

**MECHANICAL COMPACTION OF FLOUR - EFFECTS ON ITS SORPTION
HYSTERESIS AND ON THE RHEOLOGICAL BEHAVIOUR
OF A FLOUR-WATER DOUGH**

BY

SANTHANAGOPALAN RAMANATHAN

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ABSTRACT

The compaction behaviour of two grades of flour, a low extraction (64% extraction) patent flour and a high extraction (79% extraction) straight grade flour, and its effects on water sorption by flour and rheological properties of a flour-water dough were investigated.

The compaction behaviour of two grades of flour at three different moisture contents (13.1, 13.9, and 14.6% wet basis) and three different loading rates (5, 50, and 100 mm/min) was studied under axial compression using a Universal Testing Machine. The density-pressure relationship was used to characterize the compaction behaviour. The moisture content and loading rate had little influence on the compaction patterns in the range investigated.

The influence of flour compaction on the moisture sorption hysteresis patterns of two grades of flour, at three different temperatures, 10, 25, and 40°C, was studied using a Novasina water activity meter. In general, compaction of flour had little effect on its moisture sorption isotherms at all temperatures studied, i.e., at any given equilibrium moisture content, the compacted flour pellets equilibrated at nearly the same water activity as the non-compacted flour. The equilibration temperature had a significant (95% level) influence on the moisture sorption isotherms of flour. The hysteresis between the desorption and adsorption isotherms was not significantly altered (95% level), by any of the temperatures, for either the non-compacted flour or compacted flour pellets. However, the extent of hysteresis was more pronounced in the non-compacted flour than in the compacted flour pellets. The extent of hysteresis also decreased with

an increasing temperature. A non-linear regression was performed to estimate the parameters of the Modified Halsey equation, which predicted the isotherms with a reasonably good accuracy. Thermodynamic changes, such as Gibb's free energy change of sorption and isosteric heat of sorption using Clausius-Clapeyron equation and BET (Brunauer-Emmett-Teller) monolayer values, were not influenced by the compaction of flour.

The fundamental and empirical rheological properties of a flour-water dough, prepared using the two grades of flour from non-compacted flour and the flour reconstituted after compaction, were studied. The fundamental rheological properties were studied using a capillary rheometer in terms of flow behaviour index and consistency coefficient. The empirical rheological properties were studied using Farinograph, Mixograph, Alveograph, and Amylograph. The rheological properties of the dough prepared using the flour reconstituted after compaction were not significantly different from those prepared using the non-compacted flour. The reconstituted flour did not exhibit a significantly higher starch damage, wet gluten content, and bread loaf volume as compared to the non-compacted flour.

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1. INTRODUCTION

1.1 Overview of the Problem

Statistics given by the Food and Agricultural Organizations, United Nations (FAO Trade, 1992) state that the Canadian exports of wheat flour during the five year period from 1988-1992 accounts for nearly 165.3 thousand tonnes. This represents nearly 2.25% of the total world exports of 7330.4 thousand tonnes. The Canadian exports during the period from 1983-1987 accounts for 328.9 thousand tonnes which is nearly 5.1% of the total world exports of 6458.7 thousand tonnes. Thus, the Canadian exports during the last five years showed a decreasing trend comparing to the exports from 1983-1987. This decrease in the Canadian exports of wheat flour was attributed to the increased availability of milling techniques in developing countries and also to the price distortions (Dexter, 1994). However, the average Canadian exports of wheat and its products to tropical countries is more than 60% of that produced domestically. Hence, a technique for reducing the space requirement for storage and transportation and for enhancing the mechanical handling of wheat and its products, is of prime importance to countries like Canada, exporting more than 50% of their production.

The storage of flour is of prime importance in tropical countries experiencing very high temperatures during summer. The flour undergoes physical and chemical transformations during storage and hence its quality is affected (Dexter 1994). One of the most commonly encountered problems during storage includes the loss of moisture from the flour and hence a net loss in the mass of the flour (Fairbrother 1929). The flour stored at relatively high temperatures is also

susceptible to enzymatic rancidity and microbial deterioration through the increased water activity. A technique to eliminate the above mentioned problems would greatly reduce the losses through transportation and storage.

For hundreds of years, dehydration of foods has been used for reducing the weight of foods as well as extending their shelf life without the need for refrigeration. This technique has been used to reduce the activity of undesirable enzymes and microorganisms in the food which will otherwise cause food spoilage. Dehydration also reduces other deteriorative changes in foods, such as chemical oxidation, indicated by the undesirable changes in color and taste. However, it does not result in any appreciable increase in densities of foods (Hayes 1966). The bulk density of food solids and powders is an important physical attribute and is of considerable concern to the food industry. The density of a given food determines the filled weight of the containers and plays a vital role in handling and processing operations. The density of a food depends not only on its chemical composition, particle size, and moisture but also on its processing and handling history (Peleg et al. 1973). Hence, a technique to compact the dehydrated foods will result in increased density and in reduced volume than the natural foods. Information on compressibility of foods under axial loads (loads applied only in one direction) is very important in the development of pelleted foods.

Dehydration of flour to a low moisture content may cause some changes in the functional properties of flour (Dexter 1994). Hence, the compaction of flour at commercially available moisture content (approximately 14% wet basis) would give a solution to the above mentioned

problems. Flour is compressible and a considerable increase in density can be caused by static pressure, mechanical compaction, and exposure to mechanical vibration. Compaction of agricultural materials to 50% of their original volume would facilitate relatively easier mechanical handling and reduce the space requirement for storage and transportation. Compaction may prevent or slow down the diffusion of oxygen into the flour and thereby reduce the enzymatic activity within the flour and infestations of flour by mites and other microorganisms (McNulty et al. 1977). This will in turn extend the shelf life and keeping quality of flour during storage.

1.2 Need for this Research

Biological products, such as flour, are extremely hygroscopic and they tend to adsorb or desorb moisture depending on the relative humidity (RH) of the surrounding environment. Consequently, change in surrounding RH can lead to an increase or decrease in the moisture content of the products. An increase in the moisture content will result in an increase in water activity within the product which is conducive to microbial growth and enzyme activity. Also, it is well known that moisture absorption by flour can cause significant changes in its physical characteristics, most notably a decrease in its bulk density and an increase in its cohesiveness (Peleg 1977). The increase in cohesiveness will in turn lead to caking and hence affect the storage stability and cause handling problems. The decrease in moisture content will result in a net loss in the mass of the flour. Hence, it is important that the moisture sorption properties of flour be maintained when it is subjected to such a processing technique as compaction.

In the general classification of food powders (Peleg 1977), flour is generally considered as non-cohesive (or relatively free flowing) under normal storage conditions and that the inter-particle forces of attraction (or cohesive forces) between the different particles of flour (which contain mainly starch and protein) are negligible. However, such forces can develop under special conditions such as moisture absorption, elevated storage temperature or static pressure. As a result, flour is looked upon as a dynamic system with properties that may progressively change with time.

The rheological properties of a flour-water dough are very important in bread-making. The quality of the finished loaf of bread depends on the rheological properties of the dough which in turn depend on the flour quality. Hence, it is imperative that the quality of the flour be preserved in order to ensure the high quality baked foods. It is hypothesized that the flour quality might change during the compaction and reconstitution stages and that these changes would be reflected in the rheological properties of a dough and hence in bread making.

The moisture sorption by flour and the rheological behaviour of a flour-water dough have been studied extensively. One of the first attempts to account for the sorptive capacity of the flour on the basis of its physical and chemical characteristics was reported by Bushuk and Winkler (1957). They reported that the rate of moisture adsorption by flour was not affected by packing it more compactly but they did not make an attempt to find the equilibrium water activity of flour as influenced by flour compaction. Sharma et al. (1993) employed a capillary extrusion rheometer to determine the flow characteristics of a flour-water dough in terms of the

fundamental units as used in the engineering design of the pipeline and pumping systems. Weiland (1977) found a process for the treatment of cereal flour which is characterized by compressing the flour to produce a granulate with a bulk density at least 10% higher than that of the starting material. He also found that the granulate, after being ground to flour, imparted improved baking and storage properties to the baked goods. He made no attempt to study the implications of flour compaction on other important rheological measurements and analytical determinations. Hence, the present study is mainly focused on the lacunae created in the above studies.

1.3 Hypotheses

Bushuk and Winkler (1957) found that the rate of moisture adsorption by flour was not affected by packing it more compactly. However, they made no attempt to find the equilibrium water activity value as influenced by compaction. Weiland (1977) reported that the compacted flour granulate, after being ground to flour, imparted improved baking properties to the baked goods. But, he did not validate his findings from the rheological and analytical determinations of flour and dough. It is hypothesized in the current research that:

- (1) Compaction of flour to granulates alters the sorptive capacity of the flour and hence its moisture sorption hysteresis patterns, and
- (2) The reconstituted flour after compaction exhibits some changes in its functional properties which would alter the rheological properties of a flour-water dough and hence the quality of the baked foods.

1.4 Objectives

The main objectives of this research are to:

- (1) study the density-pressure relationship of flour as influenced by different loading rates and different moisture contents,
- (2) investigate the moisture sorption hysteresis patterns of flour at three different temperatures 10, 25, and 40°C as influenced by flour compaction,
- (3) validate the data using the three parameter Modified Halsey equation,
- (4) determine the thermodynamic changes, such as the Gibb's free energy change of sorption, the heat of sorption values using the Clausius-Clapeyron equation, as influenced by flour compaction,
- (5) determine the BET monolayer values as influenced by flour compaction, and
- (6) determine the fundamental and empirical rheological properties of a flour-water dough prepared using the untreated flour and the flour reconstituted after compaction.

2. REVIEW OF LITERATURE

2.1 Compaction Behaviour of Various Products

2.1.1 Compaction Behaviour of Metal Powders

The compaction behaviour of metal powders has been studied extensively in the past (Cooper and Eaton 1962, Heckel 1961a,b, Schwarzkopf 1947, Seelig and Wulff 1946). Seelig and Wulff (1946) obtained the density-pressure relationship of metal powders by pressing the powder at different pressures. Measurement of the density of the powder compact was made after it was removed from a die. This method, however, had serious disadvantages: (i) Due to the large number of pressing operations required, the method was rather slow. (ii) Because density was measured after the specimen was removed from the die, it corresponded to an over estimation of pressure. (iii) The measurement of "at-pressure" compact density in the die was not possible. Seelig and Wulff (1946) also discussed the process of compaction by three basic mechanisms: sliding, elastic and plastic deformation, and fragmentation.

Schwarzkopf (1947) suggested that the terms "compactibility" and "compressibility" have been used to rank powders qualitatively according to their compaction characteristics. He proposed that "compactibility" be defined as the minimum pressure needed to produce a compact of given density and "compressibility" be used to indicate the extent to which the density of a given powder is increased under a given pressure.

Heckel (1961a) developed a method to obtain the relationship of both the "at-pressure" powder compact density and the "zero-pressure" compact density to the applied pressure from the continuous measurements of punch movement during a single compaction operation. He also suggested that the compaction of powdered metals was mainly carried out to increase the bulk density of the material and that this process was used for the attainment of minimum porosity and maximum densification. From his results, he concluded that the rate of increase of density with applied pressure at any given pressure was proportional to the volume fraction of pores.

Heckel (1961b) analyzed the compaction of metal powders through the density-pressure curves as a three-stage process - die filling, individual particle motion and rearrangement of particles, and gross compact deformation after the interparticle bonding has become appreciable. He suggested that the amount of densification in the first and second stages is a function of particle geometry and that taking place in the third stage is dependent on the material.

Cooper and Eaton (1962) studied the compaction behaviour of four ceramic powders of different hardness, but of same particle size, by measuring the travel of a laboratory hydraulic press. They concluded that softer powders yielded a greater fractional volume compaction at a given pressure than harder ones. They also described the two processes of compaction based on the size of the voids that are being filled relative to the size of the particles. The first process was the filling of voids which are of the same size as the original particles. The second process was the filling of voids which were substantially smaller than the original particles.

2.1.2 Compaction Behaviour of Food Powders

The compaction behaviour of various food powders have been studied by various researchers (Kumar 1973, Peleg et al. 1973, Peleg and Mannheim 1973, Peleg 1977, Rumpf 1961). Information on compressibility of powder foods under axial loads (loads applied only in one direction) is important in the development of pelleted foods. Such information could also predict the density of a given powdered food at any given pressure.

Kumar (1973) studied the density-pressure relationship under axial compression for ground whole corn and endosperm. He developed a theory of compaction on the basis of bulk compressibility of the materials. From the density-pressure data, he found that the change of density with pressure at any given pressure was proportional to the density of the given powder food. He also reported that his data were adequately described by an exponential relationship between the density and the pressure given as follows:

$$\ln D = B_{cf} P + A \quad (2.1)$$

where: A = Packing factor,
 B_{cf} = Compressibility factor,
 D = Bulk density, and
 P = Applied normal pressure.

Kumar (1973) stated that the packing factor, A, was the measure of low pressure densification due to the filling of the die, individual particle motion, and initial load. The compressibility factor, B_{cf} , was a measure of the ability of the powdered food to densify by plastic deformation. However, the equation that he proposed was valid only up to a pressure of 0.5 MPa.

Peleg et al. (1973) studied the flow properties of food powders under two broad classifications, namely non-cohesive powders that are relatively free flowing (e.g., flour and starch), and cohesive powders that have the tendency to cake during storage and that cause flow problems (e.g., powdered fruits and vegetables). The non-cohesive powders have less inter-particle attractive forces between them and hence are characterised by higher initial bulk density and lower compressibility. The cohesive powders have more inter-particle attractive forces between them characterised by open liquid bridges and hence have lower initial bulk density and higher compressibility. Rumpf (1961) stated that the cohesive forces depend on the distance between the particles and the number of contact points. When compressing a powder, the number of contact points increases; solid bridges are formed which cause the powder to harden and cake. The liquid bridge is formed when there is a liquid phase on the particle surface and can be the result of moisture absorption in the case of hygroscopic materials (Peleg 1977) or moisture liberation during the crystallization of amorphous sugars (Makower and Dye 1956).

Peleg and Mannheim (1973) stated that flow problems of cohesive powders could be eliminated by drying them to low moisture content and by the addition of anticaking agents. The anticaking agents compete with host powders for moisture. The anticaking agent functions mainly as a solid barrier between the powder particles thus reducing their attractive forces and as a lubricant of the solid surface thus reducing the friction between the particles.

A review of flowability problems in food powders and the methods for their evaluation was made by Peleg (1977). He described numerous experimental methods for the determination

of angle of repose, cohesion, tensile strength, angle of internal friction, bulk density, and compressibility to characterise whether the material is freely flowing or not.

Peleg (1979) and Peleg and Moreyra (1979) studied the effect of moisture on the stress relaxation pattern of compacted powders. They reported that the stress relaxation data, besides bulk density and compressibility data, could be utilized as complementary information to characterise the properties of powders with special reference to moisture. They found that the wet powders were more deformable than the dry powders and that the time required to reach the set force (the extent to which the density of a powder is increased under a given force) was considerably longer in the case of the wet powders (allowing for more stress relaxation to occur during the deformation stage).

The stress relaxation data were processed by Peleg (1979) and were applied to powders by Peleg and Moreyra (1979). In this method, the relaxation data are normalized and linearized and can be presented graphically as straight lines in the form of:

$$\frac{F_0 t}{F_0 - F(t)} = k_1 + k_2 t \quad (2.2)$$

where: F_0 = initial force,
 $F(t)$ = force after time t at relaxation, and
 k_1, k_2 = constants.

The slope of the line must be greater than 1.0 ($1 < k_2 < \infty$) and from a rheological point of view, the slope can be considered as an indication of the dry or wet state of the powders. In the case

of dry powders, there is little stress relaxation because the stress is mainly supported by a solid matrix and hence the slope approaches infinity. In the case of wet powders, there is a greater stress relaxation during deformation and hence the slope approaches unity. Moreyra and Peleg (1980) proposed two possible ways of stress relaxation in wet powders:

- (i) reorientation by flow of liquid bridges and
- (ii) the relaxation of the solid matrix itself, due to the visco-elastic nature of the particle materials, when they are plasticized by water absorption

Moreyra and Peleg (1980) studied the compressive deformation patterns of selected food powders through compression, decompression, and stress relaxation tests up to a pressure of 6 kPa. Their data, like those of Kumar (1973), were adequately described by an exponential relationship between the density and the pressure because of the application of very low pressures in the range of 6 kPa. They found that the cohesive powders were easily deformable and that upon decompression they remained compacted while the non-cohesive powders returned to a free flowing state. They attributed the compacted nature of the wet cohesive powders to the fact that such cohesive powders have more open structure supported by interparticle forces and liquid bridges. Such structures are weak and unstable and will collapse under relatively small stresses. Thus, the compressibility of moist powders in the lower range of compressive stresses will be greater than that of dry or less cohesive powders.

The above phenomenon was also manifested in the ratio of recoverable to non-recoverable work where Moreyra and Peleg (1980) observed that much of the work needed to compress the

wet cohesive powder was non-recoverable; an indication of plastic deformation and a substantial structural change in the powder. But the work needed to compress the dry powder was mostly recoverable and the small amount of non-recoverable work might be due to internal friction and to a limited amount of particle deformation and forcing of particles into small voids.

Scoville and Peleg (1981) evaluated the effects of liquid bridges on the bulk properties of model powders with increasing particle size. The bulk properties studied were bulk density, compressibility, cohesion, angle of internal friction, and the amount of irrecoverable work. They reported that the moisture absorption by food powders increases the liquid bridges in the powder. They found that the presence of liquid bridges in the powders of small particle size decrease the bulk density and angle of internal friction and increase the compressibility and the cohesion. However, they found that the overall effect of the liquid bridges on the bulk characteristics of the powder foods diminished as particle size increased.

Moreyra and Peleg (1981) also studied the effect of equilibrium water activity on the bulk properties (namely loose bulk density, compressibility, cohesion) of selected food powders (coffee creamer, infant formula, salt, and sucrose). They found that in the crystalline powders (e.g., salt and sucrose), the physical characteristics generally followed the pattern established by the moisture content with an apparent critical water activity beyond which changes became drastic. In the coffee creamer and infant powder, which were very cohesive even in the dry form, the changes in bulk properties with water activity were generally more gradual.

Malave et al. (1985) compared the compaction characteristics of selected food powders (wheat flour, rye flour, corn starch, soy protein, and instant coffee) by vibration, tapping, and mechanical compression. They validated Sone's model (Sone 1972) for the compressibility of food powders as a result of tapping. This model gives the relationship between normalized volumetric change $\gamma(n)$ and the number of tappings (n):

$$\gamma(n) = \frac{V_0 - V(n)}{V_0} = \frac{a'b'n}{1 + b'n} \quad (2.3)$$

where: V_0 = Initial (specific) volume,
 $V(n)$ = Volume after n tappings and
 a', b' = Constants.

The validity of the Eq. (2.3) was established by testing the linearity of the transformed relationship:

$$\frac{n}{\gamma(n)} = \frac{1}{a'b'} + \frac{n}{a'} \quad (2.4)$$

Ferdinand et al. (1990) studied the compaction behaviour of two dehydrated potato granule materials in a cylindrical geometry. They found a considerable effect of moisture content on the compaction behaviour and concluded that the performance of processes such as extrusion, that involves the compaction of these materials would vary considerably with the composition and moisture content.

