

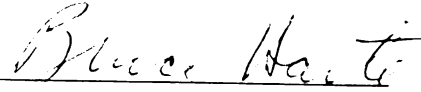




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QUANTIFICATION AND CHARACTERIZATION OF ADHESION  
BETWEEN DOUGH AND PACKAGING MATERIAL

presented by  
SHU-SHIN CHOU

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of the requirements for  
Masters degree in Packaging

  
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**QUANTIFICATION AND CHARACTERIZATION OF ADHESION  
BETWEEN DOUGH AND PACKAGING MATERIAL**

**BY**

**SHU-SHIN CHOU**

**A THESIS**

**Submitted to  
Michigan State University  
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## **ABSTRACT**

### **QUANTIFICATION AND CHARACTERIZATION OF ADHESION BETWEEN DOUGH AND PACKAGING MATERIAL**

**BY**

**SHU-SHIN CHOU**

Degree of adhesion between dough and contacting surface was studied. A wettability test was performed as a indicator of adhesion, and a modified tensile strength apparatus (Stickiforce Meter) was used to determine adhesion between dough and film. In the phase I study, adhesion between fourteen packaging materials and one flour was studied under different temperatures (23°C and 4°C). To simulate the different potential conditions for film contact with dough, the following yeast conditions were used: dough-proof, and contact with film; and dough contact with film, and proof. Wettability and Stickiforce Meter measurements were highly correlated. Proof-first-yeast dough at 4°C on PETG film formed the most adhesive bonds, and contact-first-yeast dough at 23°C on Teflon film had the least adhesive bonds. Phase II study used three different films (PE, PET, and TEFLON) and three flours (Hi-protein, bread, and pastry). The results indicated that bread flour with no-yeast dough on PE film at 4°C had the highest adhesiveness, and pastry flour with contact-first-yeast dough on Teflon film at 23°C had the lowest adhesive properties. Film type, yeast condition, flour type, and temperature all effected dough stickiness.

To my mother, Yi-Ling Chen and my father, Hsing-Ling Chow

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## **CHAPTER 1**

### **INTRODUCTION**

Adhesiveness or stickiness between the food stuff and the packaging surface is a complex phenomenon. The sticking of food to packaging surface can be desirable or undesirable to both the processor and the consumer. For example, the keeping quality of sausage products is known to be closely related to the degree of adhesion of meats to the casing. On the other hand, the adhering of a food product to the contact surface can result in product loss, and in some cases, poor product appearance. Examples are the adhering of sauces to the surface of squeezable bottles and the attaching of pizza cheese to the surface of folding cartons.

Lai (1985, 1987) reviewed the adhesion theories and the factors that affect stickiness. The word adhesion is broadly used to describe the sticking together of two materials with or without an intermediate layer. It is an interfacial phenomenon which, in the food-packaging system, generally involves the liquid-solid interface or solid-solid interface.

Only a few methods have been used to measure food stickiness. Most procedures use tensile testing

instruments. The negative curve obtained during the measurement has been interpreted to be proportional to the work needed to overcome the stickiness. Other stickiness testing devices which use a similar principle are the Struct-O-Graph (Gaines, 1982), Tensile Adhesion Tester (Yokoyama, 1966), and Adhesion Test Balance (Kumar, et al., 1975). Another common procedure employed to measure stickiness involves weighing the material adhered to the contacted surface (Motegi, 1979; Yokoyama, 1966; Taguchi, et al., 1979). The degree of stickiness is correlated to the weight of the product adhered.

Though much attention has been given to many of the chemical and physical properties of food products and polymers including adhesion, little is known about the role of the interface in food product-packaging material interaction, in particular adhesion between a foodstuff and packaging material. Lai (1988) demonstrated that different semi-solid foods interacted differently with plastic surfaces. Furthermore, surface properties of plastics are known to vary due to differences in polymer melt, modification by additives, or chemical reactions (Frisch, et al., 1976; Hair, 1967).

As the food industry continues to move toward convenience and pre-cooked foods, increasing numbers of food products are processed at centralized locations in large volumes. Gene Hoffman (Williams, 1990), the senior vice

president of SuperValu stores, observed that pre-cooked foods have been identified as a major channel through which retailers and wholesalers can get into serious food marketing. Refrigerated foods offer the consumer the greatest potential for convenience and quality. Many of these products are microbiologically sensitive and have short shelf life. Refrigerated dough is one such product (Pomeranz, 1964). It is often presheeted and cut into shape, requiring minimum preparation by the customer. To avoid spoilage of dough, the product must be kept below 40°F. Quality control of ingredients and processing conditions must be rigorously maintained during production to minimize mold, yeast, and bacteria in growth in the dough. Careful consideration must also be given to packaging of the final product. Sticking of dough to the surface of its container can result in the loss of dough, poor product appearance, and customer dissatisfaction.

The objectives of this study are:

1. To evaluate a device for determining adhesion between refrigerated dough and the contacting surface.
2. To determine the influence of flour type, yeast condition, and temperature on adhesion between dough and plastic films.

## **CHAPTER 2**

### **LITERATURE REVIEW**

#### **2.1. Theory of Adhesion**

##### **2.1.1 Definitions and classifications**

Adhesion is the phenomenon that results in surfaces being held together by interfacial forces (Bikales, 1971). The force may be mechanical, electrostatic, or due to molecular attraction. Which type of force depends on whether the interfacial forces results from interlocking action, from the attraction of electrical charges, or from valence forces. Adhesion (ASTM D 907-55, 1958), is defined as the attraction between surfaces being held together by valence forces of the same type as those that cause cohesion, while a substance capable of holding materials together by surface attachment could be defined as an adhesive.

Eley (1961) stated that in physical chemistry, attraction between a solid surface and a second phase is called adhesion. Electrostatic forces, van der Waals forces, or chemical valence forces may all promote adhesion. The technical process of producing adhesion between two solids is called adhesive bonding. In many situations, this process is irreversible.

A brittle adhesive will fracture cohesively on impact loading because the fracture stress is surpassed (Houwink et al, 1965). A rapidly loaded rubbery adhesive behaves much like a tough plastic. The adherend will tend to break or deform. But, by using an interlayer of metal oxides to form a boundary, the same rubber will break cohesively.

In order to form an adhesive joint, the adhesive must move into the bond area and remain there until the bond is complete (Dr. Alfrey, 1948). Therefore, the rheology of polymer systems has a significant role in adhesion. The measurement and understanding of intermolecular forces responsible for adhesion and cohesion is quite important for chemists and engineers whose work involves adhesion.

#### MECHANICAL INTERLOCKING

A mechanical interlocking of an adhesive with the surface structure of the adherend on a scale which could be easily recognized and discerned is the oldest and most simple theory concerning adhesion (Booth 1990). However, as adhesive technologies began to incorporate more rigid materials with smooth surface, the overall concept of mechanical bonding became inadequate. In recent times the following four broad areas have been used to explain the normal adhesive phenomena: diffusion process, electrostatic interactions, mechanical interlocking, and adsorption or specific interactions. A fifth category, viscous flow and pressure-sensitive adhesive is used in the particular case

of pressure-sensitive adhesives.

### DIFFUSION THEORY

Voyutskii (1963) initiated work on the diffusion theory of adhesion. Voyutskii's looked at the adhesion of layers of rubbery materials to each other. This lead to 'autohesion', or the process of self adhesion. Autohesion occurs when the macromolecules are mobile. Portions of the long chain molecules interdiffuse if the two polymer surfaces are in close contact at a temperature above the Glass Transition Temperature ( $T_g$ ).

Vasenin's (1965) concept of adhesion was developed in a more quantitative form. He provided formula expressions, although the mathematics were complex, for the force required to separate two polymer surfaces. This force was directly proportional to the rate of separation and to the fourth root of the time that the surfaces were in contact, as well as inversely proportional to the two-thirds root of the molecular weight to express the force of peeling separation.

Campion (1975) considered the roll free volume played within the structure of polymers. He correlated autohesion properties with the cross-sectional area of these holes in the structure. A certain amount of free space exists close to the polymer chain because of the geometry of the molecules. As Young (1805) and others have shown, diffusion is a reasonable and useful explanation of adhesion. When

the two polymer surfaces are above their Glass Transition Temperature, their molecular chains have same mobility.

Derjaguin (1955) and co-workers developed an explanation for some of the properties of pressure-sensitive tape based upon the concept of an electrostatic double layer at the interface. The peeling apart of the adhered surfaces was identified as an electrical capacitance, with the electrical energy stored within. Derjaguin (1969) emphasized that the force of attraction between the plates of a condenser is independent of their distance from each other. Once the process of separation has commenced and the separation of the two parts of a joint increased, the electrostatic force will become much more significant.

Mechanical interlocking in structural joints has had a great importance in the aerospace industry and in the production of motor vehicles. It is phenomenal that abrading a surface frequently results in stronger bonds. Abrasion takes away a whole range of materials such as dirt and dust, grease or films of oil, poorly adhering layers of oxide etc., so that a relatively clean surface can be obtained and enables a stronger bond to be occur.

Valency forces are usually described as the mechanical strength of any solid material which originates from the various forces of attraction between the ultimate particles (Booth, 1990). Van der Waals' forces are always present no matter which of these forces are significant in any

particular material, and their numbers depends upon the chemistry involved.

### 2.1.2 Wetting and surface tension

Basic to Young's (1805) concept of adhesion is the contact angle  $\theta$  between a drop of liquid and a solid surface (Fig 1). The liquid is static when  $\theta > 0^\circ$ . At a rate depending on the viscosity and surface roughness, the liquid will wet the solid completely, and then spread freely over the surface. Therefore, contact angle,  $\theta$ , not only is a good inverse measure of wetting and spreadability, but of adhesion as well.

Young (1805) initiated the theory that three surface tensions,  $\gamma_{sv}^\circ$ ,  $\gamma_{sl}^\circ$ , and  $\gamma_{lv}^\circ$ , existing at the phase boundaries of a drop of liquid at rest on a solid surface (Fig 1), form a system in static equilibrium. If the molecules that make up the surface are more polar, the surface is easier to wet and bond with a polar liquid adhesive. Consider two glass plates between water for example. The polar groups in the glass plate's surface attract the water molecules and the liquid spreads over the surface. The force of adhesion becomes evident when you try to pull the glass plates apart. Therefore, the essentials for good bond formation would be a liquid adhesive which gives close molecular contact, wets the surfaces to be adhered, and solidifies the liquid between the surfaces.

The energy involved in the relationship between a

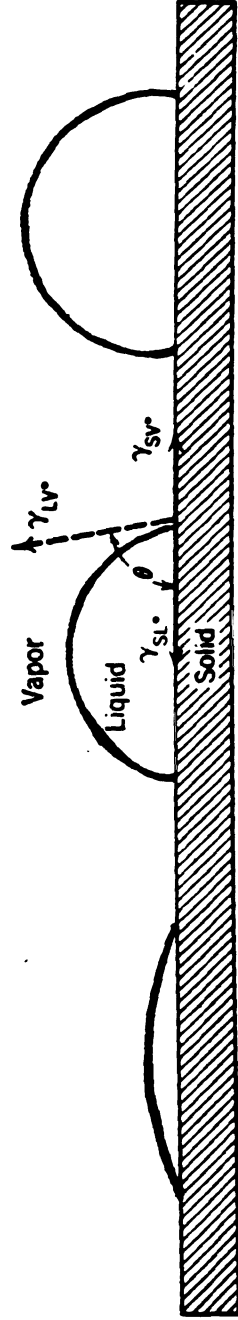


Figure 1. Adhesion depends on wetting of the surfaces.

liquid and a solid in contact has to be taken into consideration. Young (1805) considered that a drop of liquid on a solid surface is in equilibrium which resulted in the following equation:

$$\gamma_{sv} = \gamma_{sl} + \gamma_{lv} (\cos\theta) \quad (1)$$

Dupre (1869) also expressed the reversible thermodynamic work of adhesion to separate two phases which were originally in contact with each other using the following equation:

$$WA = \gamma_1 + \gamma_2 - \gamma_{12} \quad (2)$$

where  $\gamma$  = surface tension

$\theta$  = contact angle conventionally measured through the liquid

WA = the work of adhesion

In Young's equation, the solid and the liquid were in equilibrium with the vapor, but in Dupre's equation both surfaces were clean and the solid was not in equilibrium with vapor. There is an additional term needed, which is the spreading pressure to express the difference in energy between a clean solid surface in a vacuum and the same solid surface in equilibrium with the vapor of a liquid. The equation is:

$$WA = \gamma_{lv} (1 + \cos\theta) + \pi \quad (3)$$

The contact angle indicates the extent a liquid wets and spreads spontaneously on a solid. The liquid will remain a droplet if the temperature is close to 180°F (see figure 1);

but will wet and spread on the solid surface if  $\theta$  is small. This proves that the smaller the contact angle the better the solid will wet. External pressure can also be applied to the solid to increase the wetting and spreading.

The spreading of a liquid over a solid, occurs through the condensation of vapor from the liquid over the solid and the subsequent spreading of the bulk of the liquid over the film of condensed vapor (Burdon, 1949). Spreading is the process that involves a reduction in free energy. A solid surface is easily contaminated with foreign matter in a thin layer unless special precautions are taken. When left unprotected in ordinary air even for a short while, a film of greasy material will be detected on a solid surface. Quincke (1859) found that when the surfaces are considerably torn and scratched, sliding becomes impossible unless great force is applied. The ease of wetting is greatly decreased by traces of grease which in turn increase the contact angle against water.

Dupre's equation for a solid and a liquid:

$$W_{SL} = \gamma_{SA} + \gamma_{LA} + \gamma_{SL} \quad (4)$$

where  $\gamma_{SA}$  = surface tension of the solid against air

$\gamma_{SL}$  = surface tension of the solid against liquid

$\gamma_{LA}$  = surface tension of the liquid

Equation (4) shows the relative strengths of the adhesion of the liquid to the solid, and from it can be determined the contact angle. When the liquid attracts the

solid as much as it attracts itself or when the liquid attracts the solid more than it attracts itself, the contact angle will be zero,  $W = 2\gamma_{LA}$ . When the attraction of the liquid for the solid is half that of itself, the contact angle will be  $90^\circ$ , and when there is no adhesion between the liquid and the solid, the contact angle will be  $180^\circ$ .

If two microscopic glass slides are laid on top of each other with a few drops of water in between, it will be very difficult to separate them (Booth 1990). Two kinds of energy must exist in order to separate them. First, the entire thin, liquid layer between the slides must flow into a smaller area to allow for the increased distance which separates them. In order to overcome the viscosity of liquid flowing through a small gap, an expenditure of energy is required. This increases the thickness which in turn increases the surface area around the periphery. Because of the surface free energy of the additional new surface, additional energy will be required. Such a phenomenon explains the adhesion of pressure-sensitive tape. The backing is thin and flexible, allowing it to conform closely when it is applied to a surface and pressed down. By coating the backing with a thin film of a viscous liquid, adhesion can occur because the viscous liquid flows across and penetrates the irregular surface when pressure is applied. The ability of the backing to conform and the liquid to penetrate is dependent upon the pressure applied

while applying the tape.

In order to obtain proper wetting between two substances, A and B, at least one has to be applied in the liquid or plastic state, or in the highly elastic state (DeBruyne et al, 1951). When volume and temperature are constant, the following equation is true (Houwink, 1965):

$$\Delta F = \Delta U - T\Delta S \quad (5)$$

Where  $\Delta F$  = free energy

$U$  = internal energy

$S$  = entropy

$\Delta F$  controls the change in internal energy and the change in entropy. In order for wetting to occur, the solvent must be in contact with the molecules of the solid.

According to equation (5), as the solid's molecules attract those of the solvent, heat is liberated, if  $\Delta U$  is negative, wetting will occur with certainty. When the solvent attracts its own molecules more than the solid's molecules attract those of the solvent, then wetting may or may not occur, depending on the magnitude of  $(\Delta U - T\Delta S)$ .

Equation (6) shows that wetting results when the surface of the solid disappears and the interface appears:

$$\Delta U_w = \Delta U_s + \Delta U_L - \Delta U_{SL} \quad (6)$$

where  $U_w$  = energy of wetting

$U_s$  = energy of solid surface

$U_L$  = energy of liquid surface

$U_{SL}$  = energy of interface

Initially, because of van der Waals forces between the atoms in the two surfaces, the materials adhere to each other. The real strength of the adhering materials is much weaker than the interfacial strengths. The absence of weak boundary layers also brings the two surfaces together.

Conventional techniques, such as surface oxidation by corona discharge or flame treatment, are believed to be effective since they create wettable polar surfaces so that the adhesive may spread spontaneously and provide extensive interfacial contact (Bikerman, 1968).

The subject of wetting has been dealt with extensively in the literature (Zisman, 1962, 1963, 1964; Good, 1960, 1964). Several studies have been done, but the mechanisms and criteria proposed for establishing relationships between wetting and adhesion have not been consistent. Zisman (1962, 1963, 1964) recognized many of the pertinent relationships which occur with adhesion and discussed them in detail. He did not however establish the importance of these relationships to the problem of adhesion. Sharp, and Schonhorn (1964) emphasised the role spreading has on adhesion and concluded that in order to form a satisfactory adhesive bond, the adhesive must exhibit a surface tension at bonding temperature.

Johnson and Dettre (1964) have shown that for most practical coating or adhesive systems, the adhesive will exhibit contact angles with the solid substrate which are

less than  $90^\circ$ ,  $\cos\theta$  will be positive, and thermodynamic equilibrium will correspond to a wetted state. Several factors may affect the rate of wetting, the main factor, likely being the viscosity of the liquid adhesive. Another is interfacial topography, which influences the resistance to flow. Yet another, relates the dimensions of the interstices directly to the flow rates in the interfacial interstices.

#### SURFACE TENSION AND WETTABILITY

Surface tension is the measure of the tendency of the boundaries between liquids and gases or between two different liquids to contract (Bikerman 1968). A liquid-liquid interface is often referred to as interfacial tension. For this research, wettability of solid polymers is a far more important property than surface tension of liquid polymers or of polymer solutions. Whether a polymer is suitable for a raincoat, can be printed upon, or has easy gluability depends on its wettability. Contact angle is a quantitative measure of wettability. The following are methods used to measure the contact angle: direct measurement on a drop (Ray et al, 1985), the drop dimensions (Allan, 1959), angle of sliding (Bikerman, 1950), (Kawasaki, 1960, 1970), and wetting hysteresis (Bikerman, 1958). Different methods are employed, depending on the different kinds of materials and solutions.

Fowkes (1952) stated that the basic factor in wetting

is the free energy of interaction between the liquid and solid phases across the interface. This free energy is equal to the reversible work of adhesion  $W_A$ , which is the sum of several kinds of interfacial attractions:

$$W_A = W_A^D + W_{AH} + W_A^P + W_A^\pi + W_A^E \quad (7)$$

where D = dispersion forces

H = hydrogen bonds

P = other polar interactions

$\pi$  = pi-bonds

E = electrostatic interactions

The work of adhesion can be quantified as  $W_A = \gamma_{LV} + (\gamma_{SV} - \gamma_{SL})$ . This is a useful equation for some supercooled isotropic liquids when their surface free energies are equal to the surface tensions.

Interfacial tension is the force which keeps surfaces apart or causes them to coalesce (Young, 1945). However, when contact occurs between a solid or a liquid and a gas, the forces established at the interface are called the surface tension. A substance may react differently in different situations. For example, the effect of water coming in contact with a clean leaf acts differently than when it makes contact with a dusty leaf; cotton sinks in soapy water, etc. These differences are a result of surface active agents. Surface-active agents are compounds which cause variations in either interfacial tension or surface tension.

The atomic theory states that all matter consists of atoms and, as a result of recurring constant arrangements of atoms, many familiar molecules are formed. These molecules will have average forces in all directions that are equal and the attraction forces in the interior surface of a mass of the material will be balanced in all directions, and there will be uneven forces at the surface. As the temperature of a liquid increases, the attraction between the molecules decreases. If the temperature increases to the critical point, the surface tension becomes zero. Foreign matter also impacts the adhesive ability of matter. Two drops of clean mercury fuse very readily, but the presence of dust, oil, or other foreign matter on the surface of the mercury will prevent easy union of the two drops.

A fundamental factor in wetting is the reduction of surface tension. Given two pure liquids with equal viscosity and volatility, the one with the lower surface tension will spread on a clean glass plate more rapidly. The outer layer of molecules is under contractile tension because of the unbalanced cohesive forces occurring at the surface of a liquid. Accordingly, substances such as fatty acids and alcohols containing both hydrophilic and hydrophobic groups spread out over a large area. Water wets a substance only if the forces of adhesion for the adsorbing surface are stronger than the forces of cohesion between the

water molecules.

Langmuir (1916), and Harkins et al (1917) stated that absorption takes place because of a surface force presence. Cohesion is the force which holds like molecules together, and the forces of attraction between unlike molecules is adhesion. Langmuir and Harkins also stated that in general, wetting is a chemical phenomena, and the forces related to it are those classed under adsorption. The terms "chemical attraction", "residual valencies", and "unsatisfied chemical affinities" are also used in connection with adsorption.

Bartell (1931) explained that a most common type of adsorption of liquid by solid is wetting by water. Wetting by water may consist of the following three types: (1) spreading wetting (water wets a plain surface); (2) adhesional wetting (water acts as a cementing substance between two or more solids); and (3) immersional wetting (interior capillaries of a porous substance like wheat grains are wetted by water). An example of adhesional wetting by water would be when water assembles the flour particles into the dough mass.

### **2.1.3 The interaction between packaging material/ product**

Experimental values for adhesive performance are influenced by the gross-sample geometry, the topography of the interface, the chemical nature of the materials, the mechanical responses of the solid and the viscoelastic phases, strains rates, strain geometry, temperature, and

such unknowns as the thermodynamic state of the system (Huntsberger 1963). Huntsberger developed the concept that poor performance results from poor interfacial contact. He also established that the way in which the adhesive bonds was formed was greatly influenced by the temperature dependance of the adhesive performance.

The effect of different container surfaces on the flow rate of food or beverage is an important attribute in fluid and semi-solid food (Kiosseoglou et al, 1983). They suggested that the degree of wetting of the surface is an important factor affecting spreadability.

Steele (1979) found that bakery products tend to stick to trays. It was caused by the water in dough wetting the metal surface and then drying during baking and the dissolved materials are deposited to form a bond between the metal and the product. He concluded that in order to minimize adhesion of dough to aluminum trays, there should be a high degree of wetting of the aluminum surface with an oil and a low degree of wetting of the oil surface on the tray by the water in the dough.

Lebedev et al (1975) stated that the surface roughness of the container influences adhesion. He and a co-worker found that adhesion of spaghetti dough to molds was related to the degree of unevenness of the metal surface.

Lai (1985) found that different food materials wetted the polymeric film differently. He demonstrated that food

interacts differently on plastic surfaces by studying their spreading properties. He also stated that degree of wetting was determined by the spread ratio, coefficient of wetting, and coefficient of traction.

## **2.2. Chemical and Physical Properties of Dough**

### **2.2.1 Type of flour and formation of dough**

Wheat flour is a complex system of protein and carbohydrates and is susceptible to various stress conditions (Hlynka, 1964). Wheat is unique among the cereal grains in the type of products which can be produced from it (Hoseney et al, 1978). Wheat flour is the only flour that will produce good quality bread, cakes, cookies or pasta. Although there are many different kinds of wheat grown around the world, they can be categorized into three types generally:

(A) The Bread Wheats: These are generally hard wheats and have a somewhat high protein content.

(B) The Soft Wheats: The bonding between the protein and starch is weak in this type wheat. This category produces flour with small particle size and has a low level of starch damage during milling.

(C) The Pasta Wheats: These are usually hardy wheats such as durum wheats and are preferred for making pasta.

One of the most important characteristics of a bread flour is the breadmaking quality (Hoseney et al, 1978). This quality can be defined in terms of the number of loaves

produced by the flour. Loaf volume is one important parameter in judging flour. Although a high loaf volume does not necessarily indicate good flour quality, a poor loaf volume will indicate poor flour quality. The highest loaf volume possible consistent with a good crumb grain is most desirable.

#### DOUGH DEVELOPMENT

A rubbery mass of wet lumps with little coherence, is obtained during the early mixing of dough ingredients (Pomeranez, 1964). Gradually, the coherence increases, and the dough develops elastic properties and begins to pull away from the mixing bowl which makes the dough more smooth and gives it a drier appearance. This is called dough development. The time needed for optimum development usually varies with the type and speed of the mixer, the type of flour, and the water content of the dough. However, as mixing continues, the dough eventually loss its elasticity, becomes highly extensible and sticky, and in somewhat fluid. This is usually referred to as dough breakdown.

Dough could be described as a compound colloid (Swanson, 1943). When we mix flour with water, the protein particles which form gluten unite into filaments or strands, and form a three dimensional network and thus a continuous phase or system. Starch granules are speshed in this network. Water absorbed by the protein particles and starch

granules also forms into a continuous phase. Ingredients such as salt, sugar, yeast, and other soluble materials are dissolved in the water solution and their contact with the starch and protein is in the discontinuous phase.

Hlynka (1970) suggested that mixing results in unfolding and orientation of long-chain molecules. This leads to a condition of more laminar flow in the dough, which makes the dough less resistant to extension. Hosenev and Finney (1974) speculated that this orientation of protein molecules could increase the probability of hydrogen bonding resulting in a release of water. The increase in free water in dough may be among the factors explaining the lower resistance in overmixed dough to extension as well as its wet and sticky appearance.

Dough mixing involves the combining and blending of the formula ingredients. After applying sufficient physical work to the mixture, it will be transformed into a cohesive mass with the requisite viscoelastic properties (Pyler, 1988). The actual proof time will vary depending on the dough's character. Factors such as inadequate yeast content and poor control of time or temperature during fermentation result in extended proof time.

During fermentation, proofing and baking, the walls of the dough gas cells are subjected to considerable tensile and shear stress (Pyler, 1988). Therefore, it is necessary for them to possess viscoelastic properties which will allow

sufficient expansion and also to sustain these extensions without rupture.

From observation on fermenting doughs (Matz, 1960), it was formed that there was a tendency for the starch and gluten to separate during fermentation and for the gluten to form into transparent cells. These transparent cells were drawn to the surface because the gas nucleus from which the bubbles originated is a glutinous core. As the bubbles expand, the required amount of gluten needed to satisfy surface needs is drawn from the starch-gluten matrix of the endosperm material. The properties that enable this to occur may be controlled by the viscosity and fluidity of the gluten and the amount of adhesion of the gluten to starch.

Yeast action in fermentation leads to two primary results: (1) The formation and migration of carbon dioxide culminating in a network of cellular compartments to lighten or raise the dough, thereby greatly improving its ultimate palatability; and (2) the simultaneous production and concentration of alcohols, aldehydes, ketones, and acids which contribute to bread aroma and flavor. Yeast also alters the physical properties of dough, especially the gluten elasticity, through the powerful stretching actions generated by the diffusion and accumulation of carbon dioxide throughout the dough mass.

### **2.2.2 Constituent components of dough associated with functional properties**

According to Parker and Taylor (1966), adhesion can be defined as the use of one material to bond two other materials together, and cohesion as the joining together of the same material (Cherry, 1981). Cooking builds up additional adhesive and cohesive interactions among protein, lipid, and carbohydrate components of foods.

Using the scanning electron microscope, Khoo et al (1975) observed that dough consists of starch granules held together by a matrix of hydrated gluten protein. Polar groups contribute greatly to adhesion and cohesion of protein to carbohydrates. The chemistry of adhesion involves nonpolar interactions involving long chain aliphatic or aromatic groups such as Van der Waal or London forces (Parker et al 1966). Disulfide bonds in the amino acid cystine are important to the properties of many proteins by maintaining covalent intramolecular bonds and crosslinks between protein chains (Wall 1971). Complex gluten proteins can be separated, by measuring differences in solubility, which leads to separation into many soluble proteins and an insoluble protein residue. The effect of molecular size and shape on protein cohesive strength was demonstrated by measurements of tensile strength and elongation of films cast from laboratory preparations of wheat gluten, gliadin, and glutenin (Wall et al 1969).

Glutenin consisting of larger, more asymmetric

molecules forms films with greater tensile strength than gliadin films. Gliadin films stretch further than those from glutenin because of weaker molecular associations. Gluten, a mixture of gliadin and glutenin, has intermediate film properties. Reduction of the disulfides of gliadin increases its viscosity significantly due to unfolding of the polypeptide molecule. Reduction of glutenin destroys its cohesive nature when hydrated, but the reduced proteins are very sticky and quite adhesive.

Because of the existing gluten proteins, hydrated flour can be worked into an elastic-cohesive mass by mixing. During mixing, the asymmetric glutenin molecules orient and associate, thus increasing dough strength.

Orth et al (1972) investigated the relationship between flours, the variation in dough strength, and their different protein fractions. They discovered that the mixing time requirement of dough and the tolerance to mixing correlates to the residue protein content. Stronger flours not only contain more residue protein but also more of the higher molecular weight glutenin fraction.

