

As the water activity of the bread increased, nonelectrostatic and electrostatic transfer efficiency significantly increased for 5 and 7 of the 8 salts respectively (Figure 24). When all targets were included, only one nonelectrostatically coated salt showed a significant correlation. Increasing water activity of the target significantly correlated to decreasing resistivity ($R^2=0.54$ to 0.99), but only correlated to increased adhesion for a few salts. Water activity produced a significant correlation to more salts than resistivity or adhesion, for transfer efficiency, probably because water activity decreases resistivity but increases adhesion.

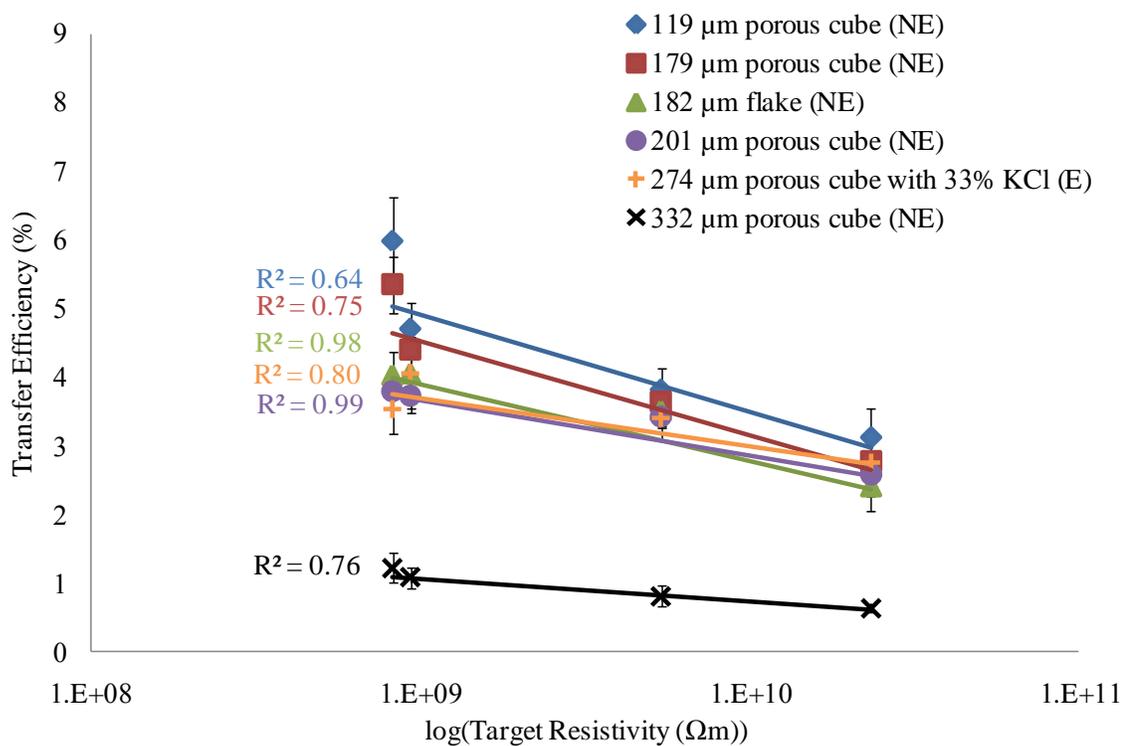


Figure 23. Transfer Efficiency (%) significantly decreased with increasing target resistivity for nonelectrostatic (NE) and electrostatic (E) coating of some salts on bread samples ($n = 3$, R^2 = correlation coefficient, error bars: standard deviation).

The charge decay time is the time required for the charge on the target surface to decrease to 10% of the original charge. Typically, targets with low resistivity have a short charge decay time (Bailey 1998, but no correlation was found in this study. The correlations between charge decay time and transfer efficiency were found only when the three large salts were electrostatically coated (Figure 25). As target charge decay time

increased, adhesion decreased for 4 of 8 salts in both nonelectrostatic and electrostatic coating (Figure 26). Shorter target charge decay time did significantly correlate with higher water activity. However, this is the opposite of what was found in a previous study (Halim and Barringer 2007). Given the weak and conflicting correlations, charge decay time does not appear to be a useful measurement.

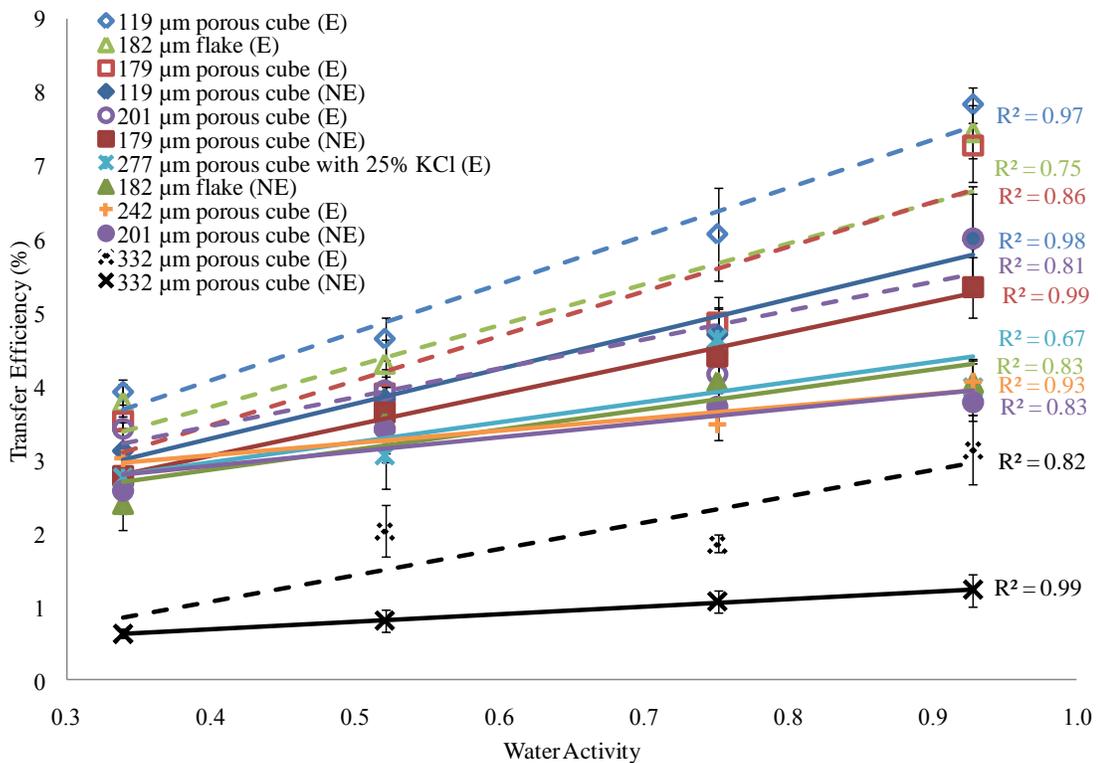


Figure 24. Transfer Efficiency (%) significantly increased with increasing bread water activity for nonelectrostatic (NE) and electrostatic (E) coating of many salts ($n = 3$, $R^2 =$ correlation coefficient, error bars: standard deviation).

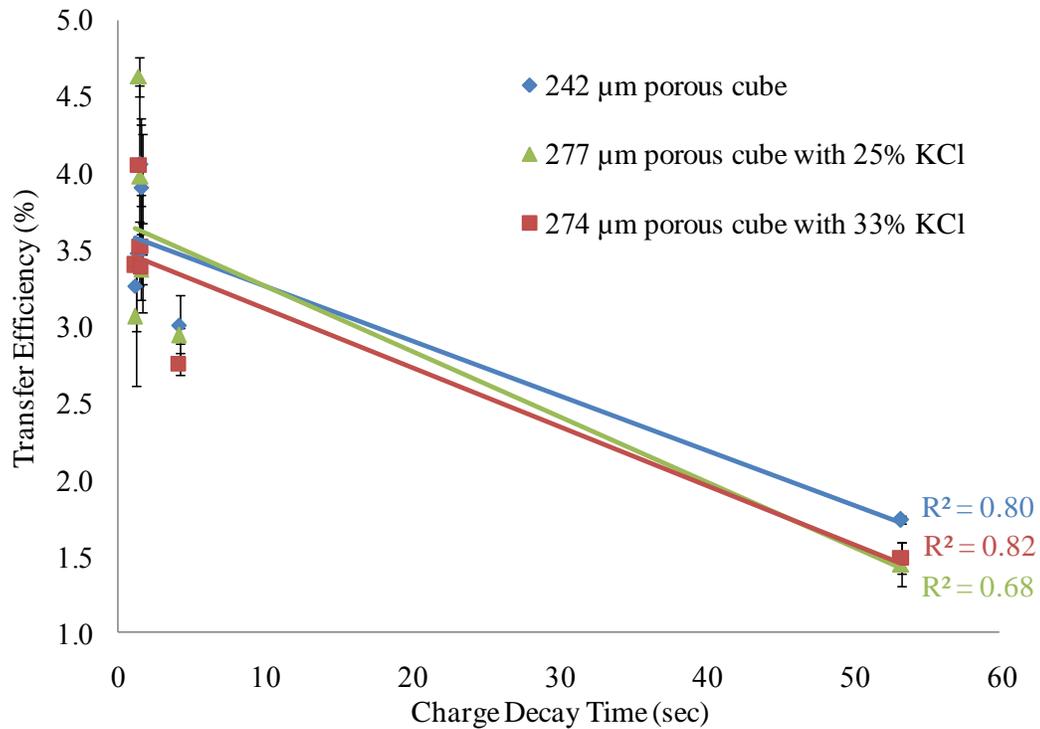


Figure 25. Transfer efficiency (%) significantly decreased with increasing target charge decay time for electrostatic coating of three large salts ($n = 3$, $R^2 =$ correlation coefficient, error bars: standard deviation).