Ramanathan and Cenkowski (1994b) made an attempt to characterize the axial compaction behaviour of two grades of flour in a cylinder-plunger arrangement. The influence of three moisture contents and three loading rates on the compaction behaviour was studied up to a pressure of 16 MPa. They concluded that compaction curves revealed curvature when log density vs. applied pressure was plotted. The results are similar to the findings of McNulty and Mohsenin (1979) for corn kernels.

2.1.3 Compaction Behaviour of Grains

McNulty and Mohsenin (1979) discussed the behaviour of low and high moisture whole corn kernels under compaction conditions leading to failure. They also discussed in detail the different density terminologies used in compaction studies. They used the punch and die set for compacting whole corn kernels. They obtained the density-pressure relationship of low and high moisture whole corn kernels at three different loading rates 0.028, 0.28, and 2.8 mm/s and found that the compaction curves revealed curvature when log density was plotted versus pressure. They found that low moisture corn kernels at 6.6% moisture content wet basis (wb) failed due to oil expression in the pressure range 140-310 MPa whereas high moisture kernels at 24.2% wb failed due to sample extrusion in the lower pressure range of 40-70 MPa. They also found that the highest temperature rise due to high pressure compaction was 8°C in case of low moisture corn at intermediate loading rate and concluded that no substantial thermally induced chemical, microbial, and /or physical changes could be anticipated in the material.

McNulty and Mohsenin (1977) also studied the compaction behaviour of whole corn kernels (1.8 - 22.0 % moisture content w.b.) by axial compression in an 18.5 mm diameter die using the Amatek Universal Testing Machine at 28°C. The maximum applied pressure was 34.6 MPa at loading rates of 0.028, 0.28, and 2.8 mm/s. They found that the samples did not fail either by oil expression or by sample extrusion probably due to the low pressure compaction. They also found that the low moisture content corn kernels (1.8% wb) experienced an extensive physical rupture and the high moisture content corn kernels (22% wb) were plastically deformed. However, they did not observe any macroscopic kernel damage. They also found that the effectiveness of compaction increased dramatically as the moisture content increased up to 22.0% and then appeared to decline gradually with further increase in moisture. The loading rates had little effect on the density-pressure profiles.

2.2 Methods of Water Activity Measurement

Labuza et al. (1976) reviewed different methods employed for the water activity determination of foodstuffs. The more common methods that have been used by researchers for the determination of moisture sorption characteristics of flour will be discussed here. These methods may be categorized as direct or indirect measurement of the water activity at a known moisture content.

2.2.1 Direct Method

In the direct method, a sample is first equilibrated to a determined moisture content and then the water activity is measured at this moisture content. This method utilizes the Lithium

Chloride (LiCl) cell, high vacuum techniques, and the wet bulb thermocouple for water activity measurement.

The LiCl cell is known commercially as a "relative humidity sensing element". This instrument is provided with a number of cells, each covering a short range, within an accuracy of $\pm 0.5\%$ relative humidity. The advantages are the short time required for each determination and the relatively low cost of the instrument. The disadvantages are its relatively poor accuracy, the necessity of calibrating the instrument periodically, and the need to equilibrate the samples to a number of moisture contents.

The direct measurement of vapor pressure by high vacuum technique is one of the most precise methods for the measurement of water activity. The evolution of respired gases by the biologically active materials during water activity determination limits its widespread use. Other disadvantages are the complexity of the instrument and the necessity of equilibrating the samples to different moisture contents.

The wet bulb thermocouple is highly precise in the water activity measurement of biological materials at high moisture levels. The disadvantages of this method are the necessity of measuring the thermocouple potential to an accuracy of 0.01 microvolt and the necessity of maintaining the sample temperature constant with a fluctuation of less than 0.001°C .

2.2.2 Indirect Method

The indirect method is the reverse of the direct method. The moisture content of the sample is equilibrated in an environment of known water activity, i.e., relative humidity, and then the moisture content is measured at this water activity.

This method employs the use of a constant relative humidity desiccator. In this method, a sample of material is allowed to equilibrate in a closed desiccator with a salt solution which produces the desired relative humidity. This method requires relatively long periods for equilibration and if high relative humidities are used, biological materials are susceptible to microbial and enzymatic spoilage.

2.3 Moisture Sorption by Flour and Its Constituents

The moisture sorption isotherms of flour, starch, and protein at different storage temperatures and their relationship to keeping quality and storage stability of flour have been considered by numerous researchers in the past.

Bailey (1920) investigated the hygroscopic moisture sorption of flour exposed to atmospheres of different relative humidity. He used the indirect method known as the constant relative humidity desiccator technique. He suggested that the moisture content of flour be obtained in atmospheres of different but constant relative humidity, after the flour comes into moisture equilibrium with the surrounding atmosphere. Such data are of use to:

- (i) shippers, purchasers and consumers who are concerned with changes in moisture content

and hence net weight of flour packages,

- (ii) baker and store-keepers who need such data to predict the effects of changing moisture content on the shelf-life of flour during a long-term storage, and
- (iii) millers wherein such data are of use in establishing a favourable atmospheric humidity in mills.

Bailey (1920) also concluded that flour at a moisture content of 12.0% wb did not lose or gain any moisture when stored at a relative humidity of 70% at a temperature of 25°C.

Fairbrother (1929) investigated the influence of atmospheric humidity on the moisture content of flour and wheat and hence on the net weight changes of flour stored in bags. He used the same technique as that employed by Bailey (1920) for creating a known relative humidity environment. He described a situation in England where a grocer was fined for selling bags of flour that showed varying shortages in weight from 1.78% to 4.01% and an average loss of 2.38%. But, the flour was packaged and stored during the hot dry weather conditions in England and inspected during the period of the lowest relative humidity of the year and hence the loss in weight.

Anker et al. (1942) investigated the net weight changes and moisture content of wheat flour packed in different sizes of cotton and paper stacks exposed to various controlled relative humidities until the equilibrium was attained. The flour used was the 83% patent flour and the different sizes of packages considered were 2.3, 4.5, and 11.1 kg. They observed that flour responds more readily to changes in relative humidity than does the bulk grain because of its

finely divided state and the method of handling. They also suggested that small packages exhibit greater and more rapid changes in net weight and moisture content than the larger ones when stored under same conditions.

Sair and Fetzer (1944) studied the relative hygroscopicity of different starches namely, cereal (wheat, rice, and corn), pith or root starches (sago, arrowroot, and sweet potato), and tuber starches (potato). They reported that the different starches differ in their amount of moisture retention which is associated with the availability of free hydroxyl (-OH) groups. They also suggested that if the -OH group is the point of attachment for water, then tuber or potato starches have more free -OH groups ready for the attachment of water molecules than have either cereal or root starches.

Pauling (1945), studying the adsorption of water by proteins, suggested that polar side chains of amino acid residues of proteins provide active polar sites for the adsorbed water molecules. He studied the amino acid composition of proteins in comparison to the monomolecular layer of adsorbed water. He concluded that initial adsorption takes place to the extent of one water molecule per side chain for some proteins.

Morey et al. (1947) used the constant RH method to study the relationship between the moisture content of flour and the atmospheric humidity (0 to 80%) at temperatures (50 to 95°C). They found that an increase in temperature caused a decrease in the time required for reaching equilibrium at any given humidity. They also concluded that the decrease in equilibrium

moisture content with an increase in temperature at any given humidity was more pronounced at higher humidities than at lower humidities.

Hellman and Melvin (1948) studied the water sorption by corn starch as influenced by preparatory procedures and time. They used the desiccator technique to obtain the desired relative humidity. They found that the moisture sorption isotherm of ten corn starches, in the range of RH's from 12 to 93%, was not altered by the various preparatory procedures for the starches.

Benson et al. (1950) determined the sorption of water vapour by proteins and based on the moisture sorption data, they have also calculated the surface area of proteins using the well-known BET theory proposed by Brunauer et al. (1938). They compared the water surface area of proteins with the nitrogen surface area and found that the water surface area is several orders of magnitude larger than the nitrogen surface area. This is due to the fact that water can act as a plasticizing agent (water molecules being small are able to get into various pores and crevices and saturate different molecules that make up the protein molecules and thereby impart flexibility, extensibility or stretchability of the protein molecules). They concluded that nitrogen adsorption is a bulk property of the material whereas the water adsorption is a specific molecular property of the protein itself.

Bushuk and Winkler (1957) used the McBain-Bakr sorption balance in conjunction with the high vacuum technique to determine the adsorption and desorption isotherms for water on

wheat flour, starch, and gluten. They made the first ever attempt to account for the sorptive capacity of flour on the basis of its physical and chemical characteristics. They found that the adsorption of water by flour is a bulk property of the material and not dependent on the particle size.

Gur-Arieh et al. (1965a) found a method for the rapid determination of moisture adsorption isotherms of solid particles. They filled different bottles with different concentrations of sulphuric acid solutions, used to create a known RH, and used a peristaltic pump to recirculate the air at constant RH through the sample tubes. Though this method is again indirect, the equilibrium was attained much faster than the constant RH desiccator technique.

Gur-Arieh et al. (1965b) also used a pressure membrane cell to determine the water activity (a_w) of high moisture flour. In this method, the flour was allowed to equilibrate with water contained in the porous membrane while under a certain constant mechanical pressure. The cell was fitted with a porous membrane which allowed transfer of moisture from and to the sample.

Gur-Arieh et al. (1967) studied the moisture adsorption by wheat flours and their cake baking performance. They found that the moisture adsorption isotherms of wheat flour was independent of the particle size distribution of flour. They concluded that water adsorption by flour is not a surface phenomenon but rather takes place on the specific active sites within the pores inside the particles. They suggested that the sorptive capacity of flour decreased with

increasing protein content and that of starch fractionated from flour was higher than that of the gluten fraction.

Udani et al. (1968) investigated the rate of moisture adsorption by wheat flour and its relation to physical, chemical, and baking characteristics. They found that the moisture adsorption rate (R) of flour decreased exponentially with time. They concluded that R increased linearly with an increase in relative humidity of the air and decreased with the protein content of flour. However, R did not change with particle size and was not related to baking quality.

Kumar (1974) studied the water vapor adsorption on whole corn flour, degermed corn flour, and corn flour containing the germ at 25 and 50°C at relative humidities in the range of 11-97%. He attempted to account for the sorptive capacity of flour on the basis of the surface area availability (using the BET equation) and the free energy change. He concluded that surface area based on water sorption was more for degermed flour than for flour containing the germ.

Henderson and Pixton (1982) used the constant RH desiccator technique to obtain the adsorption and desorption isotherms of 5 types of wheat flour at 5, 15, and 25°C. They found that the temperature had hardly any effect on the sorptive capacity of flour.

Mok and Dick (1991) studied the moisture adsorption characteristics of wheat starch, damaged by ball milling, at 30°C using the constant relative humidity desiccator technique. They found that in the water activity range of 0.097-0.462 (region where the monolayer water

adsorption takes place), the damaged starch had a lower sorptive capacity (and hence had a lower equilibrium moisture content) than the undamaged starch. They predicted that the free hydroxyl groups resulting from the cleavage of covalent bonds of amylose and amylopectin during ball milling might have had a greater tendency to form hydrogen bonds among themselves or with other hydroxyl groups in the molecules. This in turn would decrease the number of polar sites accessible to water. On the other hand, in the water activity range of 0.666-0.943 (region where multilayer adsorption and capillary condensation of water take place), the damaged starch had a higher sorptive capacity (and hence had a higher equilibrium moisture content) than the undamaged starch. This might be due to the increased amorphous region of the molecules.

Ramanathan and Cenkowski (1994a) studied the sorption isotherm of all-purpose flour as influenced by flour compaction at 25°C using a Novasina water activity meter. They observed that compaction of flour had little effect on its moisture sorption isotherm in the range of water activity studied (0.1 to 0.9).

2.4 Moisture Sorption in Pharmaceutical Applications

The influence of moisture sorption by pharmaceutical powders in relation to their compressibility has been the subject of study for many years. Scott et al. (1963) reviewed the pharmaceutical applications of the concept of equilibrium moisture content of powders. They suggested that if there are no physical changes (such as the change in particle size of the powders) or chemical reactions taking place during tableting, the equilibrium moisture content

before and after compression would remain the same. The moisture adsorption rate of the tablets is governed by their hardness. The increased hardness of the tablets results in a decreased moisture transfer within the solid and hence in a decreased rate of moisture gain. Thus, the stability of the tablets is increased by an increase in their hardness.

Shotton and Rees (1966) studied the compaction properties of sodium chloride in the presence of moisture. They concluded that at increased pressures, the dry materials produced compacts of greater strength than did the wet materials.

Down and McMullen (1985) and Kontny et al. (1987) have studied the moisture sorption of water-soluble crystalline substances (mainly sodium chloride) subjected to various pre-treatments such as compaction and grinding. They suggested that the compaction process disrupts the surface structure of sodium chloride which permits enough water vapor sorption to produce a metastable state of dissolved sodium and chloride ions. Subsequent recrystallization of the sodium chloride compact, upon standing and drying, increases its hardness.

2.5 Rheological Behaviour of a Flour-Water Dough

The rheological measurements of a flour-water dough may be divided into two main categories:

- 1) The fundamental rheological testing
- 2) The empirical and imitative rheological testing.

2.5.1 Fundamental Tests

The fundamental rheological testing includes cone-and-plate, parallel plate, and capillary tube viscometers. The use of capillary viscometers for determining the flow properties of foodstuffs is the main subject of this review.

The importance of the fundamental rheological properties of the dough and the methods to evaluate the same have been described (Bushuk 1985; Faubion and Hosney 1989; Hibberd and Parker 1975; Hosney 1985; Loh 1985; Menjivar 1989).

Bushuk (1985) reviewed the theory and applications of fundamental rheological considerations to a wheat-flour dough. He stated that such information gives a knowledge of the inter-relationships between the molecular structure of materials and their rheological properties in solid or solution state. This, in turn, helps a great deal in the control of raw materials and hence in the quality of the final baked product. Such information can also be used as a guide in the design of machines for industrial processes. Measurements of the rheological properties of dough are widely used for the selection of new cultivars in breeding, for quality control in mills and bakeries, and for the design of adaptations of processes in the milling and baking industry (Bloksma 1990).

Hosney (1985) studied the rheology of fermenting dough and discussed the need to prepare the dough to an optimum water absorption and an optimum mixing time. He discussed two reasons that would complicate the study of the rheology of a fermenting dough. First is the

drop in pH value due to the formation of carbonic acid as CO_2 produced during fermentation dissolves in water. Second is the entry of CO_2 into the preexisting air cells causing the leavening of dough which in turn would change the geometry of the dough piece.

Loh (1985) discussed the fundamental tests for the rheology of soft wheat products, that are useful in the textural characterization of baked foods made from soft wheat flours. He used creep, relaxation, and dynamic strain application tests to characterize viscoelasticity.

Blake and Moran (1975) developed a capillary extrusion rheometer to study the flow characteristics of safflower, soybean, rapeseed, and castor oils representing Newtonian fluids, and honey, peanut butter, shortening, mayonnaise, and molasses representing non-Newtonian fluids. The capillary is enclosed in a hollow plunger and the plunger is driven at a series of fixed rates into a barrel containing the test sample. There is no motion of the fluid relative to the walls of the barrel and frictional forces between the fluid and the walls of the barrel are avoided. There is no air entrapment between the sample and the plunger thus preventing gas compression effects.

Mackey and Ofoli (1990) assessed the validity of using the model, proposed by Mackey et al. (1989), for predicting the viscosity of whole wheat flour extrudates. They used an Instron capillary rheometer and a Baker Perkins MPF-50 D/25 corotating twin-screw food extruder to collect viscosity data of flour extrudates. The model proposed by Mackey et al. (1989) has been shown to predict accurately the viscosity of pure starches. This model incorporates the effects of shear rate, moisture content, temperature, time-temperature history, and strain history.

However, this model failed to predict the viscosity of whole wheat flour extrudates. Mackey and Ofoli (1990) attributed the lack of accuracy in the model to the presence of flour components such as bran, lipids, and proteins, which were not accounted for by the model and which most probably altered the starch gelatinization kinetics.

Dail and Steffe (1990) used the tube viscometer system to determine the rheological parameters (namely the flow behaviour index and the consistency coefficient) for the waxy maize starch solutions at three different concentrations (2.72, 3.14, and 3.59 g of dry starch per 100 g water) at 121.1°C. Dilatancy behaviour (i.e., increase in the apparent viscosity with increasing rates of shear) was observed in starch solutions under low acid aseptic processing conditions.

A capillary extrusion rheometer was developed to obtain basic information on the flow characteristics, namely the flow behaviour index and the consistency coefficient, of a wheat flour dough (Sharma and Hanna 1989; Sharma et al. 1990; Sharma and Hanna 1992; Sharma et al. 1993). A knowledge of such information would help in further automation of the baking industry. The authors monitored the dough flow rate and the force needed to extrude the dough out of the capillary. They concluded that the dough exhibited pseudo-plastic or shear thinning behaviour (i.e., decrease of coefficient of viscosity with increasing shear rate) in the shear rate range from 9 to 5000 s⁻¹ with an average flow behaviour index of 0.34 and consistency coefficient of 2395 Pa-s^{0.34} and that the flow curves were independent of capillary dimensions. They also observed that the flow behaviour index of dough was not affected by water content of dough (35, 45, and 55%) and protein content of flour (20, 30, and 40% - higher protein

content flours were obtained by mixing bakery flour and gluten in a mixer). But the consistency coefficient of dough decreased with increasing water content of dough and increased with protein content of flour.

2.5.2 Empirical and Imitative Rheological Tests

Various dough handling instruments, such as Farinograph, Mixograph, Alveograph, Visco-Amylograph are used to characterize the rheological properties of a flour-water dough but in terms of arbitrary units such as Brabender Units (BU). Various attempts have been made to express these units in terms of the Engineering units as used in the design and selection of various pieces of equipment for dough handling. The advantages of these empirical methods are:

- 1) They are relatively easy and fast to perform and the methods are well established. This makes them very practical.
- 2) The instruments needed to perform the tests are relatively inexpensive.
- 3) They are close simulators (rather than model systems) of the dough mixing process and dough elasticity requirements for the baking process.

Kunerth and D'Appolonia (1985) studied the extensive use of the physical dough testing instruments, such as the Farinograph and Mixograph, for wheat quality evaluation studies. They stated that mixogram characteristics tended to reflect the baking strength of a flour because of the high correlation between loaf volume and protein, and the correlation between protein and mixogram characteristics.

Rasper et al. (1985) discussed the use of alveography in quality evaluation of soft wheat flours. They suggested that the biaxial extension produced during the dough bubble inflation is well linked with the gas cell expansion in the rising dough during fermentation and early stage of baking and therefore this may serve as a valid tool in assessing the overall baking quality of the tested flour. They also made an attempt to compare the data obtained using the standard procedure, i.e., using doughs of constant water content, with results on doughs mixed to an identical consistency under the conditions of a variable flour/water ratio.

Dexter et al. (1985) studied the relationship of four flour starch damage levels (10, 23, 33, and 55 Farrand units) and five flour protein levels, %N X 5.7, (11.2, 11.8, 12.5, 12.7, and 13.5%) to the quality of Hearth bread, made using a short straight dough process, and Pan bread, made using the Grain Research Laboratory remix process, produced from Hard Red Spring Wheat. The flour was milled to the same extraction. They found that hearth bread quality was mainly affected by starch damage and remix bread quality was mainly affected by protein content. Evaluation of the physical dough properties was based on Farinograph, Extensigraph, and Alveograph tests.