Glutenin molecules, (large asymmetric shape) have considerable surface area with numerous exposed functional groups to permit strong association by noncovalent forces. Fragments of highly crosslinked residue proteins contribute lateral cohesion and resistance to laminar flow (Hoseney et al 1969). Gluten's cohesive-elastic character holds

ingredients and provides a chewy texture which is the basis for many vegetarian-simulated meat products.

Most globular or albumin plant proteins exhibit little cohesive or adhesive properties in their native state. At a pH of 9 or above, however, disulfide bonds cleave, protein unfolding occurs, and functional groups previously associated within the molecule become available for external binding.

Adhesion and cohesion are properties of many polymeric substances including proteins. Protein's high molecular weight and random coil structure result in more associations and the refore enhance adhesive and cohesive properties.

The functional properties of proteins in foods are determined by the molecular composition and structure of the individual proteins and their interactions with one another and with other substances (Wall, 1979). Altering the constituent proteins or adding other proteins could improve or modify food characteristics such as viscosity, texture, water absorption, or fat emulsification.

Proteins of wheat flour govern the plastic and elastic properties of bread doughs and some types of batters to a large extent (Pomerane, 1964). The relationship is most prominent in bread doughs, but the influence of flour proteins on the physical properties of the doughs and of batters is much less prominent and less understood. Protein consists of alpha-amino acids linked by peptide bonds

between the carboxyl group of one amino acid and the alpha-amino group of a second amino acid. This peptide backbone structure constitutes the primary structure of proteins.

Dough derives its properties from the constituent components (Pomerane, 1964). The major and most important group of constituents is the proteins. The wedge and adhering proteins in the intact grain, or the derived fractions such as gluten, globulins, albumins, and lipoproteins are included in this group. The carbohydrates are the most abundant group, including starches, sugars, and soluble and insoluble polysaccharides. The lipids form a small but significant part of the flour. Water plays a key role in dough formation. Air forms the nuclei of the gas cells, and the oxygen acts as an improver.

The amount of protein determines the density while the quality of protein determines the behavior of the three dimensional gluten network which permeates the dough (Swanson, 1943). Using higher protein flours, with the same quality but higher protein content, will result in larger loaf volumes. If flour has a high percentage of protein, the gluten mesh-work will be denser.

Osborne (1907) separated the wheat protein into four distinct proteins: leucosin, water soluble; globulin, salt soluble; gliadin, alcohol soluble; and glutenin, insoluble. Based on a protein's structure, composition, solubilities, and other characteristics, it is classified as a simple

protein, conjugated protein, or derived protein.

The most prominent component, gluten, is composed of two proteins, gliadin and glutenin. These two proteins effect baking quality. Upson et al (1916) studied the swelling of wheat gluten. Wheat proteins swell in water and especially in solutions of dilute acids because of the entrance of water between the protein molecules and the molecular structure. During the swelling process, gluten becomes softer, more flexible, and due to diminished cohesion, increases in weight and volume.

Phosphate protein interactions could be responsible for lowering hydrogen bonding activity and subsequent reduction in water binding, extensibility, and cohesion (DeMan et al, 1976). The increase in free water may be among the factors explaining lower resistance to extension of overmixed dough as well as its wet and sticky appearance. The viscoelastic properties of wheat dough are primarily attributed to gluten proteins. These proteins form a network of linear macromolecules bound together by various cross-links during the process of dough development. Because of the high concentration of glutamine in gluten-forming proteins (about 30%), a great number of hydrogen bonds form, rendering this protein fraction insoluble.

Using microscopic examination, three phases, starch, protein, and gas cells, can be distinguished in an unleavened dough, and yeast cells constitute a fourth phase

in a leavened dough (DeMan et al, 1976). All starch kernels retain their identity in dough and are embedded in a continuous matrix of protein. A fine, vesicular structure with expanding gas cells is developed and maintained during fermentation and proofing until heat fixes the texture in bread by protein denaturation and starch gelatinization in the oven.

The tendency of dough, and particularly gluten, is to spread into films having great stability and with time-dependent surface viscoelastic properties, while still being highly compressible. Glutenin forms a very tough, rubbery mass when fully hydrated, while gliadin produces a viscous, fluid mass upon hydration (Pyler et al, 1973).

Glutenin is a prime contributor to the functional properties of gluten and dough (Bietz et al, 1973). Wheat dough's characteristics of viscoelasticity and loaf volume are primarily due to the gluten protein consisting of glutenin, gliadin, and small amounts of albumins and globulins. Hydrated glutenin is tough and cohesive, but less elastic than the whole gluten, while hydrated gliadin yields only a viscous mass. Accordingly, the unique structure and composition of gluten produces the rubberlike properties of dough. Glutenin constitutes approximately 30 to 40 per cent of the protein in wheat flour, and about half that in gluten.

Because of cystine residues, disulfide linkages can

occur either within (intra-) or between (inter-) protein chains (Bietz et al, 1973). These linkages determine the functional properties of native molecules. On the other hand, if all disulfides were of the inter-chain variety, the resulting highly branched polymer would not allow suitable alignment of proteins to form a dough. To form a dough an optimum balance of inter- and intra- chain disulfide bonds is essential for good glutenin performance.

Glutenin occurs only in the endosperm, and probably serves both as structural protein and as reserve material for the seed. Too much glutenin may prevent expansion of gas cells during fermentation (Mecham, 1973). An appropriate combination of glutenin and gliadin is essential for good dough performance and loaf volume. Glutenin molecules, because of their high molecular weight, shape, and their amino acid composition, impart toughness and strength to gluten. These molecules are favorable for hydrogen and hydrophobic bonding and provide relatively large surface areas which are suitable for molecular association. However, if glutenin is the only protein fraction in dough, the dough would be too resistant to expand during fermentation and baking. To assure good performance of the dough, gliadin, with its small and symmetrical molecules must be present to modify the glutenin.

Gliadins are mixtures of tightly folded globular

proteins with hydrophobic regions buried within the molecules and the hydrophilic H bonding regions exposed on the surfaces (Mecham, 1973). This structure may explain the sticky nature of gliadins.

The factors that contribute to the strength of an adhesive joint (Gent, 1982) range from weak Van der Waals interactions to covalent chemical bonding, which plays an important role in the attractive forces at the interface. Besides these, the dissipative properties of the adhering materials are also contributing factors. On the other hand, perfectly-elastic and non-dissipative materials have the lowest adhesion strength. There are also geometrical factors in joint strength. When the adhesive layer is extremely thin, it cannot release much energy in the internal deformation processes, which keeps its contribution to the observed strength to a minimum.

The energy required for joint rupture is stored elastically in the bonded parts (Gent, 1982). When energy is expended, this makes a small detached region grow in size. The result of this action shows that the breaking stress depends upon the elastic properties of the system (the elastic modules and dimensions of the various components), and the size of the debonded zone. At least three sciences contribute to the strength of adhesion: interfacial chemistry, rheology of inelastic materials, and the mechanics of fracture of composite systems. The first

phase of dough formation primarily involves the moistening of the flour particles. The movement of the mixer elements an/or bowl disperses the dough water between the flour particles. The hydrophilic properties of the particles cause liquid to be absorbed onto their surface. This results in the shearing elements of the mixer countering the forces of adhesion.

### **2.2.3 Measurement of physical/functional properties of dough**

The most distinctive property of dough made from wheat flour is its ability to retain the gas formed within its mass either due to the growth of yeast or the action of an acid on sodium carbonate (Swanson, 1943). This property results from the water films absorbed on the filaments of gluten and the enmeshed starch. There is a certain amount of elasticity and plasticity in gluten strands, enabling recoil after stretching and if the stretching goes beyond the elastic limit, it results in permanent elongation.

The Brabender farinograph is used to measure the water absorption properties of flour and the mixing characteristics of a standard flour-water dough. Because of the complex nature of the functional properties of wheat flour, an evaluation of wheat flour dough must be performed under actual or simulated baking conditions. The brabender farinograph and extensigraph are the basic instruments used to characterize the gluten proteins under the simulated conditions existing within the bakeshop.

Dough consistency plays an important role in achieving proper mechanical development. It also influences the gas retention properties of the dough (Sietz, 1978). Absorption values are used extensively in the calculations involved in developing new formulations. Generally, flour with high absorption values are desired because it increases unit yields.

### **2.3. Correlation of measurement in stickiness with food**

The force to tear products apart per cross-sectional area or per unit weight of sample is a function of a material's tensile strength. Tensile strength was used to measure the adhesion strength of chunked and molded products (Trout and Schmidt, 1987). Tensile strength is often measured using an Instron Universal Testing Machine, or a device such as the Food Technology Corporation's Texture recorder, which measures an apparent tensile strength.

The Instron Testing machine was introduced in 1949 (Hindman & Burr) as a general purpose material testing machine, and is used for testing textiles, paper, plastics, rubber, and other flexible materials. It is suitable for laboratory use because of the sophisticated controls and sensitivity of measurement possible. Its most useful function is in the application of classical material test methods to establish fundamental material properties.

The Instron Tensile Tester, model TM (Instron Engineering Corp., Quincy, M.A.) consists of two horizontal,

parallel plates, with a constant rate-of-jaw-separation on the top plate. A device attached to the bottom plate measures the force pressing (positive) or pulling (negative) against the plate (ASTM D 882-83, 1983). The crosshead is set and moves at a constant rate during the test. The stickiness value equals the grams adhesive force and cm crosshead movement times the appropriate factor.

The Instron Tester is an excellent tool to evaluate stickiness because it focuses on the surface properties most desirable to measure. Batcher et al (1963) stated that cooked rice had similar palatability characteristics no matter what cooking method or medium was chosen. Ferrel et al (1960) used a screening device constructed so as to determine if different preparation methodologies would change the rice stickiness. Mossman et al (1975) studied different treatments that accelerated the aging of sticky rice. In each case the Instron Tester was chosen to measure stickiness levels and to differentiate the type of stickiness.

Mossman et al concluded that time and water content had varying impact on the stickiness of the sample. Increasing cooling time resulted in a small increase in the stickiness value, while an increase in the amount of cooking water caused a proportionately greater increase in the stickiness value.

### 2.3.1 Methods of measurement in stickiness

Cooked spaghetti must be firm, resilient, and nonsticky to meet maximum consumer acceptance. Voisey et al (1978) used the Instron Universal Tester to measure the stickiness of cooked spaghetti. D'Egidio et al (1982) concluded that spaghetti stickiness is related to the amount of surface material that can be washed from drained cooked spaghetti.

Compression testing utilizes a compression cell which is composed of parallel plates between which test products can be compressed. Apparent stress at failure and apparent strain at failure can be calculated (Diehl et al., 1979), while true stress and strain cannot be calculated because a uniform cylinder is not maintained during compression. Strain is highly correlated to sensory and texture profile analysis (TPA) cohesiveness, while stress to fail (compressive force to failure) is correlated to TPA hardness and sensory firmness (Montejano et L., 1983).

Dexter et al (1980) modified the GRL compression tester (Kilborn et al, 1982) to test for cooked spaghetti stickiness. Dexter's process used sample sizes as small as 6g of spaghetti. The cooked spaghetti was compressed under a plunger and the force of adhesion of the spaghetti to the plunger was measured upon lifting the plunger.

Dexter demonstrated that the quality of the cooking water and spaghetti drying procedure have a significant influence on stickiness of cooked spaghetti. Spaghetti

stickiness is also related to cooking time and elapsed time after cooking. Other factors such as gluten strength, sprout damage, semolina granulation, and extrusion conditions are all associated with cooked spaghetti stickiness. Dexter concluded that protein content has no significant correlation to spaghetti stickiness.

Dexter et al (1983) studied factors that influence stickiness and their relationship to other cooking quality characteristics. He concluded that each spaghetti sample proved to be stickier and lost more solids during cooking when cooked in tap water compared to deionized water. Comparing spaghetti processed under high temperature and low temperature drying conditions, spaghetti cooked at high temperature was less sticky. Stickiness was lightly influenced by cultivar, wheat class, raw material granulation, and protein content, but was not related to sprout damage. Even when all the factors were included in a step-up regression analysis, less than 50% of the variance in stickiness could be predicted.

Gaines et al (1982) studied the influence of temperature, humidity, and flour moisture content on stickiness in sugar-snap cookie dough. Dough stickiness measurements were conducted with a Struct-O-Graph.

Gaines concluded that the most desirable ambient conditions for evaluating soft wheat cultivars with the micro-method III (Finney et al 1950) procedure are

temperatures between 20-21°C and 30-50% relative humidity. This allows "standardization" of dough consistency and also prevents stickiness problems. No combination of flour moisture content and dough water absorption level caused stickiness problems at 21°C and 50% relative humidity. At optimum dough consistency, doughs made from flours having high moisture contents were less sticky and easier to work with because they tolerated changes in the level of dough water absorption better.

Gaines (1981) used a Struct-O-Graph (C. W. Brabender, South Hackensack, N.J.) to measure the stickiness of cake crumb and to find out if flour chlorination correlated with cake crumb stickiness. A Struct-O-Graph was fitted with a 2,000-cmg spring and a 30-mm diameter plastic disk plunger that moved at a rate of 132 mm/min. The pen arm was activated at the 500-BU chart line, when the cake crumb piece had been compressed for 1 minute, and stopped at the 1,000-BU chart line. At this point compression was relieved and the stickiness measurement was taken. This can be described as one compression/stickiness measurement cycle.

When compressing the sample, the pen arm will travel above the 500-BU line if the sample adheres to the disk and platen. The distance above the 500 line is recorded as the amount of crumb stickiness in centimeter grams. The mean of the stickiness measurements is then calculated. Higher mean stickiness values indicate greater crumb stickiness.

Neither flour chlorination rate nor the flour pH, cake volume, or batter liquid level were correlated with an objective stickiness measurement.

Taguchi et al (1979) studied the factors affecting the adhesion of canned mackerel meat. Measurement of adhesion consisted of weighing the meats that adhered to the inner container. Taguchi used the following for his study:

Different retorting time: 80 minutes and 60 minutes

Salt: 2.5% NaCl, 2.5% NaCl + 0.2% pyrophosphate, or 2.5% NaCl + 0.1% CaCl<sub>2</sub>;

Heating times, 40° to 100°C for 30 minutes.

He concluded that the freshness of the raw meat influences the degree of adhesion while the heating temperature affects the formation of adhesion bonds. The most noticeable adhesion occurred when the internal temperature reached 60°C. Adhesion increased greatly as the heating temperature reached 80°C without NaCl or with 2.5% NaCl. The amount of adhesion greatly decreased with the addition of CaCl<sub>2</sub>.

Curley et al (1983) studied the effect of corn sweeteners on dough stickiness. Dough stickiness was measured with the Instron Universal Testing Machine (model 1122) in the tension mode. He concluded that the dough with 0% dissolved sucrose was firm and manageable, while the one with 100% dissolved sucrose was very sticky and unmanageable. When 50% of the granular sucrose was replaced

with High-fructose corn syrup, the resultant dough was as sticky and unmanageable as the dough made with 100% dissolved sucrose.

Noguchi et al (1976) studied the correlation of dough stickiness with various quality parameters. A texturometer (General Food Corp. New York) was used to determine dough stickiness and consistency. Dough was mixed with a 1.5% sodium chloride solution in a pin-type mixer and the dough was shaped into a sheet (15 x 5 x 1 cm) by the Chopin alveograph mixer and then set on the texturometer.

Noguchi concluded that dough stickiness correlated very highly with the sulfhydryl (SH) content of the protein and with proteinase activity. The protein content, however, did not correlate with adhesiveness, but possibly those with a high sulfhydryl content might well be expected get involved.

In the butter industry, the term stickiness refers to the property which permits butter to remain attached to solid surfaces. This physical characteristic, which involves both adhesion and cohesion, has been described by the term 'hesion' (Claassens, 1958).

Thomasos et al (1963) studied some factors which influence butter stickiness. He pointed out that the characteristic crystal structure affected the hesion values, and homogenization of butters significantly increased hesion readings. An increase in gas content caused a decrease in hesion values with more butter remaining on the adherend.

Thomasos also stated that the crystal structure would change the adhesive property of butter and the gas content would influence the cohesive property.

Kumar et al (1975) compared a balance and a sieve to test the stickiness of cooked rice. Juliano et al (1965) concluded that stickiness related to the amylose content of rice. Sanjiva (1938) stated that rice stickiness is strongly affected by its age, that freshly harvested cooked rice is moist and sticky, but that aged cooked rice is dry and flaky.

20 g of cooked rice was placed on the top sieve (6.7 mm). The two sieves were rotated by hand, with a firm tap at the end of each rotation. After sieving, the rice retained on each sieve was carefully collected and weighed. To conduct the adhesion test, 10 g of cooked rice was placed in a stainless steel cup, gently levelled with a spatula and then pressed for 4 minutes with a 1.5 kg metal pressure weight, and passed through a vertical guide to give a uniform surface. A polished stainless steel cylindrical test body hanging from the left arm of a balance, and counter-balanced exactly on the right arm, was then gently lowered onto the rice and pressed with another small pressure weight for exactly one minute. The weight was removed, the balance released, and sand was added in a stream onto the right pan until the test body was released from the rice.

Kumar came to the conclusion that the sieve test gave a very good indication of the stickiness of cooked rice. The adhesion test, although providing indication of the stickiness of cooked rice had a low correlation and the test procedures needed further improvement. He concluded that the consistency of cooked rice has a negative correlation with stickiness but the water insoluble amylose content of rice seemed to have significant correlation.

## **CHAPTER 3**

### **MATERIALS AND METHODS**

This research is divided into two parts. In part 1, studies were designed to evaluate the stickiforce meter as a device to measure adhesion between dough and the contacting surface. In part 2, studies were designed to characterize adhesion between plastic films and doughes of different flour types and protein levels.

#### **3.1 Materials**

##### **3.1.1 Flour Samples**

A dry mix (flour plus premix), provided by a Pizza Manufacturing Company, was used for the phase I study. In the phase II study, three different flours were selected. Bleached, enriched bromated flour and unbleached, unenriched pastry flour were purchased from Food stores at MSU. The flours were manufactured by General Mills, Inc., Minneapolis, Minnesota. Premium, high gluten, bleached, bromated flour was purchased from Food Stores at MSU and was manufactured by Bay State Milling Co., Quincy, Mass.

##### **3.1.2 Film Samples**

Film samples were obtained from several suppliers and the pizza manufacturing company for phase I study. (Table 1) In the phase II study, three different films were selected

Table 1. Film samples used in Wettability and Stickiforce Meter - Phase I study

Material	Film thickness	Sources
1 HDPE (High Density Polyethylene)	20 MIL	DOMINO'S Pizza
2 PVC (Polyvinyl Chloride)	15 MIL	DOMINO'S Pizza
3 PET/RELEASE AGENT (Polyester)	14 MIL	DOMINO'S Pizza
4 PETG (Polyethylene Terephthalate Glycol)	8 MIL	DOMINO'S Pizza
5 2% EVA/VEGETABLE OIL (Ethylene Vinyl Acetate)	2.5 MIL	DOMINO'S Pizza
6 2% EVA/1.5% PAM SPRAY (Ethylene Vinyl Acetate)	2.5 MIL	DOMINO'S Pizza
7 2% EVA/MYV 9-40 (Ethylene Vinyl Acetate)	2.8 MIL	DOMINO'S Pizza
8 TRI-EXTRUDED PE	0.7 MIL	DOMINO'S Pizza
9 KRAYTON	14.5 MIL	DOMINO'S Pizza
10 60 lb. SEMI-BLEACHED SILICONE COATED RELEASE LINER	4.0 MIL	MEAD RELEASE PRODUCTS
11 TEFLON FEP	1.0 MIL	DU PONT/DURAFILM
12 SILICONE RELEASED PAPER	3.5 MIL	MEAD RELEASE PRODUCTS
13 SILICONE ON SUPER CALENDARED DENSIFIED KRAFT	*	EASTERN FINE PAPER
14 SILICONE ON CLAY- CALENDARED DENSIFIED KRAFT	*	EASTERN FINE PAPER

\* Silicone coated materials - thickness unknown

to provide a broad range of surface wettability and morphology (Table 2). Both PET and Teflon films were obtained from E. I. Du Pont De Nomours & Co., Inc. (Circleville, Ohio). LDPE film was obtained from Tredegar Film Products (Manchester, Iowa).

### 3.2 Analytical Measurements

#### 3.2.1 Determination of the Initial Flour Moisture Content (IMC)

Moisture analysis were performed on the Hi-protein flour, bread flour, and pastry flour. Triplicate 2.0 (≈2.000) gram samples were weighed on an analytical balance, then dried to a constant weight in a Hotpack Vacuum Oven, Model 633 (Hotpack Corp., Philadelphia, PA.) at 80°C in a partial vacuum of 30 mm of Hg for 6 hours, according to AACC Method 44-40 (1983).

The dried samples were transferred to a desiccator until cooled to 25°C and then weighed to the closest 0.0001 gram using a Mettler AE 166 Balance (Mettler Instruments Corp., Hightstown, N.Y.).

The samples were reweighed to determine weight loss due to loss of moisture. IMC was determined (dry basis) according to the equation:

$$(W_i - W_f) / W_f * 100\% = \% \text{ Dry Wt}$$

Where  $W_i$ : Initial weight of product sample  
 $W_f$ : Final weight of product sample after drying

Table 2. Film samples used in Wettability and Stickiforce Meter - Phase II study

<u>Film</u>	<u>Thickness</u>
Low Density Polyethylene (LDPE)	2 MIL
Polyester (PET)	1 MIL
Teflon	2 MIL

### 3.2.2 Determination of the Protein Content

Protein content of the pastry, bread, and high gluten flours were performed using the Microkjeldahl method (according to AACC method 46-13) for total nitrogen determination. Duplicate 0.5 gram samples were digested in sulfuric acid, sodium sulfate, and copper sulfate at 400 - 500°C until digestion was completed ( $\approx$ 2 hours). Samples were transferred to a distillation apparatus (Buchii Kjeldahl Machine, Brinkman Instruments) and distilled according to AOAC Methods 2.057, 14.026 and 14.068 (1980). Total protein was calculated based on percent nitrogen in the sample multiplied by a factor of 5.7.

### 3.2.3 Determination of the Water Absorption Properties -Farinograph

Flours were evaluated for water absorption using a Farinograph, manufactured by C.W. Brabender Instruments, Inc. (Model PL-2H, Dynameter number 2092). A Thermobath

(Type P 60-B) maintained at  $30 \pm 0.1^{\circ}\text{C}$  was used to regulate temperature of the mixing bowl.

The test was run according to AACC Method 54-21 A (1983). Moisture content of the pastry, bread, and Hi-protein flours were determined as described previously.

#### **3.2.4 Stickiforce Meter Determination**

Lai et al. (1985) applied a modified tensile strength apparatus to measure powder cohesiveness. Similar principle was used by Lai in developing the Stickiforce Meter.

The base, dough holder cube, and cube stopper were constructed from plastic (Acrylic) (Fig. 2). The cube was open at both ends. During measurement, a small piece (2" x 2.5") of test film surface is placed under the cube opening. The cube is then filled with dough sample ( $\approx 27.5$  g) and placed sideways against the stopper of the apparatus. The test film material is attached to a string with a small cup at the other end. The test film surface is made to break away from the dough surface by the weight of water added into the cup from a burette (50ml). The weight-force per unit area of separation (Wtforce) is recorded. In addition, the weight of the contact surface before and after each test was also measured. This was reported as weight-mass per unit area (Wtmass).

#### **3.2.5 Inclined Plane Determination**

The wettability of a surface can be determined by the sliding of a drop of fluid along a tilted surface (Kawasaki,

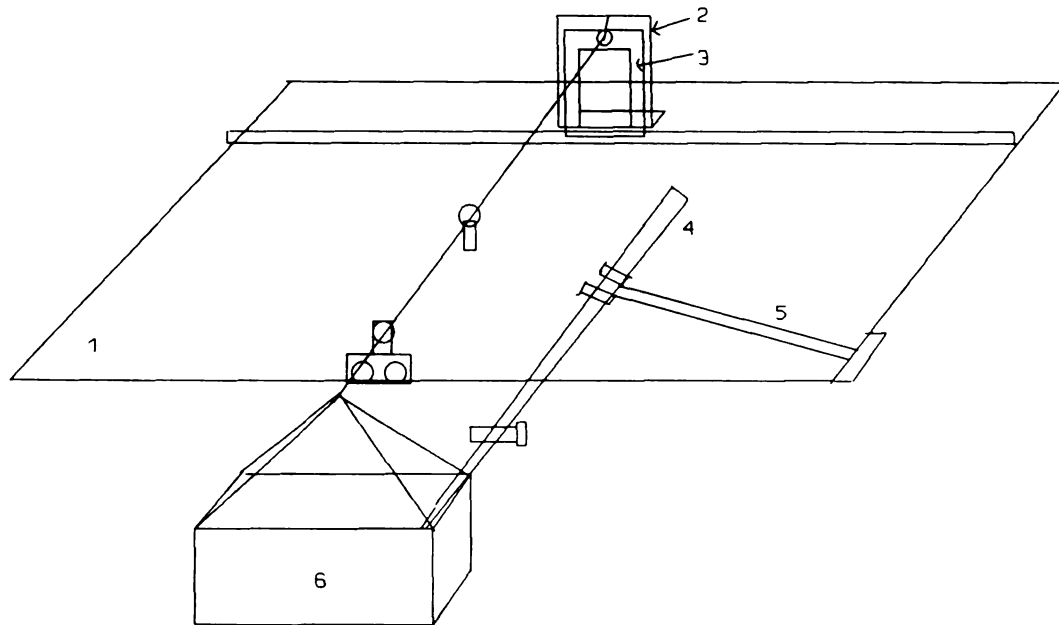


Figure 2. Diagram of Stickiforce Meter.

Components: 1. Base (Acrylic) 2. Dough holder cube 3. Test film  
4. Burette 5. Burette holder 6. Cup  
7. Cube stopper

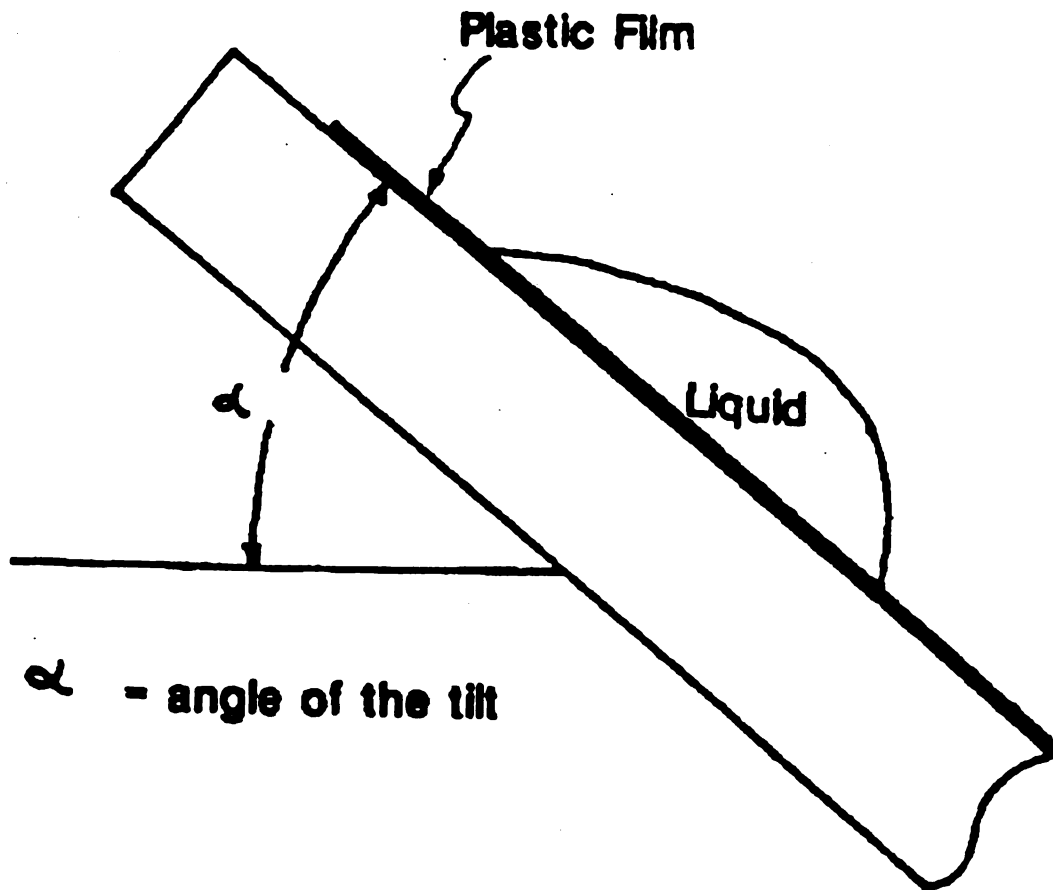
1960; Bikerman, 1966). The tilted plane procedure described by Lai (1988) was adapted for use in this study (Fig. 3). It provided a measure of the wettability of the plastic surface. A droplet of distilled water and 50% ethyl alcohol were placed separately on the test surface which lay on a horizontal platform. The droplet was allowed to spread until arrest occurred. The platform was then carefully tilted at a rate of one degree per second. Angle of slide was defined as the limiting angle between the platform and the horizontal plane at which the droplet moved at a uniform rate of one degree per second.