Percent side coverage did not significantly correlate to water activity of the target, target resistivity, or target charge decay time for either nonelectrostatic or electrostatic coating.

In addition to water activity, resistivity, and charge decay time, target surface roughness and adhesiveness may cause differences in adhesion during powder coating since roughness indicates the actual contact area of the target surface and whether the powder particles are trapped in the cracks or pores of the targets (Katainen and others

2006; Halim and Barringer 2007). Carrots and lettuce had higher moisture content, resulting in a more adhesive surface, while crackers and bread had pores and cracks to trap salts inside. Therefore, these food targets may have produced better adhesion than wood and paper, which were smoother and had lower moisture content. However, further study is needed to understand how specific food target surfaces affect coating performance.

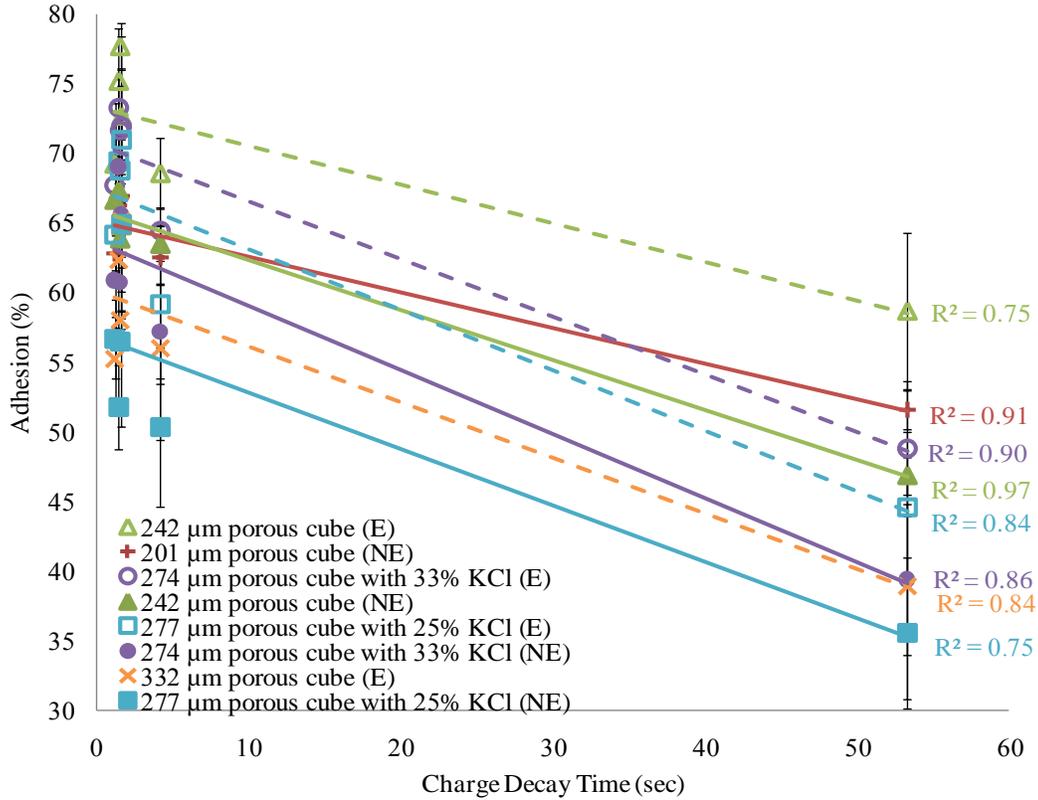


Figure 26. Adhesion (%) significantly decreased with increasing target charge decay time for nonelectrostatic (NE) and electrostatic (E) coating of some salts ($n = 3$, $R^2 =$ correlation coefficient, error bars: standard deviation).

3.5.4 Effect of salt shape and %KCl

Differences in salt shape (porous cube versus flake with similar particle size) affected transfer efficiency and adhesion on 2 and 4 of the 9 targets respectively (Table 5). When there was a significant difference between the coating performances of porous cube and flake salts, porous cube produced significantly higher transfer efficiency and adhesion than flake salt. Previous studies have found conflicting results, with either porous cubes (Buck and Barringer 2007) or flake salts (Miller and Barringer 2002) producing better electrostatic transfer efficiency and adhesion.

Percent KCl did not consistently affect transfer efficiency, adhesion, or side coverage.

3.5.5 Effect of electrostatic coating

For every type of salt and target, electrostatic coating produced the same or significantly higher percent transfer efficiency, adhesion, and side coverage as compared to nonelectrostatic coating. Figure 27 shows the improvement with electrostatic coating on percent transfer efficiency, adhesion, and side coverage for bread (a_w 0.3), but the other targets produced similar results. The charge produced by electrostatics produces better adhesion between food powders and the target surface according to Coulomb's law (Hughes 1997), resulting in improvement of adhesion and better percent side coverage. With electrostatics, charged powders are attracted to the target surface, instead of remaining in the air as dust (Ricks and others 2002; Biehl and Barringer 2003); therefore, producing better transfer efficiency. The greatest improvement in transfer efficiency, adhesion, and side coverage was observed when salts

with larger particle size and lower cohesiveness were coated on targets with lower water activity, higher resistivity, and longer charge decay time. These types of salts and targets produced poor nonelectrostatic coating performance, so there was more improvement with electrostatics.

Table 5. Average transfer efficiency (%) and adhesion (%) from nonelectrostatic and electrostatic coating of 182 μm flake salts and 179 μm porous cubic salts on each target.

Target	Nonelectrostatic coating				Electrostatic coating			
	Transfer Efficiency (%)		Adhesion (%)		Transfer Efficiency (%)		Adhesion (%)	
	Flake	Porous Cube	Flake	Porous Cube	Flake	Porous Cube	Flake	Porous Cube
Wood	0.92 ^A	0.95 ^A	47.90 ^A	57.86 ^A	1.38 ^A	1.50 ^A	66.46 ^A	71.34 ^A
Paper	0.59 ^A	0.96 ^A	45.62 ^A	47.94 ^A	1.12 ^B	1.50 ^A	63.15 ^A	64.15 ^A
Cracker	2.35 ^A	2.32 ^A	58.88 ^A	68.49 ^A	3.37 ^A	3.74 ^A	71.03 ^A	73.16 ^A
Lettuce	3.05 ^A	3.02 ^A	79.43 ^A	82.52 ^A	3.31 ^A	3.69 ^A	83.08 ^A	84.42 ^A
Carrot	3.43 ^A	3.78 ^A	68.15 ^B	88.13 ^A	4.73 ^A	4.87 ^A	78.08 ^B	92.55 ^A
Bread (a_w 0.3)	2.40 ^A	2.77 ^A	64.16 ^B	74.63 ^A	3.77 ^A	3.52 ^A	72.13 ^B	81.34 ^A
Bread (a_w 0.5)	3.51 ^A	3.65 ^A	67.24 ^A	73.80 ^A	4.31 ^A	3.91 ^A	74.56 ^A	83.45 ^A
Bread (a_w 0.7)	4.05 ^A	4.40 ^A	67.27 ^B	78.14 ^A	4.56 ^A	4.86 ^A	77.74 ^A	80.98 ^A
Bread (a_w 0.9)	4.04 ^B	5.34 ^A	65.75 ^B	74.90 ^A	7.46 ^A	7.27 ^A	82.94 ^A	82.88 ^A

^A and ^B indicate significant difference between flake salts and porous cubic salts ($n = 3$, $p = 0.05$).