Dexter et al. (1994) studied the effects of protein content and starch damage of flour on the physical dough properties and bread making quality of Canadian Durum Wheat. Durum wheat finds extensive use in some countries for the production of leavened bread. However, in Canada, it is mainly used for the production of pasta products. Recently, Canadian breeding programs made it imperative that strong gluten be one of the prerequisites in durum wheat for

good pasta making quality. Thus, the use of durum wheat for the production of bread became possible for the Canadian Durum wheat importers. They concluded that depending on protein content, starch damage, and baking procedure, durum wheat loaf volume was 65 to 85% of that achieved for a high quality Canadian bread wheat of comparable protein content.

Weiland (1977) patented a process for the treatment of cereal flour by compressing the flour to produce a granulate with higher initial bulk density than that of the original flour. In this process, the flour was moistened to a moisture content of 13% (wet basis) by passing steam and was heated to a temperature of at least 25°C, before compression. The flour was then ground and tested for baking quality. It was concluded that the baking quality and storage properties of the baked foods prepared from the treated material was improved. It was shown that the heat generated during compaction resulted in the modification of the gluten characteristics. Weiland (1977) also showed that such a treatment could improve the baking quality of the flour, obtained from a cereal damaged by severe sprouting.

3. ANALYSIS OF DATA

3.1 Calculations from Sorption Data

The following notations will be used in this work:

a_w = water activity, decimal fraction

M = equilibrium moisture content, % db (dry basis)

T = temperature, K

R = universal gas constant, 8.314 J/mol•K

3.1.1 BET Isotherm

The isotherm model with the greatest popularity in all fields of interest is the BET isotherm equation after the work of Brunauer et al. (1938) (Labuza 1968). The basic assumptions made are:

- (i) the heat of sorption of water for the first layer, Q_1 , is constant and is equal to the sum of the total heat of vaporization (ΔH_v) and heat due to site interaction (Q_s).
- (ii) the heat of sorption of water for all layers above the monolayer is equal to the heat of vaporization.
- (iii) sorption occurs only on specific sites.

The BET equation is

$$\frac{a_w}{(1-a_w)M} = \frac{1}{M_m C} + \frac{a_w(C-1)}{M_m C} \quad (3.1)$$

where: M_m = monolayer moisture content: the moisture content at which all the active sorptive sites are covered by a monomolecular layer of water,

C = constant related to the net heat of adsorption.

A plot of $a_w/\{(1-a_w)M\}$ vs a_w gives a straight line and from the slope and intercept of this line the monolayer coverage values (M_m and C) can be calculated.

Because of the over simplified assumptions of the BET isotherm equation, the equation generally holds only between water activities from about 0.1 to 0.5, but this gives enough data so that the parameters M_m and C can be calculated (Labuza 1968). The value of M_m can be used to calculate the specific surface area of the adsorbent (S_o , m^2/g) based on an assumption that the monolayer moisture is adsorbed on the entire surface of the adsorbent. The determination of the monolayer values gives additional information on the availability of sorptive sites in the flour due to compaction. The specific surface area can be calculated as:

$$S_o = M_m \frac{1}{M_{H_2O}} N_o A_{H_2O} \quad (3.2)$$

where:

S_o = specific surface area of the adsorbent, m^2/g

M_{H_2O} = molecular weight of water, 18 g/mole

N_o = Avogadro's number, 6×10^{23} molecules/mole

A_{H_2O} = Area of water molecule, $10.6 \times 10^{-20} m^2$

3.1.2 Temperature Effects and Heat of Sorption

When water vapor is adsorbed on a surface, a quantity of heat called the heat of adsorption is released. When adsorbed water vapor is desorbed, a quantity of heat is taken up

(the heat of desorption). This quantity is a measure of the heat or energy that must be added to break the inter molecular attractive force (typical of a dehydration step). Thus, heat of sorption indicates the binding energy or the inter molecular attractive forces between the molecules of water vapor and the surface of the adsorbent (Chung and Pfost 1967).

In general, sorption phenomena obey the Clausius-Clapeyron relationship shown in the equation below (Labuza 1968):

$$\frac{d(\ln a_w)}{d(1/T)} = -\frac{Q_s}{R} \quad (3.3)$$

where: Q_s is the heat due to site interaction, kJ/mol.

If isotherms are made at several temperatures, they should show a decrease in the amount of water adsorbed with an increase of temperature at constant water activity. This effect is more pronounced at lower to intermediate water activities. From the data obtained in this manner, when the values of a_w are plotted vs. the reciprocal of absolute temperature (at constant moisture content), a straight line is obtained and the slope of the line is $-Q_s/R$. The value of Q_s signifies the amount of energy that must be supplied to desorb the last traces of water. As the moisture content increases, this heat is almost equal to the heat of vaporization of water and hence the slope of the line decreases to zero. The determination of heat of sorption values is quite important in the process of dehydration as these values give an indication of the amount of heat, in addition to the heat of vaporization, which must be added to achieve the drying process.

3.1.3 Free Energy Changes of Sorption

Free energy change is the energy required to transfer the water molecules from the vapor state to the solid surface or from the solid surface to the vapor state (Chung and Pfof 1967). This can be considered as a measure of the work done by the system to accomplish the adsorption or desorption process.

The free energy change of sorption can be expressed by (Chung and Pfof 1967):

$$\Delta F = R T \ln(a_w) \quad (3.4)$$

where: ΔF = free energy change of sorption, kJ/mol.

The free energy change of desorption is always greater than that of adsorption. Chung and Pfof (1967) stated that the free energy change of sorption decreases continually with increase in moisture content and proposed the following relationship:

$$-\Delta F = A \exp(-BM) \quad (3.5)$$

where A and B are constants. The constants, however, are temperature dependent.

3.1.4 Isotherm Equation

There are a number of isotherm equations available to model the sorption phenomena. Young and Corwell (1962) remarked that a theoretical equation which may account for the complete sorption phenomena is too complex and has too many parameters to be of any use. The isotherm equation employed here is a three-parameter Modified Halsey equation proposed and

shown to be valid for wheat flour, from the data obtained by Bushuk and Winkler (1957), by Iglesias and Chirife (1976). This equation takes into account the effect of temperature on the water sorption isotherms of flour. The main reason for choosing the above equation is to analyze the parameters for their test of significance to identify if isotherms of non-compacted flour are different from those of compacted flour pellets in the range of temperatures (10, 25, and 40°C) studied.

Modified Halsey Equation

Iglesias and Chirife (1976) empirically modified the Halsey equation (Halsey 1948) to the following form:

$$a_w = \exp[-\exp(A + BT)M^{-C}] \quad (3.6)$$

where A, B, and C are constants. The constants are temperature independent.

3.2 Calculations For Dough Flow Behaviour

The wall shear stress is calculated as (Toledo 1991):

$$\tau = \frac{\Delta P r}{2L} \quad (3.7)$$

where: τ = shear stress (Pa),

ΔP = pressure drop across the capillary (Pa),

r = radius of the capillary (m), and

L = length of capillary (m).

The pressure drop data are obtained by dividing the force values by the cross-sectional area of the plunger.

The apparent shear rate is calculated as (Toledo 1991):

$$\dot{\gamma} = \frac{4Q}{\pi r^3} \quad (3.8)$$

where: $\dot{\gamma}$ = shear rate (s^{-1}),

Q = volumetric flow rate through the capillary (m^3/s), and

r = radius of the capillary (m).

The Rabinowitsch-Mooney correction is applied to obtain the corrected shear rate data (Toledo 1991):

$$CF = \frac{3}{4} + \frac{1}{4n} \quad (3.9)$$

where:

CF is the correction factor and n is the slope of the plot of log shear stress and log shear rate.

The corrected shear rate ($\dot{\gamma}_w$) is then obtained by multiplying the apparent shear rate ($\dot{\gamma}$) by CF (Toledo 1991);

$$\dot{\gamma}_w = \dot{\gamma} CF \quad (3.10)$$

Using the power law model (Toledo 1991),

$$\tau = K \dot{\gamma}_w^n \quad (3.11)$$

both, the flow behaviour index, n , and the consistency coefficient, K , can be determined.

4. MATERIALS AND METHODS

4.1 Description of Samples

Flour used in this study was milled from Grade No.1 Canadian Western Red Spring (CWRS) wheat of the 1993 harvest crop in a pilot scale flour mill at the Grain Research Laboratory, Canadian Grain Commission, Winnipeg. The protein content, ash content, and falling number of wheat were 12.0%, 1.58%, and 420 s, respectively. Flour was milled using wheats conditioned to three different moisture contents 14.5, 15.8, and 16.8% wb. Two different grades of flour were used in this investigation: a low extraction (64%) patent flour and a high extraction (79%) straight grade flour, of three different moisture contents, 13.1, 13.9 and 14.6% wb. The above flour moisture contents were chosen to simulate the flour available in the market for commercial and domestic applications (Dexter 1994).

4.2 Experimental Apparatus

4.2.1 Determination of Compaction Behaviour of Flour

The apparatus for studying the compaction behaviour of flour, consisting of a cylinder-plunger arrangement, is shown in Fig. 4.1. The plunger, made of steel, was connected to the load cell of the ATS Universal Testing Machine (Applied Test Systems, Inc., Butler, PA). The cylinder was firmly attached to the supporting collar. The entire assembly was placed on a stand which in turn was positioned under the crosshead of the Universal Testing Machine (UTM).

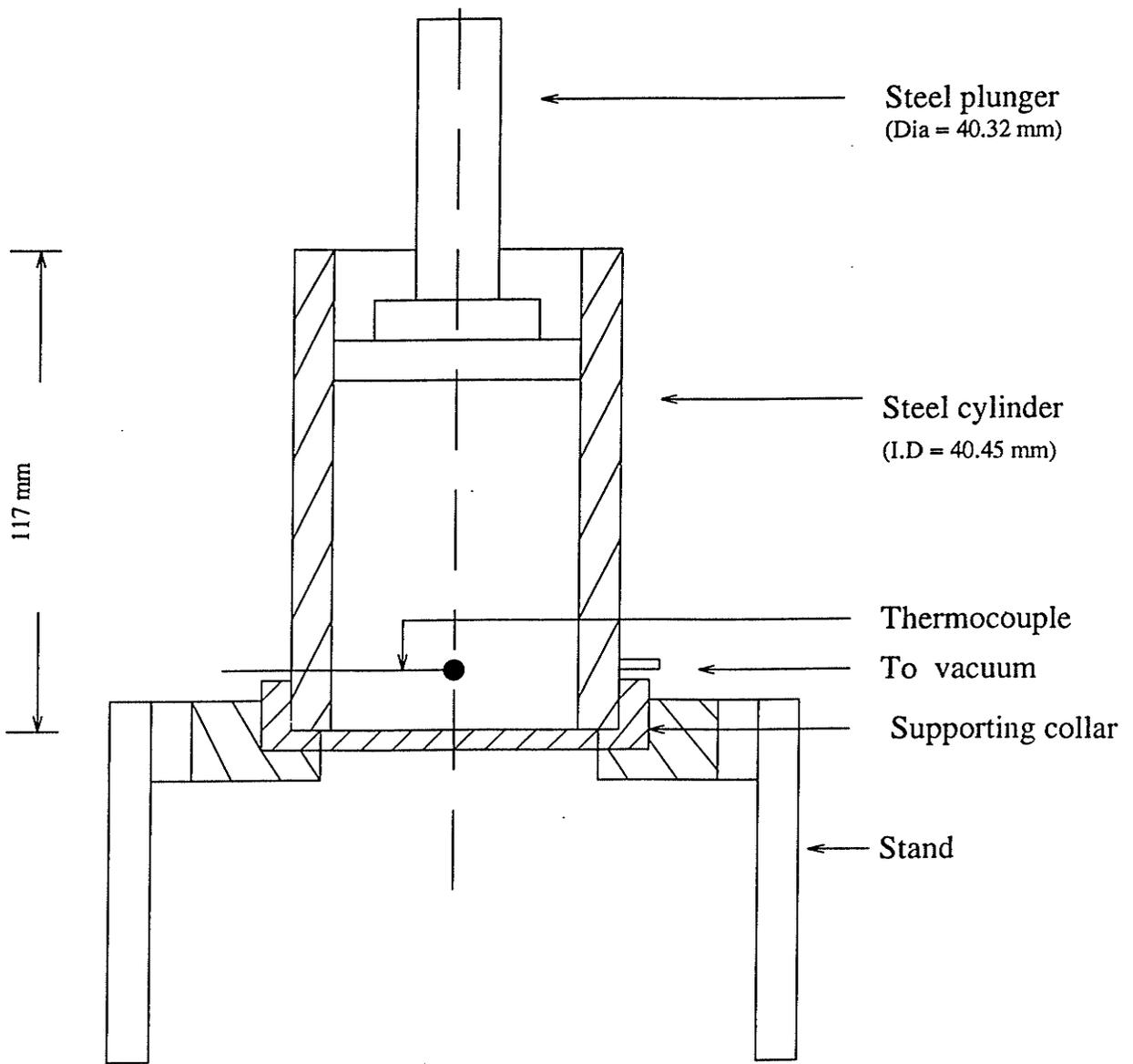


Fig. 4.1 A cylinder-plunger arrangement used for compaction of flour

The top portion of the stand had an opening whose diameter was 0.25 mm larger than the outside diameter of the cylinder. A groove was cut in the opening to a depth of 5 mm and its diameter was 0.2 mm more than the inside diameter of the cylinder. Once the flour was compacted to a given deformation, the supporting collar was removed and the cylinder was positioned on the groove cut on the top of the stand. This was to ensure that the compacted flour granulate was safely ejected out of the cylinder through the opening on the top of the stand. A small hole, just wide enough to insert a thermocouple wire, was drilled 10 mm above the bottom of the cylinder. The purpose was to measure temperature changes due to compaction. The cylinder was connected to a vacuum pump in order to remove air from interparticle space.

4.2.2 Determination of Water Activity of Flour

Novasina a_w meter (Model: a_w center, Novasina Type, Switzerland) was used for the determination of water activity (a_w) of the flour. The calibration and operation of the instrument were according to manufacturer's instructions.

The Novasina a_w meter is used to perform the temperature controlled water activity measurements. The internal temperature of the a_w meter can be digitally set to any value between 0°C and 50°C according to the table on the temperature regulator. The maximum difference between the set value and the value shown by the digital display is less than 0.2°C. According to the manufacturer's specifications, the ambient temperature changes would affect the internal temperature of the a_w box by a maximum of 0.04°C/°C.

4.2.3 Capillary Rheometer for Dough Flow Behaviour

A capillary extrusion rheometer, quite similar to the one employed by Sharma et al. (1993), was used to obtain the flow characteristics of a flour-water dough. By this, the flow behaviour index and the consistency coefficient of the dough, in standard units as used in engineering design, were determined (Sharma et al. 1993). The rheometer consisted of a cylinder-plunger and a capillary arrangement (Fig. 4.2). The cylinder made of brass had an inside diameter of 19.1mm and a length of 136mm. The capillary also made of brass had a diameter of 3.15mm and a length of 101.2mm. The cylinder was screwed into the top portion and the capillary into the bottom portion, of the supporting collar so that the bottom surface of the cylinder would be flush with the top surface of the capillary. Also the supporting collar would taper from the diameter of the cylinder to the diameter of the capillary at the point of contact of the cylinder and the capillary. A brass plunger made of brass had an outside diameter of 18.8mm and was connected to the load cell of the ATS Universal Testing Machine. The plunger was used to push the dough through the capillary.

The supporting collar, with the cylinder at the top and the capillary at the bottom, was placed on a stand and the entire assembly was positioned under the crosshead of the Universal Testing Machine. The cylinder was exactly in line with the plunger and when the crosshead moved down, the plunger would slide down inside the cylinder smoothly thereby extruding the dough through the capillary.

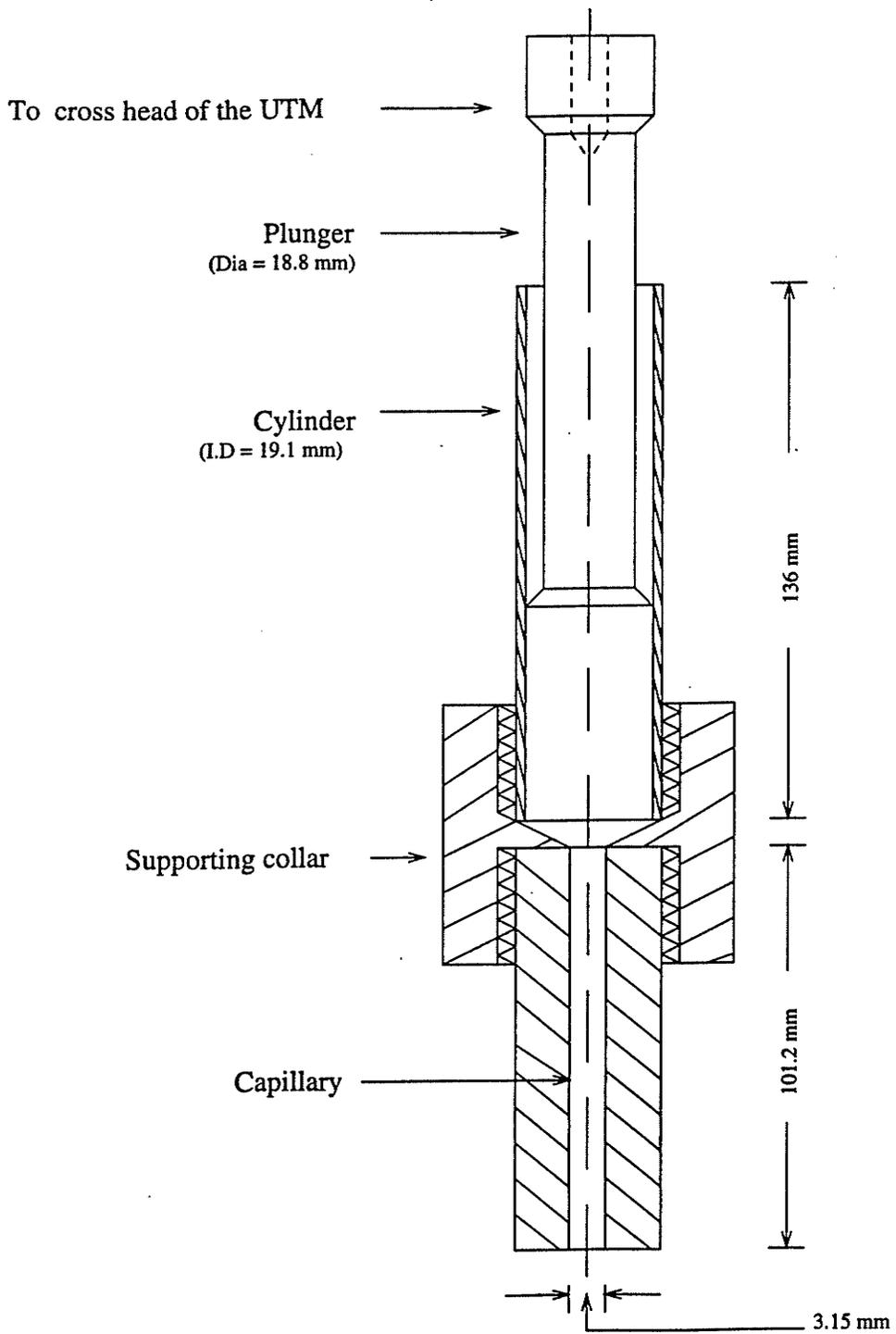


Fig. 4.2 Schematic diagram showing a capillary rheometer

4.3 Preparation of Test Samples

For the preparation of adsorption and desorption isotherm test samples, the intermediate moisture content flour namely, the flour at 13.9% moisture content (m.c.) wet basis, was used for both grades of flour.