### **3.3 Experimental Design**

#### **3.3.1 Phase I Study**

Studies were designed to evaluate the stickiforce meter as a device to measure adhesion between dough and the contacting surface. The Stickiforce meter was then used to determine if different dough temperature and contacting films would effect sticking of dough to film. Films (Table 2) were cut into 1" x 8" strips using a JDC Precision Sample Cutter Model JDC 25 (Thwing - Albert Instrument Company, Philadelphia).

Fresh dough was prepared by weighing out 0.91 grams of active dry yeast, and 481 grams of 35°C tap water. A small portion of the water was slurried with the dry yeast in a mixing bowl. One package of the prepared dry mix (flour



**Figure 3.** Sliding of a drop of fluid along tilted plane.

plus premix) was then added to the bowl, along with the remaining water. These materials were then mixed in a Kitchen Aid K-5ss Mixer (Hobart Corporation, Troy Ohio, speed range 2) at room temperature for seven minutes or until the dough was fully developed. After development, the surface was dry and shiny and the texture very fine. When the dough was stretched by hand, there was a good deal of elasticity and stretchiness.

#### **Treatment Variables**

A. Fresh prepared dough was placed in contact with film surface. Following refrigeration ( $4^{\circ}\text{C}$ ) for 24 hours, each sample was allowed to reach ambient temperature ( $23^{\circ}\text{C}$ ) and proofed to twice its original size (approximately 4 hours) before performing the Stickiforce Meter Measurement.

B. Same as treatment A except that the dough was put inside a plastic bag (LDPE) under refrigeration for 24 hours. The dough was then placed into contact with the film surface, and proofed to double its size before performing the Stickiforce Meter Measurement.

C. Same as treatment A except that dough was not refrigerated. Stickiforce Meter Measurement was performed after the dough was proofed to twice its size (approximately 2.5 hours) at ambient temperature ( $23^{\circ}\text{C}$ ).

D. Fresh dough was proofed first at room temperature ( $23^{\circ}\text{C}$ ) and then placed in contact with the film surface for Stickiforce Meter Measurement.

These four treatment combinations were arranged in a 2 x 2 factorial design. The effect of yeast condition (proofing), and temperature were evaluated, as well as the interaction of these factors. A split plot design was used. Treatment variables served as the whole plot factor and film as the split of a randomized complete block design.

### **3.3.2 Phase II Study**

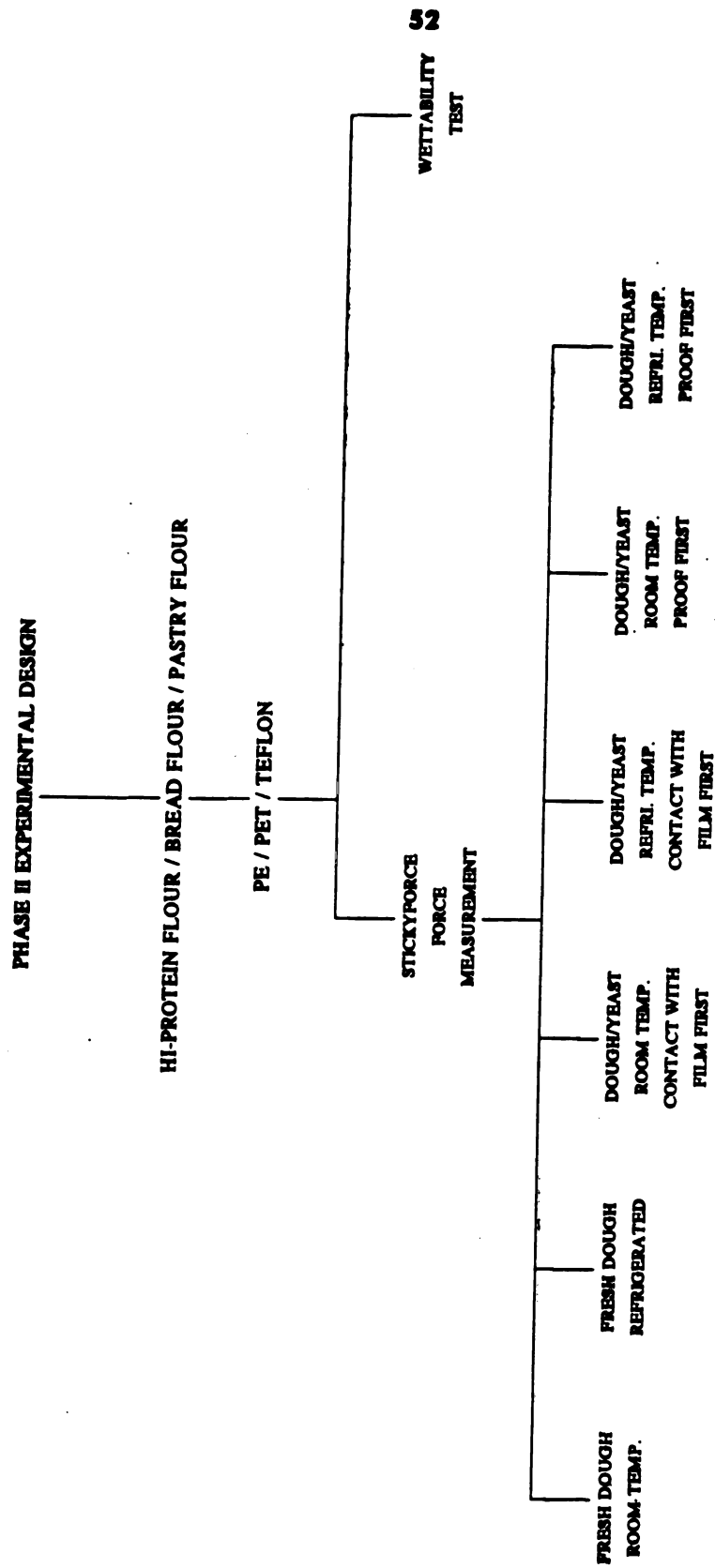
Studies were designed to characterize adhesion between plastic films and doughes of different flour types and protein levels (Fig. 4). Several plastic packaging materials were selected from Part 1 to provide a broad range of surface wettability.

Flour of different protein and starch contents were obtained and blended with water to form model systems. These were deposited onto the surface of selected packaging materials. The influence of protein concentration on adhesion was also determined. In addition, the flour protein was fractionated and studies of adhesion conducted with individual proteins.

Dough was also prepared at several protein concentrations and adhesion characterized for fresh dough, refrigerated, and proofed dough systems.

#### **Dough Preparation**

Three-hundred grams of flour were mixed in a Kitchen Aid K-5SS mixer (Hobart Corporation, Troy Ohio) at room temperature. Red Star quick-rise active dry yeast (1%) was



**Figure 4. Flow chart of Phase II study**

purchased from Food Stores at MSU (Red Star Yeast & Products., Milwaukee, Wisconsin) and was dissolved in distilled water. The amount of water used in making of the dough was determined from farinograph absorption. These ingredients were then mixed for three to seven minutes, depending upon the protein content.

#### **Treatment Variables**

E. Fresh dough was first put into a container, following refrigeration (4°C) for 6 hours. The dough was allowed to reach ambient temperature (23°C) and proofed to twice its original size (approximately four hours). Then it was placed in contact with the film surface prior to performing the Stickiforce Meter Measurement.

F. Fresh prepared dough (with yeast) was first allowed to proof to double its size (approximately two and half hours) at room temperature (23°C). Then it was placed in contact with the film surface for Stickiforce Meter Measurement.

G. Fresh prepared dough (with yeast) was placed in contact with film surface. Following refrigeration (4°C) for 6 hours, each sample was allowed to reach 23°C and proofed to twice its original size (approximately four hours) before performing the Stickiforce Meter Measurement.

H. Same as treatment G except that it was not refrigerated. Stickiforce Meter Measurement was performed after the dough was proofed to twice its original size

(approximately two and half hours).

I. Fresh prepared dough (no yeast) was placed in contact with film surface. Following refrigeration ( $4^{\circ}\text{C}$ ) for 4 hours, each sample was allowed to reach room temperature ( $23^{\circ}\text{C}$ ) before performing the Stickiforce Meter Measurement.

J. Same as treatment E except that it was not refrigerated. Stickiforce Meter Measurement was performed after the dough was made.

The six treatment combinations were arranged in a  $3 \times 2$  factorial design. The main effects were yeast condition (proofing), and temperature, as well as the interaction of these factors. Treatments served as the main plots, film as the sub-plots, and the flour type as the sub-subplots of a randomized complete block split-split plot design.

### **3.4 Statistics Analysis**

#### **3.4.1 Phase 1**

Statistical analyses were performed on a CompuAdd 212 computer utilizing MSTAT-C statistical packages (Microcomputer Program for the Design, Management, and Analysis of Agronomic Research Experiments). The two factor randomized complete block, split plot for data was computed using a MSTAT ANOVA procedure. Differences between means were determined using the Tukey's Honestly Significant Difference Test method.

### **3.4.2 Phase II**

All statistical analyses were performed on a CompuAdd 212 computer utilizing MSTAT-C statistical packages (Microcomputer Program for the Design, Management, and Analysis of Agronomic Research Experiments). The two factor randomized complete block split-split plot for data was computed using an MSTAT ANOVA procedure. Differences between means were determined using the Tuckey's Honestly Significant Difference Test method.

## **CHAPTER 4**

### **RESULTS AND DISCUSSION**

#### **4.1 Phase I study**

##### **4.1.1 Inclined Plane Determination**

Surface chemists use the term "wettability" or "wetting" to describe the degree of adhesive contact between liquid and solid. In this study, angle of slide was employed to measure the wettability of the film surface. Table 3 presents the results obtained.

The angle of slide ranged from 24.6 to 46.0 degrees for the surfaces studied. The smallest angle of slide required to move the water droplet was on the Teflon surface (24.6°). The small angle indicates poor wetting of the material by the water. On the other hand, the angle required for the water droplet to slide on the PETG surface was almost double that of the Teflon (46.0°).

##### **4.1.2. Stickiforce Meter Determination**

The dough was made of high protein wheat flour. Dough is a rather sticky and elastic substance once it is fully developed. Table 4 shows the effect of dough treatments on the weight-mass (Wtmass) gained (per unit contact area) as determined by the Stickiforce procedure. Treatment A

Table 3. Wettability of distilled water and 50% ethyl alcohol on different material surfaces (Phase I study).

<u>Sample</u>	<u>Distilled water</u> <u>Angle of slide</u>		<u>50% ethyl alcohol</u> <u>Angle of slide</u>	
	<u>Machine</u> <sup>*1</sup> <u>Direct.</u>	<u>Cross</u> <sup>*2</sup> <u>Direct.</u>	<u>Machine</u> <u>Direct.</u>	<u>Cross</u> <u>Direct.</u>
HDPE	41.8±2.04	41.4±2.15	34.4±2.45	34.2±1.60
PVC	39.8±3.32	41.5±2.68	28.6±1.85	34.0±1.60
PET/RELEASE AGENT	30.3±1.67	35.2±1.94	30.0±1.83	34.8±2.32
PETG (Polyethylene Terephthalate Glycol)	46.0±1.72	43.0±2.24	31.8±1.62	37.2±3.61
2% EVA/VEGETABLE OIL	26.0±0.63	29.4±6.95	42.4±1.36	39.0±3.72
2% EVA/1.5% PAM SPRAY	29.3±3.66	28.6±1.47	42.8±0.98	42.0±2.45
2% EVA MYV 9-40 RELEASE ADDED	23.0±2.28	20.0±0.63	26.4±1.02	30.4±1.20
KRAYTON	31.5±1.94	28.6±2.80	29.8±6.37	27.4±4.31
TRI-EXTRUDED PE HIGH SLIP	38.8±2.80	38.8±4.36	37.3±3.71	40.2±4.47
ISC-60 <sup>*3</sup>	35.4±1.36	37.4±1.94	22.8±2.28	26.6±2.14
TEFLON	24.6±1.02	22.5±0.80	21.5±1.67	22.5±1.33
SILICONE PAPER	38.0±1.20	37.3±1.20	22.3±0.98	25.5±0.80
80C146A <sup>*4</sup>	41.5±1.60	39.0±1.50	27.8±0.98	23.6±2.58
78G98 <sup>*5</sup>	40.8±1.20	45.5±2.50	22.3±0.80	28.3±1.36

<sup>\*1</sup> The direction of forward movement on the paper/plastic machine.

<sup>\*2</sup> The direction at right angles to the direction of running paper/plastic machine.

<sup>\*3</sup> 60 lb. Semi-bleached Silicone Coated Release Liner

<sup>\*4</sup> Silicone on Super-calendared Densified Kraft

<sup>\*5</sup> Silicone on Clay-calendared Densified Kraft

Table 4. The effect of treatment on the Weight-mass gain per unit ( $\text{gm}/\text{cm}^2$ ) determined by Stickiforce Meter

<u>Sample</u>	<u>TREATMENT</u> (Unit = $10^{-3}$ )			
	A*	B*	C*	D*
HDPE	71.90±34.63	0.31±0.32	1.52±0.63	0.50±0.35
PVC	102.71±59.03	7.21±4.36	0.95±0.35	0.50±0.33
PET/RELEASE AGENT	44.78±35.82	1.61±0.53	1.10±0.28	1.09±0.58
PETG	182.75±42.69	1.69±5.33	0.92±0.29	1.09±0.21
2% EVA/VEG OIL	11.11±12.29	1.23±0.48	0.57±0.19	0.26±0.53
2% EVA/1.5% PAM SPRAY	1.14±0.53	0.69±0.15	0.96±1.15	0.21±0.12
2% EVA/MYV 9-40	1.49±0.76	1.09±0.60	0.48±0.17	0.22±0.10
TRI-EXTRUDED PE	0.93±0.59	0.82±0.16	0.50±0.26	0.43±0.23
KRAYTON	2.20±0.77	1.30±0.23	0.84±0.09	0.56±0.16
ISC-60	122.94±256.78	7.92±2.95	5.03±2.26	2.53±2.01
TEFLON	1.57±0.36	0.82±0.17	0.33±0.08	0.17±0.12
SILICONE PAPER	85.45±161.97	4.83±1.95	3.45±0.76	2.14±0.80
80C146A	14.61±11.28	6.77±2.93	2.43±0.84	2.51±3.85
78G98	78.27±139.53	23.84±26.44	6.89±4.23	23.42±1.38

\*A. Dough was placed in contact with film surface. Refrigerated ( $4^{\circ}\text{C}$ ) for 24 hours, each sample was allowed to reach ambient temperature ( $23^{\circ}\text{C}$ ) and proofed to twice its original size before performing the test.

\*B. Dough was placed inside a plastic bag (LDPE), refrigerated for 24 hours. The dough was then placed into contact with the film surface, and proofed to double its size before performing the test.

\*C. Dough was placed in contact with film surface. Test was performed after the dough was proofed to twice its size at ambient temperature ( $23^{\circ}\text{C}$ ).

\*D. Dough was proofed first at room temperature ( $23^{\circ}\text{C}$ ) and then placed in contact with the film surface for the test.

samples (dough was placed in contact with film surface, refrigerated for 24 hours, allowed to reach room temperature and proofed to twice its original size before performing the test) had the highest Wtmass per unit area for all contact surfaces (Table 4). This suggests that cooling and proofing of the dough may enhance the sticking phenomenon. With Wtmass equal to  $0.15 \times 10^{-3}$ , Teflon was found to be the best surface for good release of dough. The smaller the angle the less the dough adhered to the film surface.

Weight-force (Wtforce) per unit area data obtained from the Stickiforce Meter is listed in Table 5. Wtforce results were correlated with the Wtmass results ( $R^2 = 7.24$ ). Treatment A had highest Wtforce values. This showed that treatment A produced the most dough adhesiveness. In general, Teflon and 2% EVA film with vegetable oil were found to have the least adhesiveness to dough (Inclined Plane method and Stickiforce Meter method). The films studied in Phase I are listed in Table 6 in order of their stickiness to dough. A ranking of 1 was considered to have the best release properties.

Adhesion of dough to a contact surface can sometimes be avoided by the use of 'adhesives', such as release agents. These are added to resins or used as coatings which are applied to a solid to prevent, or decrease, adhesion of another solid. For example, sample 5 (2% EVA) had a layer of vegetable oil on its surface, or sample 7 (2% EVA) had

Table 5. The effect of treatment on the Weight-force gain per unit ( $\text{gm}/\text{cm}^2$ ) determined by Stickiforce Meter

Sample	A*	B*	TREATMENT		D*
			C*		
HDPE	15.07±2.35	6.51±1.61	3.59±1.51		3.20±1.34
PVC	21.21±6.37	4.68±2.79	3.51±1.63		4.21±1.43
PET/RELEASE AGENT	19.13±6.35	6.40±2.97	5.74±1.36		4.18±1.41
PETG	25.89±4.88	4.28±2.07	5.94±1.54		4.76±1.04
2% EVA/VEG OIL	11.48±6.83	6.84±2.96	3.81±0.95		2.98±0.66
2% EVA/1.5% PAM SPRAY	5.04±1.95	3.42±1.00	3.60±0.71		3.39±1.58
2% EVA/MYV 9-40	4.20±1.29	2.32±1.23	3.00±1.22		2.64±0.91
TRI-EXTRUDED PE	4.02±2.65	3.32±0.84	3.16±0.81		2.93±0.53
KRAYTON	7.15±1.84	3.80±1.01	3.70±0.65		3.48±0.63
ISC-60	23.89±7.06	12.53±3.37	12.18±4.15		8.20±2.76
TEFLON	3.01±0.61	2.17±0.69	3.31±0.74		2.52±0.59
SILICONE PAPER	24.99±5.45	22.83±6.47	14.25±1.86		10.68±2.36
80C146A	16.60±5.78	16.44±4.91	10.28±2.51		7.89±1.64
78G98	28.19±4.78	12.96±3.02	13.42±1.75		10.50±1.73

\*A. Dough was placed in contact with film surface. Refrigerated ( $4^{\circ}\text{C}$ ) for 24 hours, each sample was allowed to reach ambient temperature ( $23^{\circ}\text{C}$ ) and proofed to twice its original size before performing the test.

\*B. Dough was placed inside a plastic bag (LDPE), refrigerated for 24 hours. The dough was then placed into contact with the film surface, and proofed to double its size before performing the test.

\*C. Dough was placed in contact with film surface. Test was performed after the dough was proofed to twice its size at ambient temperature ( $23^{\circ}\text{C}$ ).

\*D. Dough was proofed first at room temperature ( $23^{\circ}\text{C}$ ) and then placed in contact with the film surface for the test.

Table 6. Ranking of dough stickiness to films samples with different treatments, the rank of 1 was considered to have the best release properties.

Sample	<u>GRAM MASS</u> <u>TREATMENT</u>				<u>GRAM FORCE</u> <u>TREATMENT</u>			
	A*	B*	C*	D*	A*	B*	C*	D*
HDPE	9	1	10	6	7	9	5	5
PVC	12	12	7	7	10	7	4	9
PET/RELEASE AGENT	8	8	9	9	9	8	9	8
PETG	14	9	6	10	13	6	10	10
2% EVA/VEG OIL	6	6	4	4	6	10	8	4
2% EVA/1.5% PAM	2	2	8	2	4	4	6	6
2% EVA/MYV 9-40	3	5	2	3	3	2	1	2
TRI-EXTRUDED PE	1	4	3	5	2	3	2	3
KRAYTON	5	7	3	5	5	5	7	7
ISC-60 (SILICONE COATED)	13	13	13	13	11	11	12	12
TEFLON	4	3	1	1	1	1	3	1
SILICONE PAPER	11	10	12	11	12	14	14	14
80C146A (SILICONE COATED)	7	11	11	12	8	13	11	11
78G98 (SILICONE COATED)	10	14	14	14	14	12	13	13

\*A. Dough was placed in contact with film surface. Refrigerated for 24 hours, each sample was allowed to reach ambient temperature (23°C) and proofed to twice its original size before performing the test.

\*B. Dough was placed inside a plastic bag (LDPE), refrigerated for 24 hours. The dough was then placed into contact with the film surface, and proofed to double its size before performing the test.

\*C. Dough was placed in contact with film surface. Test was performed after the dough was proofed to twice its size at ambient temperature (23°C).

\*D. Dough was proofed first at room temperature (23°C) and then placed in contact with the film surface for the test.

MYV 9-40 release agent added to its surface. To minimize adhesion of dough to its container, there should be a high degree of wetting of the contact surface with oil or a release agent and a low degree of wetting of the oil surface in contact with the water and dough.

#### **4.1.3 Wtforce/Wtmass Results**

The effects of temperature, yeast condition and film on Wtforce and Wtmass were studied. Mean squares from the analyses of variance of Wtforce and Wtmass values are given in Table 7 and 8. Each film has its own characteristics and wettability. A small angle of slide indicates poor wettability of the material. Angle of slide varied significantly among films with different coating materials - silicone release agent, pam spray, vegetable oil, and MYV 9-40 - and different surface polarities). The type of film had significant effect on both measures of Wtmass ( $F = 2.190$ ,  $p = 0.019$ ) and Wtforce ( $F = 69.605$ ,  $p = 0.000$ ).

#### **Wtmass**

Tukey's Honestly Significant Difference test result of Wtmass (Appendix B, Table 15) indicates that there was no significant difference among all films. Tukey's method provides for the comparison of any or all pairs of treatment means after observation of the data with a probability of falsely rejecting the hypothesis of equality on at least one comparison equal to a probability  $\alpha$ . It's a much more conservative method compared to other methods (Mendenhall,

Table 7 Analysis of variance for Wtforce values of 14 different films under different yeast conditions at various temperature.

Source of variation	Degree of freedom	Sum of squares	Mean Square	F values	P
Film (F)	13	8200.200	630.785	69.6046	0.0000**
Replic (F)	70	684.676	9.781	1.0793	0.3752
Yeast (Y)	1	1524.532	1524.532	168.2262	0.0000**
FY	13	1044.181	80.322	8.8632	0.0000**
Error	70	634.368	9.062		
Temp. (T)	1	2651.489	2651.489	312.6570	0.0000**
FT	13	1282.947	98.688	11.6371	0.0000**
YT	1	745.479	745.479	87.9051	0.0000**
FYT	13	974.941	74.995	8.8433	0.0000**
Error	140	1187.271			
Total	335				

\*\* represent  $p < 0.01$

Table 8 Analysis of variance for Wtmass values of 14 different films under different yeast conditions at various temperature.

Source of variation	Degree of freedom	Sum of squares	Mean Square	F values	P
Film (F)	13	0.073	0.006	2.1900	0.0188*
Replic (F)	70	0.182	0.003	1.0793	0.4674
Yeast (Y)	1	0.048	0.048	18.9208	0.0000**
FY	13	0.061	0.005	1.8434	0.0529
Error	70	0.179	0.003		
Temp. (T)	1	0.059	0.059	23.3396	0.0000**
FT	13	0.066	0.005	2.0173	0.0234*
YT	1	0.045	0.045	18.0728	0.0000**
FYT	13	0.060	0.005	1.8406	0.0424*
Error	140	0.352	0.003		
Total	335				

\* represent  $p < 0.05$   
 \*\* represent  $p < 0.01$

1968). This might explain why it may contradict the ANOVA results.

When a contact-first-yeast dough starts to proof, the air bubbles formed from inside the dough gradually separate the dough from the film. When dough develops to double its original size, the dough on the top of the film becomes loose, and easier to pull from the film. Proof-first-yeast dough was developed and fermented. It was then put onto the film so that the bonding between dough was tighter than contact-first-yeast dough.

The interaction between film type and yeast condition was not significantly different ( $F = 1.843$ ,  $p = 0.053$ ) for Wtmass (Fig. 5). As the figure shows, Wtmass values of proof-first-yeast dough were higher than those of contact-first-yeast dough, and PETG/Proof-first had the highest Wtmass among all tested. For the films PETG, ISC-60, PVC, and silicone paper, the differences between proof-first and contact-first yeast dough were greater than the rest of the films. There were only slight differences between proof-first and contact-first yeast doughs for: EVA film with different release agent, krayton, Teflon, and tri-extruded PE. However, Tukey's more conservative Honestly Significant Difference test result of Wtmass values (Appendix B, Table 16, Fig. 6) indicates that there were no significant differences for film and yeast interaction.

Temperature has significant effect on dough stickiness.

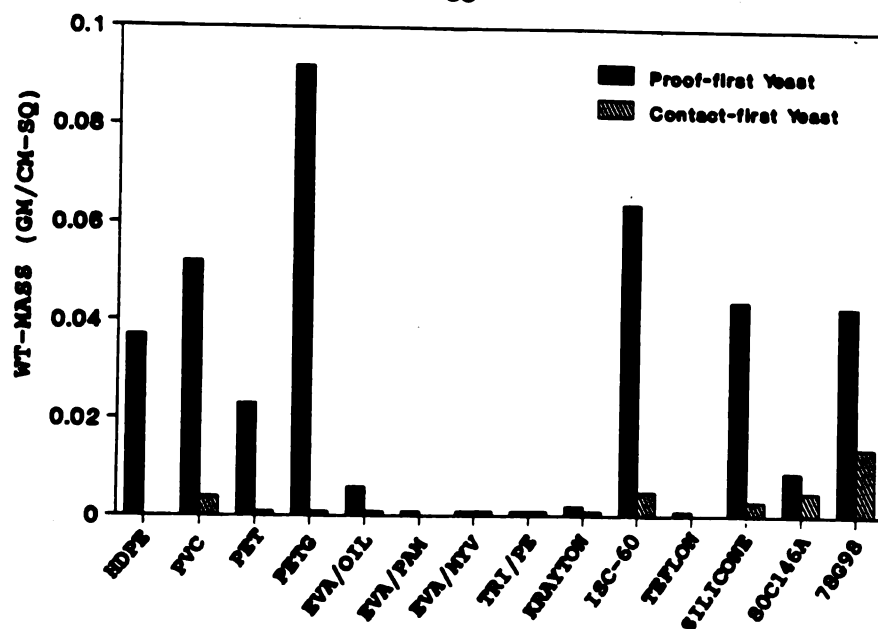


Figure 5. The Wtmass values of proof-first and contact-first yeast dough on various films.

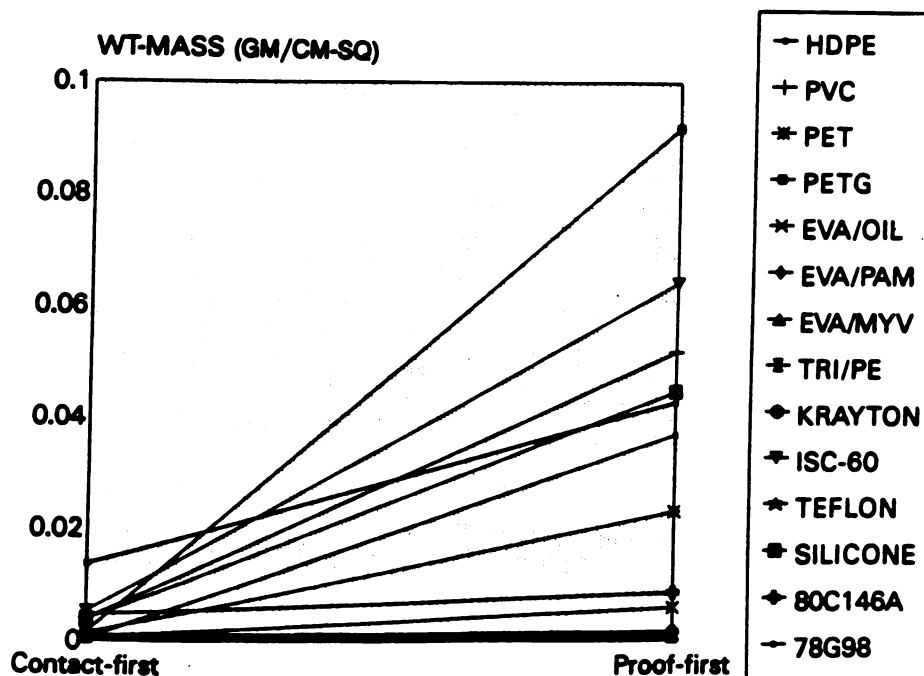


Figure 6. Tukey's HSD test Wtmass mean values of various types of material with proof-first and contact-first yeast.