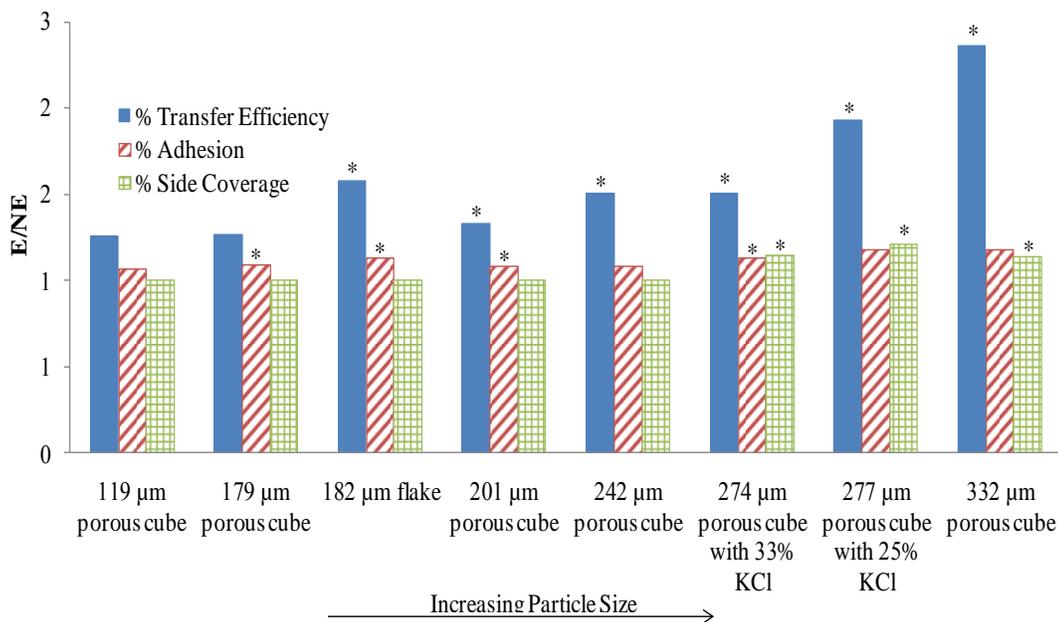


Figure 27. Improvement in transfer efficiency, adhesion, and side coverage with electrostatic (E) instead of nonelectrostatic (NE) coating (ratios of E/NE) of NaCl on bread (a_w 0.3). * indicates a significant difference between electrostatic and nonelectrostatic coating ($n = 3, p = 0.05$).

3.6 Conclusions

Smaller and more cohesive salts electrostatically coated on targets with higher water activity, lower resistivity, and shorter charge decay time produced better transfer efficiency, adhesion, and percent side coverage. Porous cube provided significantly better transfer efficiency and adhesion than flake salts on some targets. However, there was no significant effect of % KCl on coating performance. Electrostatic coating produced the

same or better transfer efficiency, adhesion, and percent side coverage as nonelectrostatic coating.

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CHAPTER 4: COMPARISON OF SOLID-LIQUID SEPARATION (SLS) AND VACUUM CONCENTRATION OF TOMATO JUICE

4.1 Abstract

Hot break, cold break and commercially canned tomato juice was concentrated by vacuum concentration and solid-liquid separation (SLS). Tomato products from vacuum concentration had higher soluble solids than those from SLS because of loss of soluble solids into the filtrate. Most volatile levels in vacuum concentration greatly decreased initially then remained constant during further concentration. In SLS, volatile levels linearly decreased with increasing concentration so SLS had greater retention of volatile compounds than vacuum concentration. Viscosity of the rediluted samples decreased with concentration, except in the hot break and commercial samples from SLS, which maintained the same viscosity. Samples from SLS were close to the original color while samples from vacuum concentration were redder due to heat induced Maillard browning. Vitamin C decreased during concentration with greater loss during SLS than vacuum concentration. SLS consumed 45 times less electric power energy than vacuum concentration, which also needed water for creating vacuum conditions.

Key words: tomato processing, tomato paste, concentration, evaporation, solid-liquid separation

4.2 Practical Applications

Solid-liquid separation (SLS) is a new technology for dewatering products, such as concentration of tomato juice. SLS does not need heat to concentrate the product; therefore, the processor may be able to reduce energy consumption while better preserving flavor and viscosity of the product.

4.3 Introduction

Tomatoes (*Lycopersicon esculentum*) are one of the most important vegetables for processing with a total value of \$926 million in the U.S. in 2010 and a U.S. consumption of approximately 67 lb processing tomatoes per capita in 2008 (Boriss and Brunke 2011). After harvesting, tomatoes are usually processed into tomato sauces, ketchup, pastes, and other products (Barringer 2004).

Total, soluble and insoluble solids, volatile compounds, viscosity, color, and vitamin C are important quality attributes that affect product quality and consumer acceptability of tomato products (Thakur and others 1996). Ripe tomatoes have an average of 94.5 % water (USDA 2011), which is a disadvantage during concentration. Half of the total solids are reducing sugars while the other half are citric, malic, and dicarboxylic amino acids, minerals, lipids, and alcohol-insoluble solids, such as pectic substances, cellulose, protein, and hemicelluloses (USDA 2011).

The volatile compounds in tomatoes determine the aroma, and together with the titratable acidity and total sugars, the taste of tomato products (Malundo and others 1995). There are 9 important compounds responsible for fresh tomato flavor: hexanal,

(*E*)-2-hexanal, (*Z*)-3-hexanal, (*Z*)-3-hexenol, (*E*)-2-(*E*)-4-decadienal, 2-isobutylthiazole, 6-methyl-5-hepten-2-one, 1-penten-3-one, and β -ionone (Petro-Turza 1986), while 11 compounds are the most important in the aroma of tomato paste: dimethyl sulfide, β -damascenone, β -ionone, 3-methylbutanal, 1-nitro-2-phenylethane, eugenol, methional, 3-methylbutyric acid, 6-methyl-5-hepten-2-one, phenylacetaldehyde, and linalool (Buttery and others 1990). Some volatile compounds, such as (*Z*)-3-hexenal, hexanal, 1-penten-3-one, and 2-phenylethanol, evaporate during heat processing, such as hot break, concentration, and pasteurization. Other compounds, such as pyrrolidone carboxylic acid and dimethyl sulfide, can be formed by the breakdown of carotenoids, amino acids, and sugars during thermal processing (Buttery and others 1990).

Viscosity, the resistance of fluid to the relative shear, is another important attribute in the quality of tomato products. Products with high quality have high viscosity (Thakur and others 1996). Factors affecting the viscosity of tomato products include tomato variety, solids content, particle size, and processing (Hayes and others 1998). During processing into juice, tomatoes can be put through either a cold break or hot break system, which greatly affect the viscosity of the final products. In the cold break method, a lower temperature of 60 – 66 °C is used to enhance the activity of pectolytic enzymes, which can degrade pectin in tomatoes into smaller molecules of acids and decrease viscosity of the juice, as well as to increase yield, and create flavor in the tomato juice (Madhavi and Salunkhe 1998). In hot break, chopped tomatoes are rapidly heated to at least 82 °C to inactivate the pectolytic enzymes in order to produce a product with high

viscosity. Most juice is processed by hot break systems since most juice is concentrated into paste and high viscosity in tomato paste is desired.

Color is important to the quality of tomato products since it is the first quality parameter observed by consumers and is presumed to predict other qualities such as flavor (Thakur and others 1996). Carotenoids, especially lycopene, create the color in tomato. They are more stable than other pigments; however, they can be degraded and lead to the loss of color (von Elbe and Schwartz 1996). The main cause of carotenoid degradation during processing is oxidation in the presence of light, oxygen, and lipoxygenase. During processing, a change in color is also caused by brown pigments in the products, which are formed due to Maillard reactions (Thakur and others 1996).

Tomatoes are a good source of vitamin C, which provide 20% of the recommended daily allowance of vitamin C per serving (90 g of fresh tomatoes) (USDA 2009). Vitamin C is very sensitive to changes in air, light, pH, and heat. During processing to paste, vitamin C content in tomatoes decreases to approximately 50% of its original value. The main processing steps responsible for the loss of vitamin C are hot break and pasteurization (Capanoglu and others 2008).

The traditional concentration of tomato juice occurs in three- or four-effect vacuum evaporators with forced circulation. At each successive effect, the juice is heated to 48 – 82 °C by using vapor collected from later effects to conserve energy. During concentration, color and flavor are preserved by the reduced pressure, which lowers the concentration temperature (Barringer 2004; Smith 2011).