4.3.1 Preparation of Samples for Adsorption Isotherm Tests

To prepare samples for the determination of adsorption characteristics, approximately 1.5 kg of the 13.9% m.c. flour was dried at 40-45°C and 10-20% relative humidity until the samples reached approximately 5% moisture content. The drying time with the above conditions was approximately 6h. The low drying temperature was selected because Bushuk and Winkler (1957) have found that heat treatment of flour at 100°C for 24h reduced its sorptive capacity by about 20%. The low moisture content flour prepared in the above manner was divided into 6 equal sub-samples and exposed to a high humidity (about 90%) environment at a temperature of 10°C for progressively increasing periods of time until the designated sub-samples reached approximately 6, 8, 10, 12, 14, and 16% moisture content. This was done by measuring the continuous increase in the mass, using a weighing balance, of the samples to get to a high final moisture content from a low initial moisture content. The moisture contents up to 10%wb were attained within 1h whereas the moisture contents above 10%wb were attained between 2 and 4h. Once the desired moisture contents were attained, the various sub-samples were transferred into plastic ziploc bags, mixed thoroughly, and stored at 10°C. The water activity of each sub-sample was measured using the Novasina a_w meter at 10, 25, and 40°C.

4.3.2 Preparation of Samples for Desorption Isotherm Tests

Samples used for the determination of desorption characteristics were prepared as follows. Approximately 1.5 kg of the 13.9% m.c. flour was hydrated by exposing a thin layer of flour to a relative humidity of 90% and a temperature of 10°C until the samples reached about 17.5% moisture content. The wetting time with the above conditions was approximately 4h. The high moisture content flour prepared in the above manner was divided into 6 equal sub-samples and exposed to a drying temperature of 40-45°C and a relative humidity of 10% for progressively increasing periods of time until the designated sub-samples reached approximately 16, 14, 12, 10, 8, and 6% moisture content. The moisture contents up to 12%wb were attained within 1h and the moisture contents below 12% were attained between 2 and 4h. Once the desired moisture contents were attained, the various sub-samples were transferred into separate plastic ziploc bags, mixed thoroughly, and stored at 10°C before they were used for the a_w measurement. The water activity of each sub-sample was measured using the Novasina a_w meter at 10, 25, and 40°C.

4.3.3 Preparation of Flour Pellets for Sorption Measurements

The flour pellets, for the determination of sorption characteristics of compacted flour, were prepared on a single-punch tableting machine (Manesty Type F3, Liverpool, England) using a 12.8 mm diameter punch and die set. A flour sample of 0.8 g was compacted in the form of a small pellet of diameter and height approximately 12.8 mm and 5.3 mm (± 0.2 mm), respectively. The original height of the flour before compaction was 11.8 mm, corresponding to a reduction in flour volume of 55% ($\pm 2\%$). Two layers of pellets, with each layer having about 7 pellets, were placed in the sample bowl for a_w determination. The same procedure for

making pellets was repeated for each of the samples conditioned for adsorption and desorption isotherm tests.

4.3.4 Preparation of Flour Granulates for Rheological Testing of Dough

Only the intermediate moisture content flour (namely the flour at 13.9% moisture content wb) was used for the rheological testing of a flour-water dough. The cylinder-plunger arrangement, as described earlier in section 4.2.1 for the determination of the compaction behaviour of flour, was used for the preparation of flour granulates. The only difference was that the dimensions of the cylinder were 27.1 mm diameter and 235 mm height and that of the plunger was 26.9 mm diameter. Approximately 65g (± 0.1 g) of flour was used for each trial. The loading rate was kept constant at 100 mm/min. The deformation was adjusted so that the compaction would result in a 55% density increase. The average pressure required to achieve this deformation was 7.62 MPa (± 0.21 MPa) in the case of the patent flour and 6.64 MPa (± 0.30 MPa) in the case of the straight grade flour. The compacted flour granulates were then stored for one month at 10°C. The compacted flour granulates were then reconstituted to investigate the rheological behaviour of a flour-water dough as prepared using the flour reconstituted after compaction.

4.4 Experimental Methods and Standards

All of the following determinations were made for the non-compacted flour and the flour reconstituted after compaction and for the dough prepared using both flours of both grades. The dough prepared using the non-compacted flour and the flour reconstituted after compaction will

be referred to as control and treated, respectively.

4.4.1 Flour Moisture Content

The moisture content of the flour was determined by drying 3 g of the sample in a convection oven at 130°C for 1 h according to the Approved Methods of the American Association of Cereal Chemists (method 44-15A, AACC 1983).

4.4.2 Farinograph

The water absorption by flour, the dough development time, mixing tolerance index, and the stability were determined using a farinograph (Brabender Instruments Inc., South Hackensack, New Jersey) according to the Approved Methods of the American Association of Cereal Chemists (method 54-21, AACC 1983).

4.4.3 Mixograph

The mixograph peak time and mixograph peak height were determined using a mixograph (Bodine Electric Co., Chicago, USA) according to the Approved Methods of the American Association of Cereal Chemists (method 54-40, AACC 1983).

4.4.4 Amylograph

The amylograph peak viscosity was determined using the visco-amylograph (Brabender Instruments Inc., South Hackensack, New Jersey) according to the Approved Methods of the American Association of Cereal Chemists (method 22-10, AACC 1983).

4.4.5 Alveograph

The alveograph is a rheological measuring instrument for the determination of the quality of wheat and flour. It measures the air pressure required for biaxial extension of a sample of

dough, an indication of dough strength. Alveograms were obtained with an MA 82 Alveograph (Chopin SA, Villeneuve-La-Garenne, France) at 50% water addition (15% flour wet moisture basis) as recommended in the ICC procedure (Standard No. 121, ICC 1980).

4.4.6 Starch Damage

The flour starch damage was determined according to the Approved Methods of the American Association of Cereal Chemists (method 76-30A, AACC 1983).

4.4.7 Flour Wet Gluten Content

The wet gluten content of flour was determined according to the ICC procedure (Standard No. 137, ICC 1980).

4.4.8 Bake Test

The remix loaf volume baking test of the Canadian Test Baking Procedures was conducted as per the method proposed by Kilborn and Tipples (1981).

4.5 Experimental Procedure

4.5.1 Compaction Behaviour of Flour

Approximately 72 g (± 0.1 g) of flour was used in the compaction tests and the same quantity was filled in the cylinder for each trial. The cylinder was firmly attached to the supporting collar and the whole assembly was placed on the stand. The plunger was connected to the load cell of the UTM and the cylinder was positioned exactly below the plunger. To ensure uniformity in testing procedure, the plunger was always lowered to the same position (17mm below the top surface of the cylinder) before the start of each test. Thus, the effective height of the sample was 100mm. This was done to ensure that the surface of the plunger was

just touching the top surface of the flour. The deformation of the plunger was so adjusted that the compaction could give an increase in density of 48%. The loading rate was kept constant at 5 mm/min and when the crosshead moved down, the plunger was allowed to slide down inside the cylinder thus compacting the flour to a known level. The force-deformation profile was monitored during compaction. After the known compaction level was reached, the plunger was drawn up, the supporting collar was removed, the cylinder was positioned on the groove cut on the top of the stand, and again the plunger was allowed to slide down inside the cylinder to eject the compacted flour granulate (40.32 mm diameter and 52.15 mm height corresponding to a volume reduction of 48%) out of the cylinder. The percent reduction in volume was calculated using the formula $\{(V_i - V_f)/V_i\}$, where V_i is the initial volume of the sample and V_f is the final volume of the sample. The temperature rise during compaction was measured by a thermocouple. A vacuum pressure of 400 mm of Hg was applied using a vacuum pump during compaction. Three replicates were run for each trial. The experiment was repeated for 3 loading rates (5, 50, and 100 mm/min) and 3 flour moisture contents (13.1, 13.9, and 14.6% moisture content wet basis) for both grades of flour.

4.5.2 Determination of Water Activity

The a_w determination was conducted in triplicate for each of the samples conditioned for adsorption and desorption isotherm measurements. Prior to the determination of a_w of non-compacted flour and compacted flour pellets, the samples were brought to an approximate thermal equilibrium with the temperature at which they were to be tested (namely 10, 25, and 40°C) for 1 h.

The temperature of the a_w meter was set to the testing temperature. Approximately 7.5 g of flour was placed in the sample cup. The sample cup was placed in the measuring cup which in turn was positioned under the sensor inside the meter. This allowed the system to heat or cool the sample and the measuring bowl to the selected temperature. The approximate equilibrium was reached in 60 min for non-compacted flour. For compacted flour, the intermediate moisture content flours equilibrated faster (two to four hours) than the low and high moisture content flours (four to six hours). However, the waiting time in all experiments was extended for up to 6 h to ensure that the equilibrium had been achieved. The long equilibration time could be due to the fact that compaction would slow down the diffusion rate (Labuza, 1994). Once the equilibrium was reached, the a_w value of the flour was recorded and the moisture content of the sample determined. Thus, a series of adsorption and desorption isotherm data were obtained for non-compacted flour and compacted flour pellets at 3 different temperatures 10, 25, and 40°C. In the case of compacted flour pellets, subsequent to equilibration, the pellets were reconstituted using a mortar and pestle arrangement and the moisture content of the flour determined.

4.5.3 Rheometer Operation

The rheometer was used for determining the flow parameters of the dough (Fig. 4.2). The inside wall of the cylinder was lubricated with shortening to reduce friction between the walls of the cylinder and the dough. The dough, prepared using the non-compacted flour and the flour that was reconstituted after compaction, was filled in the cylinder and was pushed using a small rod so that there were no air gaps left in the cylinder. The speed of the crosshead was adjusted for 5 mm/min. When the crosshead moved down, it allowed the plunger to slide down smoothly

inside the cylinder containing the dough thus extruding the dough through the capillary.

The force required to extrude the dough through the capillary was recorded by a data acquisition system. A Mettler balance connected to a data acquisition system was placed beneath the capillary to measure the amount of extruded dough collected for a given time. This enabled the calculation of the mass flow rate of the dough. The dough density was determined by measuring the mass of a known volume of dough. Though air is incorporated during dough mixing which would decrease its density (Faubion and Hosney 1989), lack of instrumentation eliminated the possibility of measuring the changes in dough density during mixing. The volumetric flow rate of the dough was then calculated by dividing the mass flow rate of the dough by the dough density. This was also verified theoretically by multiplying the speed of the crosshead by the cross sectional area of the plunger.

To determine the pressure drop in the capillary, the force at the point of entry of the dough into the capillary was also measured. This was done by removing the capillary and by recording the force when the dough was about to extrude through the supporting collar (having the same diameter as that of the capillary). For each speed, the mass flow rate of the dough was calculated. The experiment was repeated for different crosshead speeds namely 10, 20, 50, 100, 200, 350, and 500 mm/min and for the dough prepared using the non-compacted flour and the flour reconstituted after compaction for both grades (low and high extraction) of flour.

4.6 Reconstitution Methods

Initially, the compacted flour granulates (as described in section 4.3.4) were reconstituted using different methods; the champion juicer (Model G-5 NG853S, Plastaket Manufacturing Inc.,

Lodi, CA), bran finisher (Buhler Brothers, Toronto), hammer mill (Falling Number Laboratory Mill 3100), and burr mill (Hobart Corporation, Troy, OH). The operation of these methods was according to the manufacturer's specifications. All methods were operated on a continuous basis. The pellets were continuously fed from one end and the reconstituted flour was collected in the other end. The flour reconstituted in the above manner was tested for Farinograph absorption.

4.7 Experimental Design

Two grades of flour (64% and 79% extraction flours) of three different moisture contents (13.1, 13.9, and 14.6% wb moisture content) were compacted at three different loading rates (5, 50, and 100 mm/min) to determine the compaction behaviour of flour as influenced by different moisture contents and loading rates. Three replicates were made at each level.

The adsorption and desorption isotherm measurements of non-compacted and compacted flour (with an initial moisture content of 13.9% wet basis) were carried out for two grades of flour (64% and 79% extraction flours) and at three different temperatures (10, 25, and 40°C) to determine the moisture sorption hysteresis as influenced by compaction of flour. The moisture contents ranged from 6% to 16% at 2% increments. Three replicates of a_w measurements were made at each level.

The flow characteristics of a flour-water dough namely, the flow behaviour index (n) and the consistency coefficient (K), were determined for the dough prepared using the non-compacted flour and the flour reconstituted after compaction. The empirical rheological measurements of

a dough - visco-Amylograph, Alveograph, the analytical determinations - starch damage and gluten content of flour, and the remix bake test were also performed for the non-compacted flour and the flour reconstituted after compaction. Two grades of flour (64% and 79% extraction flours) of an initial moisture content of 13.9% wb were used in this investigation.

The statistical analysis of the results was made using the SAS PC version (SAS 1985).

5. RESULTS AND DISCUSSION

5.1 Compaction Behaviour of Flour

The density-pressure relationship under axial compression was obtained for both grades of flour showing the effect of loading rates, i.e., 5, 50, and 100 mm/min, on compaction behaviour at constant moisture content. The results are presented in Figs. 5.1 and 5.2 for the patent flour and for the straight grade flour, respectively. The density of flour compact was obtained by dividing the mass of flour by the volume of flour in the cylinder. The pressure was obtained by dividing the applied force by the flour surface area.

Kumar (1973) studied the compaction behaviour of ground corn. Using a thermodynamic approach, he proposed a linear relationship between log density and pressure and found that it was valid for a pressure of up to 0.5 MPa. Similar observations were also made by Moreyra and Peleg (1980) who found that the plot between log density versus pressure for various food powders would be linear provided the maximum applied pressure did not exceed 0.5 MPa. In the present investigation, the maximum applied pressure varied between 9-16 MPa. All compaction curves revealed curvature when they were plotted in the form of log density versus pressure. This is in good agreement with the findings of McNulty and Mohsenin (1979) who obtained a similar trend in their study of the compaction behaviour of bulk corn kernels. The minimum pressure applied in their case was 40 MPa, depending on the moisture content of the corn kernels studied. They attributed this high pressure densification to such a curvilinear relationship between log density and pressure. Thus, they contradicted the findings of Kumar

(1973), who also suggested that his model could be extrapolated to pressure higher than those available in his experimental conditions.

Because of the relatively low total applied pressure in the present findings, the highest temperature rise due to compaction was 0.7°C in the low moisture flour (13.1% wb) at the low loading rate (5 mm/min). Thus, no thermally induced chemical or physical changes could be anticipated in the material. The vacuum applied during compaction (53.33 kPa) did not have any marked influence on the total pressure requirement to compact a known amount of samples to a given deformation.

Figs. 5.1 and 5.2 show the effects of different loading rates on the compaction behaviour of various constant moisture content patent and straight grade flours, respectively. The test for the homogeneity of slopes of different curves using the General Linear Model Procedure (SAS 1985) suggests that the loading rates had no significant influence on the compaction behaviour of flour at any moisture content. This is in good agreement with the findings of McNulty and Mohsenin (1977) who observed that the loading rates (1.68, 16.8, and 168 mm/min) had little influence on the density-pressure profiles in the compaction behaviour of bulk whole corn kernels. But, they did not attempt to make any statistical conclusions of their results.

However, between 5 and 50 mm/min, there is a small difference in the pressure required to deform the sample to a given deformation whereas this difference is negligible between 50 and 100 mm/min. In other words, the pressure needed to deform the sample to a given deformation

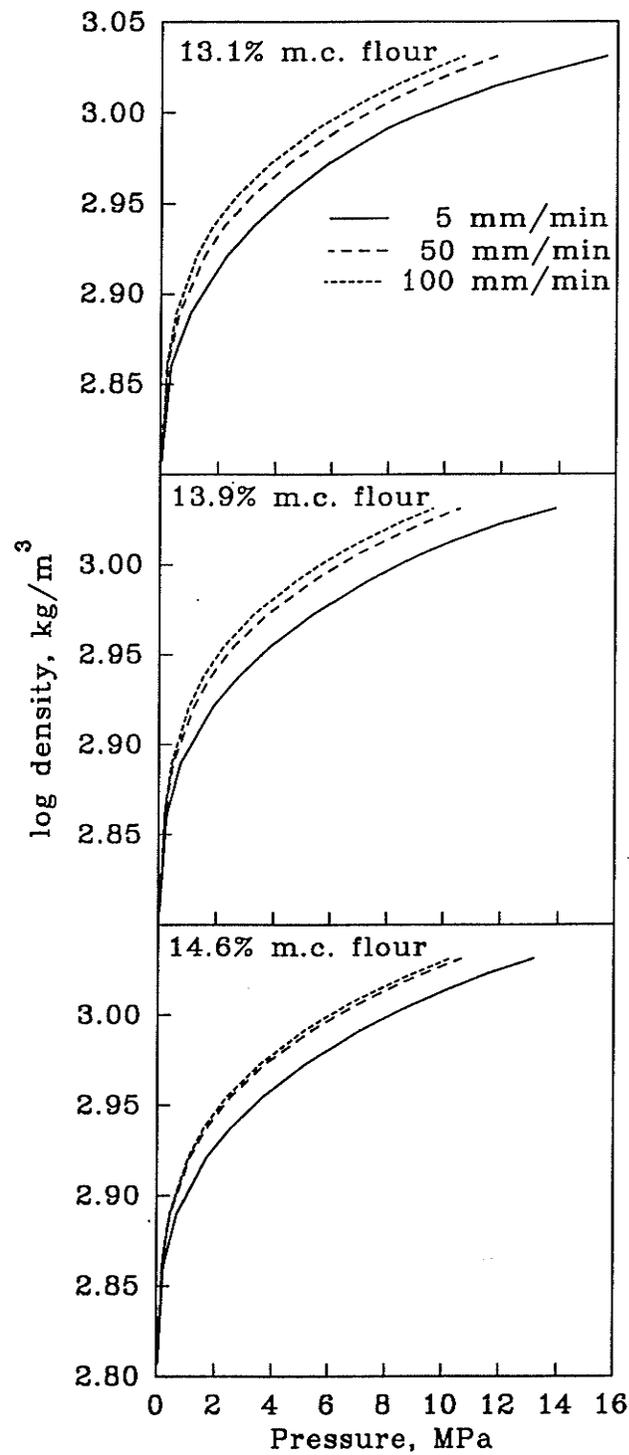


Fig. 5.1 Pressure-density relationship of patent flour as influenced by different loading rates and at various constant moisture contents.