Significant differences were found between refrigerated dough and room temperature dough for Wtmass ( $F = 23.340$ ,  $p = 0.000$ ). After the dough was mixed at room temperature ( $23^{\circ}\text{C}$ ), it was then put in the refrigerator ( $4^{\circ}\text{C}$ ) for 24 hours. When the dough was taken out of the refrigerator it may have become drier. When the dough was removed from the refrigerator and left at room temperature, sweat appeared on the surface of the dough, the cold surface causing condensation to occur. This condensation maybe at least partially why the dough was more sticky.

The effect of film type varied between the two temperature conditions. The Wtmass values at refrigerated temperature are higher than those at room temperature (Fig. 7). Figure 7 shows that PETG at refrigerated temperature has the highest Wtmass value of all films tested. Silicone coated materials (i.e., silicone release paper, ISC-60, 80C146A, and 78G98), PETG, PVC, HDPE, and PET films had the next highest values. However, Tukey's Honestly Significant Difference test results of Wtmass values (Appendix B, Table 17, Fig. 8) indicated that effect of film type did not vary by temperature.

The effect of yeast (proofing condition) on Wtmass ( $F = 18.073$ ,  $p = 0.000$ ) also depended on temperature (Fig. 9). The mean values of Wtmass at refrigerated temperature ( $4^{\circ}\text{C}$ ) were higher than those at room temperature ( $23^{\circ}\text{C}$ ), and the means of Wtmass of proof-first-yeast dough were higher than

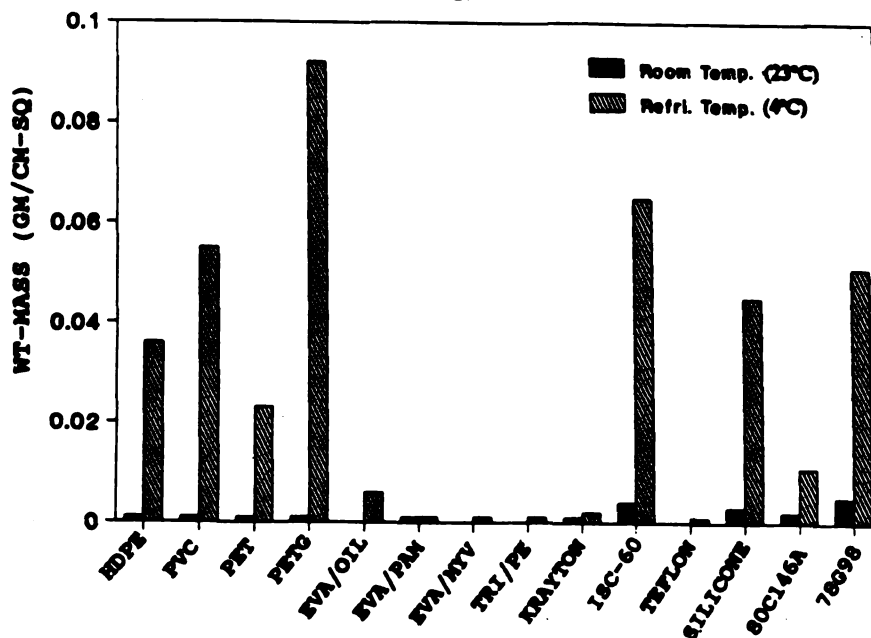


Figure 7. The Wtmass values of room and refrigerated temperature dough on various films.

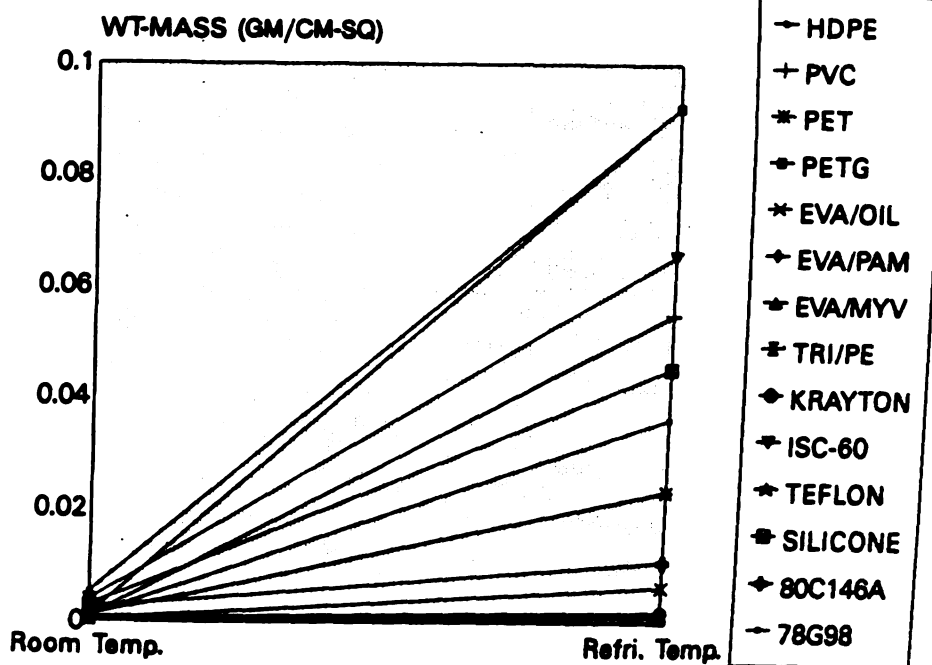


Figure 8. Tukey's HSD test Wtmass mean values of various types of material at room and refrigerated temperature.

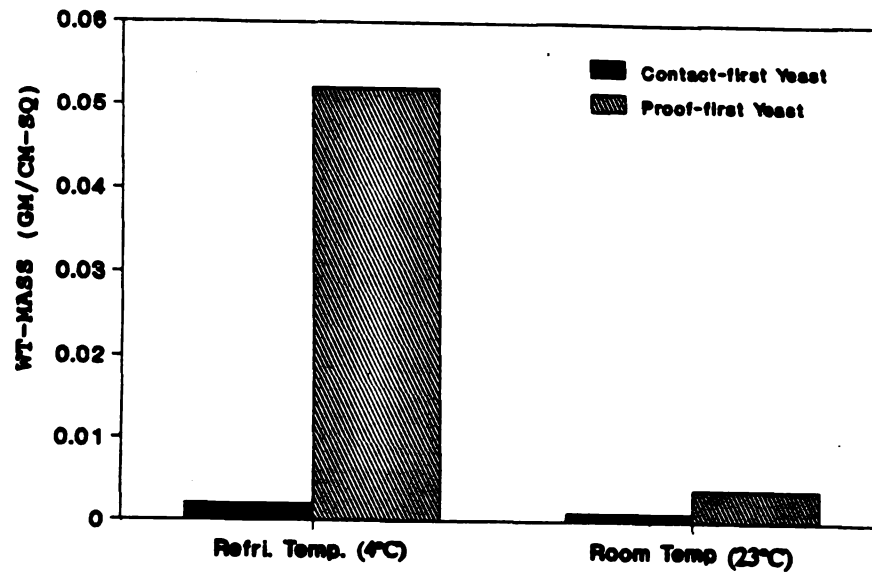


Figure 9. The Wtmass values of proof-first and contact-first yeast dough at room and refrigerated temperature.

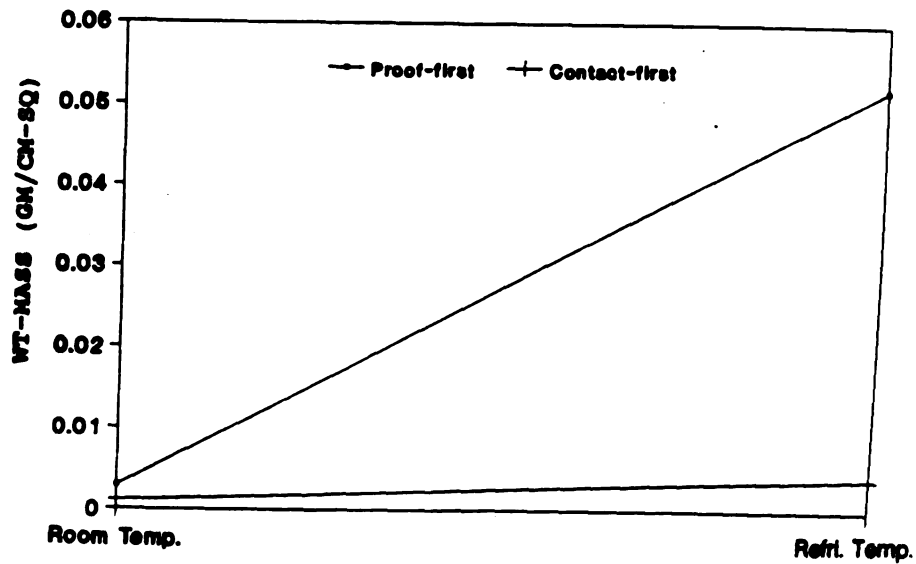


Figure 10. Tukey's HSD test Wtmass mean values of proof-first and contact-first yeast at room and refrigerated temperature.

those of contact-first-yeast dough. Tukey's Honestly Significant Difference test results of Wtmass (Appendix B, Table 18, Fig. 10) indicates that there was no significant difference for temperature and yeast interaction.

A three-way interaction of film, yeast, and temperature was significantly different ( $F = 1.841$ ,  $p = 0.042$ ) for Wtmass. This three-way interaction is illustrated in Figure 11, and 12. Dough stickiness varied when either yeast condition, temperature, or film type changed. Different films have different wetting characteristics because of difference surface polarities. Yeast condition and the change of temperature caused variation in the stickiness.

In general, stickiness was higher for dough at refrigerated temperature ( $4^{\circ}\text{C}$ ) than for dough at room temperature ( $23^{\circ}\text{C}$ ), and stickiness was higher with proof-first-yeast dough than with contact/first-yeast dough. However, there was an exception. The Wtmass value for 80C146A (silicone on super-calendared densified kraft) for contact-first-yeast dough was higher than those for proof-first-yeast dough at room temperature. The Wtmass values at refrigerated temperature with proof-first-yeast dough are higher than those at room temperature with contact-first-yeast dough. Proof-first-yeast dough on PETG at refrigerated temperature ( $4^{\circ}\text{C}$ ) had the highest Wtmass value among all film samples, and contact-first-yeast dough on Teflon at room temperature ( $23^{\circ}\text{C}$ ) had the lowest. Tukey's

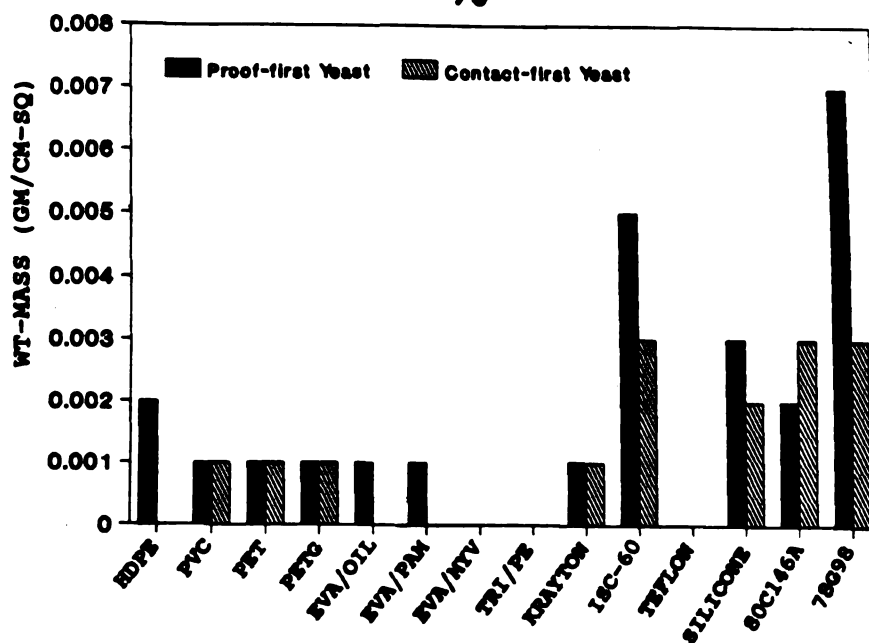


Figure 11. The Wtmass values of proof-first and contact-first yeast dough at room temperature (23°C).

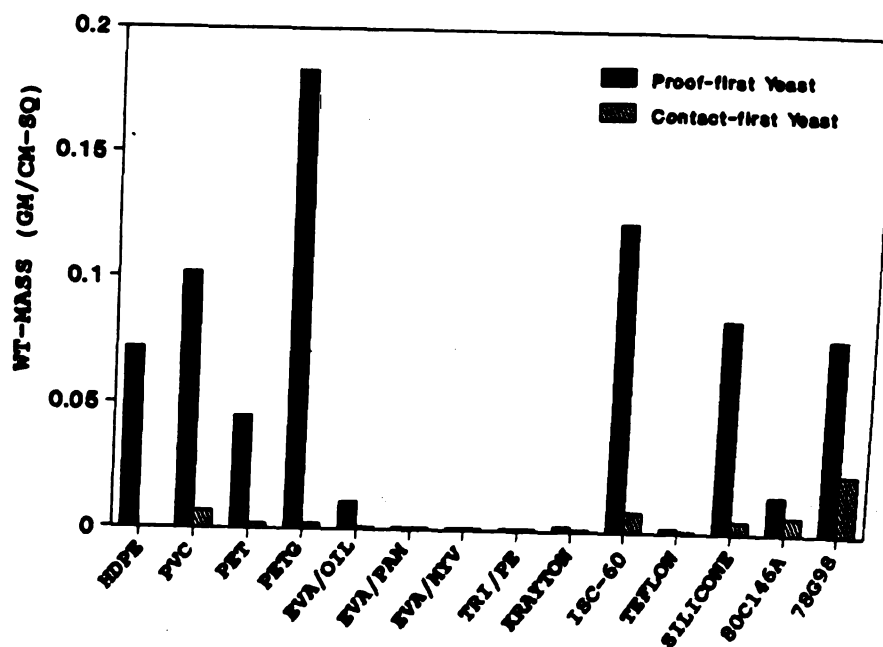


Figure 12. The Wtmass values of proof-first and contact-first yeast dough at refrigerated temperature (4°C).

Honestly Significant Difference test results of Wtmass values are listed in Appendix B, Table 19.

Proof-first and contact-first yeast dough had the same amount of yeast and the same fermentation time, but appear to yield totally different results. The reason could be that after contact-first-yeast dough reached full maturity, the web structure reveals thin-walled gluten strands that were dry and mellow and offered minimum resistance to stretching when pulled. Proof-first-yeast dough reached a fully fermented stage, and was then put in contact with the films and the test (Stickiforce Meter) was performed immediately. Thus, there wasn't any opportunity to bring about a relaxation of the stresses created within the dough by the mixer action. Apparently, the lack of a second fermentation period resulted in these stresses not being released. This may be the reason why proof-first-yeast dough proved stickier than contact-first-yeast dough.

#### **Wtforce**

The type of film has significant effect on dough stickiness. Significant differences were found among all films for Wtforce ( $F = 69.605$ ,  $p = 0.0000$ ). Tukey's Honestly Significant Difference test result of Wtforce (Appendix B, Table 20) indicated that there was no significant difference among HDPE, PVC, PET, and PETG; no significant difference among PETG, 2% EVA/OIL, 2% EVA/PAM, 2% EVA/MYV, Tri-extruded PE, Krayton, and ISC-60; and there

was no significant difference among 2% EVA/MYV, Tri-extruded PE, Krayton, ISC-60, Teflon FEP, Silicone released paper, 80C146A, and 78G98.

The interaction between film type and yeast condition was highly significant ( $F = 168.226$ ,  $p = 0.000$ ) for Wtforce (Fig. 13). The effect of film type or yeast conditions on the Wtforce value is dependant on each other. As figure 13 shows, Wtforce values of proof-first-yeast dough were higher than those of contact-first-yeast dough. Wtforce values for 78G98/Proof, PETG/Proof, ISC-60/Proof, and Silicone released paper/Proof were higher than the rest. Apparently, the silicone release agent didn't function properly.

Tukey's Honestly Significant Difference Test Result of Wtforce values (Appendix B, Table 21, Fig. 14) indicated that there was no significant difference among 78G98/Proof, PETG/Proof, Silicone/Proof, ISC-60/Proof, Silicone/Contact, and 80C146A/Proof, no significant difference among PETG/Proof, 80C146A/Proof, PET/Proof, PVC/Proof, 80C146A/Contact, 78G98/Contact, ISC-60/Contact, and HDPE/Proof. Also there was no significant difference among EVA/OIL/Proof, Krayton/Proof, PET/Contact, EVA/OIL/Contact, HDPE/Contact, PETG/Contact, PVC/Contact, EVA/PAM/Proof, Krayton/Contact, EVA/MYV/Proof, Teflon/Proof, Tri/PE/Contact, EVA/MYV/Contact, and Teflon/Contact.

The materials (Table 1) can be categorized into four groups. The EVA films with different coating materials

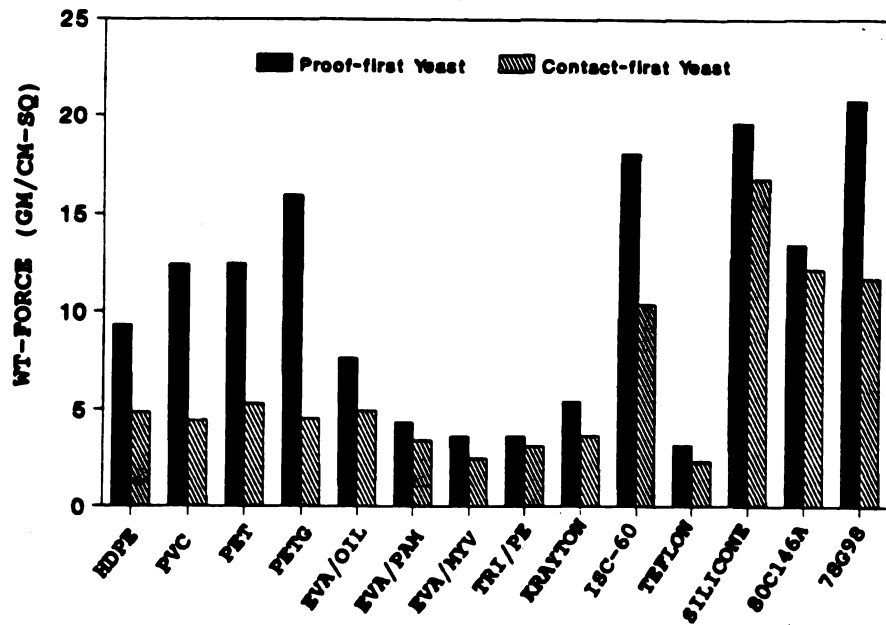


Figure 13. The Wtforce values of proof-first and contact-first yeast dough on various films.

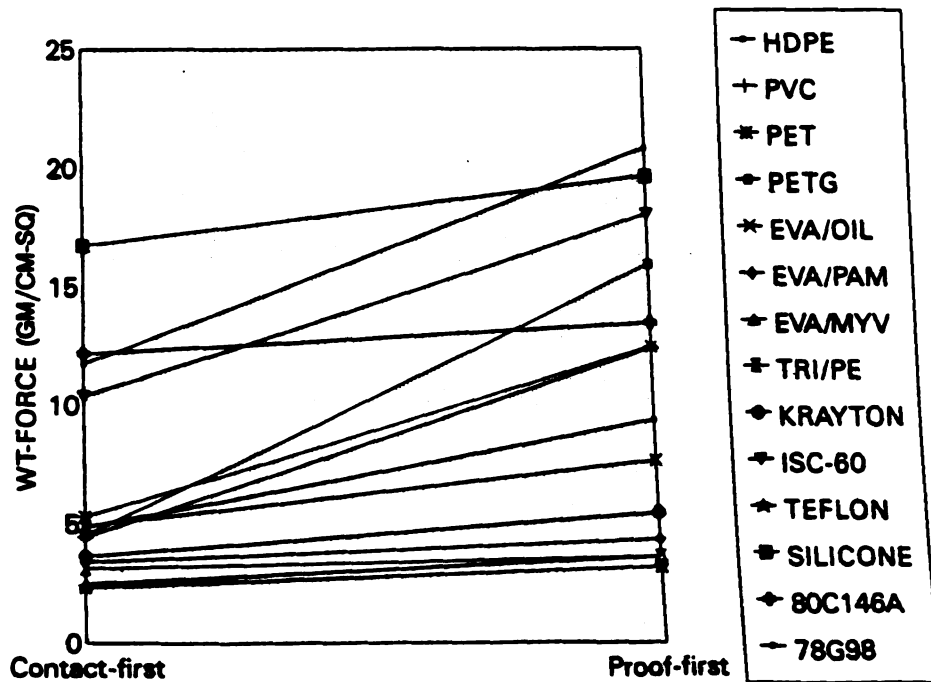


Figure 14. Tukey's HSD test Wtforce mean values of various types of material with proof-first and contact-first yeast.

(coated with vegetable oil, 1.5% pam spray, and myv 9-40), Teflon, Krayton, and tri-extruded PE high slip film as good release materials, PET with release agent as medium release material, silicone release agent coated materials (silicone released liner, silicone on super-calendared densified kraft, and silicone on clay-calendared densified kraft), and HDPE, PVC, and PETG as poor release materials.

MYV 9-40 is a grouping of food emulsifiers, dough strengtheners, and softeners, coatings. It consists of texturing/aerating agents, and lubricants, made of edible lard. Liquid MYV 9-40 materials are used to stabilize the viscosity of chocolate-flavored syrups. MYV 9-40 can provide a bland, edible, protective coating on food products to improve their shelf life, texture, appearance, and stability. They are waxy rather than greasy, and have extremely good oxidative stability and good oxygen and moisture barrier properties. When a 0.25 percent level of MYV 9-40 was sprayed on dates and raisins, it reduced their stickiness for easier handling in automatic equipment. Also these agents can be sprayed on food as surface lubricants and on processing equipment for use as a lubricant or release agent (Eastman Chemicals, 1986).

Silicone is widely used as a release coating on plastic film label stock. Small amounts of specific silicones are used as internal and external mold release agents, process aids, and flame retardants in thermoplastics (Bafford,

1987). In our study, the materials coated with silicone release agents were quite sticky compared with other groups of materials, and the reason is unknown.

PVC, PET, and PETG all have very polar surfaces. According to Young (1805), surface tensions exist at the phase boundaries of a drop of liquid at rest on a solid surface (Fig. 1). If the molecules that make up the surface are more polar, the surface is easier to wet and bond with a polar liquid adhesive. PET film was treated with release agent to decrease its stickiness. HDPE film is not as polar as PVC and PETG.

Temperature has significant effect on dough stickiness. Significant differences were found between refrigerated dough and room temperature dough for Wtforce ( $F = 312.657$ ,  $p = 0.000$ ). After the dough was mixed at room temperature ( $23^{\circ}\text{C}$ ), it was then put in the refrigerator ( $4^{\circ}\text{C}$ ) for 24 hours. As a result, when the dough was taken out of the refrigerator it may have been drier than when it was put into the refrigerator. When the dough was removed from the refrigerator and left at room temperature, sweat appeared on the surface of the cold dough, causing condensation to occur. This condensation may have been the reason why the dough was more sticky.

The effect of film type varied between the two temperature conditions. The Wtforce values at refrigerated temperature were higher than those at room temperature (Fig.

15). Figure 14 shows that 60 lb. semi-bleached densified kraft film (ISC-60) at refrigerated temperature had the highest value among all tested. Tukey's Honestly Significant Difference test results of Wtforce values (Appendix B, Table 22, Fig. 16) indicated that temperature had different effects on the different films.

The effect of yeast condition on Wtforce ( $F = 7.905$ ,  $p = 0.000$ ) also depended on temperature (Fig. 17). The mean values of Wtforce at refrigerated temperature ( $4^{\circ}\text{C}$ ) were higher than those at room temperature ( $23^{\circ}\text{C}$ ). The means of Wtforce values for proof-first-yeast dough were higher than those of contact-first-yeast dough. Tukey's Honestly Significant Difference test results of Wtforce (Appendix B, Table 23, Fig. 18) indicated that proof-first-yeast dough at refrigerated temperature differed significantly from others.

A three-way interaction of film, yeast, and temperature was highly significant ( $F = 8.843$ ,  $p = 0.000$ ) for Wtforce. This three-way interaction is illustrated in Figure 19 and 20. Dough stickiness varied when either yeast condition, temperature, or film type changed. As discussed above, different films have different wetting characteristics. Stickiness varied with yeast condition and the change in temperature.

In general, stickiness values were higher with proof-first-yeast dough than with contact/first-yeast dough. Stickiness values were higher when dough was stored at

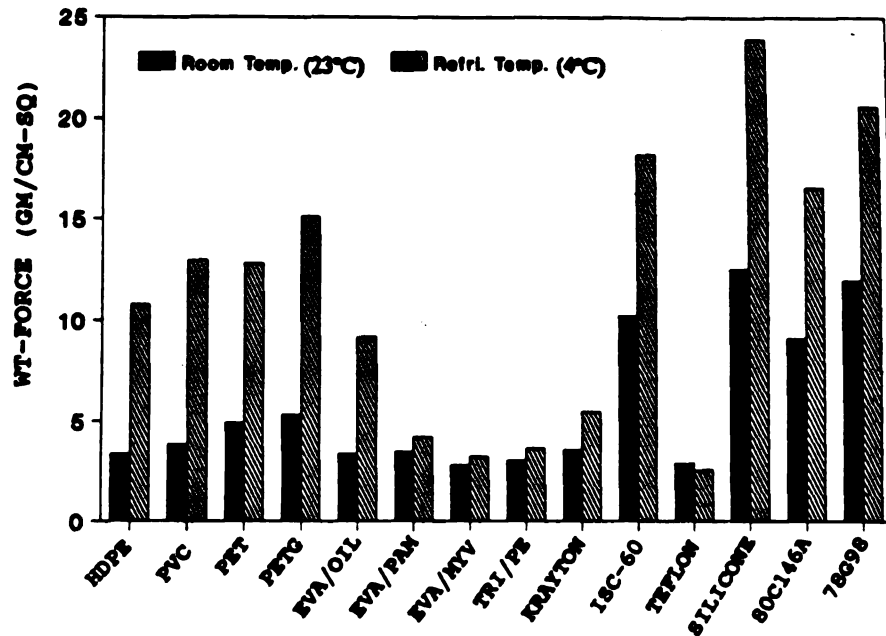


Figure 15. The Wtforce values of room and refrigerated temperature dough on various films.

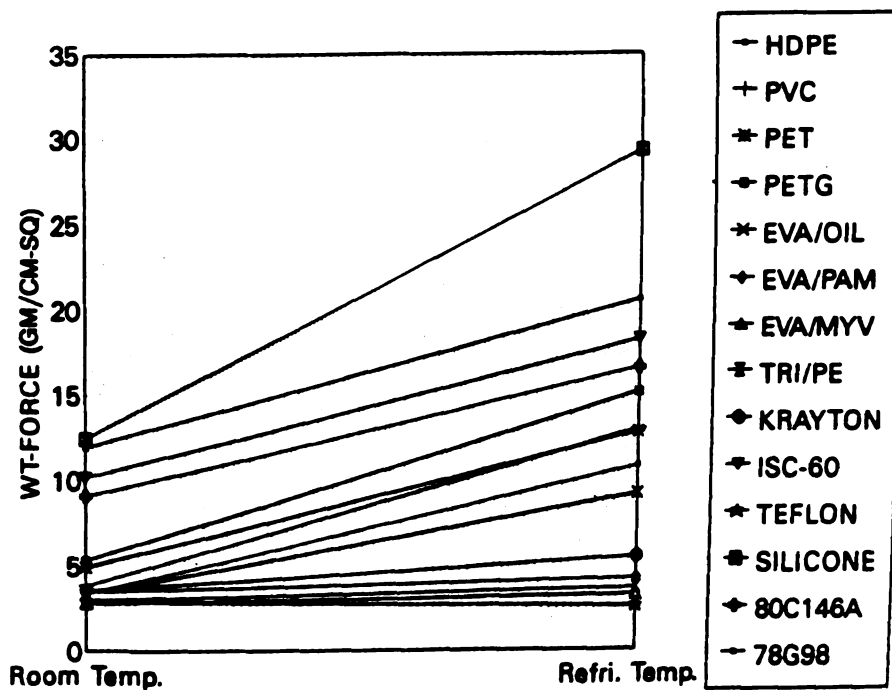


Figure 16. Tukey's HSD test Wtforce mean values of various types of material at room and refrigerated temperature.

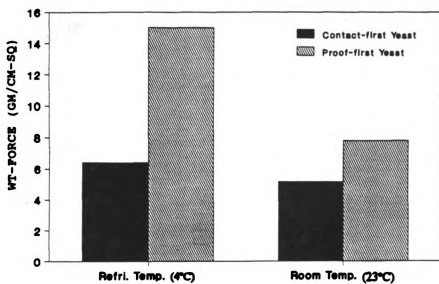


Figure 17. The Wtforce values of proof-first and contact-first dough at room and refrigerated temperature.