The solid-liquid separation (SLS) system is a new innovative technology to dewater products with low energy consumption. This equipment uses different belts, including dewatering and membrane belts, to separate liquids from solids by adhesion and cohesion. It is claimed to be capable of separating 18 – 45% solids from dilute liquids without forced drying or heating (Coho and Youngs 2010); therefore the SLS is expected to concentrate tomato product with better energy conservation and product quality preservation. The objectives of this study are to compare the quality of commercially canned, hot break, and cold break tomato juice concentrated to different total solids by vacuum evaporation and SLS.

4.4 Materials and Methods

Three types of tomato juice were used in the study: commercially canned hot break tomato juice (Hirzel Canning Co., Toledo, O.H., U.S.A.), hot break tomato juice, and cold break tomato juice. Hot break and cold break tomato juice was produced from the same batch of fresh tomatoes harvested in September 2011. After harvesting, tomatoes were washed, sorted, chopped, put through a break system, and extracted into juice. Chopped tomatoes were rapidly heated at 88 °C and 60 °C in hot break and cold break systems respectively before the extraction. For vacuum concentration, the tomato juice was evaporated using a rotary evaporator (Rotavapor R110, Buchi, Brinkmann Instruments, Inc., Subsidiary of Subron Corp., Westburg, N.Y., U.S.A) at 65 °C, 1.86×10^5 Pa for 85 – 190 min to obtain samples with total solids of approximately 11, 15, 20, 25, 30, and 35%. For concentration by solid-liquid separation (Algaeventure System,

Marysville, O.H., U.S.A.) (Figure 28), the tomato juice was concentrated at different speeds of sample input (5.7 – 20.5 g/s) and dewatering belt (12.4 – 18.6 cm/s) to obtain samples with total solids of approximately 8, 9, 10, and 11%. The speeds of membrane belt and concentrate removal were 0.51 and 25.4 cm/s respectively. Three replicates from each concentration, as well as the filtrates from the SLS, were tested for total solids content, soluble solids, volatile concentration, viscosity, color, and vitamin C within a day after concentration. All samples were diluted to approximately 5% soluble solids before testing, except when testing for total solids, soluble solids, and vitamin C, which were measured directly.

The total solids content was determined by vacuum oven. The sample (approximately 5 g) was weighed on the weighing dish (low form aluminum, Fisher Scientific, China) and placed in a vacuum oven (Precision Vacuum Oven Model 29, Winchester, V.A., U.S.A.) at 70 °C, 1.03×10^4 Pa for 24 h. The dried sample was then cooled in desiccators with desiccant before recording the weight. The total solids content was calculated by the following equation: $\% \text{ Total solids} = \left(\frac{wt_{after} - wt_{dish}}{wt_{before} - wt_{dish}} \right) \times 100$; where wt_{dish} = weight of the weighing dish, wt_{before} = weight of sample and dish before drying, and wt_{after} = weight of sample and dish after drying.

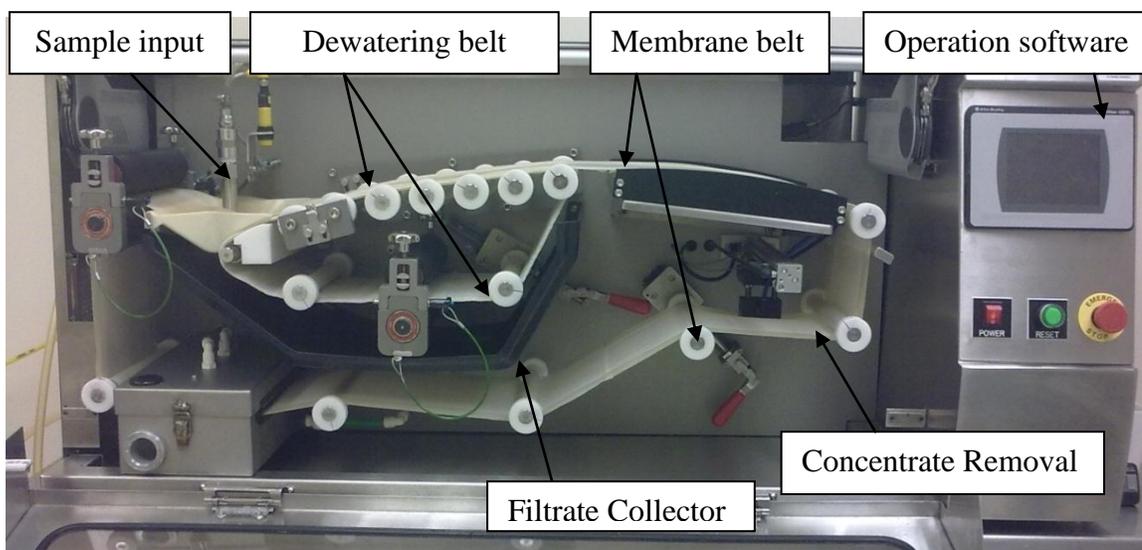


Figure 28. Solid-liquid separation system used in the concentration of tomato juice.

The soluble solids of the sample was measured by a refractometer (Mark II, Model 10480 S/N, Reichert Scientific Instruments, Division of Warner-Lambert Technologies, Inc., Buffalo, N.Y., U.S.A.) at room temperature. Samples were centrifuged at 1000 rpm for 10 min and the supernatant was collected and used for the measurement. Insoluble solids (%) was calculated by subtracting soluble solids from the total solids.

The volatile compounds of interest in the headspace of the sample were analyzed by a selected ion flow tube-mass spectrometer (SIFT-MS) (VOICE 100, Syft Technologies Ltd, Christchurch, New Zealand) instrument and Syft VOICE-100 software (v.1.4.9.17754, Syft Technologies Ltd, Christchurch, New Zealand) using the method developed by Xu and Barringer (2010). 100 g of sample was transferred to a 500 ml glass

media bottle with screw top caps and polytetrafluoroethylene-faced silicone septa (Pyrex 1395-45TS, Corning, N.Y., U.S.A.), and placed in a 50°C water bath for 60 min prior to the analysis in order to achieve equilibrium of headspace volatiles. During the analysis, an 18-gauge (3.8 cm) passivated collection needle connected to the SIFT-MS was used to collect volatiles in the headspace. A 14-gauge (15 cm) needle was used to create circulation. The short needle pierced the center of the septum and was placed at 16 cm above the bottom of the bottle. The long needle was placed at 7 cm under the cap. SIFT-MS quantifies volatile concentration using selected ion mode (SIM) based on known kinetic parameters. The concentrations of the volatiles are calculated by using the rate coefficients, k , of the volatile with the precursor ion, and the known ratios of product to precursor ion count rates (Španěl and Smith 1999). The instrument was calibrated using a given concentration of benzene, toluene, ethylbenzene, and xylene. The parameters of the SIFT-MS were set as following: sample scan time 180 sec, heated inlet temperature 120 °C, carrier gas argon pressure 2.00×10^5 Pa, helium pressure 2.07×10^5 Pa, inlet flow rate $120 \text{ cm}^3/\text{min}$, and flow tube vacuum pressure 5.07 ± 0.40 Pa.

The color of the sample was measured by a colorimeter (Minolta Chroma meter CR-300 Series and data processor DR-301, Minolta, Japan) at room temperature. 20 g of sample was weighed on a glass petri dish for the measurement. The top of the sample surface was touched by the measuring probe and CIELAB values of L^* (lightness), a^* (redness) and b^* (yellowness) were measured. Hue angle (h°) was calculated by the following equation: $h^\circ = \tan^{-1} \frac{b^*}{a^*}$.

Viscosity was measured from 300 ml of sample in a 600 ml beaker using a rotatory viscometer (Brookfield model DV-II+), LV spindle no. 3, and LV guard leg (Brookfield Engineering Labs, Inc., Stoughton, M.A., U.S.A.) at 60 rpm at room temperature. LV guard leg was also used to enhance an outer boundary of the shear action produced by the spindle in order to obtain more accurate and reliable results.