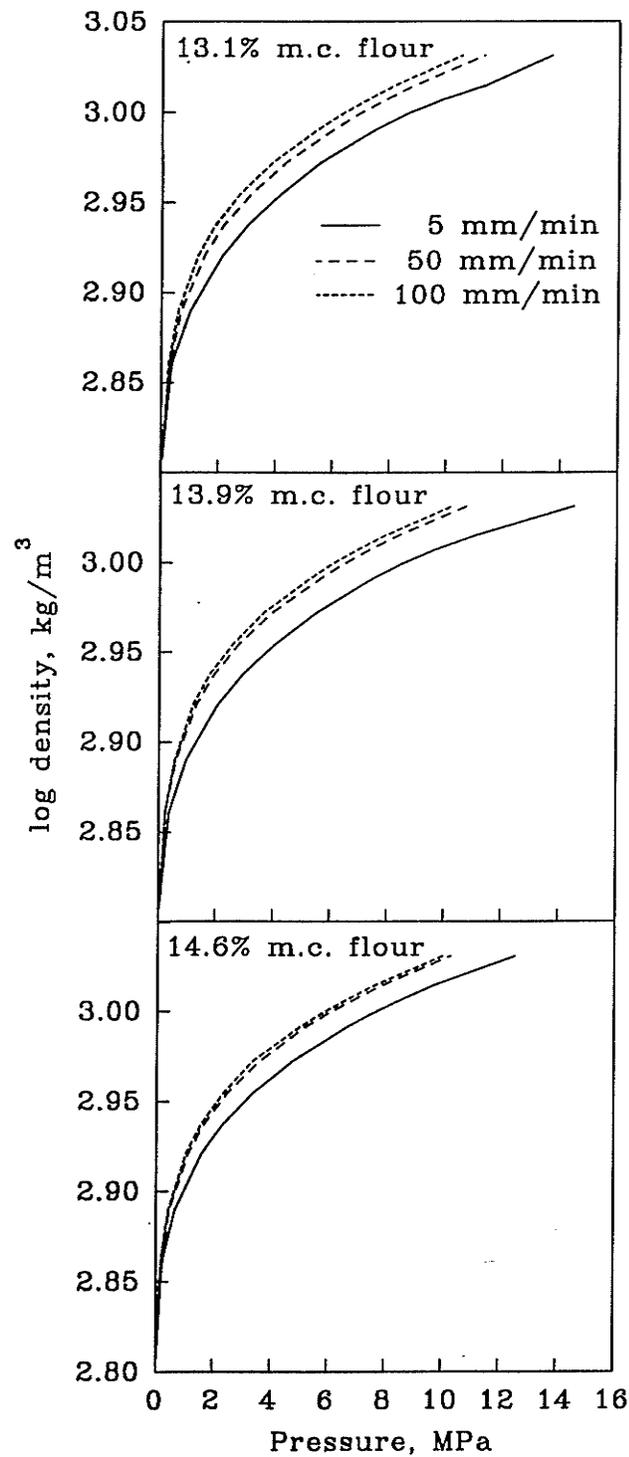


Fig. 5.2 Pressure-density relationship of straight grade flour as influenced by different loading rates and at various constant moisture contents.

is higher at low loading rate (5 mm/min) than it is at high loading rate (100mm/min). Similar trend is observed for the straight grade flour as shown by Fig. 5.2. This seems to agree well with the three stage definition of compaction proposed by Heckel (1961a, 1961b). At low loading rate, enough time was allowed for the rearrangement of particles in the cylinder during compaction and the samples to form a dense mass. At high loading rate, little time was allowed for the rearrangement of particles in the cylinder and hence the samples form a more incoherent mass. Hence, the pressure required to deform the samples plastically at low loading rate is more than that at high loading rate. This is supported by the findings of McNulty and Mohsenin (1979) who observed insufficient densification of corn kernels at high loading rate probably because of the low total applied pressure. They also observed that at high loading rate (168 mm/min), the entrapped gas was unable to escape fast enough thus leading to gas compression and hence a loud whining noise.

Figs. 5.3 and 5.4 show the effects of various moisture contents of the two grades of flour on the compaction behaviour at various constant loading rates. The test for the differences in the slopes of the curves suggests that the null hypothesis is valid, i.e., the moisture content in the tested range had no significant influence on the compaction behaviour at any given loading rate. However, the observed trend is in good agreement with the findings of Peleg (1977), who found that the compressibility of moist powders in the lower range of compressive stresses will be greater than that of dry or less cohesive powders. This could be explained on the basis of molecular adsorption of water. The slipping of the anion (-OH⁻) bond between neighbouring chains is probably the most reasonable explanation for increasing plasticity with increasing

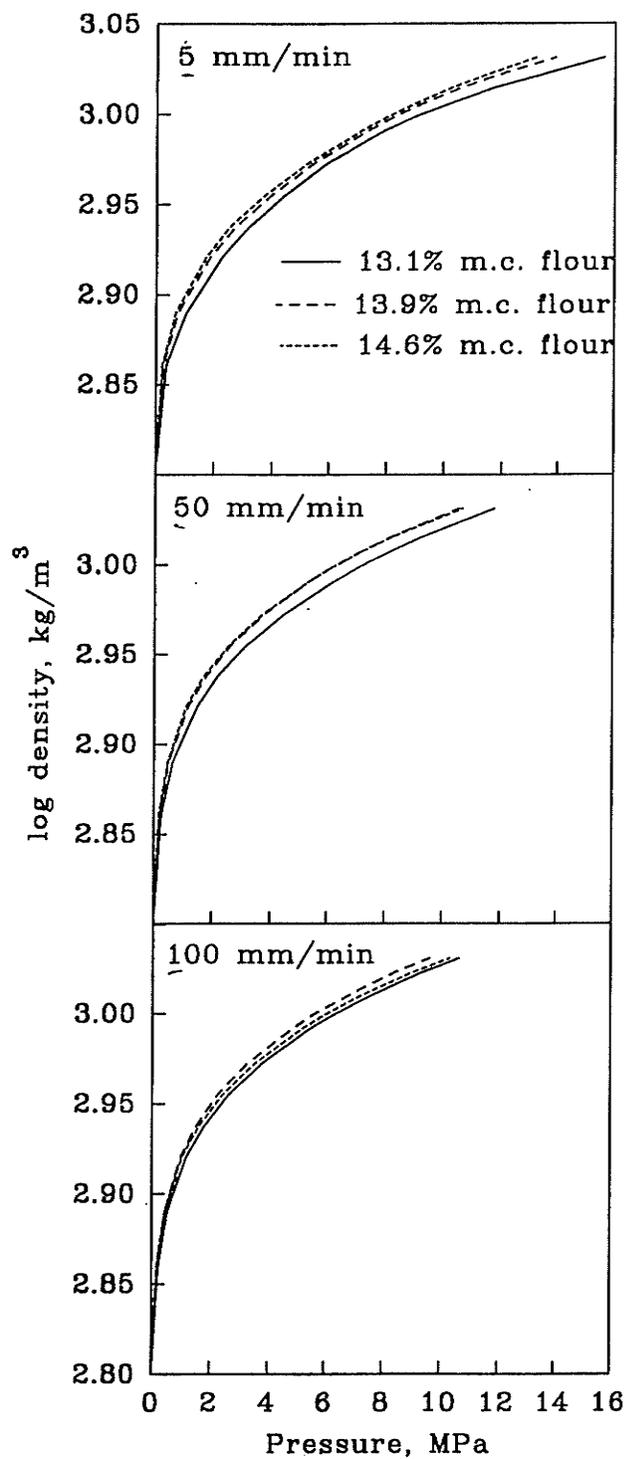


Fig. 5.3 Pressure-density relationship of patent flour as influenced by different moisture contents at various constant loading rates.

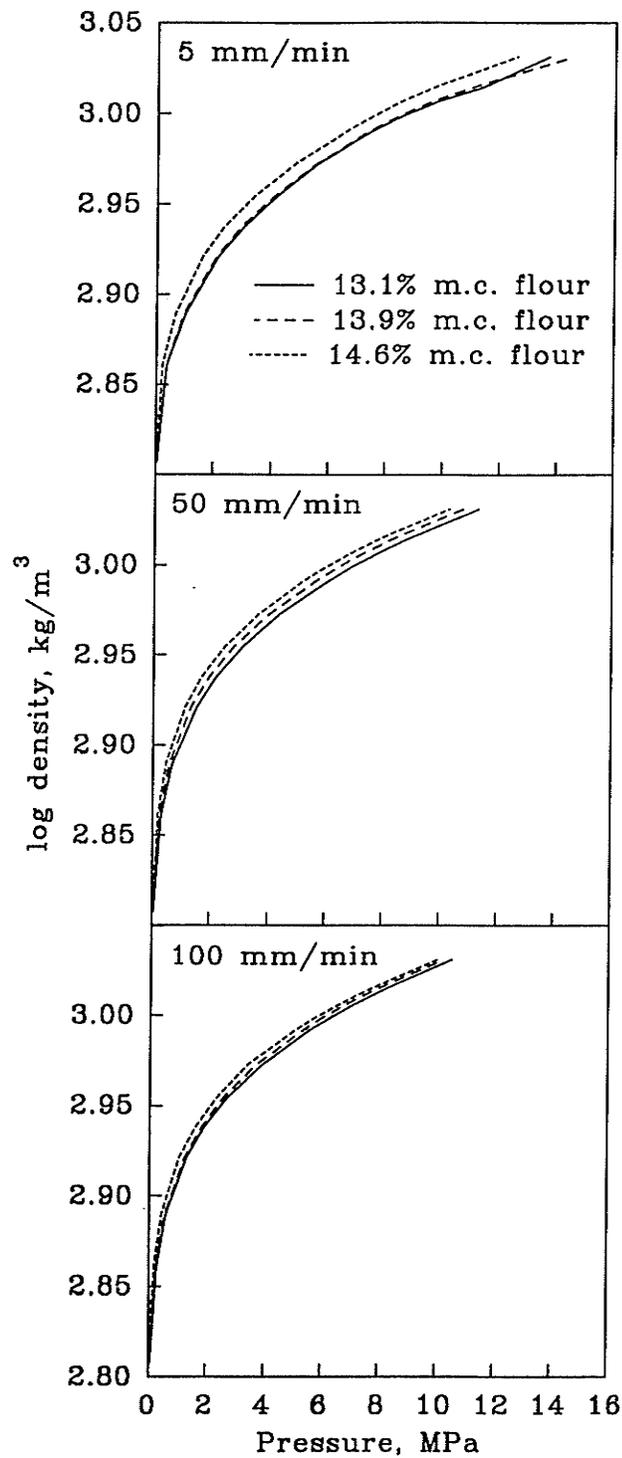


Fig. 5.4 Pressure-density relationship of straight grade flour as influenced by different moisture contents at various constant loading rates.

moisture content (Kumar 1973). Also, the increase in moisture content results in the formation of liquid bridges within the particles. Such bridges are relatively weak and can collapse under very small applied pressures. This will also account for the increased compressibility of high moisture flour (thus resulting in an increase in the plasticity).

Thus, it has been shown that the compaction of flour was feasible on a laboratory scale using a cylinder-plunger arrangement and a Universal Testing Machine. The development of a commercial piece of equipment for the commercial compaction of flour, ideal for export conditions, would be the major focus of future research.

5.2 Effect of Compaction on Moisture Sorption

5.2.1 Sorption Behaviour

The desorption and adsorption isotherms of patent non-compacted and compacted flour at three different temperatures 10, 25, and 40°C are given in Fig. 5.5. The error bars indicate the 95% confidence limits for the mean water activity value.

The statistical analysis of the results suggests that the null hypothesis is valid, i.e., the isotherms of non-compacted flour are not significantly different from those of the compacted flour (95% confidence). Though the isotherms of control and treated flour at 10°C may seem to be different at some of the individual data points, it showed no significant difference when the whole curve was tested in the range of a_w studied. Similar observations were also obtained in the case of straight grade flour (Fig. 5.6).

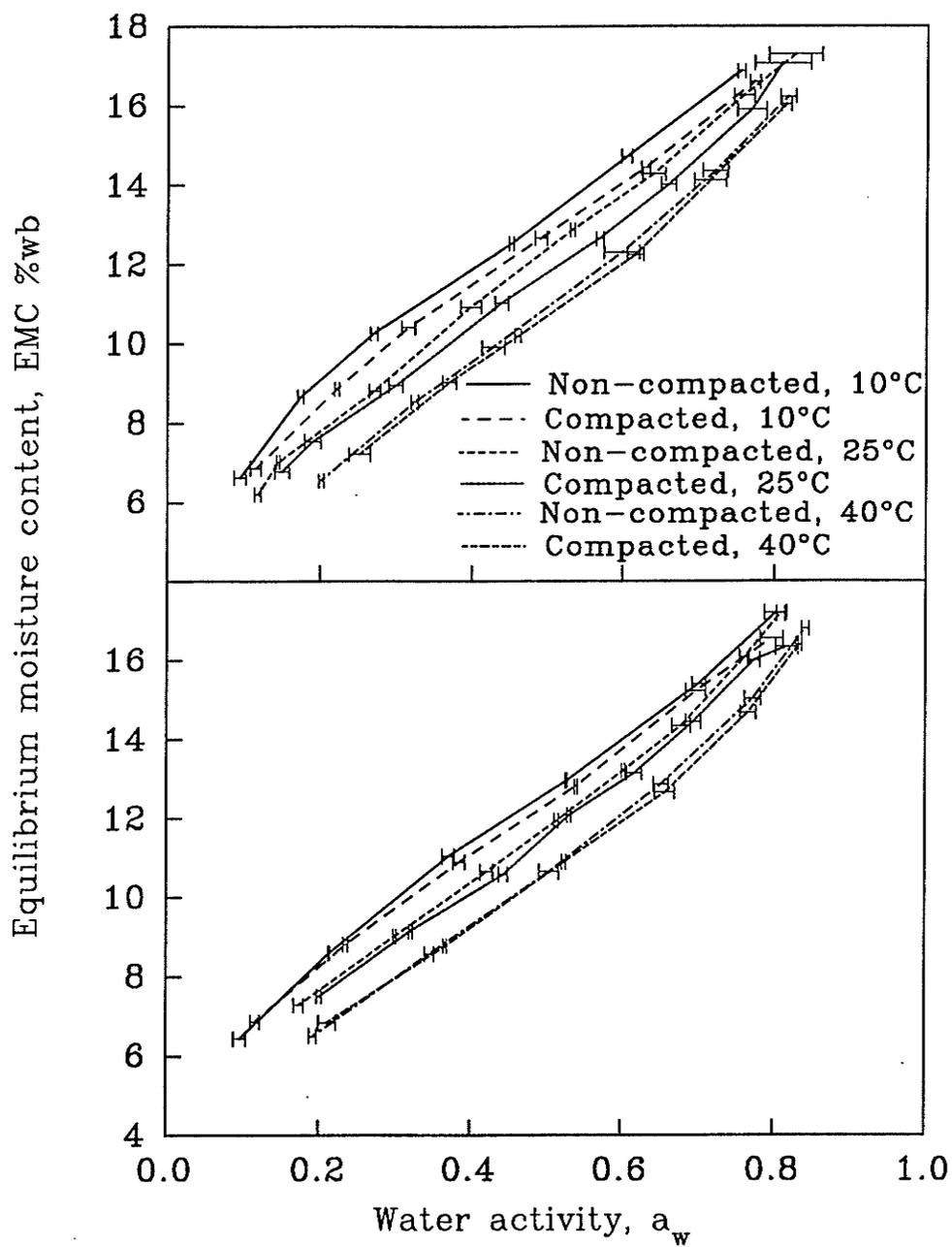


Fig. 5.5 Desorption (above) and adsorption (below) isotherms of patent flour. Error bars indicate the 95% confidence limits for the mean water activity value.

However, it can be noticed from the desorption isotherm curves at 10 and 25°C, that for any given moisture content, the compacted flour pellets equilibrated at a slightly higher water activity than did the non-compacted flour. In other words, at any given vapour pressure, the compacted flour pellets held less water than did the non-compacted flour. This suggests that the availability of sorptive sites for water molecules during compaction is reduced. However, this small difference in water activity value as influenced by compaction was not evident at 40°C; the compacted flour equilibrated at nearly the same water activity as the non-compacted flour. The adsorption isotherms of patent flour reveal that the compacted flour did not show even a small difference in a_w , in the given range of a_w studied, as compared to the non-compacted flour, except for a small increase in the a_w of the compacted flour at a given moisture content in the a_w range of 0.3 to 0.7 at 10°C. This is in good agreement with the findings of Ramanathan and Cenkowski (1994a), who observed that the sorption isotherm of all-purpose flour at 25°C was not significantly altered by compacting the flour to small pellets.

The sorptive capacity of flour is an internal hygroscopic property and hence it will be worthwhile to account for its sorptive capacity as an integrated hygroscopic properties of its constituents, mainly starch, gluten proteins, and different solubles (Gur-Arieh et al. 1967). Also, it has been reported that the moisture sorption isotherm of flour is independent of the particle size distribution of the flour (Bushuk and Winkler 1957, Gur-Arieh et al. 1967). Hence, this work supports the hypothesis that the difference in moisture sorption behaviour of compacted flour is not due to any difference in particle size that may have occurred during compaction.

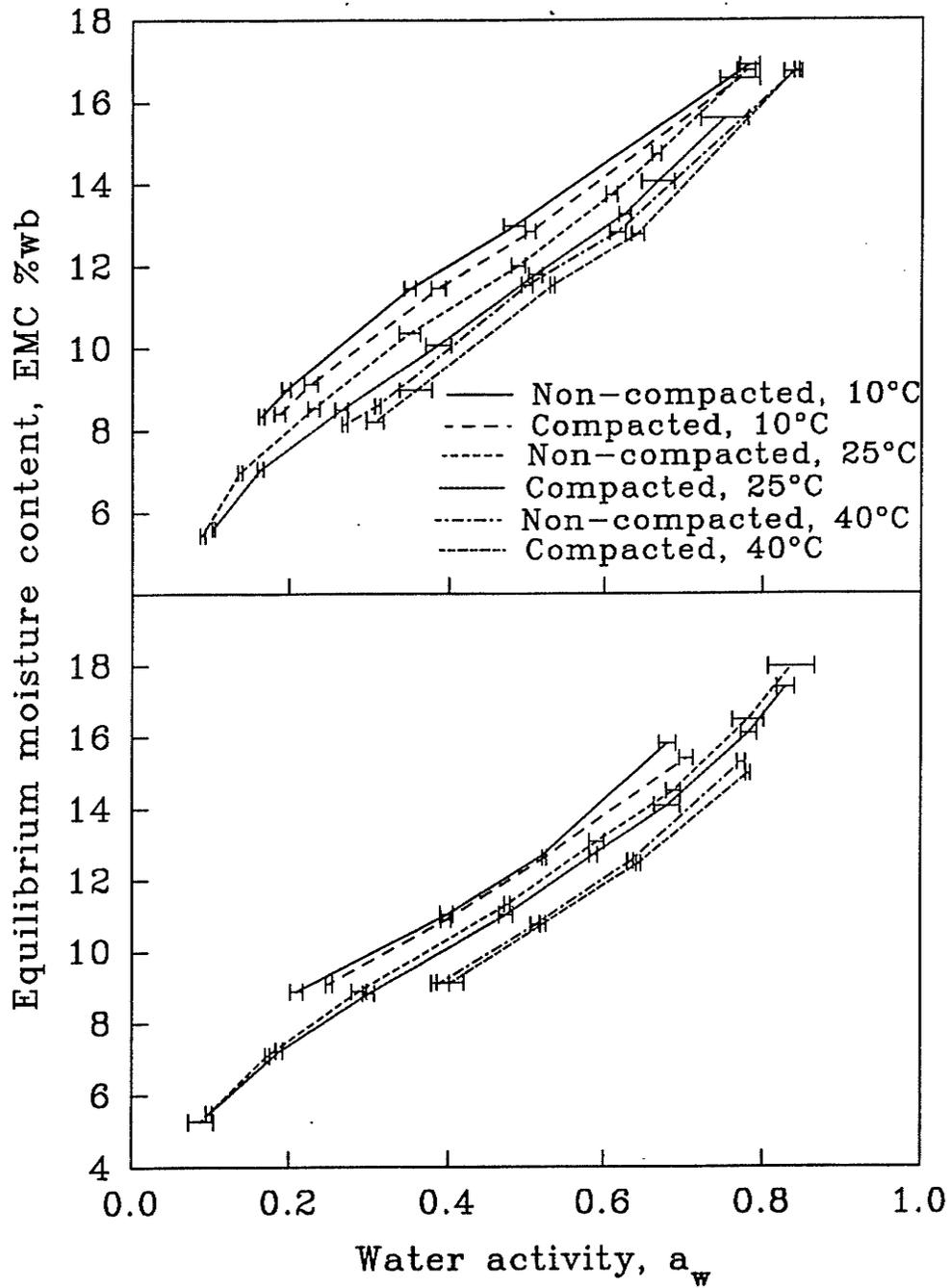


Fig. 5.6 Desorption (above) and adsorption (below) isotherms of straight grade flour. Error bars indicate the 95% confidence limits for the mean water activity value.