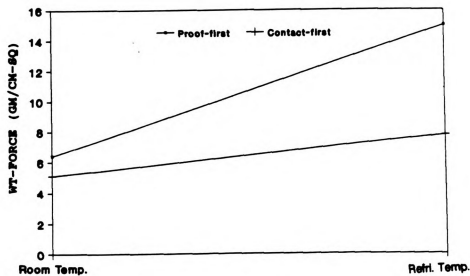


Figure 18. Tukey's HSD test Wtforce values of proof-first and contact-first yeast dough at room and refrigerated temperature.

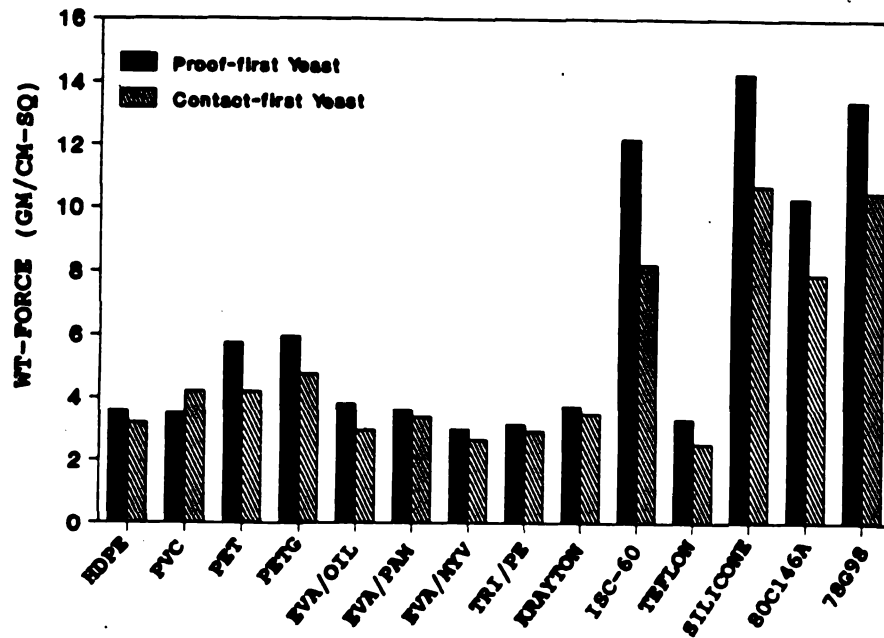


Figure 19. The Wtforce values of proof-first and contact-first yeast dough at room temperature (23°C).

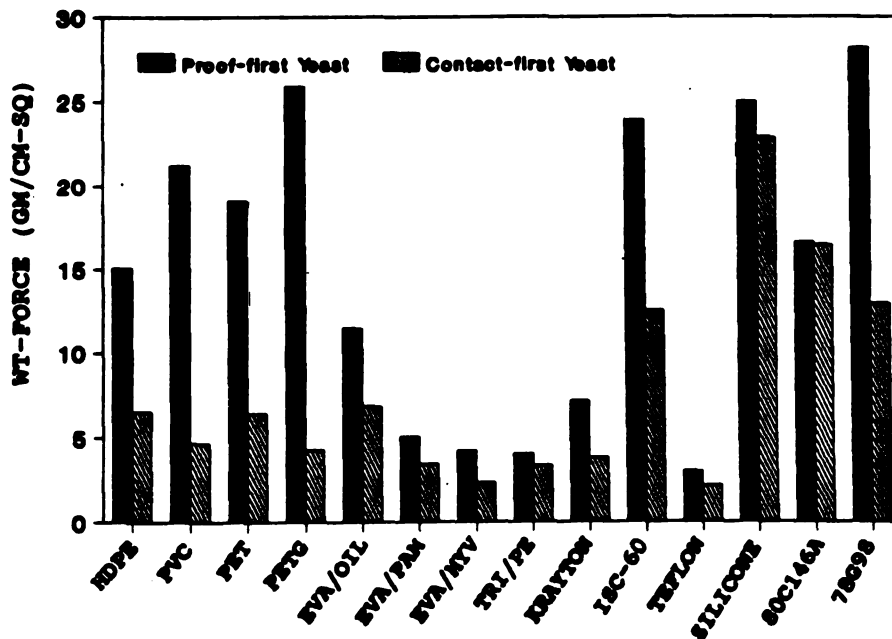


Figure 20. The Wtforce values of proof-first and contact-first yeast dough at refrigerated temperature (4°C).

refrigerated temperatures than when dough was stored at room temperatures. However, there were a few exceptions. For PVC, the Wtforce value for contact-first-yeast dough was higher than those for proof-first-yeast dough. The Wtforce values at refrigerated temperature (4°C) with proof-first-yeast dough were higher than those at room temperature (23°C) with contact-first-yeast dough. Proof-first-yeast dough on 78G98 at refrigerated temperatures had the highest Wtforce values and contact-first-yeast dough on Teflon at refrigerated temperature had the lowest Wtforce values among all tested. Comparing the Wtforce mean values with different factors, silicone coated materials had the highest Wtforce mean values. Tukey's Honestly Significant Difference test results of Wtforce values are listed in Appendix B, Table 24.

#### **4.2. Phase II study**

##### **4.2.1. Inclined Plane Determination**

In this study, the wettability of the film surface was also determined using a modified, inclined plane apparatus. Table 9 presents the results obtained for the different film systems. The resultant angle of slide for the surfaces studied ranged from 25.1 to 33.8 degrees. Teflon film had the smallest angle of slide (25.1°). This indicates poor wetting of the material. The PET surface had the largest angle of slide (33.8°).

Table 9. Wettability of distilled water and 50% ethyl alcohol on different material surfaces.

<u>Material Sample</u>	<u>Distilled water</u> <u>Angle of slide</u>		<u>50% ethyl alcohol</u> <u>Angle of slide</u>	
	<u>Machine</u> <u>Direct.</u>	<u>Cross</u> <u>Direct.</u>	<u>Machine</u> <u>Direct.</u>	<u>Cross</u> <u>Direct.</u>
LDPE	29.9±1.92	29.8±2.04	24.9±2.26	26.3±2.93
PET	33.8±1.94	32.9±2.26	32.3±2.14	32.0±3.07
TEFLON	25.1±2.46	25.7±1.40	22.3±0.87	23.9±1.28

#### 4.2.2 % Protein in Flours

Doughes of different protein concentrations were prepared by mixing water with flours of different protein contents. The amount of protein in the three different flours is shown in Table 10.

Based on the results of the Farinograph measurements, water (Appendix A) was mixed into the flour systems to optimize dough consistency. Dough mixing time was also optimized using the results of the Farinograph evaluation. Doughes from each of the different flour mixtures were then applied to the three test films (Teflon, PET, and LDPE) and tested for Wtforce and Wtmass as previously described.

#### 4.2.3. Stickiforce Meter Determination

The doughes were made of Hi-protein, bread, and pastry flour. Table 11 shows the effect of dough treatments on the

Table 10. The percentage protein in pastry, bread and Hi-protein flours - determined using Microkjeldahl method.

<u>Flour Type</u>	<u>% Protein</u>
Pastry Flour	8.76
Bread Flour	13.05
Hi-protein Flour	14.81

Table 11. The effect of treatment on the Weight-mass gain per unit ( $\text{gm}/\text{cm}^2$ ) determined by Stickiforce Meter

<u>TREATMENT</u>	<u>FILM SAMPLE</u> (Unit = $10^{-1}$ )		
	<u>LDPE</u>	<u>PET</u>	<u>TEFLON</u>
E*	2.16 $\pm$ 1.24	3.19 $\pm$ 0.71	0.20 $\pm$ 0.42
F*	2.19 $\pm$ 1.20	2.96 $\pm$ 0.73	0.29 $\pm$ 0.27
G*	0.94 $\pm$ 1.32	1.72 $\pm$ 0.48	0.22 $\pm$ 0.26
H*	2.28 $\pm$ 1.25	2.04 $\pm$ 2.27	0.37 $\pm$ 0.20
I*	6.38 $\pm$ 1.52	3.95 $\pm$ 1.13	2.09 $\pm$ 0.63
J*	6.83 $\pm$ 1.13	4.27 $\pm$ 0.97	2.53 $\pm$ 0.53

E\*. Dough was put into a container, refrigerated ( $4^{\circ}\text{C}$ ), then was allowed to reach room temperature ( $23^{\circ}\text{C}$ ) and proofed to double its original size. Then it was placed in contact with the film surface prior to the test.

F\*. Dough (with yeast) was first proof to double its size at room temperature. Then it was placed in contact with the film surface prior for test.

G\*. Dough (with yeast) was placed in contact with film surface. Refrigerated, then each sample was allowed to reach  $23^{\circ}\text{C}$  and proofed to double its size prior to the test.

H\*. Dough (with yeast) was placed in contact with film at room temperature. Test is performed after the dough was proofed to twice its original size.

I\*. Dough (no yeast) was placed in contact with film surface. refrigerated for 4 hours, then was allowed to reach room temperature before performing the test.

J\*. Dough (no yeast) was placed in contact with film surface. Test was performed after the dough was made.

weight-mass gained (per unit contact area) as determined by the stickiforce procedure. Treatment J (no-yeast dough contact with film surface, test was performed after the dough was made) had the highest weight-mass values for all contact surfaces. This suggests that dough made without yeast and held in contact under refrigerated temperature (4°C) had enhanced adhesion.

The fully hydrated protein of a mixed and kneaded dough forms a veil-like film over the external surface of starch granules. The fractured surface of the inside of the dough exposes many cleaved starch granules embedded in a protein matrix with numerous microscopic holes (Christianson, 1975). After fermentation, this protein lattice structure shows larger air cells. Many of the small air cells enmesh minute starch granules within them. The veil-like protein coating on the surface of the starch granules, stretches and rolls up into fibrils due mainly to an increase in the size of the air cells. This may have caused the yeast doughes to be less sticky than the no-yeast doughes.

Wtforce per unit area data obtained from the Stickiforce Meter is shown in Table 12. For both PET and Teflon film, treatment J had the highest Wtforce per unit area studied for all the contact surfaces. However, for PE film, treatment I (no-yeast dough contact with film, refrigerated, equilibrated to 23°C) had the highest Wtforce values.

Table 12. The effect of treatment on the Weight-force gain per unit ( $\text{gm}/\text{cm}^2$ ) determined by Stickiforce Meter

<u>TREATMENT</u>	<u>FILM SAMPLE</u>		
	<u>PE</u>	<u>PET</u>	<u>TEFLON</u>
E*	34.93 $\pm$ 8.54	35.12 $\pm$ 8.08	20.73 $\pm$ 7.37
F*	34.93 $\pm$ 10.98	34.39 $\pm$ 11.57	20.18 $\pm$ 7.95
G*	14.69 $\pm$ 7.67	17.72 $\pm$ 7.27	10.56 $\pm$ 8.58
H*	20.85 $\pm$ 9.52	20.06 $\pm$ 10.60	12.67 $\pm$ 6.01
I*	53.24 $\pm$ 9.31	49.86 $\pm$ 7.33	37.39 $\pm$ 5.93
J*	50.18 $\pm$ 6.47	50.07 $\pm$ 6.35	38.05 $\pm$ 5.46

E\*. Dough was put into a container, refrigerated ( $4^{\circ}\text{C}$ ), then was allowed to reach room temperature ( $23^{\circ}\text{C}$ ) and proofed to double its original size. Then it was placed in contact with the film surface prior to the test.

F\*. Dough (with yeast) was first proof to double its size at room temperature. Then it was placed in contact with the film surface prior for test.

G\*. Dough (with yeast) was placed in contact with film surface. Refrigerated, then each sample was allowed to reach  $23^{\circ}\text{C}$  and proofed to double its size prior to the test.

H\*. Dough (with yeast) was placed in contact with film at room temperature. Test is performed after the dough was proofed to twice its original size.

I\*. Dough (no yeast) was placed in contact with film surface. refrigerated for 4 hours, then was allowed to reach room temperature before performing the test.

J\*. Dough (no yeast) was placed in contact with film surface. Test was performed after the dough was made.

Dough treatment had a definite impact on Wtforce values. For all three flours, proofed-yeast dough had less adhesion. Pastry flour doughes were less adhesive. With pastry doughes, Teflon had better release properties. The Wtmass values also show better release for proofed-yeast dough systems. Pastry doughes had less adhesion. Dough release from Teflon was generally better than from PET and LDPE. The temperatures used to age the dough did not result in different adhesion strengths.

#### **4.2.4 Wtforce/Wtmass Results**

The effect of flour type, yeast, temperature, and film type on Wtforce and Wtmass were studied. Analyses of variance results for the Wtforce and Wtmass values are given in Table 13 and 14. All four factors had significant effect on Wtmass. All factors, except for temperature, had significant effects on Wtforce.

#### **Wtmass**

According to Tukey's Honestly Significant Difference test results for yeast conditions, there was no significant difference between contact-first-yeast and proof-first-yeast dough mean values (Appendix C, Table 25). There was no significant difference between Hi-protein and bread flour mean values (Appendix C, Table 26). When considering the flour factors, both were significantly different ( $p < 0.05$ ) from pastry flour. According to the test results, the protein content may have an effect on the stickiness of the

**Table 13 Analysis of variance for Wtforce values of 3 different types of flours under three different yeast conditions at various temperature on 3 different films**

Source of variation	Degree of freedom	Sum of squares	Mean Square	F values	P
Yeast (Y)	2	49930.570	24965.285	1587.6636	0.0000**
Flour (F)	2	38076.142	19038.071	1210.7233	0.0000**
YF	4	8661.255	2165.314	137.7028	0.0000**
Replic (YF)	45	3911.319	86.918	5.5276	0.0000**
Temp. (T)	1	51.229	51.229	3.2579	0.0778
YT	2	306.707	153.353	9.7525	0.0000**
FT	2	136.557	68.278	4.3422	0.0189*
YFT	4	177.790	44.448	2.8266	0.0356*
Error	45	707.604	15.725		
Film (M)	2	9373.635	4686.818	203.4749	0.0000**
YM	4	886.085	221.521	9.6172	0.0000**
FM	4	562.259	140.565	6.1025	0.0001**
YFM	8	1065.557	133.195	5.7826	0.0000**
TM	2	2.586	1.293	0.0561	
YTM	4	167.499	41.875	1.8180	0.1273
FTM	4	7.269	1.817	0.0789	
YFTM	8	172.960	21.620	0.9386	
Error	180	4146.100	23.034		

Total 323

\* represent  $p < 0.05$

\*\* represent  $p < 0.01$

Table 14 Analysis of variance for Wtmass values of 3 different types of flours under three different yeast conditions at various temperature on 3 different films

Source of variation	Degree of freedom	Sum of squares	Mean Square	F values	Probability
Yeast (Y)	2	5.787	2.894	352.2237	< 0.0000**
Flour (F)	2	1.493	0.747	90.8741	< 0.0000**
YF	4	2.124	0.531	64.6488	< 0.0000**
Replic (YF)	45	0.277	0.006	0.7489	
Temp. (T)	1	0.084	0.084	10.2407	< 0.0025**
YT	2	0.057	0.029	3.4913	< 0.0389*
FT	2	0.031	0.016	1.9105	> 0.1598
YFT	4	0.013	0.003	0.3841	
Error	45	0.370	0.008		
Film (M)	2	3.889	1.944	350.4445	< 0.0000**
YM	4	1.479	0.370	66.6561	< 0.0000**
FM	4	0.660	0.165	29.7597	< 0.0000**
YFM	8	1.844	0.231	41.5487	< 0.0000**
TM	2	0.035	0.017	3.1331	< 0.0460*
YTM	4	0.047	0.012	2.1335	> 0.0785
FTM	4	0.012	0.003	0.5544	
YFTM	8	0.039	0.005	0.8765	
Error	180	0.999	0.006		

Total 323

\* represent  $p < 0.05$

\*\* represent  $p < 0.01$

tested doughes. Noguchi (1976) suggested that dough stickiness correlated highly with the sulfhydryl content of the protein, but protein content itself did not correlate with adhesiveness. Protein content largely determines the grain's suitability for its intended end use.

Yeast exists and is active in air as well as in the absence of air, but its behavior will change according to the environment (Vallery-Radot, 1957). In the presence of air, yeast grow rapidly and produce little alcohol, while in the absence of air, yeast growth is slow but alcohol formation is favored. During yeast fermentation, glycerol and succinic acid will also be produced, as well as carbon dioxide.

Yeast provides flavoring compounds, affects the texture of dough and baked product, and creates carbon dioxide which decreases the density of the food (Matz, 1960). Carbon dioxide passes through the yeast cell wall as a dissolved compound, probably in the form of a bicarbonate ion. As the concentration of carbon dioxide increases in the free liquid outside the cell, gas bubbles begin to form around foci in the dough. The formation and migration of carbon dioxide in a network of cellular compartments, occupy about 120 cubic inches per pound of loaf, and serves to lighten or raise the dough. The physical properties of the dough are altered through the powerful stretching actions generated by diffusion and accumulation of carbon dioxide throughout the

dough mass.

The effect of yeast condition on Wtmass depended on the flour ( $F = 90.874$ ,  $p = 0.000$ , Fig. 21). Pastry flour (with yeast) had higher mean values than bread flour (with yeast), perhaps because the cohesion forces within pastry flour is lower than the adhesion forces to films, so the remaining dough on the film will be higher. When there is no yeast in the dough, bread flour is most sticky.

Tukey's Honestly Significant Difference Test result of Wtmass (Fig. 22, Appendix C, Table 27) showed that the effect of yeast condition varied for different types of flours. For Wtmass measures, values were highest when flours had no yeast. When flours had proof-first-yeast, both values were second highest. And when flour had contact-first-yeast, both values were the lowest. These differences appeared stronger for Hi-protein and bread flours.

There was a highly significant ( $p < 0.01$ ) difference between yeast condition and temperature for Wtmass ( $F = 3.491$ ,  $p = 0.0389$ ) (Fig. 23). The effect of yeast condition depended on the effect of temperature for Wtmass values. Mean values at refrigerated temperature were higher than those at room temperature for contact-first-yeast and no-yeast dough. Figure 23 shows that the proof-first-yeast dough at room temperature had higher values than those at refrigerated temperature. Tukey's Honestly Significant

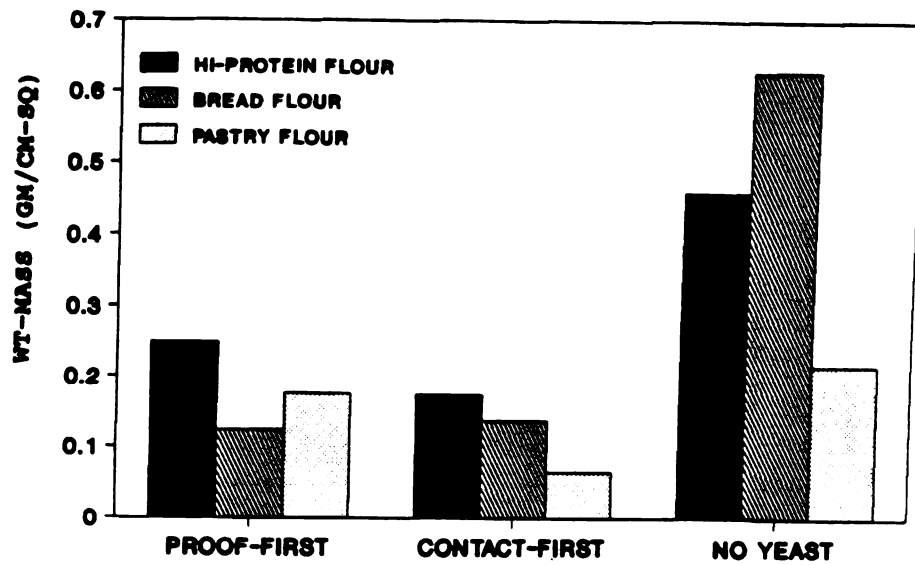


Figure 21. The Wtmass values of proof-first, contact-first, and no-yeast dough with HI-protein, bread, and pastry flour.

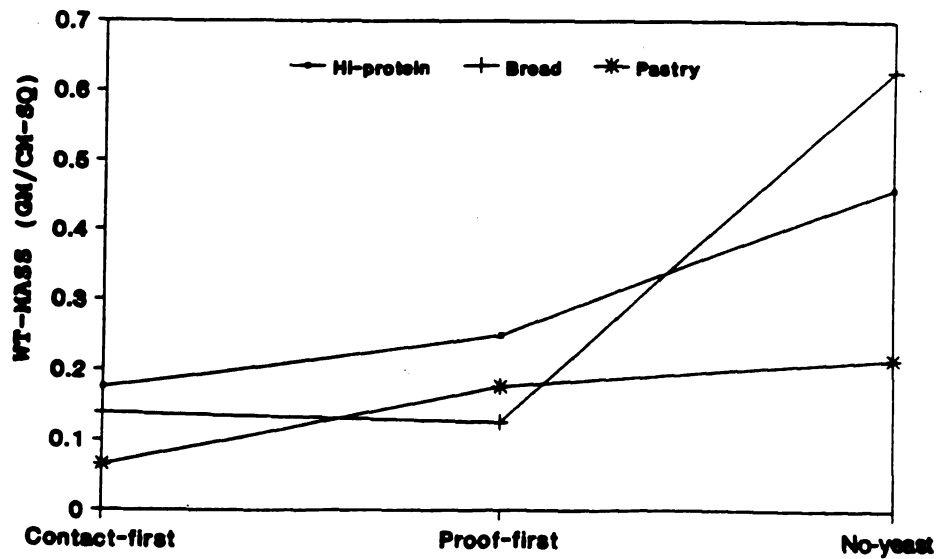


Figure 22. Tukey's HSD test Wtmass mean values of proof-first, contact-first, and no-yeast dough with HI-protein, bread and pastry flour.

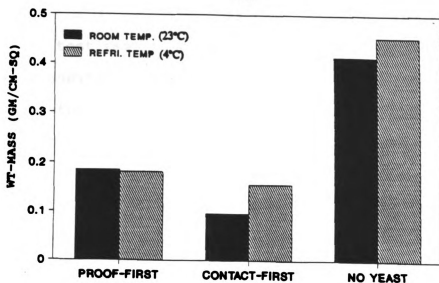


Figure 23. The Wtmass values of proof-first, contact-first, and no-yeast dough with room and refrigerated temperature.

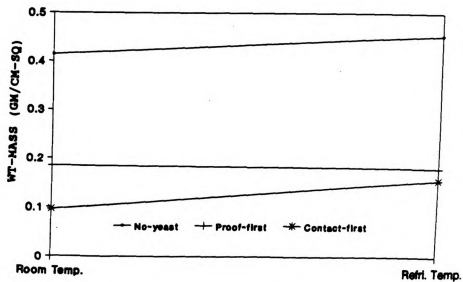


Figure 24. Tukey's HSD test Wtmass mean values of proof-first, contact-first and no-yeast dough at room and refrigerated temperature.

Difference test results of Wtmass indicated that the effect of yeast condition varied under different temperatures. However, within each yeast condition the effect of temperature was not significant (Fig. 24, Appendix C, Table 28).

The effect of flour type on Wtmass ( $F = 3.491$ ,  $p = 0.0389$ ) depended on temperature (Fig 25). Tukey's Honestly Significant Difference test results (Fig. 26, Appendix C, Table 29) showed how the effect of temperature condition varied for different types of flours. Higher values occurred for the flour at refrigerated temperature. These differences appeared stronger for Hi-protein and bread flours. When the dough was put in the refrigerator, the protein structure may have changed, and the low temperature will slow down the yeast fermentation. These factors may make the dough more sticky.

A three-way interaction of yeast, flour, and temperature was not significant for Wtmass. This three-way interaction is illustrated in Figures 27 and 28. Figure 27 shows that at room temperature, bread flour with no-yeast dough had the highest mean value, and pastry flour with contact-first-yeast dough had the lowest mean value.

Figure 28 shows that bread flour with no-yeast dough had the highest mean value, and pastry flour with contact-first-yeast dough had the lowest mean value. For both room and refrigerated temperature, bread flour with proof-first-

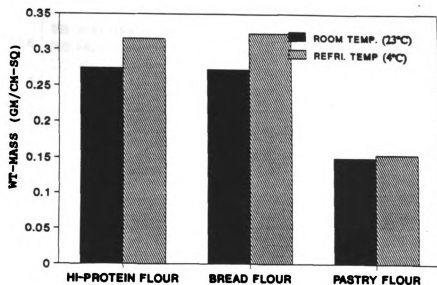


Figure 25. The Wt-mass values of HI-protein, bread, and pastry flour dough at room and refrigerated temperature.

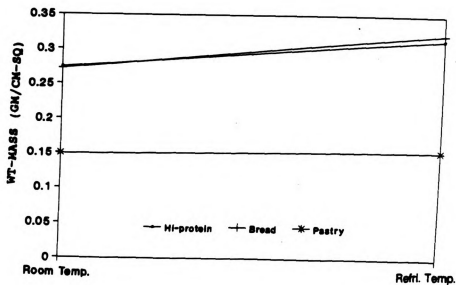


Figure 26. Tukey's HSD test Wt-mass mean values of HI-protein, bread, and pastry flour at room and refrigerated temperature.

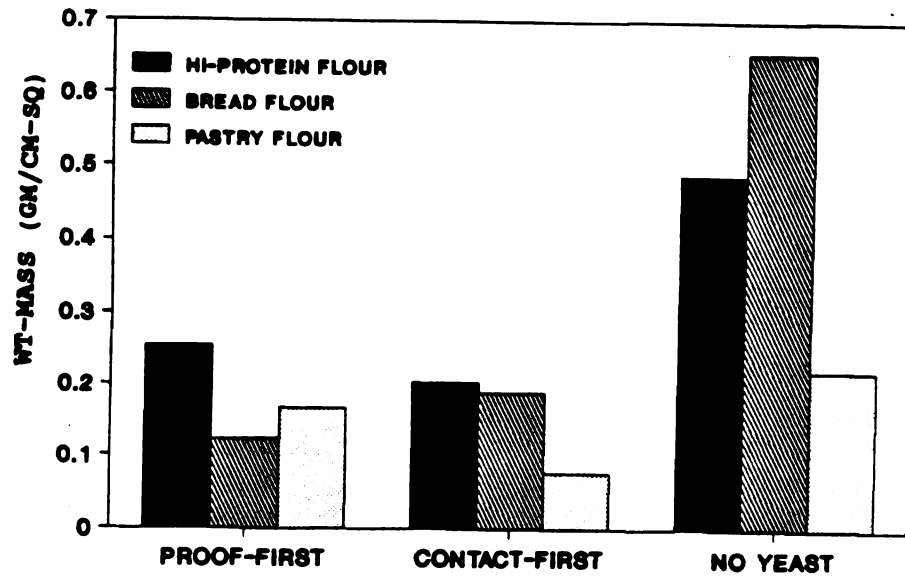


Figure 27. The Wtness values of HI-protein, bread, and pastry flour with proof-first, contact-first and no-yeast dough at room (23°C) temperature.

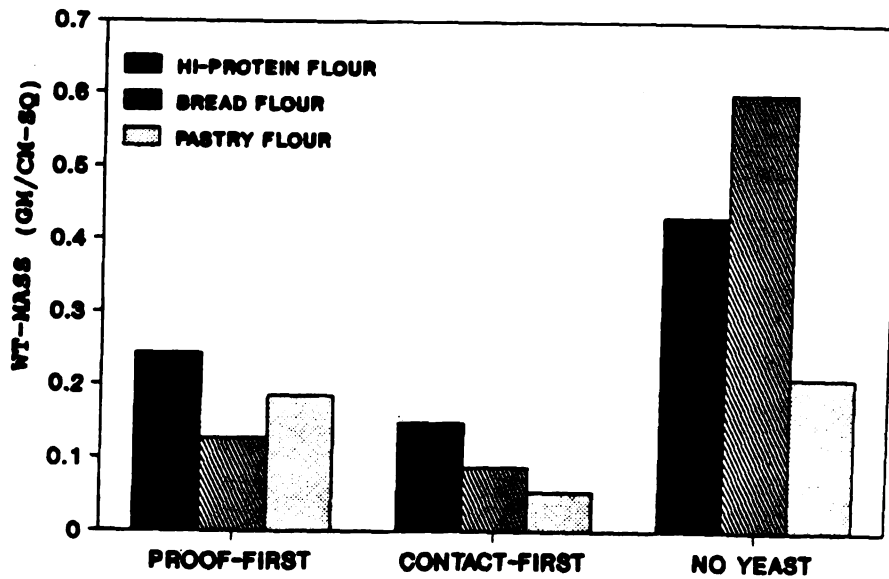


Figure 28. The Wtness values of HI-protein, bread, and pastry flour with proof-first, contact-first and no-yeast dough at refrigerated (4°C) temperature.

yeast was lower than contact-first-yeast dough. The mean value of pastry flour at room temperature was higher than flours at refrigerated temperature. The same situation occurred with Hi-protein and bread flour with proof-first-yeast dough. The Tukey's Honestly Significant Difference test results of Wtmass values are listed in Appendix C, Table 30.