The vitamin C in the sample was determined using 2,6-dichloroindophenol titration (adapted from AOAC Method 967.21, 45.1.14). A blank solution was prepared by mixing 2 ml of deionized H₂O and 5 ml of metaphosphoric acid-acetic acid extracting solution (15 g HPO₃ and 40 ml HOAc in 500 ml deionized H₂O). Ascorbic acid standard solution was prepared by mixing 2 ml aliquots of ascorbic acid standard solution (50 mg of L-ascorbic acid in 50 ml HPO₃-HOAc extracting solution) and 5 ml of HPO₃-HOAc extracting solution. Sample solution was prepared by mixing 20 ml of HPO₃-HOAc extracting solution into 20 ml of sample and filtering by filter paper no. 3 (Whatman filter paper, Whatman International Ltd., Maidstone, England). 5 ml of filtered sample solution was transferred into a 50 ml Erlenmeyer flask for the titration. Within 5 min after sample preparation, each sample was titrated with 2,6-dichloroindophenol solution (prepared by dissolving 50 mg of 2,6-dichloroindophenol sodium salt in NaHCO₃ solution (42 mg of NaHCO₃ in 50 ml deionized H₂O) and diluting the solution to 2000 ml using deionized H₂O) until it turn a light rose pink color for blank and standard solutions or a light orange color for sample solution. The titration of ascorbic acid standard solution was done to determine the concentration of the indophenols solution as mg ascorbic acid equivalents to 1.0 ml of reagent. The ascorbic acid content was calculated by the following equation:

mg ascorbic acid / g of total solid = $[(S-B) \times (F/E) \times (V/X)] / (T/100)$; where S= average ml for sample titration, B = average ml for blank titration, F = ascorbic acid mg equivalent to 1.0 ml 2,6-dichloroindophenol solution= 2 mg/average ml titrated for ascorbic acid, E = number of ml assayed = 5 ml, V = initial assay solution volume = 25 ml, X = volume of sample aliquot titrated = 50 ml, and T = % total solid of sample. All chemicals used in vitamin C determination were from Sigma-Aldrich Co. (St. Louis, M.O., U.S.A.).

Electric power consumption during concentration was measured by using an electricity consumption meter (Watts Up? Pro, Electronic Educational Devices, Denver, C.O., U.S.A.). The electric power consumption (watt-hours/g removed water) was calculated by the following equation: energy consumption (watt-hours/g removed water) = $[(\text{watts reading} \times \text{concentration time (h)}) / \text{removed water (g)}]$. During concentration by vacuum, water consumption was also calculated by the following equation: water consumption (m^3) = $[\text{water flow rate} \times \text{concentration time (sec)}] / 10^6$, where water flow rate = $95.7 \text{ cm}^3/\text{s}$.

One-way analysis of variance with Tukey's HSD (Honestly Significant Difference) test was used for statistical analysis to determine if there was a significant difference between the quality of samples from vacuum concentration and SLS, and between quality of commercial, hot break, and cold break samples, as well as to determine significant changes in quality of the samples during concentration. A p-value of 0.05 or lower indicates a significant difference between variables.

4.5 Results and Discussion

4.5.1 Total, soluble, and insoluble solids

Tomato products from vacuum concentration had significantly higher soluble solids than those from SLS at the same total solids (Figure 29) since some soluble solids were lost into the filtrate during concentration by SLS (Table 6). In the samples from SLS, the filtrates and concentrates had similar percent soluble solids. Most of the insoluble solids were retained in the concentrate for both SLS and vacuum concentration; therefore, insoluble solids were chosen as the basis on which to compare the samples in this study for other quality attributes.

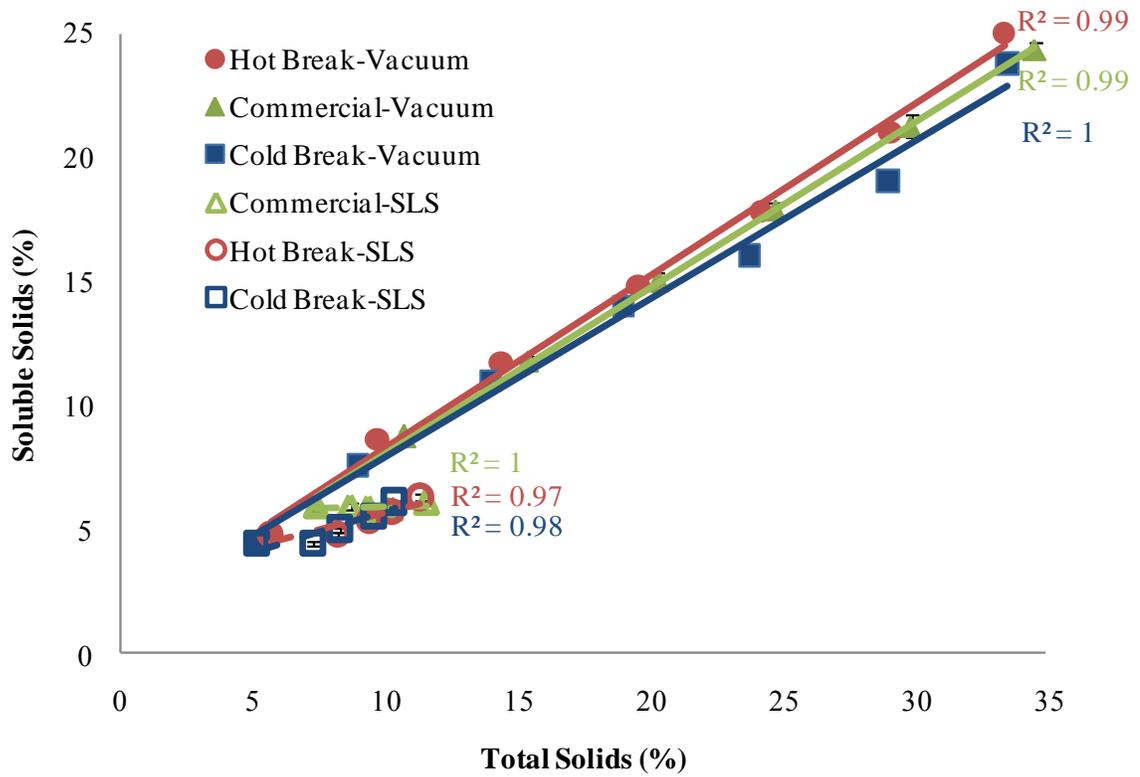


Figure 29. Soluble solids of tomato products at different total solids from vacuum concentration and solid-liquid separation (SLS).

Table 6. Quality of initial samples, concentrate and filtrate from the SLS system, and concentrate from vacuum concentration at approximately 10% total solids in the concentrate.

Sample		Total Solids (%)	Soluble Solids (%)	(Z)-3-hexenal (ppm)	Viscosity (Pa.s)	Hue Angle (°)	Ascorbic acid (mg/g of solids)
Commercial	Initial	7.42	6.52	179	0.36	1.93	1.93
	SLS Concentrate*	10.24	5.83	2	0.36	0.94	0.94
	SLS Filtrate	5.17	4.92	1	0.39	1.86	1.86
	Vacuum Concentrate*	10.77	8.72	31	0.34	1.87	1.87
Hot Break	Initial	5.72	4.76	221	0.36	2.30	2.30
	SLS Concentrate*	10.30	5.69	76	0.37	1.04	1.04
	SLS Filtrate	5.05	4.78	192	0.42	1.72	1.72
	Vacuum Concentrate*	9.76	8.60	21	0.36	1.98	1.98
Cold Break	Initial	5.21	4.39	241	0.37	2.34	2.34
	SLS Concentrate*	9.63	5.51	68	0.37	1.07	1.07
	SLS Filtrate	4.35	4.29	183	0.39	1.63	1.63
	Vacuum Concentrate*	9.05	7.55	22	0.35	2.00	2.00

*Concentrates were diluted to original solids for volatile, viscosity, and color tests.

4.5.2 Volatile Compounds

Most volatile levels decreased greatly during the first concentration step then remained almost constant during further concentration in vacuum concentration. In SLS, volatile levels linearly decreased with increasing concentration. (*Z*)-3-Hexenal is representative of the response of most of the volatile compounds in the study and is one of the important compounds in the flavor of fresh tomatoes (Figure 30) (Thakur and others 1996). Heat and vacuum cause the destruction and evaporation of volatile compounds (Hayes and others 1998). Although SLS preserved some volatile compounds in the samples, there were still some volatile compounds lost through the filtrate (Table 6). SLS concentration of cold break and hot break samples retained significantly higher concentrations of most volatiles than those from vacuum concentration (Table 7). In commercial samples, which started with significantly lower levels for many volatiles, SLS had greater retention than vacuum concentration initially, but at higher concentration, there was no significant difference between vacuum and SLS. Yildiz and others (1993) found that tomato paste concentrated by evaporation had lower sensory scores for fresh-like characteristic than those from reverse osmosis membrane concentration, which is what we would expect from these volatile results.