Sair and Fetzer (1944) accounted for the sorptive capacity of starch based on the fact that -OH group is the point of attachment for water. They observed that the cereal starch has a less percentage of free -OH groups as compared to root and tuber starches. In other words, the degree of association of cereal starch is more than that of root and tuber starch. If this degree of association were disrupted due to compaction, then the compacted flour would sorb more water than the non-compacted flour because of a high percentage of free -OH groups. However, this was not observed in our results. Hence, it can be proposed that compaction of flour does not cause starch damage of a significant magnitude to alter the moisture sorption characteristics of the flour.

Makower and Dye (1956) observed that during moisture sorption by sugars in a model system, amorphous sucrose may be converted to crystalline water soluble sucrose. This conversion will result in the released water to move to the surface which will cause an increase in water activity. However, Gur-Arieh et al. (1967) observed that the water soluble fraction of the carbohydrate (mainly maltose) did not contribute to the water activity of the flour. Hence, it can be proposed that any phase transformation of soluble sugar occurring during compaction would not have any effect on the final equilibrium water activity of wheat flour.

Bushuk and Winkler (1957) found that the rate of moisture adsorption was not affected by packing it more compactly. But, they did not make an attempt to account for the effect of compaction on the final equilibrium water activity value of flour. However, the results of the present work add to their finding that compaction of flour did not influence either the rate of

adsorption or the equilibrium adsorption.

5.2.2 Effect of Temperature on Sorption

The temperature in the range studied i.e., 10, 25, and 40°C, had a marked influence on the moisture sorption behaviour of flour (Fig. 5.5). The desorption and adsorption isotherms of non-compacted flour and compacted flour pellets are significantly (95% confidence) influenced by temperature. This contradicts the findings of Henderson and Pixton (1982) who observed that temperature (5, 15, and 25°C) had little influence on the moisture sorption isotherms of flour. Conclusions could not be drawn about the straight grade flour because not enough data points were obtained to characterize its sorption behaviour at 10 and 40°C.

5.2.3 Sorption Effects on Hysteresis

Fig. 5.7 shows the hysteresis between the adsorption and desorption isotherm in non-compacted flour and compacted flour pellets at 10, 25, and 40°C for the patent flour. The desorption isotherms are clearly above the adsorption isotherms in case of both non-compacted flour and compacted flour pellets. The hysteresis also decreases with an increase in temperature. This is in good agreement with the findings of Wolf et al. (1972) who found that the increasing temperature decreased the hysteresis. However, this hysteresis is not significant ($P = 0.05$) in either the non-compacted flour or the compacted flour pellets. An attempt was also made to show the hysteresis in straight grade flour (Fig. 5.8) with the available data but statistical conclusions could not be derived from them.

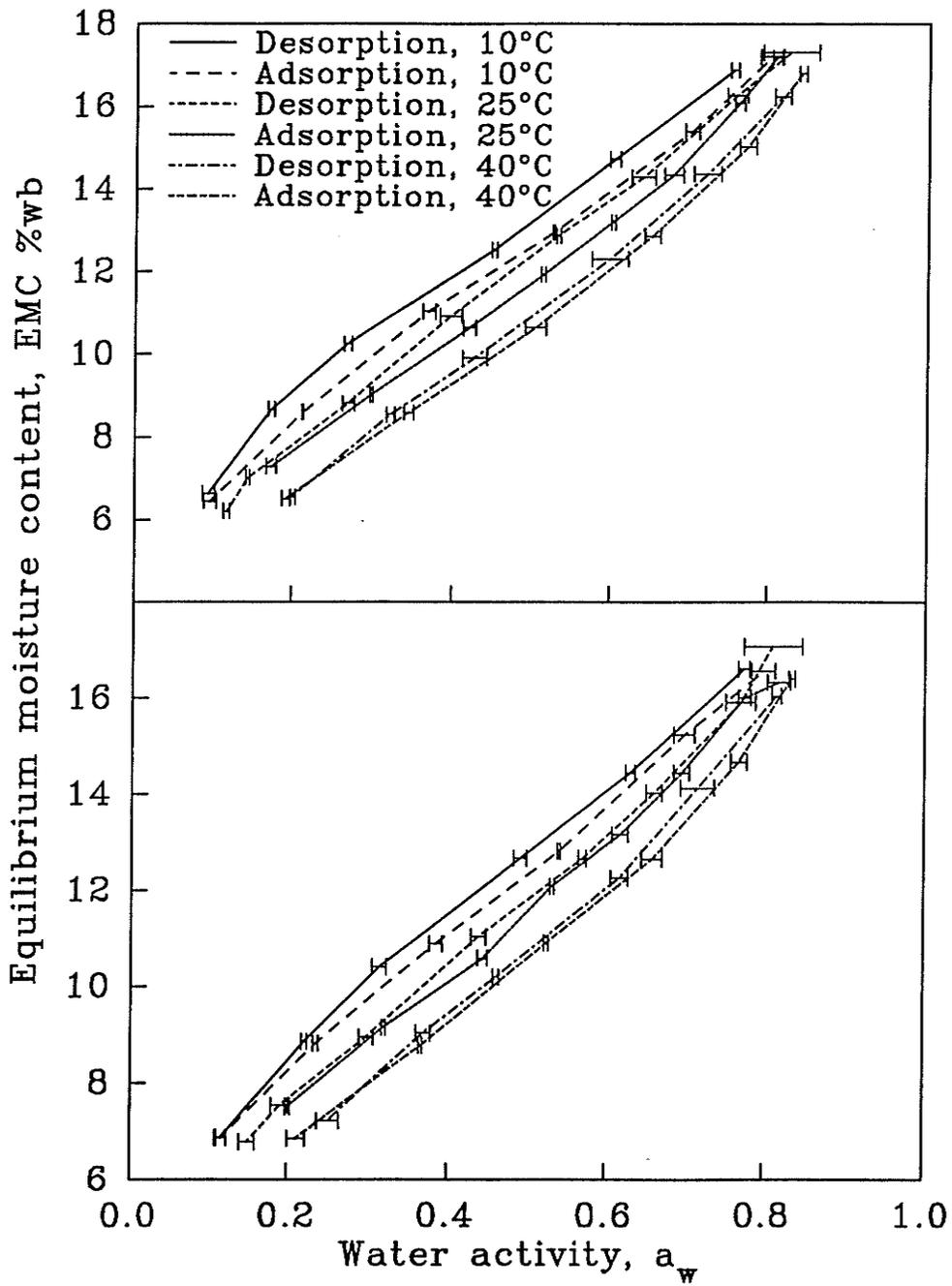


Fig. 5.7 Hysteresis in patent flour, non-compacted flour (above) and compacted flour (below). Error bars indicate the 95% confidence limits for the mean water activity value.

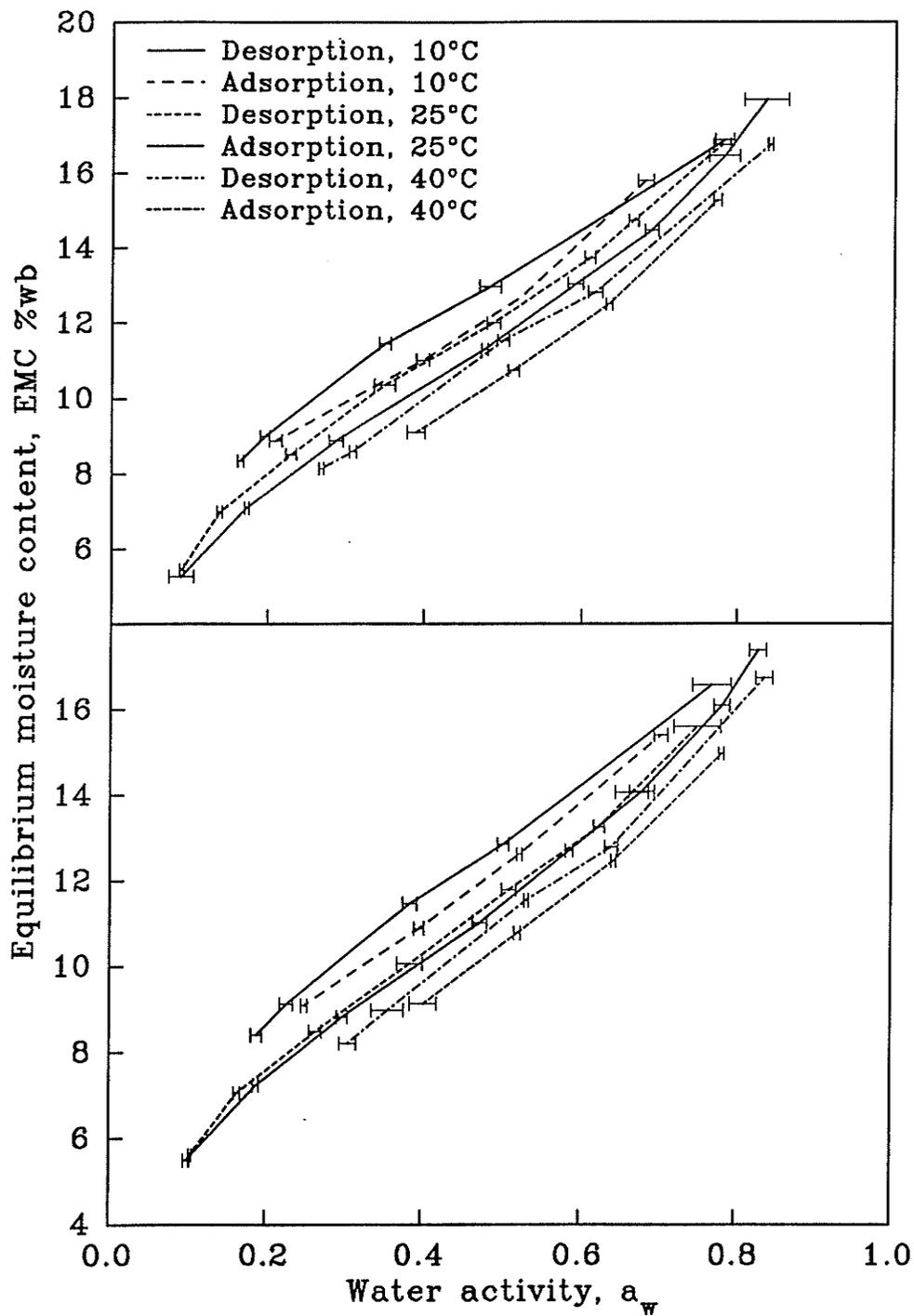


Fig. 5.8 Hysteresis in straight grade flour, non-compacted flour (above) and compacted flour (below). Error bars indicate the 95% confidence limits for the mean water activity value.

5.2.4 Isotherm Equation

Modified Halsey equation (Eq. 3.6) was used to characterize the sorption behaviour of flour. Iglesias and Chirife (1976) developed and showed this equation to be valid for flour-water systems based on the sorption data obtained by Bushuk and Winkler (1957).

Table 5.1. Estimates of parameters of Modified Halsey equation (Eq. 3.6) with their standard errors and confidence intervals for the patent flour.

Flour	Parameter	Estimate	Std.Error	95% Confidence Interval	
				Lower	Upper
Non-compacted, Desorption	A	5.22	0.156	4.91	5.54
	B	-0.0163	0.00150	-0.0193	-0.0133
	C	2.01	0.0570	1.90	2.13
Compacted, Desorption	A	5.23	0.129	4.97	5.49
	B	-0.0138	0.00120	-0.0162	-0.0114
	C	2.07	0.0480	1.97	2.16
Non-compacted, Adsorption	A	5.29	0.134	5.03	5.56
	B	-0.0138	0.00130	-0.0163	-0.0112
	C	2.09	0.0490	2.00	2.19
Compacted, Adsorption	A	5.40	0.127	5.15	5.66
	B	-0.0129	0.00120	-0.0152	-0.0105
	C	2.16	0.0470	2.07	2.26

The non-linear regression procedure (Proc NLIN, SAS 1985) was used to estimate the parameters of the equation for the adsorption and desorption isotherms of non-compacted flour and the compacted flour pellets. The parameter estimates with their standard error and confidence

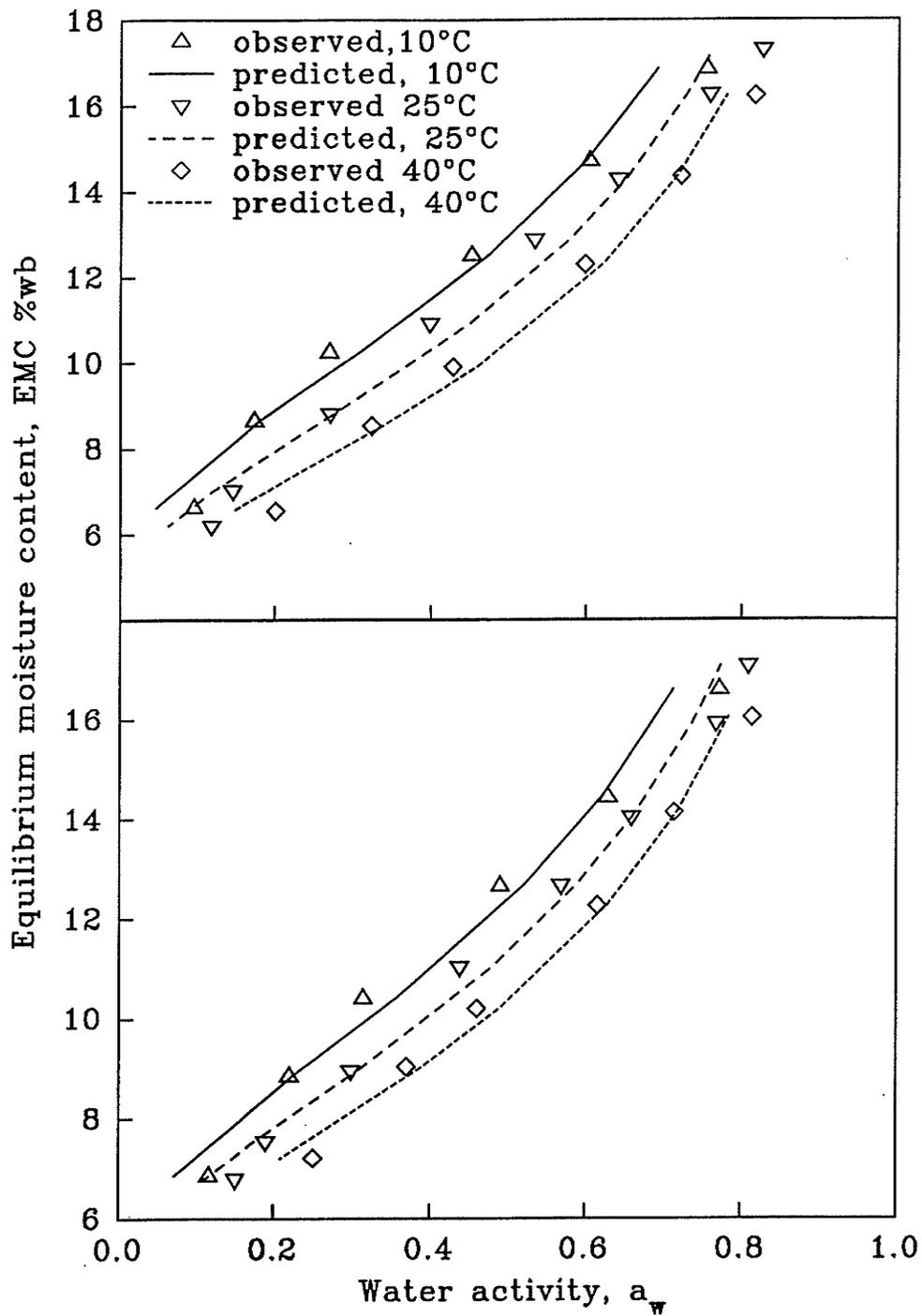


Fig. 5.9 Observed and predicted values for the desorption isotherms of patent flour, non-compacted flour (above) and compacted flour (below). Values predicted using Modified Halsey equation.