The effect of film type on Wtmass ( $F = 350.445$ ,  $p = 0.0000$ ) was highly significant among all films. Tukey's Honestly Significant Difference test indicated that there was no significant difference between PE and PET film, but both films were significantly different ( $p < 0.05$ ) from Teflon film (Appendix C, Table 31). This may be due to Teflon film's superior anti-stick/low friction properties (Dupont, 1988).

The effect of yeast condition on Wtmass varied for the different films ( $F = 350.445$ ,  $p = 0.0000$ ). The mean values of Teflon film were the lowest of all (Fig. 29). For both proof-first-yeast and contact-first-yeast dough, the mean values of PET film were higher than those of the PE films, but for no-yeast dough, the mean value of the PE film was higher than PET film. Tukey's Honestly Significant Difference test indicated that there was no significant difference between PE and PET film for proof-first-yeast and contact-first-yeast dough (Fig. 30, Appendix C, Table 32). For no-yeast dough, there was a significant difference

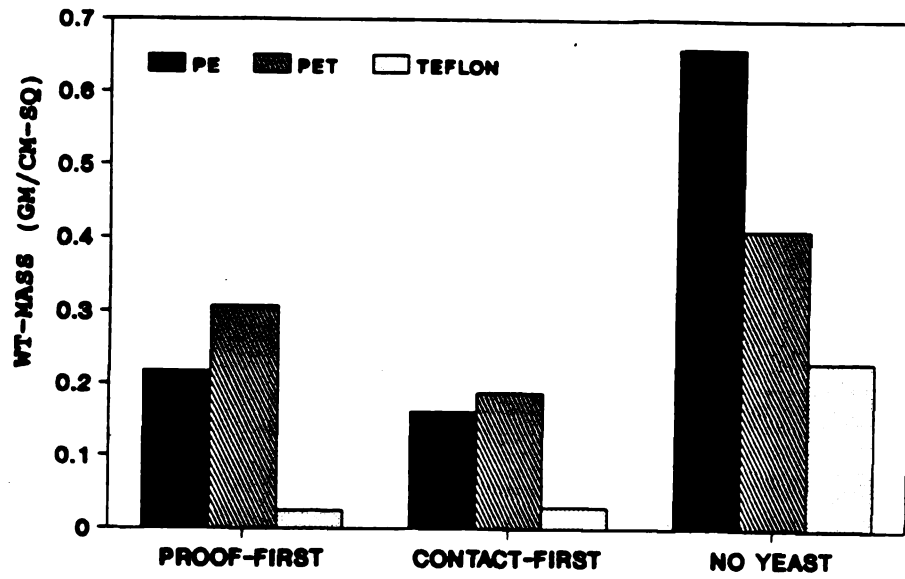


Figure 29. The Wt-mass values of proof-first, contact-first, and no-yeast dough on PE, PET, and TEFLON film.

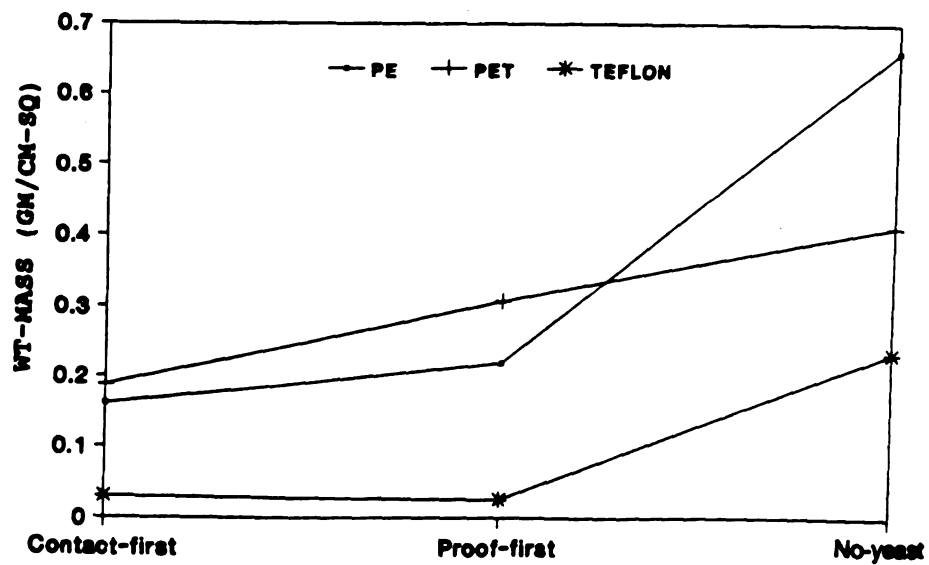


Figure 30. Tukey's HSD test Wt-mass mean values of proof-first, contact-first, and no-yeast dough on PE, PET, and TEFLON film.

between PE and PET film. PE film was slightly less sticky than PET film with yeast dough, however, with no-yeast, PE film was much more stickier than PET film.

The interaction between flour and film was highly significant on Wtmass ( $F = 29.760$ ,  $p = 0.0000$ ). The Teflon film had the lowest mean values of all. The mean value for the PET film was higher than the PE film (Fig. 31), and the mean value of PE film with bread flour was the highest. Tukey's Honestly Significant Difference test results of Wtmass (Fig. 32, Appendix C, Table 33) indicates that there was no significant difference between Hi-protein and bread flour with PE or PET film, and no significant difference between Hi-protein and bread flour with Teflon film. There was no significant difference between PE and PET with the pastry flour.

A three-way interaction of yeast, flour, and film was highly significant for Wtmass ( $F = 41.549$ ,  $p = 0.0000$ ). This three-way interaction is illustrated in Figures 33, 34, and 35. Stickiness depended on yeast condition, flour, and type of film.

Figure 33 shows that the mean values of proof-first-yeast dough on PET film were higher than those of PE and Teflon film for all three flours. Figure 34 shows that the mean value of contact-first-yeast on PET film was higher than those of PE film with bread and pastry flours, but with Hi-protein flour, the mean value of PE film was higher than

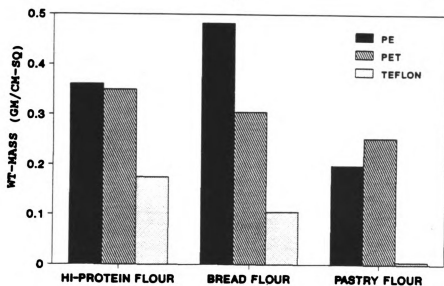


Figure 31. The Wtmass values of HI-protein, bread, and pastry flour on PE, PET, and TEFLON film.

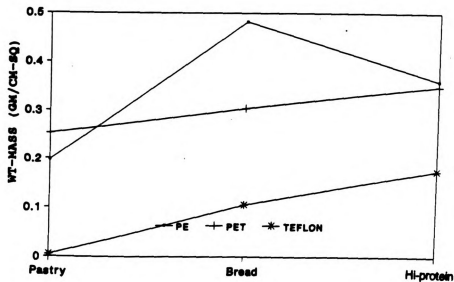


Figure 32. Tukey's HSD test Wtmass mean values of HI-protein, bread, and pastry flour on PE, PET, and TEFLON film.

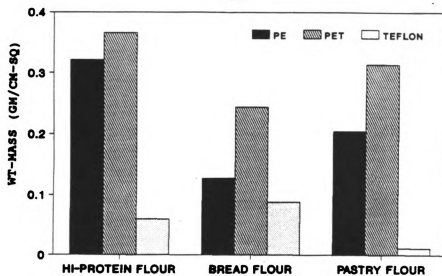


Figure 33. The Wtmass value of Hi-protein, bread, and pastry flour on PE, PET, and TEFLON film with proof-first-yeast dough.

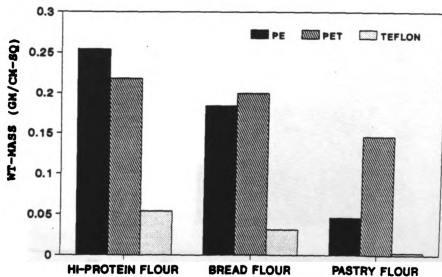


Figure 34. The Wtmass value of Hi-protein, bread, and pastry flour on PE, PET, and TEFLON film with contact-first-yeast dough.

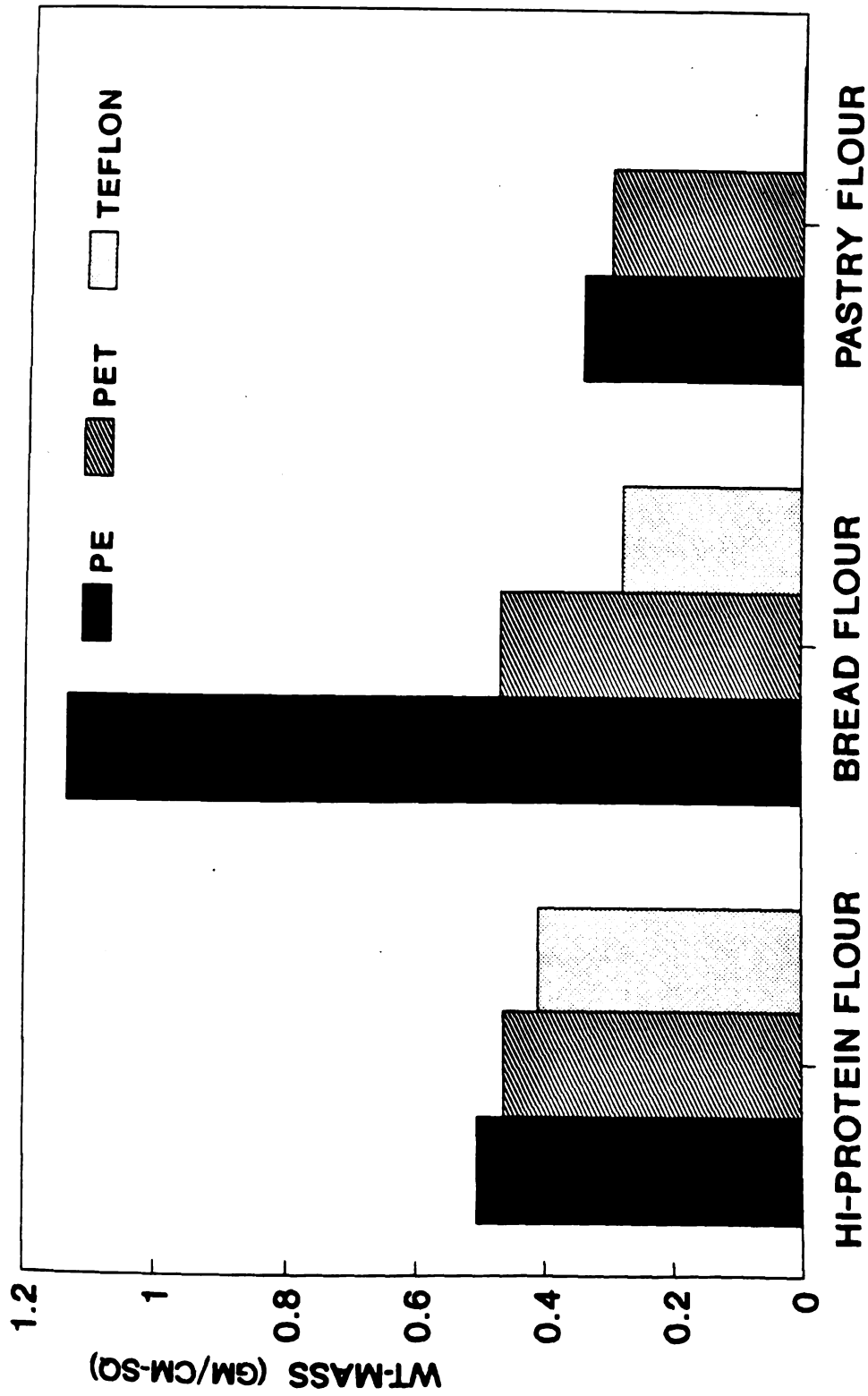


Figure 35. The Wt mass value of HI-protein, bread, and pastry flour on PE, PET, and TEFLON film with no-yeast dough.

PET film. Teflon film with pastry flour had the lowest mean value of all. Figure 35 shows that for all three flours, the mean values of PE film were higher than the mean values of PET film. These differences appeared stronger for bread flour. Tukey's Honestly Significant Difference test results of Wtmass values are listed in Appendix C, Table 34.

The interaction between temperature and film type was significant ( $F = 3.133$ ,  $p = 0.0460$ ) for Wtmass (Fig. 36). Under refrigerated temperature, Wtmass values were higher than those at room temperature. Tukey's Honestly Significant Difference test indicated that Teflon film was less sticky than both PE and PET film. Temperature had no effect on all three films for Wtmass (Fig. 37, Appendix C, Table 35).

The flour samples used were Hi-protein, bread, and pastry flour. According to the manufacturing company (General Mill, Inc., 1991), Hi-protein flour was milled from a hard spring wheat, bread flour was milled from hard winter wheat, and pastry flour was milled from hard and soft winter wheat blend.

Hard wheat is physically hard, and has relatively high protein content. The hardness is under genetic control and is thought to result from the strength of the bonding between the protein and starch in the endosperm (Hoseney, 1978). Bonding between protein and starch is weak for soft wheat. Soft wheat is low in protein. Also, dough from a

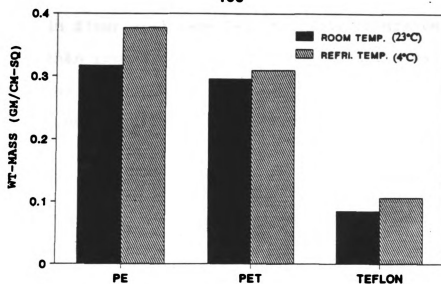


Figure 36. The Wt-mass values of room and refrigerated temperature dough on PE, PET, and TEFLON film.

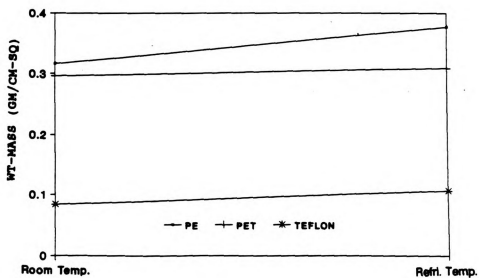


Figure 37. Tukey's HSD test Wt-mass mean values of room and refrigerated temperature dough with PE, PET, and TEFLON film.

high protein flour will have less mobility or greater stiffness than from a low protein flour with the same percentage of absorption. This may explain why the different types of flour react differently.

### **Wtforce**

According to Tukey's Honestly Significant Difference test results, there was no significant difference between proof-first-yeast and no-yeast dough for Wtforce values (Appendix C, Table 36). There was a significant difference ( $p < 0.05$ ) between Hi-protein, bread, and pastry flour for Wtforce values (Appendix C, Table 37).

The effect of yeast condition depended on the flour type for Wtforce ( $F = 137.703$ ,  $p = 0.000$ , Fig. 38). Contact-first-yeast dough had lower Wtforce mean values than proof-first-yeast dough. Pastry flour had the lowest mean values among all flours, bread flour was most sticky.

Tukey's Honestly Significant Difference Test result of Wtforce (Fig. 39, Appendix C, Table 38) showed that the effect of yeast condition varied for different type flours. Values were highest when flours had no yeast. When flours had proof-first-yeast, both measures (Wtmass and Wtforce) were second highest. And when flour had contact-first-yeast, both measures had the lowest values. These differences appeared stronger for Hi-protein and bread flours.

All gluten proteins contain disulfide groups, each

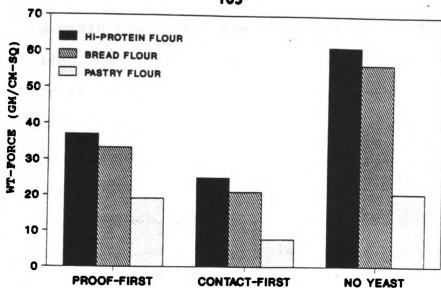


Figure 38. The Wtforce values of HI-protein, bread, and pastry flour with proof-first, contact-first, and no-yeast dough.

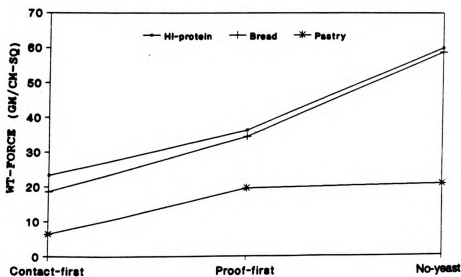


Figure 39. Tukey's HSD test Wtforce mean values of HI-protein, bread and pastry flour with proof-first, contact-first, and no-yeast dough.

polypeptide chain has an average of two (Wall, 1967).

Although gliadin and glutenin proteins possess similar amino acids, their physical behaviors differ; hydrated gliadin is a viscous mass, whereas hydrated glutenin is cohesive and elastic. In the hydration stage during dough formation, water penetrates the protein particles and associated with polar sites, overcomes forces that cause the molecules to adhere. Some of the protein molecules may be cleaved at the disulfide bonds and reassembled by oxidation to conform more effectively to the mixing stress. During proofing, the protein matrix is stretched. The ability to stretch depends upon the elastic characteristics of the gluten protein.

Yeast brings about changes in the dough in the course of fermentation. This includes depletion of fermentable substances, accumulation of waste products in the form of carbon dioxide, alcohols, acids and esters, modification of pH conditions, and a softening or mellowing of the gluten character (Pyler, 1978). According to Jackel (1969), yeast requires about 45 min in a favorable environment to attain full adaptation to fermentation. Proteolytic enzymes act upon the protein materials of the dough, the overall effect of these enzymatic reactions is a softening of the dough, due in part to a reduction in the absorption capacity of the starch material, and in part to a weakening of the gluten system.

There was a highly significant ( $p < 0.01$ ) difference

between yeast condition and temperature for Wtforce ( $F = 9.753$ ,  $p = 0.0003$ , Fig. 40). The effect of yeast condition depended on the effect of temperature for Wtforce values. Mean values at refrigerated temperature were higher than those at room temperature for contact-first-yeast dough. Figure 40 shows that the mean values of proof-first-yeast dough at room temperature were higher than those at refrigerated temperature. No-yeast dough showed higher values at room temperature than at refrigerated temperature.

Tukey's Honestly Significant Difference test results of Wtforce indicated that the effect of yeast condition varied under different temperatures. However, within each yeast condition the effect of temperature was not significant (Fig 41, Appendix C, Table 39).

The effect of flour on Wtforce ( $F = 4.342$ ,  $p = 0.0189$ ) depended on temperature (Fig 42, 43, Appendix C, Table 40). Higher Wtforce mean values occurred for flour at refrigerated temperature. These differences appeared stronger for Hi-protein and bread flours.

A three-way interaction of yeast, flour, and temperature was significantly different ( $F = 3.491$ ,  $p = 0.0356$ ) for Wtforce. This three-way interaction is illustrated in Figure 44, 45, and Appendix C Table 41. Figure 44 shows that at room temperature, pastry flour with contact-first-yeast dough had the lowest mean value. Except for bread and pastry flour with proof-first-yeast and no-

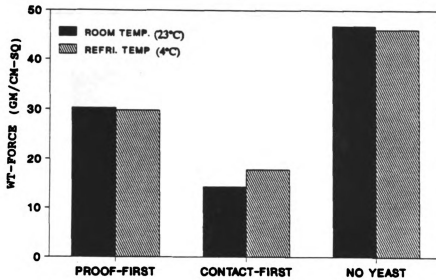


Figure 40. The Wtforce values of proof-first, contact-first, and no-yeast dough at room and refrigerated temperature.

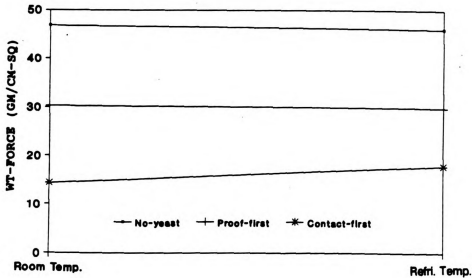


Figure 41. Tukey's HSD test Wtforce mean values of proof-first, contact-first and no-yeast dough at room and refrigerated temperature.

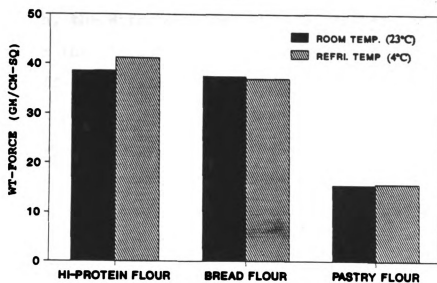


Figure 42. The Wtforce values of Hi-protein, bread, and pastry flour dough at room and refrigerated temperature.

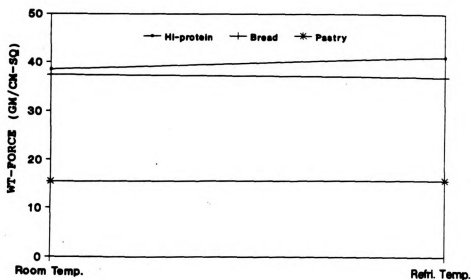


Figure 43. Tukey's HSD test Wtforce mean values of Hi-protein, bread, and pastry flour at room and refrigerated temperature.

yeast dough, the Wtforce values at refrigerated temperature were higher than those at room temperature.

The effect of film type on Wtforce ( $F = 203.475$ ,  $p = 0.0000$ ) was highly significant among all films. Tukey's Honestly Significant Difference test indicated that there was no significant difference between PE and PET film, but both films were significantly different ( $p < 0.05$ ) from Teflon film (Appendix C, Table 42).

The effect of yeast varied for the different films for Wtforce ( $F = 203.475$ ,  $p = 0.0000$ , Fig. 46). The mean values for the Teflon film were the lowest of all. For the contact-first-yeast, the mean value of the PET film was higher than that of the PE film.

Tukey's Honestly Significant Difference test indicated that there was no significant difference between PE and PET film for proof-first-yeast and contact-first-yeast dough (Fig. 47, Appendix C, Table 43). For no-yeast dough, there was no significant difference between PE and PET film. PE film was slightly less sticky than PET film with yeast dough, however, with no-yeast, PE film was much more sticky than PET film.

The interaction between flour and film was highly significant on Wtforce ( $F = 9.617$ ,  $p = 0.0000$ ). The Teflon film had the lowest mean values of all. For both Hi-protein and bread flour, the mean values of PE and PET film were close (Fig. 48), while the mean value of Teflon was lower.

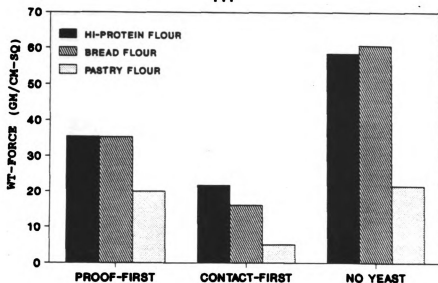


Figure 44. The Wtforce values of Hi-protein, bread, and pastry flour with proof-first, contact-first and no-yeast dough at room (23°C) temperature.

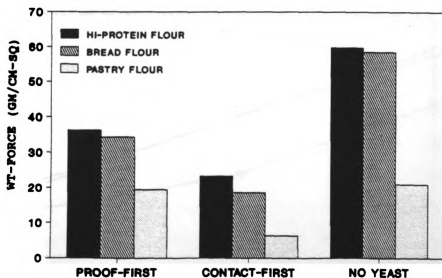


Figure 45. The Wtforce values of Hi-protein, bread, and pastry flour with proof-first, contact-first and no-yeast dough at refrigerated (4°C) temperature.

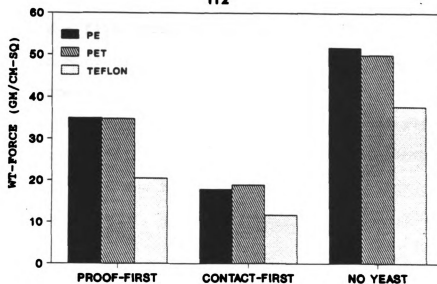


Figure 46. The Wtforce values of proof-first, contact-first, and no-yeast dough on PE, PET, and TEFLON film.

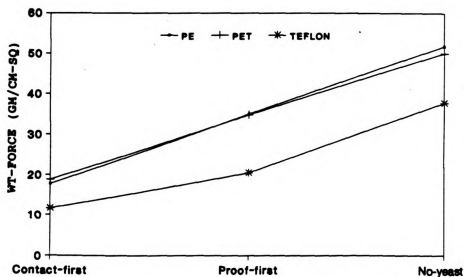


Figure 47. Tukey's HSD test Wtforce mean values of proof-first, contact-first, and no-yeast dough on PE, PET, and TEFLON film.

Pastry flour - film interactions were the lowest.

Tukey's Honestly Significant Difference test results (Fig. 49, Appendix C, Table 44) indicates that there were no significant differences for Hi-protein and bread flour with either PE or PET film, and no significant differences between Hi-protein and bread flour for Teflon film. There was no significant difference between PE and PET film for pastry flour.

A three-way interaction of yeast, flour, and film was highly significant for Wtforce ( $F = 5.783$ ,  $p = 0.0000$ ). This three-way interaction is illustrated in Figure 50, 51, and 52. Stickiness depended on yeast condition, flour, and type of film.

Figure 50 shows that the mean values of proof-first yeast dough on PET film were higher than those of PE film with Hi-protein and bread flours. Figure 51 shows that the mean value of contact-first-yeast on PET film was higher than those of PE film for all three flours. Teflon film with pastry flour had the lowest Wtforce value of all.

Figure 52 (Appendix C, Table 45) shows that for no-yeast dough the mean value of PE film with bread flour was the highest, and the Teflon film with pastry flour the lowest. For both Hi-protein and bread flour, the mean value of PE film was higher than that of PET film. For pastry flour, the mean value of PET film was higher than PE film.

The interaction between temperature and film on Wtforce

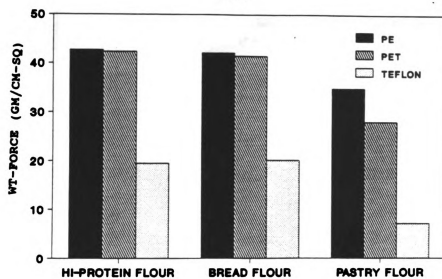


Figure 48. The Wtforce values of Hi-protein, bread, and pastry flour on PE, PET, and TEFLON film.

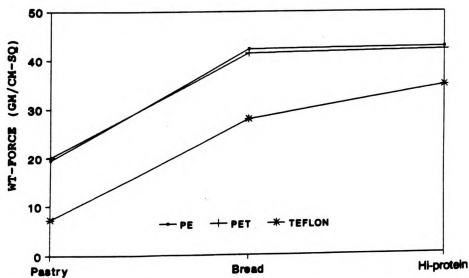


Figure 49. Tukey's HSD test Wtforce mean values of Hi-protein, bread, and pastry flour on PE, PET, and TEFLON film.

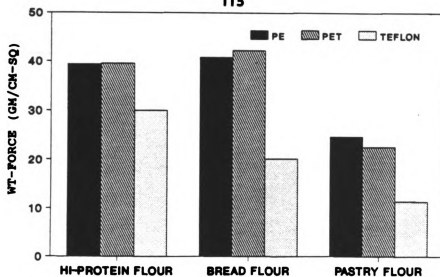


Figure 50. The Wtforce value of Hi-protein, bread, and pastry flour on PE, PET, and TEFLON film with proof-first-yeast dough.

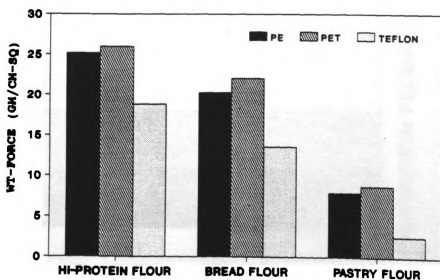


Figure 51. The Wtforce value of Hi-protein, bread, and pastry flour on PE, PET, and TEFLON film with contact-first-yeast dough.