The initial levels of volatile concentrations were different between the hot and cold break samples because of enzyme activity (Table 7). In cold break samples, enzymes of the lipoxygenase pathway are active and produce important volatiles including (*Z*)-2-hexenal, (*E*)-2-hexenal, hexanal, (*Z*)-3-hexen-1-ol, and 6-methyl-5-hepten-2-one (Madhavi and Salunkhe 1998; Boukobza and Taylor 2002). In commercial and hot break

samples, enzymes were inactivated by the heat treatment and so these volatiles are at lower levels. Some volatile compounds, such as acetone, ethanol, and dimethyl sulfide, had highest initial concentrations in the commercial samples. These volatiles are generated during heat treatment. Initial levels affected significant differences, but all of the volatiles were lost following the same pattern.

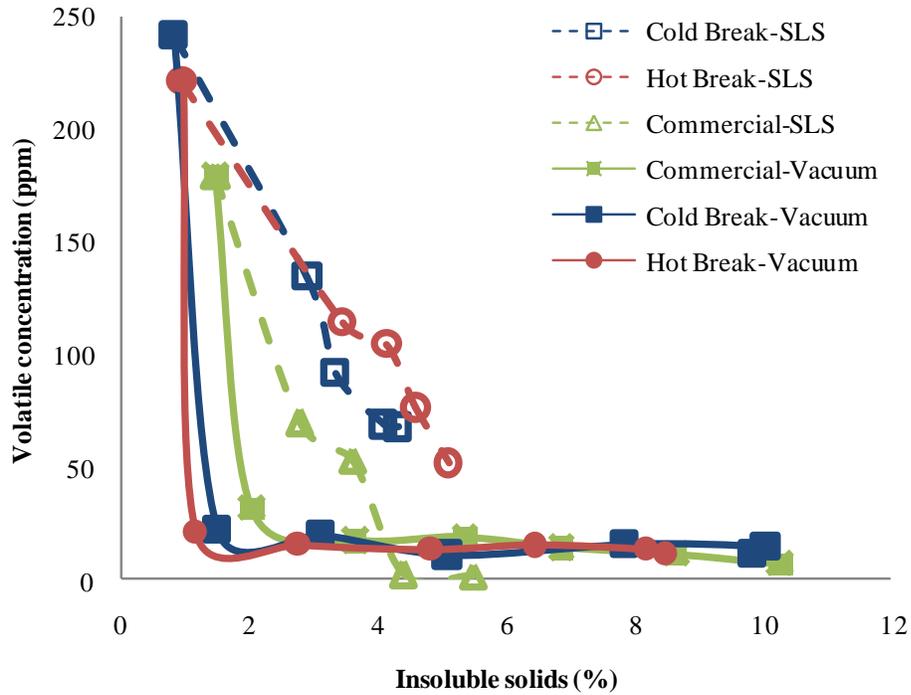


Figure 30. (Z)-3-Hexenal in tomato products concentrated to different insoluble solids by vacuum concentration and SLS then diluted to 5% soluble solids for measurement.

Table 7. Concentration of analyzed volatiles in initial samples and concentrates at approximately 3.5% insoluble solids.

Volatile compounds	Concentration (ppm)								
	Cold break			Hot break			Commercial		
	Initial	SLS	Vacuum	Initial	SLS	Vacuum	Initial	SLS	Vacuum
(<i>E</i>)-2-heptenal	18 ^B	22 ^A	3 ^D	8 ^C	6 ^{CD}	2 ^D	7 ^C	4 ^{CD}	2 ^D
(<i>E</i>)-2-hexenal	246 ^A	110 ^C	37 ^D	193 ^B	93 ^{BC}	29 ^D	177 ^B	63 ^{BD}	19 ^D
(<i>E</i>)-2-octenal	13 ^B	24 ^A	2 ^C	5 ^C	4 ^C	2 ^C	10 ^B	3 ^C	0 ^C
(<i>E</i>)-2-pentenal	88 ^A	39 ^C	5 ^F	48 ^B	20 ^E	3 ^F	30 ^D	8 ^F	1 ^F
(<i>E,E</i>)-2,4-decadienal	13 ^B	22 ^A	1 ^D	6 ^C	8 ^C	1 ^D	5 ^C	1 ^D	0 ^D
(<i>Z</i>)-3-hexenal	241 ^A	91 ^C	20 ^D	221 ^A	105 ^C	15 ^D	179 ^B	53 ^D	17 ^D
1-butanol	84 ^{AB}	88 ^A	83 ^{AB}	72 ^{AB}	85 ^A	94 ^A	74 ^{AB}	86 ^A	67 ^B
1-hexanol	28 ^A	11 ^B	4 ^{CD}	7 ^C	4 ^{CD}	1 ^{DE}	4 ^{CD}	1 ^{DE}	0 ^E
1-octen-3-ol	69 ^A	21 ^{CD}	4 ^D	37 ^{BD}	17 ^{CD}	2 ^{CD}	46 ^B	17 ^{CD}	6 ^{CD}
1-penten-3-one	67 ^A	55 ^B	2 ^E	12 ^C	7 ^D	2 ^E	6 ^{DE}	2 ^E	2 ^E
1-propanol	49 ^B	20 ^{BC}	2 ^D	33 ^{BC}	14 ^{CD}	1 ^D	112 ^A	33 ^{BC}	26 ^C
2,3-butanediol	24 ^{BC}	11 ^{CD}	9 ^D	28 ^B	13 ^{CD}	3 ^D	51 ^A	14 ^{CD}	5 ^D
2,3-butanedione	58 ^A	28 ^B	7 ^D	30 ^B	17 ^C	4 ^D	27 ^B	10 ^{CD}	8 ^{CD}
2-isobutylthiazole	18 ^B	11 ^{BC}	0 ^D	15 ^B	6 ^{CD}	0 ^D	33 ^A	6 ^{CD}	0 ^D
2-methylpropanal	201 ^A	201 ^A	74 ^C	177 ^{AB}	131 ^{BD}	66 ^D	75 ^{BD}	84 ^{BD}	52 ^D
2-pentanol	23 ^A	11 ^C	4 ^{DE}	18 ^B	8 ^{CD}	2 ^E	17 ^B	5 ^{CE}	2 ^E
2-pentanone	37 ^A	12 ^B	4 ^D	10 ^{BC}	6 ^{CD}	1 ^D	6 ^{CD}	4 ^D	1 ^D
2-pentylfuran	5 ^{BC}	16 ^A	2 ^C	3 ^{BC}	5 ^{BC}	1 ^C	9 ^B	1 ^C	0 ^C
6-methyl-5-hepten-2-one	266 ^A	123 ^C	13 ^D	133 ^C	59 ^D	17 ^D	198 ^B	35 ^D	15 ^D
acetaldehyde	1090 ^A	429 ^C	93 ^{DE}	917 ^B	394 ^{CD}	58 ^E	502 ^C	186 ^D	93 ^E
acetone	510 ^C	226 ^{DE}	441 ^C	793 ^B	344 ^{DC}	92 ^E	1757 ^A	541 ^C	207 ^{DE}