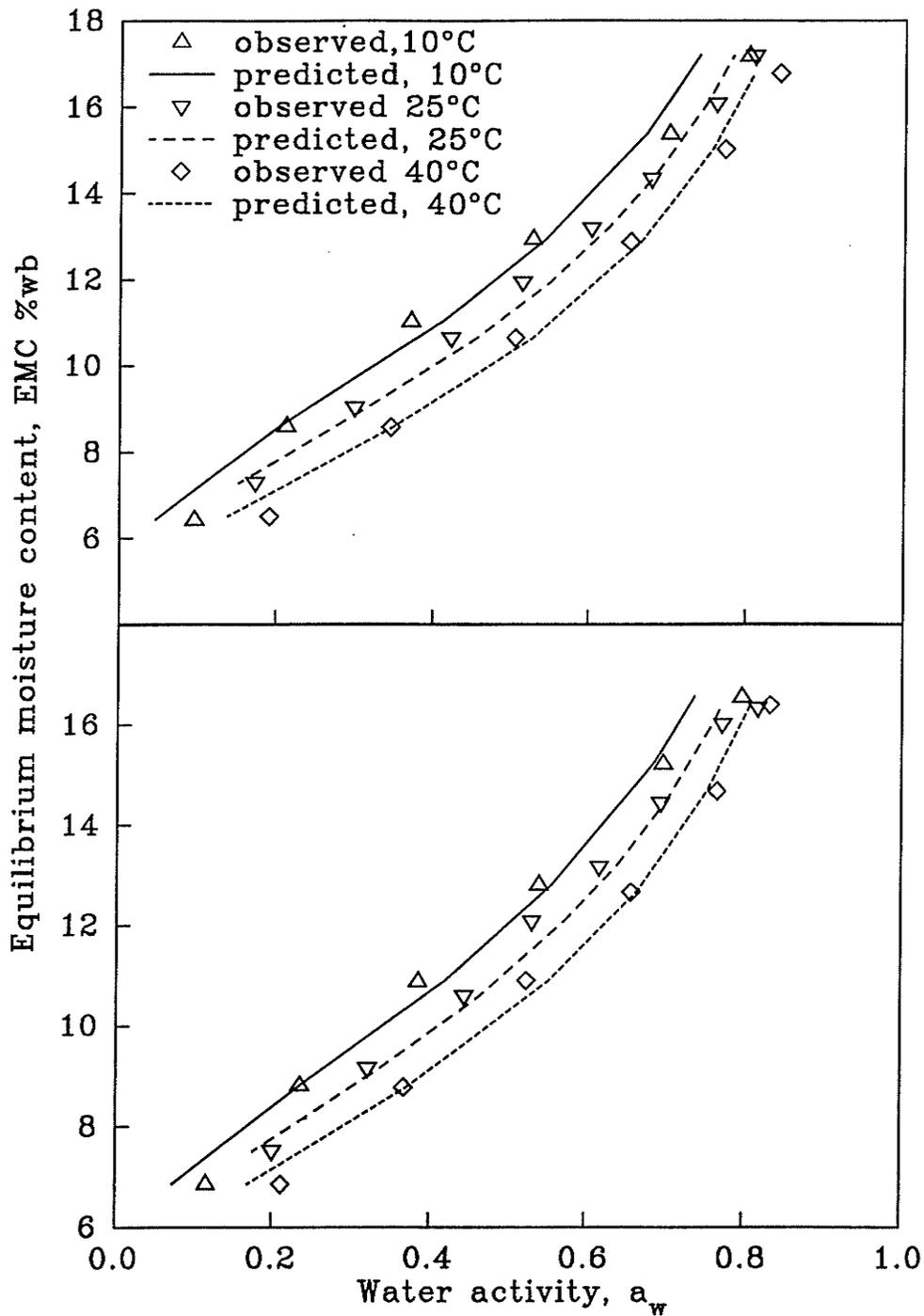


Fig. 5.10 Observed and predicted values for the adsorption isotherms of patent flour, non-compacted flour (above) and compacted flour (below). Values predicted using Modified Halsey equation.

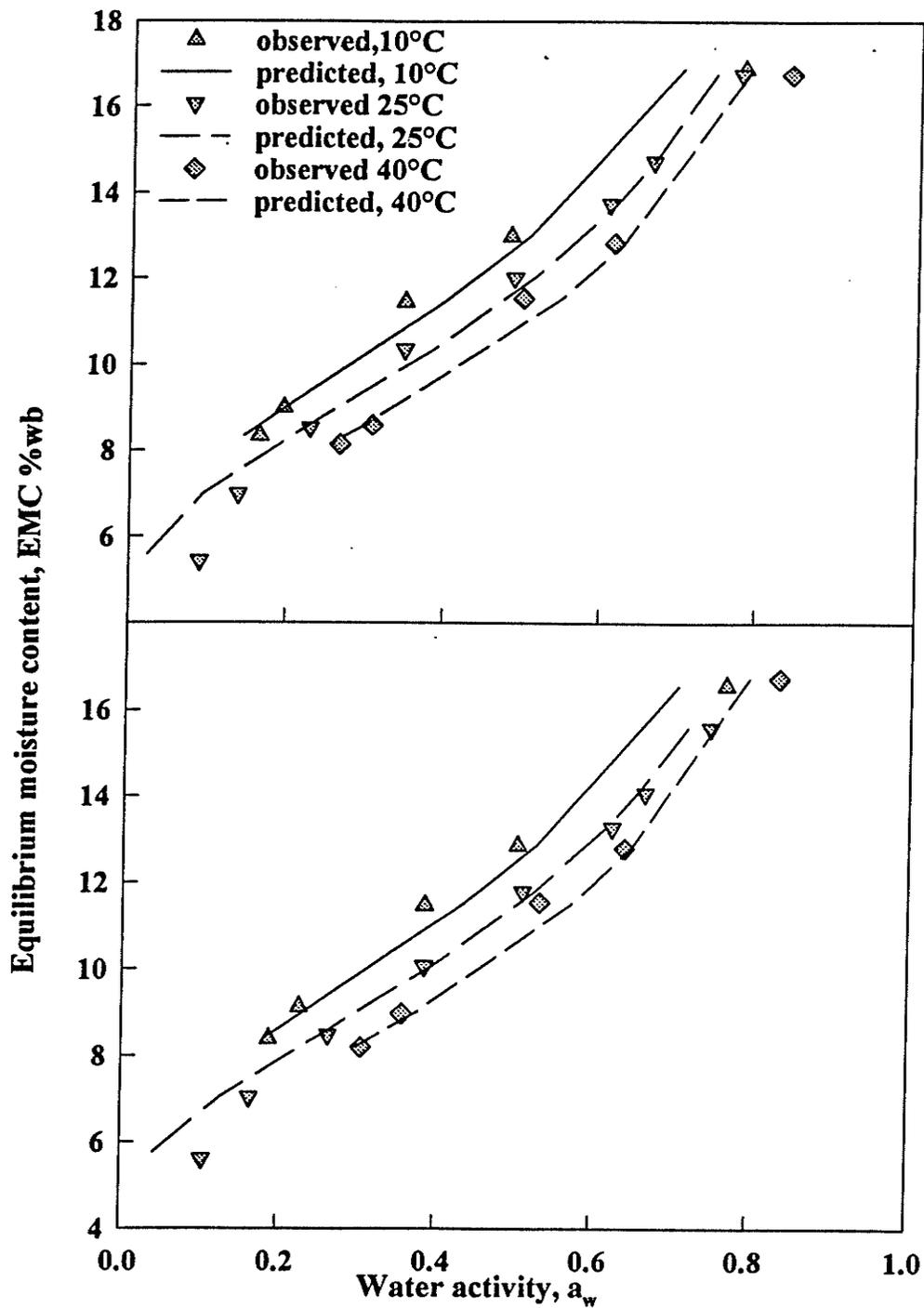


Fig. 5.11 Observed and predicted values for the desorption isotherms of straight grade flour, non-compacted flour (above) and compacted flour (below). Values predicted using Modified Halsey equation.

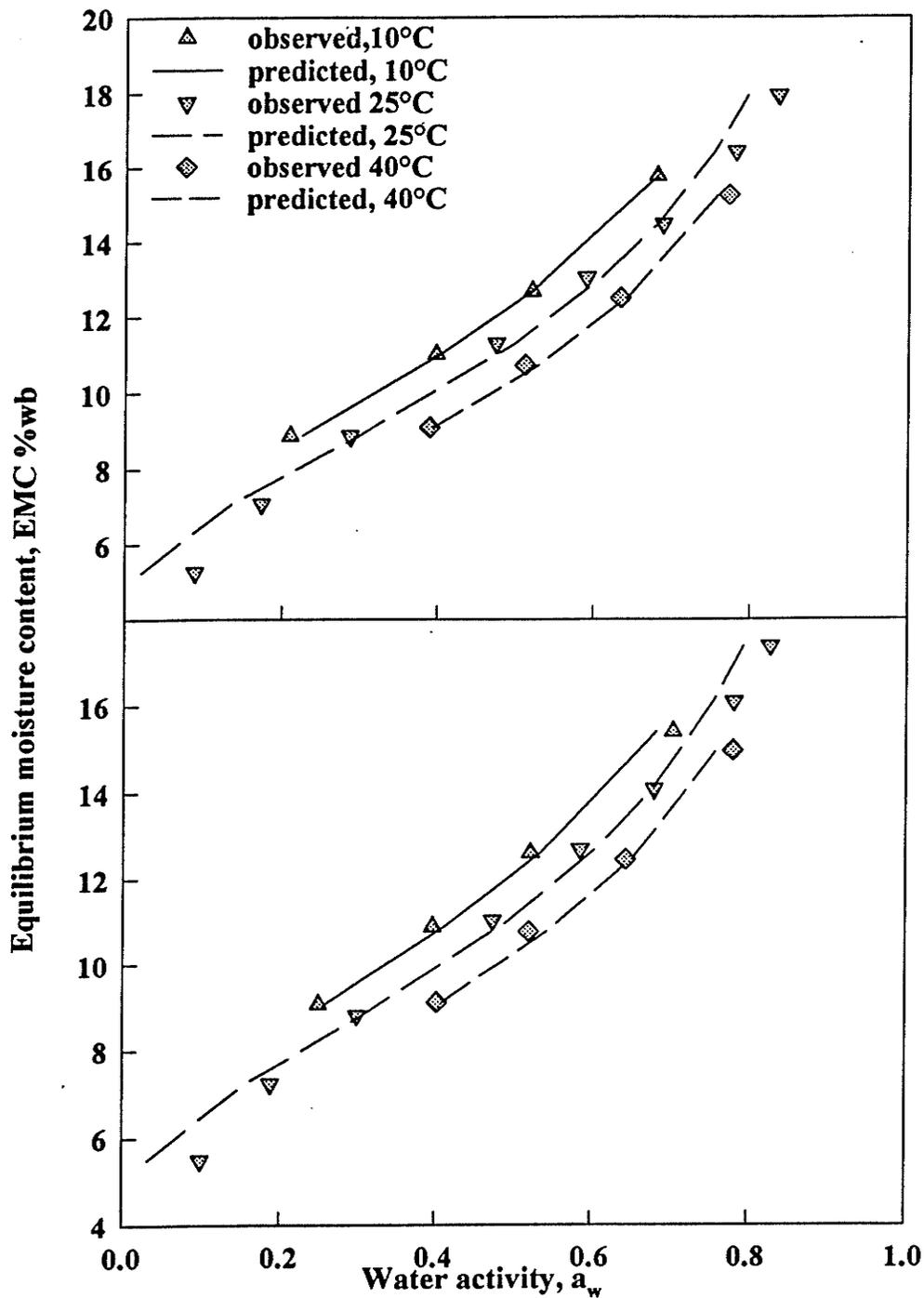


Fig. 5.12 Observed and predicted values for the adsorption isotherms of straight grade flour, non-compacted flour (above) and compacted flour (below). Values predicted using Modified Halsey equation.

intervals (95%) are indicated in Tables 5.1 and 5.2 for the patent and straight grade flour, respectively. The parameters of non-compacted flour are not significantly (95% level) different from those of compacted flour pellets in either desorption or adsorption for both flour grades. Iglesias and Chirife (1976) obtained the parameter estimates based on the experimental data of Bushuk and Winkler (1957) and the results of the present work are in excellent agreement with their findings.

Table 5.2. Estimates of parameters of Modified Halsey equation (Eq. 3.6) with their standard errors and confidence intervals for the straight grade flour.

Flour	Parameter	Estimate	Std.Error	95% Confidence Interval	
				Lower	Upper
Non-compacted, Desorption	A	5.46	0.180	5.10	5.82
	B	-0.0135	0.00160	-0.0167	-0.0104
	C	2.11	0.0670	1.98	2.25
Compacted, Desorption	A	5.26	0.154	4.96	5.57
	B	-0.0135	0.00130	-0.0161	-0.0108
	C	2.07	0.0590	1.95	2.19
Non-compacted, Adsorption	A	5.24	0.133	4.97	5.50
	B	-0.0139	0.00110	-0.0162	-0.0116
	C	2.06	0.050	1.96	2.16
Compacted, Adsorption	A	5.28	0.137	5.01	5.56
	B	-0.0134	0.00120	-0.0157	-0.0111
	C	2.10	0.0520	2.00	2.21

Using the parameter estimates, the a_w values were predicted using Eq. 3.6 for non-compacted

flour and compacted flour pellets conditioned for desorption and adsorption in both flour grades. A reasonably good agreement was obtained between the observed and predicted values (Figs. 5.9 to 5.12) in the water activity range of 0.2 to 0.7. Below and above this range at 10°C, the observed values of a_w deviate markedly from the predicted values for patent flour.

5.2.5 Free Energy Changes of Adsorption and Desorption

The observed free energy change (calculated using Eq. 3.4) and predicted free energy change (calculated using Eq. 3.5) of desorption and adsorption, as a function of moisture content, for non-compacted flour and compacted flour pellets in patent flour are given in Figs. 5.13 and 5.14.

The free energy change of adsorption or desorption decreases with an increase in moisture content. This suggests that, with an increase in moisture content, water will start condensing in the capillaries and the forces of attraction between the active sorptive sites (polar sites) and water molecules are significantly decreased. Thus, water behaves like free water and is more available for chemical and microbiological deterioration because it increases the solute mobility.

The plot of free energy change of sorption vs. moisture content (Figs. 5.13 to 5.16) followed a linear relationship, as was observed by Chung and Pfoest (1967). The free energy change of non-compacted flour is not significantly (95% level) greater than that of compacted flour pellets for any given moisture content. It can also be observed that the free energy change of desorption is not significantly (95% level) greater than that of adsorption. This confirms with the earlier findings that the hysteresis between desorption and adsorption in both non-compacted flour and compacted flour pellets is not significant (95% level) at all temperatures.

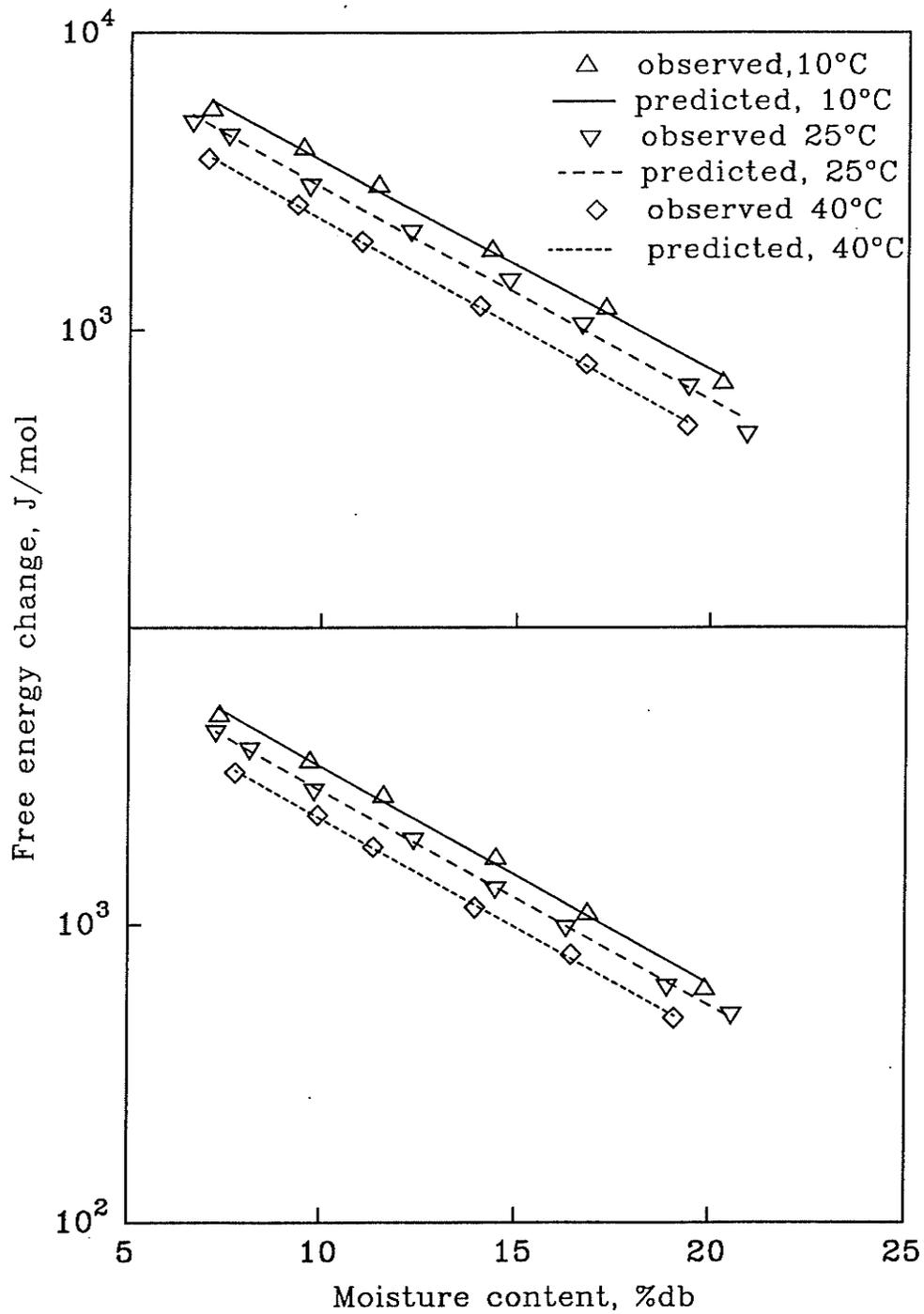


Fig. 5.13. Observed and predicted free energy change of desorption of patent flour, non-compacted flour (above) and compacted flour (below).

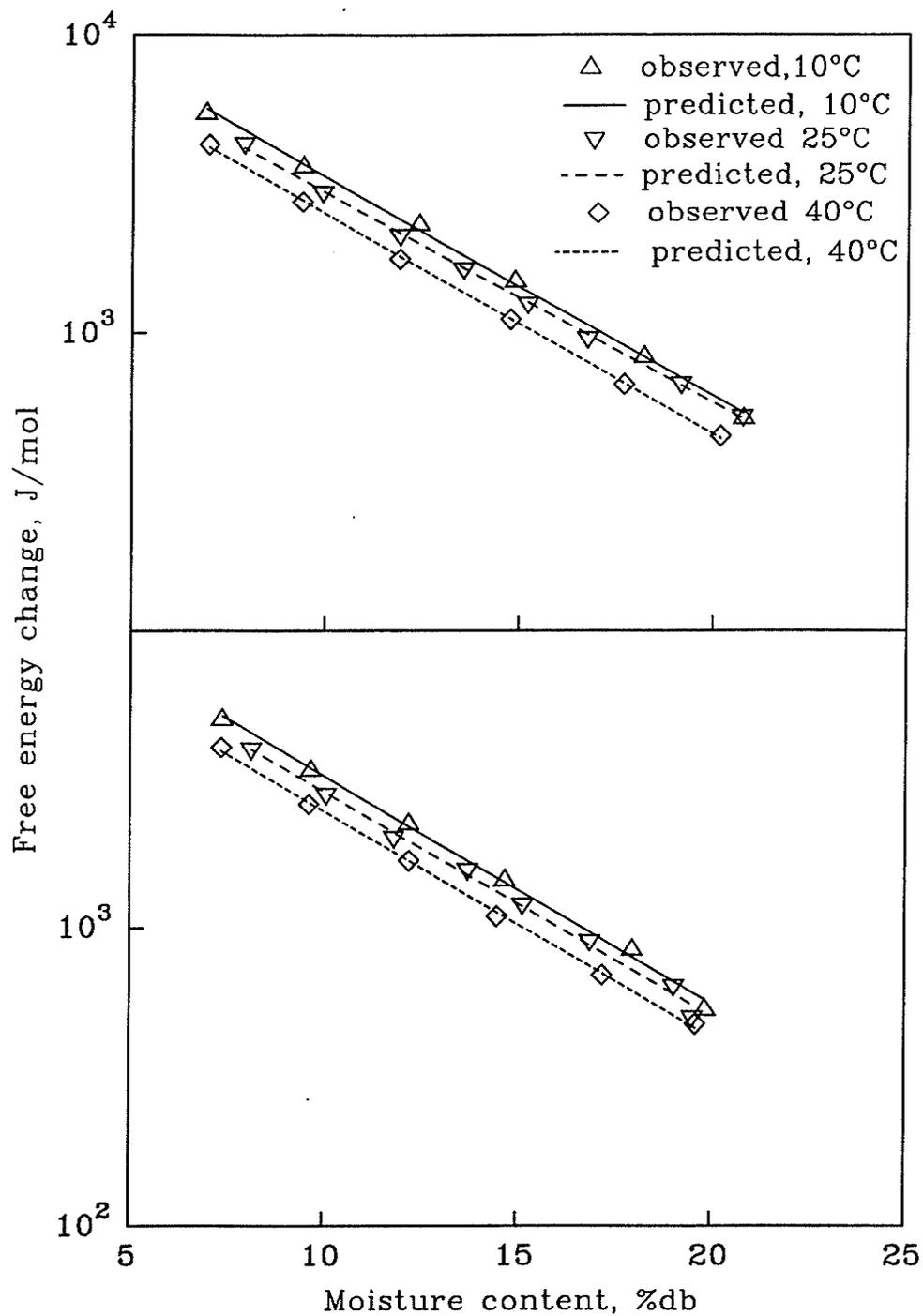


Fig. 5.14. Observed and predicted free energy change of adsorption of patent flour, non-compacted flour (above) and compacted flour (below).