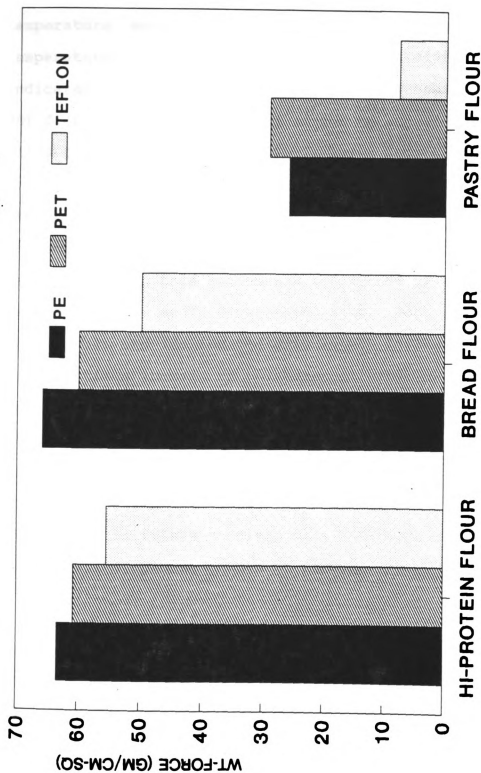


Figure 52. The Wtforce value of HI-protein, bread, and pastry flour on PE, PET, and TEFLON film with no-yeast dough.

was not significant (Fig. 53). For doughes at refrigerated temperature, mean values were higher than those at room temperature. Tukey's Honestly Significant Difference test indicated that Teflon film was less sticky than both PE and PET film. Temperature had no effect on any of the three films (Fig. 54, Appendix C, Table 46).

In the original study, we reported force per unit area at separation which implies that force is proportional to the contact area between the film and the dough. In a separate study, this hypothesis was tested by incorporating various lengths and widths of the film. Doubling the width of the film and leaving the same length increased the force by 1.75 times instead of two times as expected. Doubling the length of the film and leaving the same width only increased the force 1.2 times instead of two times as expected. Therefore, force is not proportional to area, nor is it proportional to length or width of the film. Therefore, in future studies, only the force of a given standardized opening should be reported.

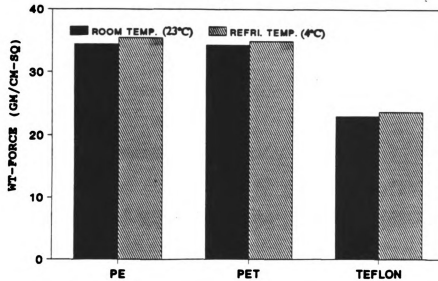


Figure 53. The Wtforce values of room and refrigerated temperature dough on PE, PET, and TEFLON film.

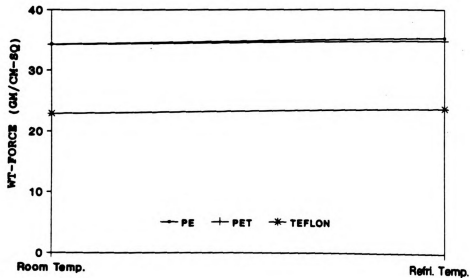


Figure 54. Tukey's HSD test Wtforce mean values of room and refrigerated temperature dough on PE, PET, and TEFLON film.

## **CONCLUSIONS**

This study was designed to:

1. Evaluate a device for determining adhesion between refrigerated dough and the contacting surface, and
2. To determine the influence of flour type, yeast condition, and temperature on adhesion between dough and plastic films.

The major findings of the study are summarized below:

### (1) Phase I Study:

#### **Wettability Test**

A. Wettability was characterized using the inclined plane method, and the results show that PETG had the highest angle and Teflon had the lowest angle.

#### **Stickiforce Meter Test**

B. Proof-first-yeast dough on PETG film at 4°C had the highest Wtmass mean value, and contact-first-yeast dough on Teflon film at 23°C had the lowest Wtmass mean value. As for the Wtforce mean values, proof-first-yeast dough on film 78G98 at 4°C had the highest mean value, and contact-first-yeast dough on Teflon film at 4°C had the lowest.

#### Wtmass

PETG was found to be the most adhesive material, and

Tri-extruded PE found to be the least adhesive material. When we consider both film and yeast factors, PETG with proof-first-yeast dough remained the most adhesive interaction, and HDPE with contact-first-yeast dough was found to be the least adhesive. Considering various films and the different temperatures, PETG at 4°C proved to be the most adhesive, and Teflon at 23°C was found to be the least. With all three factors combined, PETG with proof-first-yeast dough at 4°C was found to be the most adhesive material, and Teflon with contact-first-yeast dough at 23°C was found to be the least adhesive.

#### Wtforce

The Wtforce values of Silicone release paper are a result of that material requiring the highest amount of water to break away the test film from the dough surface. Teflon film used the least amount of water. When type of film and the yeast factor are considered, film 78G98 with proof-first-yeast dough was found to be the material that required the greatest force, and Teflon film the least. If we combine film type with the temperature factor, Silicone release paper at 4°C required the most force and Teflon at 4°C required the least. When we combine all three factors together, film 78G98 with proof-first-yeast dough at 4°C was found to be the material that used the highest amount of water to break the test film away from the dough surface, and the Teflon film with contact-first-yeast dough at 4°C

used least amount of water. Each factor contributed to and affected the dough stickiness.

(2) Phase II Study:

**Wettability Test**

A. The results from performing the Inclined plane method show that PET had the highest angle, and Teflon had the lowest angle.

**Stickiforce Meter Test**

B. Bread flour with no-yeast dough on PE film at 4°C had the highest Wtmass mean value, and pastry flour with no-yeast dough on Teflon film at 23°C had the lowest Wtmass mean value. As for the Wtforce mean values, bread flour with no-yeast dough on PE film at 23°C had the highest mean value, and pastry flour with contact-first-yeast dough on Teflon film at 4°C had the lowest.

Wtmass

PE film found to be the most adhesive material, and Teflon film found to be the least adhesive material. Bread flour had the highest adhesive property, and pastry flour had the lowest. When yeast conditions are combined with the flour factor, bread flour with no-yeast dough had the highest adhesiveness, and pastry flour with contact-first-yeast dough had the lowest. Considering yeast conditions, flour factor and temperature factor together, bread flour with no-yeast dough at 4°C had the highest adhesiveness, and pastry flour with contact-first-yeast dough at 23°C had the

lowest. Combine the four factors (Yeast, flour, temperature, and film) together, bread flour with no-yeast dough at 4°C on PE film was the most adhesive, and pastry flour with contact-first-yeast dough on Teflon film at 4°C was the least adhesive.

#### Wtforce

The Wtforce mean values showed that PE film was the material that used the most force to break the test film away from the dough surface and Teflon film the least. Considering both film and yeast factors, PE film with no-yeast dough used the highest amount of water to break away from the film, and Teflon film with contact-first-yeast dough the least. When film type and flour factors are considered, Hi-protein flour with PE film required the most force, and pastry flour with Teflon film required the lowest. Considering film type with yeast and flour factors, bread flour with no-yeast dough on PE film required the highest amount of water, and pastry flour with contact-first-yeast dough on Teflon film the least. Looking at the mean values of film types, and both flour and temperature factors, Hi-protein flour on PE film at 4°C found was the highest, and pastry flour on Teflon film at 23°C the lowest. Combining all factors, bread flour with no-yeast dough on PE film at 23°C was the combination that used the highest amount of water to break the test film away from the dough surface, and pastry flour with contact-first-yeast dough on

Teflon at 4°C was the combination that used the least.

(3) PVC, PET, and PETG all have a polar surface. That may be one reason why these three films stuck to the dough more than the other materials. The use of release agents, added as resins or used as coatings applied to a solid, may prevent/decrease adhesion of another solid. However, the silicone-based materials did not provide the required release properties. This was true in all cases.

(4) Good correlation was observed between the Wtforce (gram-force) and Wtmass (gram-mass) results.  $R^2 = 0.80$  for Phase I study, and  $R^2 = 0.92$  for Phase II study.

(5) Yeast provides flavoring compounds, affects the texture of dough, and creates carbon dioxide. The physical properties of the dough are altered through the powerful stretching actions generated by diffusion and accumulation of carbon dioxide throughout the dough mass, and the web structure reveals that thin-walled gluten strands offer minimum resistance to stretching when pulled. This might explain why yeast dough was less sticky than no-yeast dough.

(6) Hard wheat has relatively high protein content. Greater stickiness of this type of dough is thought to result from the strength of the bonding between the protein and starch in the endosperm. The high protein dough has less mobility and greater stiffness. Bonding between protein and starch is weak for soft wheat, which usually is low in protein content. This may explain why different

types of flour reacted differently in the tests performed.

(7) The original hypothesis that the force is required for separation is proportional to the contact area between the film and the dough was proven false by testing various lengths and widths of film. Doubling the width and length of the film increases the force by only 1.75 and 1.2 times respectively, rather than the expected 2 times.

## **RECOMMENDATIONS**

The instrumental technique using a Stickiforce Meter appears useful for measuring dough stickiness. The following are recommendations for future research:

1. The testing results obtained from a Farinograph will differ if test factors are strictly controlled. The variation of dough development will change the mixing tolerance index. Normally, relative humidity and temperature are controlled factors when using a Farinograph. When the experiment environment changes, the instrument changes, and results will vary considerably if there is no viable control.
2. Standardize the test conditions to obtain meaningful comparisons, which requires selection and control of temperature, relative humidity, and fermentation time.
3. The use of commercial flour instead of the pure variety did not enable us to differentiate what kind of protein and starch are in the flour. We need to do more chemical compound testing to obtain the ingredient analysis of each flour type: That is, the percentage of each component of protein and starch, in order to do a more comprehensive study on the cause of stickiness in dough.

## APPENDICES

## **APPENDIX A**

## APPENDIX A

## Absorption at 14% moisture content

$$A = 86 \frac{B+M}{100-M} - 14$$

	Pastry Flour	Bread Flour	Hi-protein Flour
M : Flour Moisture	9.38%	9.27%	9.08%
A : Absorption 14% mb			
B : Absorption, as-is mb			
H <sub>2</sub> O Added	29.52 ml	32.65 ml	33.20 ml
Flour Weight	50.48 g	47.50 g	46.80 g
Pastry Flour			

$$B = \frac{100 \times 29.52}{50.48} - 58.48\%$$

$$A = 86 \times \frac{58.48 + 9.38}{100 - 9.38} - 14 = 50.4$$

## Bread Flour

$$B = \frac{100 \times 32.5}{47.5} - 68.4\%$$

$$A = 86 \times \frac{68.4 + 9.27}{100 - 9.27} - 14 = 59.62$$

## Hi-protein Flour

$$B = \frac{100 \times 33.2}{46.8} - 70.94\%$$

$$A = 86 \times \frac{70.94 + 9.08}{100 - 9.08} - 14 = 61.69$$

## APPENDIX B

## Appendix B

Tukey's Honestly Significant Difference Test of  
Wtforce and Wtmass Results for Phase I Study

Table 15. Wtmass mean values of the test materials.

Film Type	Mean <sup>1</sup>
PETG	4.7 x 10 <sup>-2a</sup>
ISC-60	3.5 x 10 <sup>-2a</sup>
78G98	2.8 x 10 <sup>-2a</sup>
PVC	2.8 x 10 <sup>-2a</sup>
SILICONE PAPER	2.4 x 10 <sup>-2a</sup>
HDPE	1.9 x 10 <sup>-2a</sup>
PET	1.2 x 10 <sup>-2a</sup>
80C146A	6.6 x 10 <sup>-3a</sup>
2% EVA/VEG OIL	3.3 x 10 <sup>-3a</sup>
KRAYTON	1.2 x 10 <sup>-3a</sup>
2% EVA/MYV 9-40	8.2 x 10 <sup>-4a</sup>
2% EVA/1.5% PAM	7.5 x 10 <sup>-4a</sup>
TEFLON FEP	7.2 x 10 <sup>-4a</sup>
TRI-EXTRUDED PE	6.7 x 10 <sup>-4a</sup>

<sup>1</sup> means followed by the same letter are not significantly different at  $p < 0.05$  by Tukey's HSD Test

Table 16. Wtmass mean values of the test materials with proof-first and contact-first yeast dough.

Treatment	Mean <sup>1</sup>
PETG/Proof-first	9.2 x 10 <sup>-2a</sup>
ISC-60/Proof-first	6.4 x 10 <sup>-2a</sup>
PVC/Proof-first	5.2 x 10 <sup>-2a</sup>
SILICONE/Proof-first	4.4 x 10 <sup>-2a</sup>
78G98/Proof-first	4.3 x 10 <sup>-2a</sup>
HDPE/Proof-first	3.7 x 10 <sup>-2a</sup>
PET/Proof-first	2.3 x 10 <sup>-2a</sup>
78G98/Contact-first	1.4 x 10 <sup>-2a</sup>
80C146A/Proof-first	8.5 x 10 <sup>-3a</sup>
EVA/OIL//Proof-first	5.8 x 10 <sup>-3a</sup>
ISC-60/Contact-first	5.3 x 10 <sup>-3a</sup>
80C146/Contact-first	4.6 x 10 <sup>-3a</sup>
PVC/Contact-first	3.9 x 10 <sup>-3a</sup>
SILICONE/Contact-first	3.7 x 10 <sup>-3a</sup>
KRAYTON/Proof-first	1.5 x 10 <sup>-3a</sup>
PETG/Contact-first	1.4 x 10 <sup>-3a</sup>
PET/Contact-first	1.3 x 10 <sup>-3a</sup>
EVA/PAM/Proof-first	1.1 x 10 <sup>-3a</sup>
EVA/MYV/Proof-first	9.9 x 10 <sup>-4a</sup>
TEFLON/Proof-first	9.5 x 10 <sup>-4a</sup>
KRAYTON/Contact-first	9.3 x 10 <sup>-4a</sup>
EVA/OIL/Contact-first	7.4 x 10 <sup>-4a</sup>
TRI/PE//Proof-first	7.1 x 10 <sup>-4a</sup>
EVA/MYV/Contact-first	6.5 x 10 <sup>-4a</sup>
TRI/PE/Contact-first	6.3 x 10 <sup>-4a</sup>
TEFLON/Contact-first	5.0 x 10 <sup>-4a</sup>
EVA/PAM/Contact-first	4.5 x 10 <sup>-4a</sup>
HDPE/Contact-first	4.0 x 10 <sup>-4a</sup>

<sup>1</sup> means followed by the same letters are not significantly different at p < 0.05 by Tukey's HSD Test

Table 17. Wtmass mean values of the test materials at room (23°C) and refrigerated (4°C) temperature.

Treatment	Mean <sup>1</sup>
PETG/Refri	9.2 x 10 <sup>-2a</sup>
ISC-60/Refri	6.5 x 10 <sup>-2a</sup>
PVC/Refri	5.5 x 10 <sup>-2a</sup>
78G98/Refri	5.1 x 10 <sup>-2a</sup>
SILICONE/Refri	4.5 x 10 <sup>-2a</sup>
HDPE/Refri	3.6 x 10 <sup>-2a</sup>
PET/Refri	2.3 x 10 <sup>-2a</sup>
80C146A/Refri	1.1 x 10 <sup>-2a</sup>
EVA/OIL/Refri	6.2 x 10 <sup>-3a</sup>
78G98/Room	5.2 x 10 <sup>-3a</sup>
ISC-60/Room	3.8 x 10 <sup>-3a</sup>
SILICONE/Room	2.8 x 10 <sup>-3a</sup>
80C146A/Room	2.5 x 10 <sup>-3a</sup>
KRAYTONE/Refri	1.7 x 10 <sup>-3a</sup>
EVA/MYV/Refri	1.3 x 10 <sup>-3a</sup>
TEFLON/Refri	1.2 x 10 <sup>-3a</sup>
PET/Room	1.1 x 10 <sup>-3a</sup>
HDPE/Room	1.0 x 10 <sup>-3a</sup>
PETG/Room	1.0 x 10 <sup>-3a</sup>
EVA/PAM/Refri	9.2 x 10 <sup>-4a</sup>
TRI/PE/Refri	8.7 x 10 <sup>-4a</sup>
PVC/Room	7.8 x 10 <sup>-4a</sup>
KRAYTON/Room	7.0 x 10 <sup>-4a</sup>
EVA/PAM/Room	5.8 x 10 <sup>-4a</sup>
TRI/PE/Room	4.6 x 10 <sup>-4a</sup>
EVA/OIL/Room	4.2 x 10 <sup>-4a</sup>
EVA/MYV/Room	3.5 x 10 <sup>-4a</sup>
TEFLON/Room	2.5 x 10 <sup>-4a</sup>

<sup>1</sup> means followed by the same letter are not significantly different at  $p < 0.05$  by Tukey's HSD Test

Table 18. Wtmass mean values of proof-first and contact-first yeast dough at room (23°C) and refrigerated (4°C) temperature.

Treatment	Mean <sup>1</sup>
Proof/Refri	5.2 x 10 <sup>-2a</sup>
Contact/Refri	4.3 x 10 <sup>-2a</sup>
Proof/Room	2.9 x 10 <sup>-2a</sup>
Contact/Room	1.1 x 10 <sup>-2a</sup>

<sup>1</sup> means followed by the same letter are not significantly different at  $p < 0.05$  by Tukey's HSD Test

Table 19. Wtmass mean values of the test materials with proof-first and contact-first yeast dough at room (23°C) and refrigerated (4°C) temperature.

Treatment	Mean <sup>1</sup>
PETG/Proof-first/Refri	1.8 x 10 <sup>-1a</sup>
ISC-60/Proof-first/Refri	1.2 x 10 <sup>-1ab</sup>
PVC/Proof-first/Refri	1.0 x 10 <sup>-1ab</sup>
SILICONE/Proof-first/Refri	8.5 x 10 <sup>-2ab</sup>
78G98/Proof-first/Refri	7.8 x 10 <sup>-2ab</sup>
HDPE/Proof-first/Refri	7.2 x 10 <sup>-2ab</sup>
PET/Proof-first/Refri	4.5 x 10 <sup>-2b</sup>
78G98/Contact-first/Refri	2.4 x 10 <sup>-2b</sup>
80C146A/Proof-first/Refri	1.5 x 10 <sup>-2b</sup>
EVA/OIL/Proof-first/Refri	1.1 x 10 <sup>-2b</sup>
ISC-60/Contact-first/Refri	7.9 x 10 <sup>-3b</sup>
PVC/Contact-first/Refri	7.2 x 10 <sup>-3b</sup>
78G98/Proof-first/Room	6.9 x 10 <sup>-3b</sup>
80C146A/Contact-first/Refri	6.8 x 10 <sup>-3b</sup>
ISC-60/Proof-first/Room	5.0 x 10 <sup>-3b</sup>
SILICONE/Contact-first/Refri	4.8 x 10 <sup>-3b</sup>
SILICONE/Proof-first/Room	3.5 x 10 <sup>-3b</sup>
78G98/Contact-first/Room	3.4 x 10 <sup>-3b</sup>
ISC-60/Contact-first/Room	2.5 x 10 <sup>-3b</sup>
80C146A/Contact-first/Room	2.5 x 10 <sup>-3b</sup>
80C146A/Proof-first/Room	2.4 x 10 <sup>-3b</sup>
KRAYTON/Proof-first/Refri	2.2 x 10 <sup>-3b</sup>
SILICONE/Contact-first/Room	2.1 x 10 <sup>-3b</sup>
PETG/Contact-first/Refri	1.7 x 10 <sup>-3b</sup>
PET/Contact-first/Refri	1.6 x 10 <sup>-3b</sup>
TEFLON/Proof-first/Refr	1.6 x 10 <sup>-3b</sup>
HDPE/Proof-first/Room	1.5 x 10 <sup>-3b</sup>
EVA/MYV/Proof-first/Refri	1.5 x 10 <sup>-3b</sup>
KRAYTON/Contact-first/Refri	1.3 x 10 <sup>-3b</sup>
EVA/OIL/Contact-first/Refri	1.2 x 10 <sup>-3b</sup>
EVA/PAM/Proof-first/Refri	1.1 x 10 <sup>-3b</sup>
PET/Proof-first/Room	1.1 x 10 <sup>-3b</sup>
EVA/MYV/Contact-first/Refri	1.1 x 10 <sup>-3b</sup>
PETG/Contact-first/Room	1.1 x 10 <sup>-3b</sup>
PET/Contact-first/Room	1.1 x 10 <sup>-3b</sup>
EVA/PVM/Proof-first/Room	1.0 x 10 <sup>-3b</sup>
PVC/Proof-first/Room	1.0 x 10 <sup>-3b</sup>
TRI/PE/Proof-first/Refri	9.3 x 10 <sup>-4b</sup>
PETG/Proof-first/Room	9.2 x 10 <sup>-4b</sup>
KRAYTON/Proof-first/Room	8.4 x 10 <sup>-4b</sup>
TRI/PE/Contact-first/Refri	8.2 x 10 <sup>-4b</sup>
TEFLON/Contact-first/Refri	8.2 x 10 <sup>-4b</sup>
EVA/PAM/Contact-first/Refri	6.9 x 10 <sup>-4b</sup>
PVC/Contact-first/Room	6.0 x 10 <sup>-4b</sup>

Table 19: (cont'd)

Treatment	Mean <sup>1</sup>
EVA/OIL/Proof-first/Room	5.7 x 10 <sup>-4b</sup>
KRAYTON/Contact-first/Room	5.6 x 10 <sup>-4b</sup>
HDPE/Contact-first/Room	5.0 x 10 <sup>-4b</sup>
TRI/PE/Proof-first/Room	5.0 x 10 <sup>-4b</sup>
EVA/MYV/Proof-first/Room	4.8 x 10 <sup>-4b</sup>
TRI/PE/Contact-first/Room	4.3 x 10 <sup>-4b</sup>
TEFLON/Proof-first/Room	3.3 x 10 <sup>-4b</sup>
HDPE/Contact-first/Refri	3.1 x 10 <sup>-4b</sup>
EVA/OIL/Contact-first/Room	2.6 x 10 <sup>-4b</sup>
EVA/MYV/Contact-first/Refri	2.2 x 10 <sup>-4b</sup>
EVA/PAM/Contact-first/Room	2.1 x 10 <sup>-4b</sup>
TEFLON/Contact-first/Room	1.7 x 10 <sup>-4b</sup>

<sup>1</sup> means followed by the same letters are not significantly different at  $p < 0.05$  by Tukey's HSD Test

Table 20. Wtforce mean values of the test materials.

Film Type	Mean <sup>1</sup>
SILICONE PAPER	18.19 <sup>a</sup>
78G98	16.27 <sup>a</sup>
ISC-60	14.20 <sup>ab</sup>
80C146A	12.80 <sup>abc</sup>
PETG	10.22 <sup>bcd</sup>
PET	8.86 <sup>bcde</sup>
PVC	8.40 <sup>bcdef</sup>
HDPE	7.09 <sup>cdef</sup>
2% EVA/VEG OIL	6.28 <sup>def</sup>
KRAYTON	4.53 <sup>def</sup>
2% EVA/1.5% PAM	3.86 <sup>ef</sup>
TRI-EXTRUDED PE	3.36 <sup>ef</sup>
2% EVA/MYV 9-60	3.04 <sup>ef</sup>
TEFLON FEP	2.75 <sup>f</sup>

<sup>1</sup> means followed by the same letters are not significantly different at  $p < 0.05$  by Tukey's HSD Test

Table 21. Wtforce mean values of the test materials with proof-first and contact-first yeast dough.

Treatment	Mean <sup>1</sup>
78G98/Proof-first	20.80 <sup>a</sup>
SILICONE/Proof-first	19.62 <sup>ab</sup>
ISC-60/Proof-first	18.03 <sup>abc</sup>
SILICONE/Contact-first	16.75 <sup>abcd</sup>
PETG/Proof-first	15.92 <sup>abcde</sup>
80C146A/Proof-first	13.44 <sup>bcdef</sup>
PET/Proof-first	12.43 <sup>cdef</sup>
PVC/Proof-first	12.36 <sup>cdef</sup>
80C146A/Contact-first	12.16 <sup>cdef</sup>
78G98/Contact-first	11.73 <sup>cdefg</sup>
ISC-60/Contact-first	10.36 <sup>defgh</sup>
HDPE/Proof-first	9.33 <sup>efghi</sup>
EVA/OIL/Proof-first	7.64 <sup>fghij</sup>
KRAYTON/Proof-first	5.43 <sup>ghij</sup>
PET/Contact-first	5.29 <sup>ghij</sup>
EVA/OIL/Contact-first	4.91 <sup>hij</sup>
HDPE/Contact-first	4.86 <sup>hij</sup>
PETG/Contact-first	4.52 <sup>hij</sup>
PVC/Contact-first	4.44 <sup>hij</sup>
EVA/PAM/Proof-first	4.32 <sup>hij</sup>
KRAYTON/Contact-first	3.64 <sup>ij</sup>
EVA/MYV/Proof-first	3.60 <sup>ij</sup>
TRI/PE/Proof-first	3.59 <sup>ij</sup>
EVA/PAM/Contact-first	3.40 <sup>ij</sup>
TEFLON/Proof-first	3.16 <sup>ij</sup>
TRI/PE/Contact-first	3.12 <sup>ij</sup>
EVA/MYV/Contact-first	2.48 <sup>j</sup>
TEFLON/Contact-first	2.34 <sup>j</sup>

<sup>1</sup> means followed by the same letters are not significantly different at  $p < 0.05$  by Tukey's HSD Test

Table 22. Wtforce mean values of the test material at room (23°C) and refrigerated (4°C) temperature.

Treatment	Mean <sup>1</sup>
SLICONE/Refri	29.31 <sup>a</sup>
78G98/Refri	20.57 <sup>ab</sup>
ISC-60/Refri	18.21 <sup>abc</sup>
80C146A/Refri	16.52 <sup>bcd</sup>
PETG/Refri	15.09 <sup>bcde</sup>
PVC/Refri	12.94 <sup>cde</sup>
PET/Refri	12.76 <sup>cde</sup>
SILICONE/Room	12.46 <sup>cde</sup>
78G98/Room	11.96 <sup>cdef</sup>
HDPE/Refri	10.79 <sup>defg</sup>
ISC-60/Room	10.19 <sup>defgh</sup>
EVA/OIL/Refri	9.16 <sup>efghi</sup>
80C146A/Room	9.09 <sup>efghi</sup>
KRAYTON/Refri	5.48 <sup>fghi</sup>
PETG/Room	5.35 <sup>ghi</sup>
PET/Room	4.96 <sup>ghi</sup>
EVA/PAM/Refri	4.23 <sup>ghi</sup>
PVC/Room	3.86 <sup>hi</sup>
TRI/PE/Refri	3.67 <sup>hi</sup>
KRAYTON/Room	3.59 <sup>i</sup>
EVA/PAM/Room	3.50 <sup>i</sup>
HDPE/Room	3.40 <sup>i</sup>
EVA/OIL/Room	3.39 <sup>i</sup>
EVA/MYV/Refri	3.26 <sup>i</sup>
TRI/PE/Room	3.04 <sup>i</sup>
TEFLON/Room	2.92 <sup>i</sup>
EVA/MYV/Room	2.82 <sup>i</sup>
TEFLON/Refri	2.59 <sup>i</sup>

<sup>1</sup> means followed by the same letters are not significantly different at  $p < 0.05$  by Tukey's HSD Test

Table 23. Wtforce mean values of proof-first and contact-first yeast dough at room (23°C) and refrigerated (4°C) temperature.

Treatment	Mean <sup>1</sup>
Proof/Refri	14.99 <sup>a</sup>
Contact/Refri	7.75 <sup>b</sup>
Proof/Room	6.39 <sup>b</sup>
Contact/Room	5.11 <sup>b</sup>

<sup>1</sup> means followed by the same letter are not significantly different at  $p < 0.05$  by Tukey's HSD Test

Table 24. Wtforce mean values of the test materials with proof-first and contact-first yeast dough at room (23°C) and refrigerated (4°C) temperature.