Table 7 Continued

benzaldehyde	53 ^A	35 ^B	6 ^D	52 ^A	21 ^{CE}	8 ^{CF}	31 ^{BC}	9 ^{DE}	4 ^F
benzene ethanol	9 ^{AB}	6 ^{BC}	3 ^C	8 ^{BC}	6 ^{BC}	4 ^C	10 ^{AB}	5 ^C	3 ^C
benzyl alcohol	55 ^B	83 ^A	13 ^{CD}	36 ^{BC}	39 ^{BE}	7 ^D	103 ^A	14 ^{DE}	4 ^D
citral	52 ^B	85 ^A	4 ^{DE}	23 ^C	29 ^C	5 ^{DE}	20 ^{CD}	4 ^{DE}	1 ^E
cyclic terpenes	12 ^B	19 ^A	3 ^C	6 ^{BC}	6 ^{BC}	3 ^C	12 ^B	4 ^C	1 ^C
dimethyl disulfide	445 ^A	82 ^C	11 ^D	318 ^B	77 ^C	6 ^D	118 ^C	27 ^D	4 ^D
dimethyl sulfide	738 ^B	390 ^C	86 ^C	994 ^B	246 ^C	58 ^C	9240 ^A	1427 ^B	41 ^C
ethanol	627 ^B	341 ^{BC}	66 ^D	492 ^{CD}	294 ^{CD}	37 ^D	3180 ^A	1270 ^A	69 ^D
ethyl acetate	74 ^A	26 ^C	5 ^E	46 ^B	18 ^{CD}	3 ^E	72 ^A	17 ^D	3 ^E
furfural	25 ^A	15 ^B	2 ^C	14 ^B	5 ^C	1 ^C	25 ^A	5 ^C	1 ^C
guaiacol	5 ^B	3 ^B	1 ^B	5 ^B	4 ^B	2 ^B	90 ^A	3 ^B	1 ^B
hexanal	537 ^A	262 ^C	60 ^{EF}	385 ^B	151 ^D	22 ^{FG}	72 ^E	44 ^{FG}	8 ^G
hexen-1-ol	91 ^A	60 ^B	11 ^D	42 ^C	31 ^C	6 ^D	46 ^{BC}	13 ^D	3 ^D
hexenal	1100 ^A	720 ^B	69 ^D	618 ^B	387 ^C	73 ^D	7 ^D	4 ^D	1 ^D
isobutanal	201 ^A	201 ^A	74 ^D	177 ^{AB}	131 ^{BC}	66 ^D	75 ^{CD}	84 ^{CD}	52 ^D
isobutyl alcohol	77 ^{ABC}	81 ^{AB}	76 ^{ABC}	66 ^B	78 ^{AB}	86 ^A	68 ^B	79 ^{AB}	61 ^{ABC}
methanol	133900 ^A	45833 ^C	2407 ^E	86133 ^B	36833 ^C	2907 ^E	45100 ^C	19067 ^D	220 ^E
methional	81 ^A	29 ^C	3 ^{DE}	44 ^B	15 ^D	3 ^{DE}	14 ^D	6 ^{DE}	1 ^E
methyl benzoate	165 ^A	78 ^B	6 ^{DE}	66 ^B	26 ^D	3 ^{DE}	39 ^C	12 ^{DE}	0 ^E
methyl salicylate	50 ^A	29 ^B	2 ^D	22 ^{BC}	21 ^{BC}	1 ^D	14 ^{CD}	5 ^D	0 ^D
methylbutanal	141 ^A	85 ^B	12 ^D	86 ^B	43 ^C	7 ^D	49 ^C	18 ^D	7 ^D
methylbutanoic acid	19 ^A	5 ^C	2 ^C	13 ^B	4 ^C	2 ^C	18 ^{AB}	5 ^C	1 ^C
nonanal	68 ^A	62 ^{AB}	12 ^D	40 ^{BC}	31 ^{CD}	11 ^D	30 ^{CD}	20 ^{CD}	13 ^D
octanal	12 ^{BC}	23 ^A	6 ^{BC}	11 ^{BC}	11 ^{BC}	8 ^C	21 ^{AB}	10 ^{BC}	2 ^{BC}
phenylacetaldehyde	20 ^A	12 ^{BCD}	7 ^{CD}	13 ^{BC}	7 ^{CD}	6 ^D	18 ^{AB}	5 ^D	14 ^{ABC}

Table 7 Continued

propanal	62 ^A	34 ^B	9 ^{EF}	31 ^B	17 ^D	6 ^{EF}	24 ^C	12 ^{DE}	5 ^F
propanoic acid	81 ^A	64 ^{AB}	24 ^C	65 ^A	54 ^{BD}	35 ^{CD}	40 ^{CD}	40 ^{CD}	21 ^C

A-G indicate significant difference of volatile concentration between the samples on the same row.

4.5.4 Viscosity

Viscosity significantly decreased with concentration when samples were diluted back to their original concentration, except in the hot break and commercial sample from SLS (Figure 31). During vacuum concentration, the heat treatment decreases the size of pectin due to the breakdown of pectin polymers (Ciruelos and others 2001; Cámara and others 2002). The hot break sample concentrated by SLS showed only a small decline in viscosity since less destruction of pectin structure occurred because no heat was applied in SLS. Hot break samples initially had significantly higher viscosity than the cold break samples since pectolytic enzymes, such as polygalacturonase and pectin methylesterase, were inactivated (Figure 31). In the cold break samples, pectin was degraded by the pectolytic enzymes. Cold break samples, therefore, had lower initial viscosity than hot break samples. In the commercial samples, the disruption of structure due to severe heat during canning or the use of different tomato varieties resulted in the lower initial viscosity. In the vacuum concentration and hot break samples, the viscosity of samples initially decreased greatly and then there was no significant change in viscosity after concentrating to approximately 2 – 3 % insoluble solids.

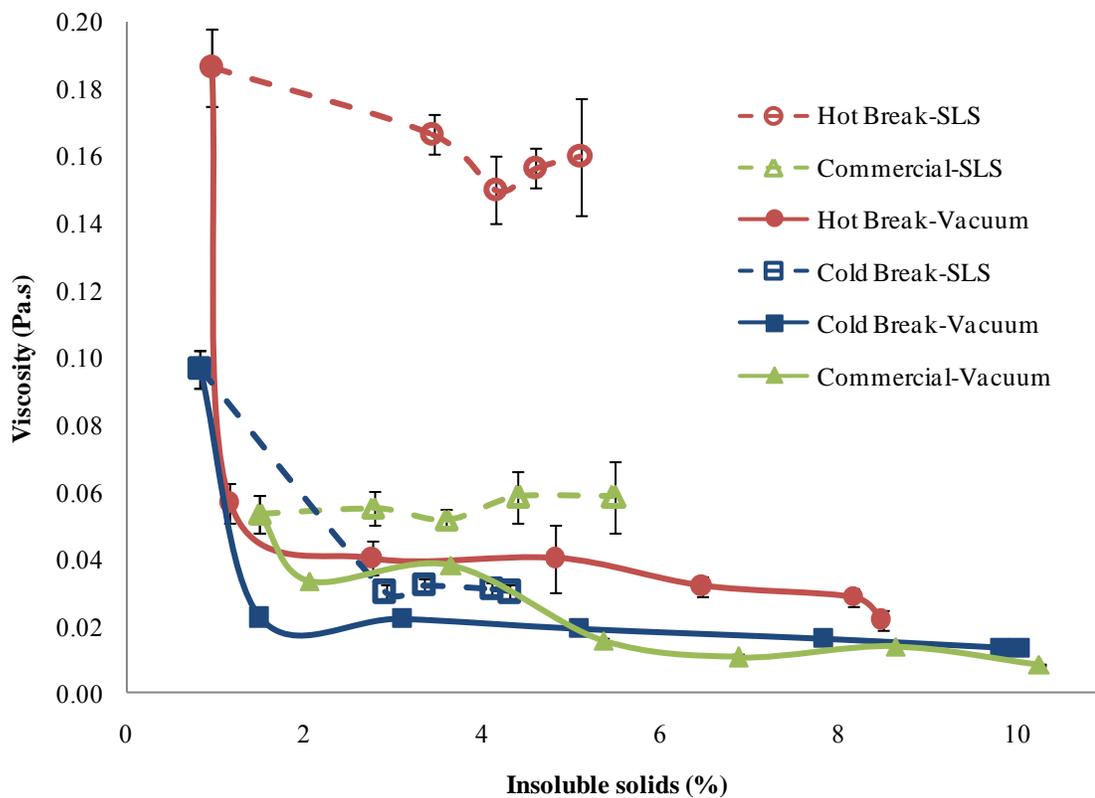


Figure 31. Viscosity of tomato products concentrated to different insoluble solids by vacuum concentration and SLS then diluted to 5% soluble solids for measurement.

4.5.5 Color

The color of tomato products can be used to indicate the stability and retention of carotenoids (Yildiz and others 1993). Hue angle in the final samples decreased as the insoluble solids increased during concentration in vacuum concentration, except the color of commercial samples (Figure 32). The color change from vacuum concentration is likely caused by browning due to the Maillard reaction and degradation of ascorbic acid,

which occurs during thermal treatments (Mudahar and others 1986; Eichner and others 1996). The color of SLS samples did not significantly change (Figure 32); however, some color was lost through the filtrate during concentration (Table 6).

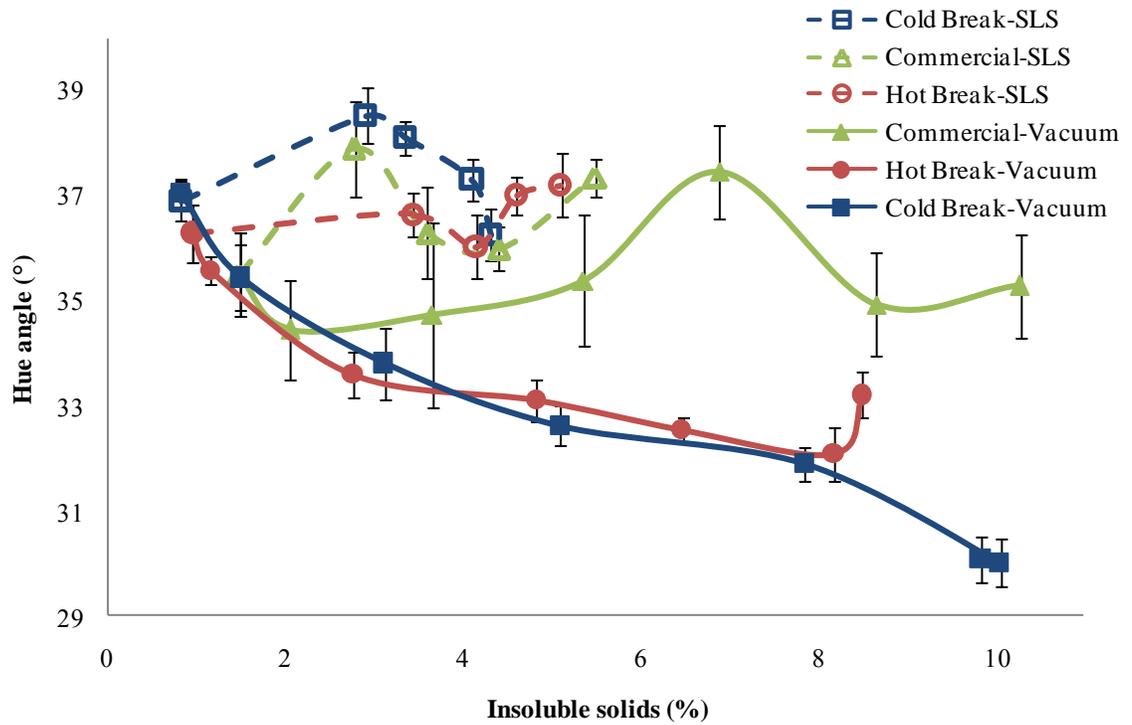


Figure 32. Color of tomato products concentrated to different insoluble solids by vacuum concentration and SLS then diluted to 5% soluble solids for measurement.

4.5.6 Vitamin C

Vitamin C decreased linearly during concentration in both vacuum concentration and SLS ($R^2 > 0.78$) (Figure 33). In vacuum concentration, the heat applied during evaporation significantly destroyed vitamin C in the sample. In the samples from SLS, vitamin C, which is water-soluble, was lost through the filtrate (Table 6). The loss of vitamin C in the SLS samples at 5% insoluble solids was 53 - 60% of the initial value, which was greater than that in the vacuum concentrated samples (19 - 46% of the initial value). Vitamin C content of SLS samples was significantly lower than that of vacuum concentrated samples at the same insoluble solids. Therefore, vacuum concentration retained more vitamin C than SLS even though the SLS was expected to preserve vitamin C in the samples better than vacuum concentration due to the lower temperature. Reverse osmosis retains more vitamin C in tomato concentrates than traditional evaporation since it requires less heat treatment and processing time (Yildiz and others 1993).

4.5.7 Energy consumption

In laboratory scale systems, SLS consumed significantly less electric power energy (approximately 45 times) than vacuum concentration, which also needed water for creating vacuum conditions (Figure 34). The SLS requires very low energy since there is no heat application needed during concentration. Moreover, the SLS works much faster than vacuum concentration; for example, it took only 30 min to concentrate 2 L of tomato juice to 10% total solids while vacuum concentration took more than hour to concentrate the same sample to the same total solids.

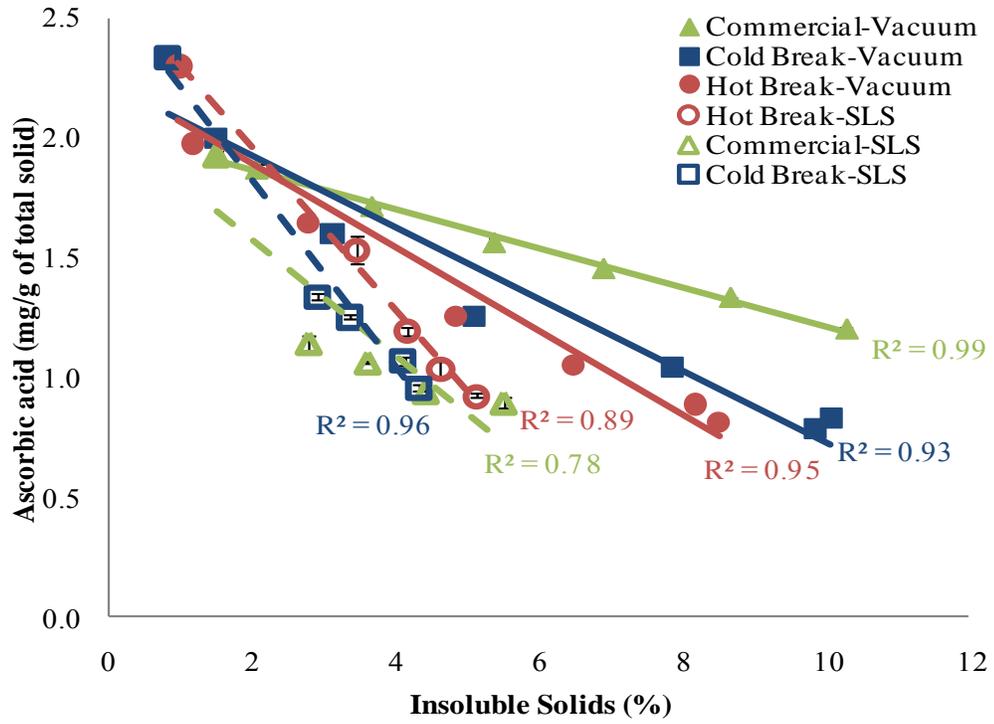


Figure 33. Vitamin C in tomato products at different insoluble solids from vacuum concentration and SLS.

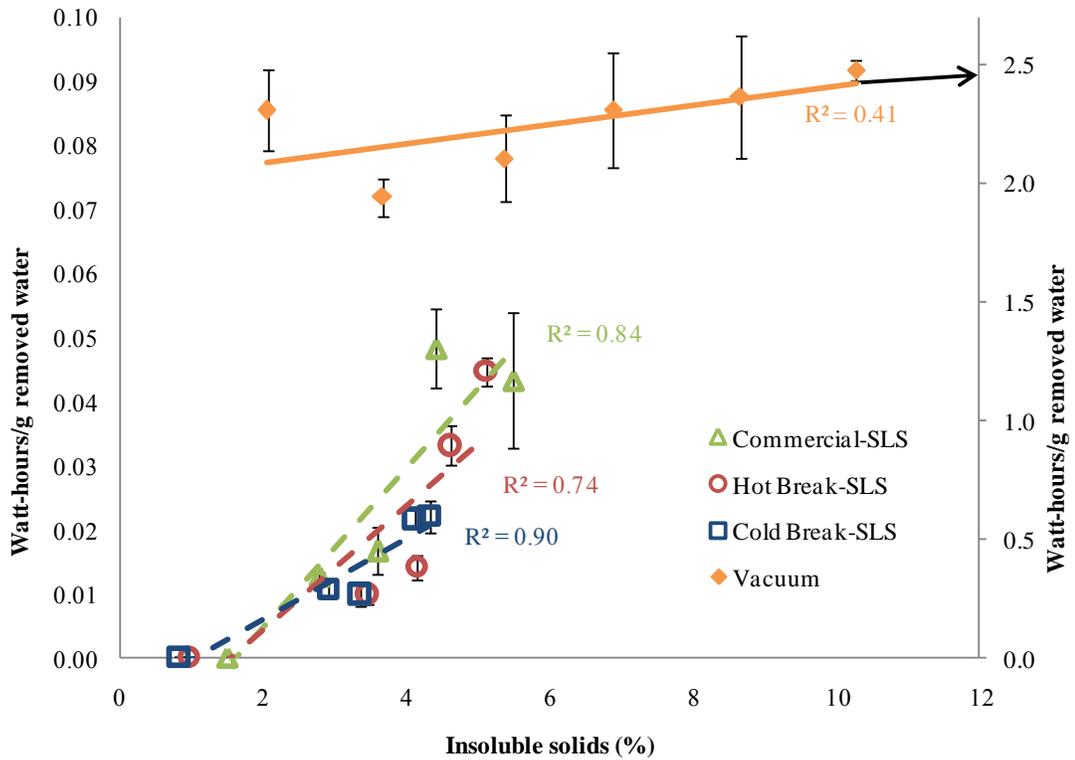


Figure 34. Energy consumption of vacuum concentration and SLS at different insoluble solids. The energy consumption of vacuum concentration is on the right hand axis.

4.6 Conclusions

The application of SLS in concentration of tomato juice can produce products with higher levels of volatile compounds and viscosity when compared to concentration using vacuum concentration, especially in hot break tomato juice. A big advantage of SLS over vacuum concentration was that it consumed up to 45 times less energy during operation. Nevertheless, SLS still needs to be improved in the preservation of quality

attributes since some compounds were lost through the filtrate during concentration, most notably the soluble solids, vitamin C, and carotenoids which determine the color.

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