Treatment	Mean <sup>1</sup>
78G98/Proof-first/Refri	28.19 <sup>a</sup>
PETG/Proof-first/Refri	25.89 <sup>ab</sup>
SILICONE/Proof-first/Refri	24.99 <sup>ab</sup>
ISC-60/Proof-first/Refri	23.89 <sup>abc</sup>
SILICONE/Contact-first/Refri	22.83 <sup>abcd</sup>
PVC/Proof-first/Refri	21.21 <sup>abcde</sup>
PET/Proof-first/Refri	19.13 <sup>bcdef</sup>
80C146A/Proof-first/Refri	16.60 <sup>cdefg</sup>
80C146A/Contact-first/Refri	16.44 <sup>defg</sup>
HDPE/Proof-first/Refri	15.07 <sup>efgh</sup>
SILICONE/Proof-first/Room	14.25 <sup>efghi</sup>
78G98/Proof-first/Room	13.42 <sup>fghij</sup>
78G98/Contact-first/Refri	12.96 <sup>fghijk</sup>
ISC-60/Contact-first/Refri	12.53 <sup>fghijk</sup>
ISC-60/Proof-first/Room	12.18 <sup>fghijkl</sup>
EVA/OIL/Proof-first/Refri	11.48 <sup>ghijklm</sup>
SILICONE/Contact-first/Room	10.68 <sup>ghijklmn</sup>
78G98/Contact-first/Room	10.50 <sup>ghijklmno</sup>
80C146A/Proof-first/Room	10.28 <sup>ghijklmnop</sup>
ISC-60/Contact-first/Room	8.20 <sup>hijklmnopq</sup>
80C146A/Contact-first/Room	7.89 <sup>hijklmnopq</sup>
KRAYTON/Proof-first/Refri	7.15 <sup>ijklmnopq</sup>
EVA/OIL/Contact-first/Refri	6.84 <sup>jklmnopq</sup>
HDPE/Contact-first/Refri	6.51 <sup>jklmnopq</sup>
PET/Contact-first/Refri	6.40 <sup>jklmnopq</sup>
PETG/Proof-first/Room	5.94 <sup>klmnopq</sup>
PET/Proof-first/Room	5.74 <sup>klmnopq</sup>
EVA/PAM/Proof-first/Refri	5.04 <sup>lmnopq</sup>
PETG/Contact-first/Room	4.76 <sup>mnopq</sup>
PVC/Contact-first/Refri	4.68 <sup>mnopq</sup>
PETG/Contact-first/Refri	4.28 <sup>mnopq</sup>
PVC/Contact-first/Room	4.21 <sup>mnopq</sup>
EVA/MYV/Proof-first/Refri	4.20 <sup>mnopq</sup>
PET/Contact-first/Room	4.18 <sup>mnopq</sup>
TRI/PE/Proof-first/Refri	4.02 <sup>nopq</sup>
EVA/OIL/Proof-first/Room	3.81 <sup>nopq</sup>
KRAYTON/Contact-first/Refri	3.80 <sup>nopq</sup>
KRAYTON/Proof-first/Room	3.70 <sup>nopq</sup>
EVA/PAM/Proof-first/Room	3.60 <sup>nopq</sup>
HDPE/Proof-first/Room	3.59 <sup>nopq</sup>
PVC/Proof-first/Room	3.51 <sup>nopq</sup>
KRAYTON/Contact-first/Room	3.48 <sup>nopq</sup>
EVA/PAM/Contact-first/Refri	3.42 <sup>nopq</sup>
EVA/PAM/Contact-first/Room	3.39 <sup>nopq</sup>

Table 24: (Cont'd)

Treatment	Mean <sup>1</sup>
TRI/PE/Contact-first/Refri	3.32 <sup>opq</sup>
TEFLON/Proof-first/Room	3.31 <sup>opq</sup>
HDPE/Contact-first/Room	3.20 <sup>opq</sup>
TRI/PE/Proof-first/Room	3.16 <sup>pq</sup>
TEFLON/Proof-first/Refri	3.01 <sup>pq</sup>
EVA/MYV/Proof-first/Room	3.00 <sup>pq</sup>
EVA/OIL/Contact-first/Room	2.98 <sup>pq</sup>
TRI/PE/Contact-first/Room	2.93 <sup>q</sup>
EVA/MYV/Contact-first/Room	2.64 <sup>q</sup>
TEFLON/Contact-first/Room	2.52 <sup>q</sup>
EVA/MYV/Contact-first/Refri	2.32 <sup>q</sup>
TEFLON/Contact-first/Refri	2.17 <sup>q</sup>

<sup>1</sup> means followed by the same letters are not significantly different at  $p < 0.05$  by Tukey's HSD Test

## APPENDIX C

## APPENDIX C

**Tukey's Honestly Significant Difference Test for  
Wtforce and Wtmass Results for Phase II Study**

**Table 25. Wtmass mean values of proof-first, contact-first, and no-yeast dough.**

Yeast Type	Mean <sup>1</sup>
None	4.3 x 10 <sup>-1b</sup>
Proof-first	1.8 x 10 <sup>-1b</sup>
Contact-first	1.3 x 10 <sup>-1b</sup>

<sup>1</sup> means followed by the same letter are not significantly different at  $p < 0.05$  by Tukey's HSD Test

**Table 26. Wtmass mean values of Hi-protein, bread, and pastry flour.**

Flour Type	Mean <sup>1</sup>
Bread	3.0 x 10 <sup>-1a</sup>
Hi-protein	2.9 x 10 <sup>-1a</sup>
Pastry	1.5 x 10 <sup>-1b</sup>

<sup>1</sup> means followed by the same letter are not significantly different at  $p < 0.05$  by Tukey's HSD Test

Table 27. Wtmass mean values of proof-first, contact-first, and no-yeast dough with Hi-protein, bread, and pastry flour.

Treatment	Mean <sup>1</sup>
None/Bread	6.3 x 10 <sup>-1a</sup>
None/Hiprotein	4.6 x 10 <sup>-1b</sup>
Proof/Hiprotein	2.5 x 10 <sup>-1c</sup>
None/Pastry	2.1 x 10 <sup>-1c</sup>
Proof/Pastry	1.8 x 10 <sup>-1cd</sup>
Contact/Hiprotein	1.8 x 10 <sup>-1cd</sup>
Contact/Bread	1.4 x 10 <sup>-1cd</sup>
Proof/Bread	1.2 x 10 <sup>-1cd</sup>
Contact/Pastry	6.5 x 10 <sup>-2d</sup>

<sup>1</sup> means followed by the same letters are not significantly different at  $p < 0.05$  by Tukey's HSD Test

Table 28. Wtmass mean values of room (23°C) and refrigerated (4°C) temperature with proof-first, contact-first, and no-yeast dough.

Treatment	Mean <sup>1</sup>
None/Refri	4.5 x 10 <sup>-1a</sup>
None/Room	4.1 x 10 <sup>-1a</sup>
Proof-first/Room	1.9 x 10 <sup>-1b</sup>
Proof-first/Refri	1.8 x 10 <sup>-1b</sup>
Contact-first/Refri	1.6 x 10 <sup>-1b</sup>
Contact-first/Room	9.6 x 10 <sup>-2b</sup>

<sup>1</sup> means followed by the same letter are not significantly different at  $p < 0.05$  by Tukey's HSD Test

Table 29. Wtmass mean values of room (23°C) and refrigerated (4°C) temperature with Hi-protein, bread, and pastry flour.

Treatment	Mean <sup>1</sup>
Bread/Refri	3.2 x 10 <sup>-1a</sup>
Hiprotein/Refri	3.1 x 10 <sup>-1a</sup>
Hiprotein/Room	2.7 x 10 <sup>-1ab</sup>
Bread/Room	2.7 x 10 <sup>-1ab</sup>
Pastry/Refri	1.8 x 10 <sup>-1b</sup>
Pastry/Room	1.5 x 10 <sup>-1b</sup>

<sup>1</sup> means followed by the same letters are not significantly different at  $p < 0.05$  by Tukey's HSD Test

Table 30. Wtmass mean values of proof-first, contact-first, and no-yeast dough with Hi-protein, bread, and pastry flour at room (23°C) and refrigerated (4°C) temperature.

Treatment	Mean <sup>1</sup>
Bread/None/Refri	6.6 x 10 <sup>-1a</sup>
Bread/None/Room	6.0 x 10 <sup>-1ab</sup>
Hiprotein/None/Refri	4.9 x 10 <sup>-1bc</sup>
Hiprotein/None/Room	4.3 x 10 <sup>-1c</sup>
Hiprotein/Proof/Refri	2.5 x 10 <sup>-1d</sup>
Hiprotein/Proof/Room	2.4 x 10 <sup>-1de</sup>
Pastry/None/Refri	2.2 x 10 <sup>-1def</sup>
Pastry/None/Room	2.1 x 10 <sup>-1defg</sup>
Hiprotein/Contact/Refri	2.0 x 10 <sup>-1defg</sup>
Bread/Contact/Refri	1.9 x 10 <sup>-1defg</sup>
Pastry/Proof/Room	1.8 x 10 <sup>-1defg</sup>
Pastry/Proof/Refri	1.7 x 10 <sup>-1defg</sup>
Hiprotein/Contact/Room	1.5 x 10 <sup>-1defg</sup>
Bread/Proof/Room	1.3 x 10 <sup>-1defg</sup>
Bread/Proof/Refri	1.2 x 10 <sup>-1defg</sup>
Bread/Contact/Room	8.8 x 10 <sup>-2efg</sup>
Pastry/Contact/Refri	7.7 x 10 <sup>-2fg</sup>
Pastry/Contact/Room	5.3 x 10 <sup>-2g</sup>

<sup>1</sup> means followed by the same letters are not significantly different at  $p < 0.05$  by Tukey's HSD Test

Table 31. Wtmass mean values of PE, PET, and TEFLON film.

Film Type	Mean <sup>1</sup>
PE	$3.5 \times 10^{-1a}$
PET	$3.0 \times 10^{-1a}$
TEFLON	$9.5 \times 10^{-2b}$

<sup>1</sup> means followed by the same letter are not significantly different at  $p < 0.05$  by Tukey's HSD Test

Table 32. Wtmass mean values of proof-first, contact-first, and no-yeast dough on PE, PET, and TEFLON film.

Treatment	Mean <sup>1</sup>
None/PE	$6.6 \times 10^{-1a}$
None/PET	$4.1 \times 10^{-1b}$
Proof-first/PET	$3.1 \times 10^{-1bc}$
None/TEFLON	$2.3 \times 10^{-1cd}$
Proof-first/PE	$2.2 \times 10^{-1cd}$
Contact-first/PET	$1.9 \times 10^{-1cd}$
Contact-first/PE	$1.6 \times 10^{-1de}$
Contact-first/TEFLON	$3.0 \times 10^{-2e}$
Proof-first/TEFLON	$2.5 \times 10^{-2e}$

<sup>1</sup> means followed by the same letters are not significantly different at  $p < 0.05$  by Tukey's HSD Test

Table 33. Wtmass mean values of Hi-protein, bread, and pastry flour on PE, PET, and TEFLON film.

Treatment	Mean <sup>1</sup>
PE/Bread	4.8 x 10 <sup>-1a</sup>
PE/Hi-protein	4.0 x 10 <sup>-1ab</sup>
PET/Hi-protien	3.5 x 10 <sup>-1ab</sup>
PET/Bread	3.0 x 10 <sup>-1bc</sup>
PET/Pastry	2.5 x 10 <sup>-1bc</sup>
PE/Pastry	2.0 x 10 <sup>-1cd</sup>
TEFLON/Hi-protein	1.7 x 10 <sup>-1cd</sup>
TEFLON/Bread	1.1 x 10 <sup>-1de</sup>
TEFLON/Pastry	5.1 x 10 <sup>-3e</sup>

<sup>1</sup> means followed by the same letters are not significantly different at  $p < 0.05$  by Tukey's HSD Test

Table 34. Wtmass mean values of Hi-protein, bread, and pastry flour with proof-first, contact-first and no-yeast dough on PE, PET, and TEFLON film.

Treatment	Mean <sup>1</sup>
Bread/None/PE	1.1 x 10 <sup>0a</sup>
Hiprotein/None/PE	5.0 x 10 <sup>-1b</sup>
Bread/None/PET	4.7 x 10 <sup>-1bc</sup>
Hiprotein/None/PET	4.6 x 10 <sup>-1bc</sup>
Hiprotein/None/TEFLON	4.1 x 10 <sup>-1bcd</sup>
Hiprotein/Proof/PET	3.7 x 10 <sup>-1bcde</sup>
Pastry/None/PE	3.4 x 10 <sup>-1bcdef</sup>
Hiprotein/Proof/PE	3.2 x 10 <sup>-1cdef</sup>
Pastry/Proof/PET	3.1 x 10 <sup>-1cdefg</sup>
Pastry/None/PET	3.0 x 10 <sup>-1cdefg</sup>
Bread/None/TEFLON	2.8 x 10 <sup>-1cdefgh</sup>
Hiprotein/Contact/PE	2.5 x 10 <sup>-1defgh</sup>
Bread/Proof/PET	2.4 x 10 <sup>-1defgh</sup>
Hiprotein/Contact/PET	2.2 x 10 <sup>-1efghi</sup>
Pastry/Proof/PE	2.0 x 10 <sup>-1efghij</sup>
Bread/Contact/PET	2.0 x 10 <sup>-1efghij</sup>
Bread/Contact/PE	1.8 x 10 <sup>-1fghijk</sup>
Pastry/Contact/PT	1.5 x 10 <sup>-1ghijkl</sup>
Bread/Proof/PE	1.3 x 10 <sup>-1hijkl</sup>
Hiprotein/Proof/TEFLON	5.9 x 10 <sup>-2ijkl</sup>
Hiprotein/Contact/TEFLON	5.4 x 10 <sup>-2ijkl</sup>

Table 34: (cont'd)

Treatment	Mean <sup>1</sup>
Pastry/Contact/PE	4.6 x 10 <sup>-2ijkl</sup>
Bread/Contact/TEFLON	3.1 x 10 <sup>-2jkl</sup>
Pastry/Proof/TEFLON	1.1 x 10 <sup>-2kl</sup>
Bread/Proof/TEFLON	4.0 x 10 <sup>-3l</sup>
Pastry/Contact/TEFLON	3.1 x 10 <sup>-3l</sup>
Pastry/None/TEFLON	1.3 x 10 <sup>-3l</sup>

<sup>1</sup> means followed by the same letters are not significantly different at  $p < 0.05$  by Tukey's HSD Test

Table 35. Wtmass mean values of room (23°C) and refrigerated (4°C) temperature with PE, PET, and TEFLON film.

Treatment	Mean <sup>1</sup>
PE/Refri	3.8 x 10 <sup>-1a</sup>
PE/Room	3.2 x 10 <sup>-1a</sup>
PET/Refri	3.1 x 10 <sup>-1a</sup>
PET/Room	3.0 x 10 <sup>-1a</sup>
TEFLON/Refri	1.1 x 10 <sup>-1b</sup>
TEFLON/Room	8.3 x 10 <sup>-2b</sup>

<sup>1</sup> means followed by the same letter are not significantly different at  $p < 0.05$  by Tukey's HSD Test

Table 36. Wtforce mean values of proof-first, contact-first, and no-yeast dough.

Yeast Type	Mean <sup>1</sup>
None	39.80 <sup>a</sup>
Proof-first	37.19 <sup>a</sup>
Contact-first	15.61 <sup>b</sup>

<sup>1</sup> means followed by the same letter are not significantly different at  $p < 0.05$  by Tukey's HSD Test

Table 37. Wtforce mean values of Hi-protein, bread, and pastry flour.

Flour Type	Mean <sup>1</sup>
Hi-protein	46.47 <sup>a</sup>
Bread	30.05 <sup>b</sup>
Pastry	16.09 <sup>c</sup>

<sup>1</sup> means followed by the same letter are not significantly different at  $p < 0.05$  by Tukey's HSD Test

Table 38. Wtforce mean values of none, proof-first, contact-first, and no-yeast dough with Hi-protein, bread, and pastry flour.

Treatment	Mean <sup>1</sup>
None/Hiprotein	59.84 <sup>a</sup>
None/Bread	58.61 <sup>b</sup>
Proof/Hiprotein	36.27 <sup>c</sup>
Proof/Bread	34.37 <sup>c</sup>
Contact/Hiprotein	23.30 <sup>cd</sup>
None/Pastry	20.95 <sup>cd</sup>
Proof/Pastry	19.50 <sup>cd</sup>
Contact/Bread	18.59 <sup>cd</sup>
Contact/Pastry	6.38 <sup>d</sup>

<sup>1</sup> means followed by the same letters are not significantly different at  $p < 0.05$  by Tukey's HSD Test

Table 39. Wtforce mean values of room (23°C) and refrigerated (4°C) temperature with proof-first, contact-first, and no-yeast dough.

Treatment	Mean <sup>1</sup>
None/Room	46.83 <sup>a</sup>
None/Refri	46.10 <sup>a</sup>
Proof-first/Room	30.26 <sup>b</sup>
Proof-first/Refri	29.83 <sup>b</sup>
Contact-first/Refri	17.86 <sup>c</sup>
Contact-first/Room	14.32 <sup>c</sup>

<sup>1</sup> means followed by the same letter are not significantly different at  $p < 0.05$  by Tukey's HSD Test

Table 40. Wtforce mean values of room (23°C) and refrigerated (4°C) temperature with Hi-protein, bread, and pastry flour.

Treatment	Mean <sup>1</sup>
Hiprotein/Refri	41.10 <sup>a</sup>
Hiprotein/Room	38.51 <sup>a</sup>
Bread/Room	37.41 <sup>a</sup>
Bread/Refri	36.96 <sup>a</sup>
Pastry/Refri	15.73 <sup>b</sup>
Pastry/Room	15.49 <sup>b</sup>

<sup>1</sup> means followed by the same letter are not significantly different at  $p < 0.05$  by Tukey's HSD Test

Table 41. Wtforce mean values of proof-first, contact-first, and no-yeast dough with Hi-protein, bread, and pastry flour at room (23°C) and refrigerated (4°C) temperature.

Treatment	Mean <sup>1</sup>
Hiprotein/None/Refri	61.32 <sup>a</sup>
Bread/None/Room	60.67 <sup>a</sup>
Hiprotein/None/Room	58.35 <sup>a</sup>
Bread/None/Rrfri	56.54 <sup>a</sup>
Hiprotein/Proof/Refri	37.07 <sup>b</sup>
Hiprotein/Proof/Room	35.48 <sup>b</sup>
Bread/Proof/Room	35.40 <sup>b</sup>
Bread/Proof/Refri	33.34 <sup>bc</sup>
Hiprotein/Contact/Refri	24.91 <sup>cd</sup>
Hiprotein/Contact/Room	21.70 <sup>d</sup>
Pastry/None/Room	21.47 <sup>d</sup>
Bread/Contact/Refri	21.01 <sup>d</sup>
Pastry/None/Refri	20.44 <sup>d</sup>
Pastry/Proof/Room	19.90 <sup>d</sup>
Pastry/Proof/Refri	19.09 <sup>d</sup>
Bread/Contact/Room	16.16 <sup>de</sup>
Pastry/Contact/Refri	7.67 <sup>ef</sup>
Pastry/Contact/Room	5.10 <sup>f</sup>

<sup>1</sup> means followed by the same letters are not significantly different at  $p < 0.05$  by Tukey's HSD Test

Table 42. Wtforce mean values of PE, PET, and TEFLON film.

Film Type	Mean <sup>1</sup>
PE	34.80 <sup>a</sup>
PET	34.54 <sup>a</sup>
TEFLON	23.26 <sup>b</sup>

<sup>1</sup> means followed by the same letter are not significantly different at  $p < 0.05$  by Tukey's HSD Test

Table 43. Wtforce mean values of proof-first, contact-first and no-yeast dough on PE, PET, and TEFLON film.

Treatment	Mean <sup>1</sup>
None/PE	51.71 <sup>a</sup>
None/PET	49.97 <sup>a</sup>
None/TEFLON	37.72 <sup>b</sup>
Proof-first/PE	34.93 <sup>b</sup>
Proof-first/PET	34.76 <sup>b</sup>
Proof-first/TEFLON	20.45 <sup>c</sup>
Contact-first/PET	18.89 <sup>c</sup>
Contact-first/PE	17.77 <sup>c</sup>
Contact-first/TEFLON	11.61 <sup>c</sup>

<sup>1</sup> means followed by the same letter are not significantly different at  $p < 0.05$  by Tukey's HSD Test

Table 44. Wtforce mean values of Hi-protein, bread, and pastry flour on PE, PET, and TEFLON film.

Treatment	Mean <sup>1</sup>
PE/Hi-protein	42.62 <sup>a</sup>
PE/Bread	42.28 <sup>a</sup>
PET/Hi-protein	42.07 <sup>a</sup>
PET/Bread	41.42 <sup>a</sup>
TEFLON/Hi-protein	34.73 <sup>ab</sup>
TEFLON/Bread	27.85 <sup>bc</sup>
PET/Pastry	20.12 <sup>c</sup>
PE/Pastry	19.51 <sup>c</sup>
TEFLON/Pastry	7.21 <sup>d</sup>

<sup>1</sup> means followed by the same letters are not significantly different at  $p < 0.05$  by Tukey's HSD Test

Table 45. Wtforce mean values of Hi-protein, bread, and pastry flour with proof-first, contact-first and no-yeast dough on PE, PET, and TEFLON film.

Treatment	Mean <sup>1</sup>
Bread/None/PE	65.85 <sup>a</sup>
Hiprotein/None/PE	63.33 <sup>a</sup>
Hiprotein/None/PET	60.74 <sup>a</sup>
Bread/None/PET	60.02 <sup>ab</sup>
Hiprotein/None/TEFLON	55.44 <sup>ab</sup>
Bread/None/TEFLON	49.95 <sup>bc</sup>
Bread/Proof/PET	42.23 <sup>c</sup>
Bread/Proof/PE	40.79 <sup>c</sup>
Hiprotein/Proof/PET	39.53 <sup>cd</sup>
Hiprotein/Proof/PE	39.35 <sup>cd</sup>
Hiprotein/Proof/TEFLON	29.95 <sup>de</sup>
Pastry/None/PET	29.14 <sup>def</sup>
Pastry/None/PE	25.95 <sup>ef</sup>
Hiprotein/Contact/PET	25.94 <sup>ef</sup>
Hiprotein/Contact/PE	25.18 <sup>ef</sup>
Pastry/Proof/PE	24.65 <sup>ef</sup>
Pastry/Proof/PET	24.51 <sup>efg</sup>
Bread/Contact/PET	22.02 <sup>efgh</sup>

Table 45 : (cont'd)

Treatment	Mean <sup>1</sup>
Bread/Contact/PE	20.22 <sup>efgh</sup>
Bread/Proof/TEFLON	20.09 <sup>efgh</sup>
Hiprotein/Contact/TEFLON	18.79 <sup>fghi</sup>
Bread/Contact/TEFLON	13.52 <sup>ghij</sup>
Pastry/Proof/TEFLON	11.33 <sup>hijk</sup>
Pastry/Contact/PET	8.71 <sup>ijk</sup>
Pastry/Contact/PE	7.92 <sup>jk</sup>
Pastry/None/TEFLON	7.77 <sup>jk</sup>
Pastry/Contact/TEFLON	2.53 <sup>k</sup>

<sup>1</sup> means followed by the same letters are not significantly different at  $p < 0.05$  by Tukey's HSD Test

Table 46. Wtforce mean values of room (23°C) and refrigerated (4°C) temperature with PE, PET, and TEFLON film.

Treatment	Mean <sup>1</sup>
PE/Refri	35.32 <sup>a</sup>
PET/Refri	34.84 <sup>a</sup>
PE/Room	34.29 <sup>a</sup>
PET/Room	34.23 <sup>a</sup>
TEFLON/Refri	23.64 <sup>b</sup>
TEFLON/Room	22.89 <sup>b</sup>

<sup>1</sup> means followed by the same letter are not significantly different at  $p < 0.05$  by Tukey's HSD Test

Table 47. Wtmass mean values of proof-first, contact-first, and no-yeast dough at room (23°C) and refrigerated (4°C) temperature with PE, PET, and TEFLON film.

Treatment	Mean <sup>1</sup>
None/Refri/PE	6.8 x 10 <sup>-1a</sup>
None/Room/PE	6.4 x 10 <sup>-1a</sup>
None/Refri/PET	4.3 x 10 <sup>-1b</sup>
None/Room/PET	4.0 x 10 <sup>-1bc</sup>
Proof/Room/PET	3.2 x 10 <sup>-1bcd</sup>
Proof/Refri/PET	3.0 x 10 <sup>-1bcd</sup>
None/Refri/TEFLON	2.5 x 10 <sup>-1cde</sup>
Contact/Refri/PE	2.3 x 10 <sup>-1de</sup>
Proof/Refri/PE	2.2 x 10 <sup>-1de</sup>
Proof/Room/PE	2.2 x 10 <sup>-1de</sup>
None/Room/TEFLON	2.1 x 10 <sup>-1de</sup>
Contact/Refri/PET	2.0 x 10 <sup>-1de</sup>
Contact/Room/PET	1.7 x 10 <sup>-1def</sup>
Contact/Room/PE	9.4 x 10 <sup>-2ef</sup>
Contact/Refri/TEFLON	3.7 x 10 <sup>-2f</sup>
Proof/Refri/TEFLON	2.9 x 10 <sup>-2f</sup>
Contact/Room/TEFLON	2.2 x 10 <sup>-2f</sup>
Proof/Room/TEFLON	2.0 x 10 <sup>-2f</sup>

<sup>1</sup> means followed by the same letters are not significantly different at  $p < 0.05$  by Tukey's HSD Test

Table 48. Wtforce mean values of proof-first, contact-first, and no-yeast dough at room (23°C) and refrigerated (4°C) temperature with PE, PET, and TEFLON film.

Treatment	Mean <sup>1</sup>
None/Room/PE	53.24 <sup>a</sup>
None/Refri/PE	50.18 <sup>ab</sup>
None/Refri/PET	50.07 <sup>bc</sup>
None/Room/PET	49.86 <sup>c</sup>
None/Refri/TEFLON	38.05 <sup>d</sup>
None/Room/TEFLON	37.39 <sup>de</sup>
Proof/Room/PET	35.12 <sup>def</sup>
Proom/Room/PE	34.93 <sup>defg</sup>
Proof/Refri/PE	34.93 <sup>defg</sup>
Proof/Refri/PET	34.39 <sup>defg</sup>
Contact/Refri/PE	20.85 <sup>defg</sup>
Proof/Room/TEFLON	20.73 <sup>defg</sup>
Proof/Refri/TEFLON	20.18 <sup>defg</sup>
Contact/Refri/PET	20.06 <sup>defg</sup>
Contact/Room/PET	17.72 <sup>defg</sup>
Contact/Room/PE	14.69 <sup>efg</sup>
Contact/Refri/TEFLON	12.67 <sup>fg</sup>
Contact/Room/TEFLON	10.55 <sup>g</sup>

<sup>1</sup> means followed by the same letters are not significantly different at  $p < 0.05$  by Tukey's HSD Test

Table 49. Wtmass mean values of Hi-protein, bread, and pastry flour at room (23°C) and refrigerated (4°C) temperature with PE, PET, and TEFLON film.

Treatment	Mean <sup>1</sup>
Bread/Refri/PE	5.3 x 10 <sup>-1a</sup>
Bread/Rm/PE	4.4 x 10 <sup>-1ab</sup>
Hiprotein/Refri/PE	4.0 x 10 <sup>-1abc</sup>
Hiprotein/Refri/PET	3.5 x 10 <sup>-1bcd</sup>
Hiprotein/Rm/PET	3.4 x 10 <sup>-1bcde</sup>
Hiprotein/Rm/PE	3.2 x 10 <sup>-1bcdef</sup>
Bread/Refri/PET	3.2 x 10 <sup>-1bcdef</sup>
Bread/Rm/PET	2.9 x 10 <sup>-1bcdef</sup>
Pastry/Rm/PET	2.5 x 10 <sup>-1cdefg</sup>
Pastry/Refri/PET	2.5 x 10 <sup>-1cdefgh</sup>
Pastry/Refri/PE	2.0 x 10 <sup>-1defgh</sup>
Pastry/Rm/PE	1.9 x 10 <sup>-1defgh</sup>
Hiprotein/Refri/TEFLON	1.9 x 10 <sup>-1efgh</sup>
Hiprotein/Rm/TEFLON	1.6 x 10 <sup>-1fghi</sup>
Bread/Refri/TEFLON	1.2 x 10 <sup>-1ghi</sup>
Bread/Rm/TEFLON	9.0 x 10 <sup>-2hi</sup>
Pastry/Refri/TEFLON	8.0 x 10 <sup>-3i</sup>
Pastry/Rm/TEFLON	2.3 x 10 <sup>-3i</sup>

<sup>1</sup> means followed by the same letters are not significantly different at  $p < 0.05$  by Tukey's HSD Test

Table 50. Wtforce mean values of Hi-protein, bread, and pastry flour at room (23°C) and refrigerated (4°C) temperature with PE, PET, and TEFLON film.

Treatment	Mean <sup>1</sup>
Hiprotein/Refri/PE	44.05 <sup>a</sup>
Hiprotein/Refri/PET	43.26 <sup>ab</sup>
Bread/Refri/PE	42.43 <sup>ab</sup>
Bread/Room/PE	42.14 <sup>ab</sup>
Bread/Room/PET	41.89 <sup>ab</sup>
Hiprotein/Room/PE	41.18 <sup>ab</sup>
Bread/Refri/PET	40.95 <sup>ab</sup>
Hiprotein/Room/PET	40.88 <sup>ab</sup>
Hiprotein/Refri/TEFLON	35.99 <sup>abc</sup>
Hiprotein/Room/TEFLON	33.47 <sup>bc</sup>
Bread/Room/TEFLON	28.19 <sup>cd</sup>
Bread/Refri/TEFLON	27.52 <sup>cd</sup>
Pastry/Refri/PET	20.31 <sup>d</sup>
Pastry/Room/PET	19.93 <sup>d</sup>
Pastry/Room/PE	19.53 <sup>d</sup>
Pastry/Refri/PE	19.48 <sup>d</sup>
Pastry/Refri/TEFLON	7.40 <sup>e</sup>
Pastry/Room/TEFLON	7.01 <sup>e</sup>

<sup>1</sup> means followed by the same letters are not significantly different at  $p < 0.05$  by Tukey's HSD Test

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