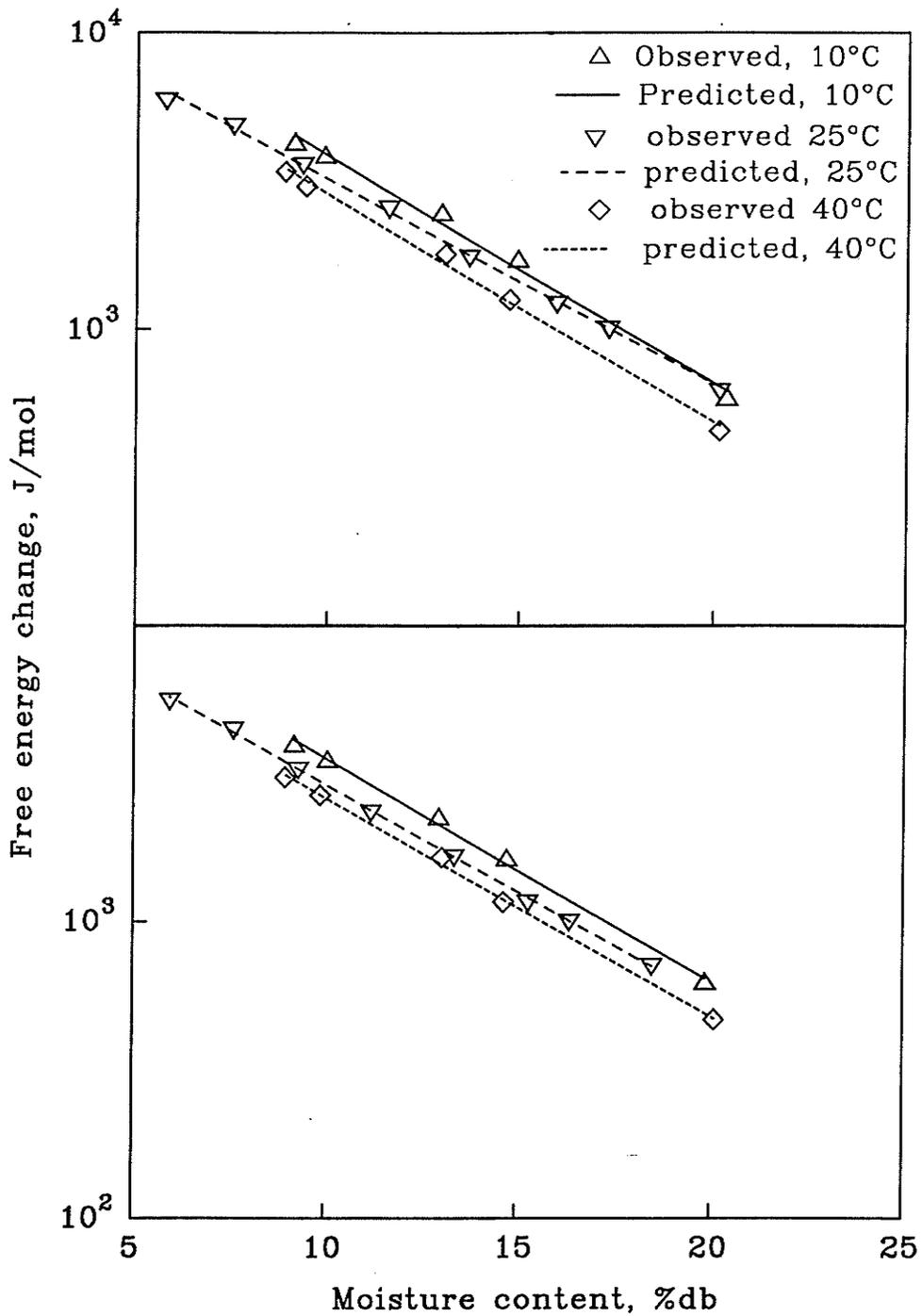


Fig. 5.15. Observed and predicted free energy change of desorption of straight grade flour, non-compacted flour (above) and compacted flour (below).

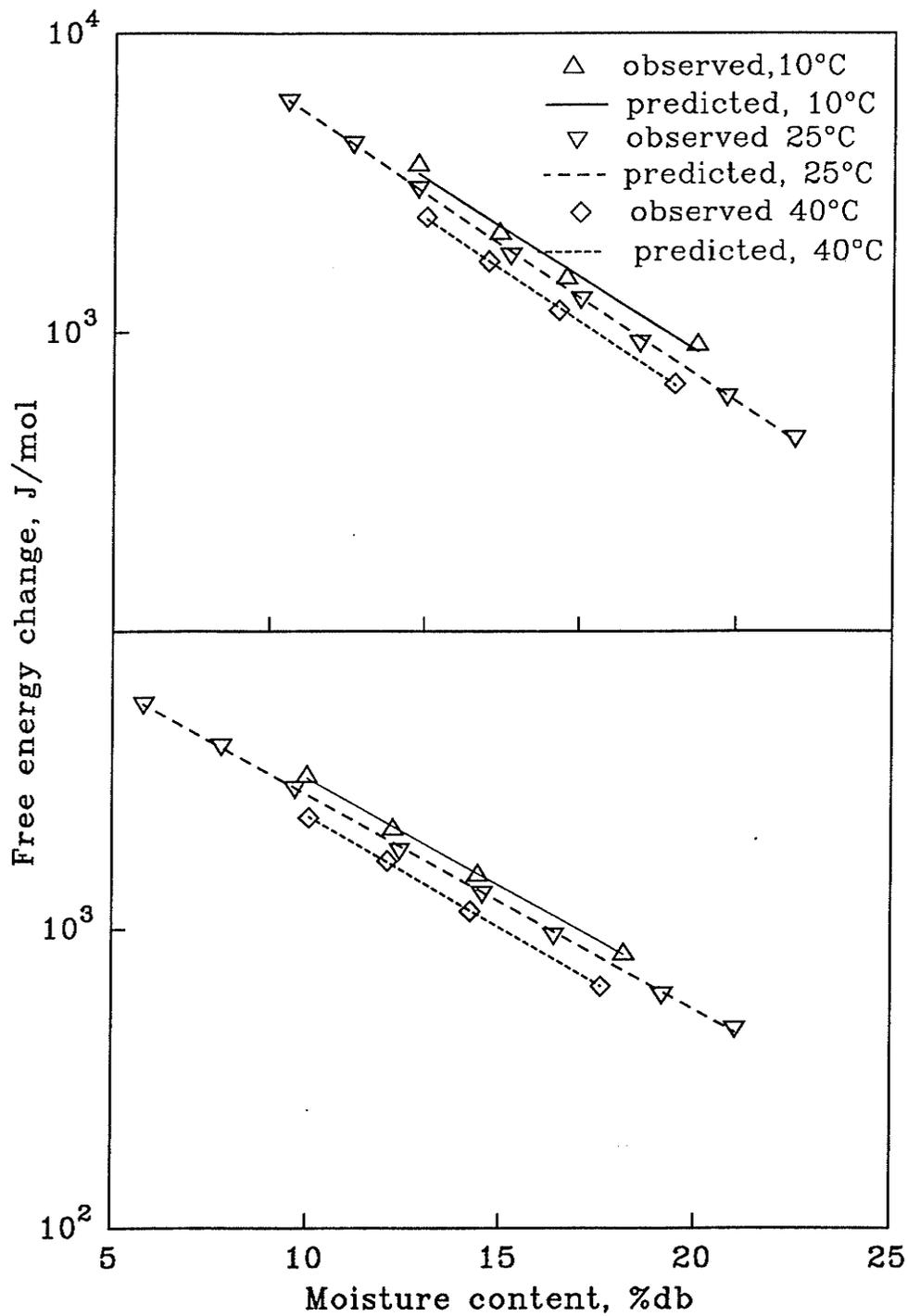


Fig. 5.16. Observed and predicted free energy change of adsorption of straight grade flour, non-compacted flour (above) and compacted flour (below).

Table 5.3. Estimates of parameters of Eq. 3.5 with their standard error and confidence intervals for the patent flour.

Material	Temp. °C	Constant	Estimate	Std. error	95% Confidence Intervals	
					Lower	Upper
Non-compacted, Desorption	10	A	1.91×10^4	686	1.77×10^4	2.06×10^4
		B	0.162	0.003	0.157	0.168
	25	A	1.60×10^4	579	1.48×10^4	1.72×10^4
		B	0.161	0.003	0.156	0.167
	40	A	1.36×10^4	291	1.30×10^4	1.42×10^4
		B	0.165	0.002	0.162	0.169
Compacted, Desorption	10	A	1.83×10^4	677	1.69×10^4	1.98×10^4
		B	0.167	0.003	0.161	0.173
	25	A	1.59×10^4	313	1.52×10^4	1.65×10^4
		B	0.167	0.002	0.164	0.170
	40	A	1.34×10^4	390	1.25×10^4	1.42×10^4
		B	0.167	0.002	0.162	0.172
Non-compacted, Adsorption	10	A	1.81×10^4	567	1.69×10^4	1.93×10^4
		B	0.168	0.002	0.163	0.173
	25	A	1.53×10^4	270	1.47×10^4	1.59×10^4
		B	0.164	0.001	0.161	0.167
	40	A	1.39×10^4	235	1.34×10^4	1.44×10^4
		B	0.171	0.001	0.168	0.174
Compacted, Adsorption	10	A	1.85×10^4	640	1.72×10^4	1.99×10^4
		B	0.174	0.003	0.168	0.179
	25	A	1.58×10^4	588	1.46×10^4	1.70×10^4
		B	0.171	0.003	0.165	0.177
	40	A	1.47×10^4	287	1.41×10^4	1.53×10^4
		B	0.178	0.002	0.174	0.181

Table 5.4. Estimates of parameters of Eq. 3.5 with their standard error and confidence intervals for straight grade flour.

Material	Temp. °C	Constant	Estimate	Std. error	95% Confidence Intervals	
					Lower	Upper
Non-compacted, Desorption	10	A	2.15×10^4	1482	1.83×10^4	2.47×10^4
		B	0.172	0.005	0.161	0.183
	25	A	1.62×10^4	342	1.55×10^4	1.69×10^4
		B	0.161	0.002	0.157	0.164
	40	A	1.55×10^4	799	1.38×10^4	1.73×10^4
		B	0.170	0.004	0.161	0.179
Compacted, Desorption	10	A	1.94×10^4	927	1.74×10^4	2.14×10^4
		B	0.170	0.004	0.162	0.178
	25	A	1.56×10^4	286	1.50×10^4	1.61×10^4
		B	0.168	0.002	0.165	0.171
	40	A	1.40×10^4	618	1.27×10^4	1.53×10^4
		B	0.168	0.004	0.160	0.176
Non-compacted, Adsorption	10	A	1.58×10^4	1479	1.25×10^4	1.91×10^4
		B	0.156	0.007	0.141	0.172
	25	A	1.48×10^4	196	1.44×10^4	1.53×10^4
		B	0.162	0.001	0.160	0.164
	40	A	1.25×10^4	350	1.17×10^4	1.33×10^4
		B	0.163	0.002	0.158	0.168
Compacted, Adsorption	10	A	1.70×10^4	571	1.57×10^4	1.83×10^4
		B	0.167	0.003	0.161	0.172
	25	A	1.51×10^4	188	1.47×10^4	1.55×10^4
		B	0.168	0.001	0.165	0.170
	40	A	1.35×10^4	540	1.23×10^4	1.47×10^4
		B	0.173	0.003	0.166	0.180

The parameter estimates for the relationship between free energy change of sorption and moisture content (Eq. 3.5), as proposed by Chung and Pfoest (1967), were also calculated for non-compacted flour and compacted flour pellets conditioned for desorption and adsorption in both flour grades and are given in Tables 5.3 and 5.4. Proc. NLIN (SAS 1985) was used to estimate the parameters with their standard error and confidence intervals.

The parameter estimates (refer to Tables 5.3 and 5.4) of non-compacted flour are not significantly different (95% level) from those of compacted flour pellets in either desorption or adsorption for both flour grades. This suggests that sorption isotherms are not significantly influenced by compaction. But, the parameter estimates of non-compacted and compacted flour decrease significantly with increasing temperatures from 10 to 40°C. The parameter estimates of non-compacted flour conditioned for desorption are not significantly different (95% level) from those conditioned for adsorption at any temperature. This suggests that hysteresis is not significant in non-compacted flour. A similar trend was also observed for compacted flour pellets.

5.2.6 Heats of Adsorption and Desorption

The isosteric heat of sorption (Q_s) is a measure of the excess heat of sorption in addition to the heat of vaporization or the heat of condensation to account for the desorption or adsorption process, respectively.

When the values of $\ln(a_w)$ are plotted as a function of the reciprocal of absolute temperature ($1/T$) at constant moisture content, a straight line is obtained (Labuza 1968). The slope of the line according to Eq. 3.3 is directly proportional to the isosteric heat of sorption. Thus, the calculation of heat of sorption values requires that sorption isotherm measurements be made at several temperatures (minimum of two).

Figs. 5.17a, 5.17b and 5.18a, 5.18b show the plots of $\ln(a_w)$ vs. $1/T$ at constant moisture content for the non-compacted flour and compacted flour pellets of the patent flour equilibrated using the desorption and adsorption conditions, respectively. From the slope of the curve, the isosteric heat of sorption is calculated. The figures also show that the slope of the curve decreases with an increase in moisture content. This suggests that the isosteric heat of sorption decreases continuously with an increase in moisture content. Such a trend is also shown in Figs. 5.17 c and 5.18 c, where the total heat of sorption i.e., Q_s + heat of vaporization, in case of desorption, or Q_s + heat of condensation, in case of adsorption, is plotted as a function of moisture content. The total heat of sorption decreases with an increase in moisture content, approaching the heat of vaporization or the heat of condensation (43.5 kJ/mol). At low moisture contents, water adheres tightly to the polar sites thus increasing the heat of sorption necessary to disrupt the intermolecular attractive forces. At higher moisture contents, water is sorbed on multilayers and hence it behaves almost like pure water. Thus, the heat equal to the heat of vaporization would be just sufficient to desorb the water molecules.

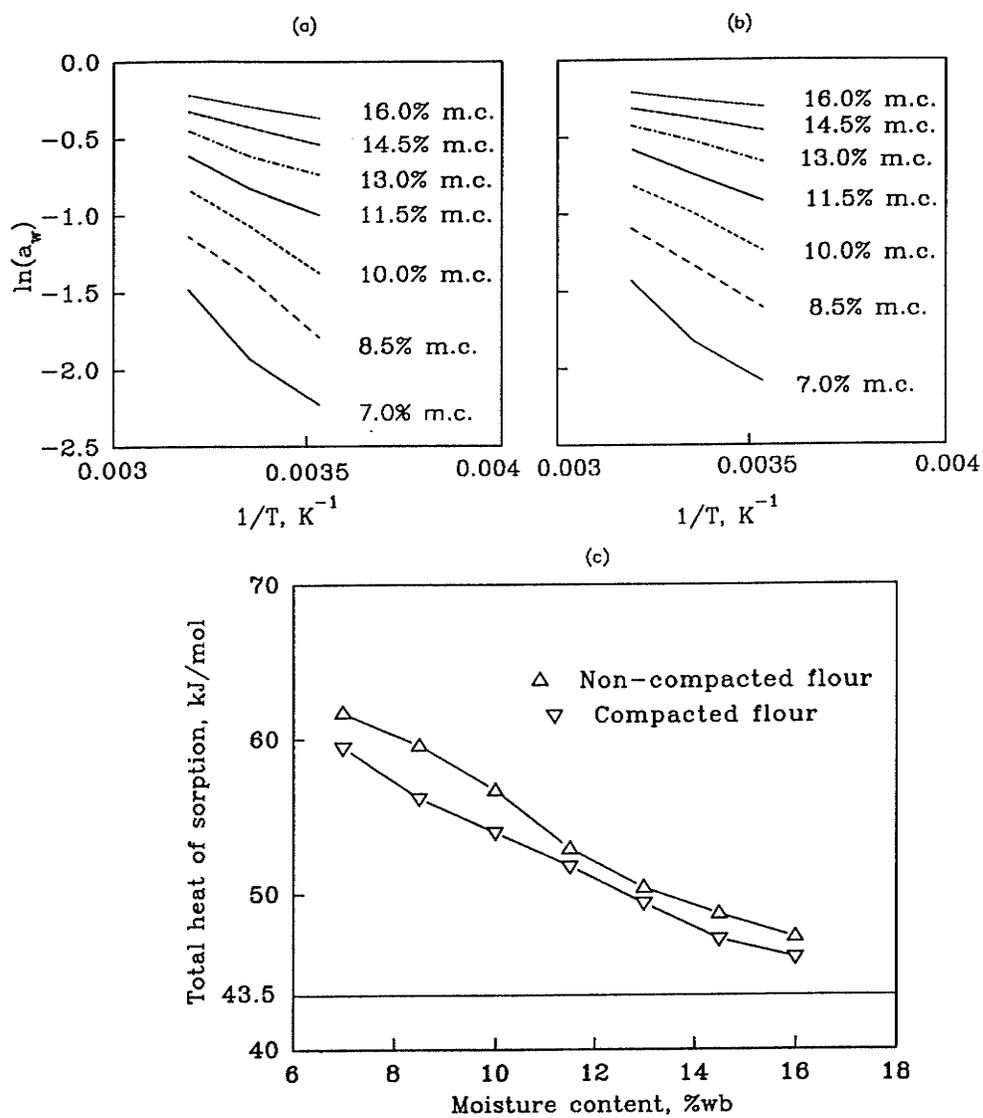


Fig. 5.17. Isosteric heat of sorption for (a) non-compacted flour and (b) compacted flour and (c) heat of sorption as a function of moisture content - for the desorption isotherm of patent flour.

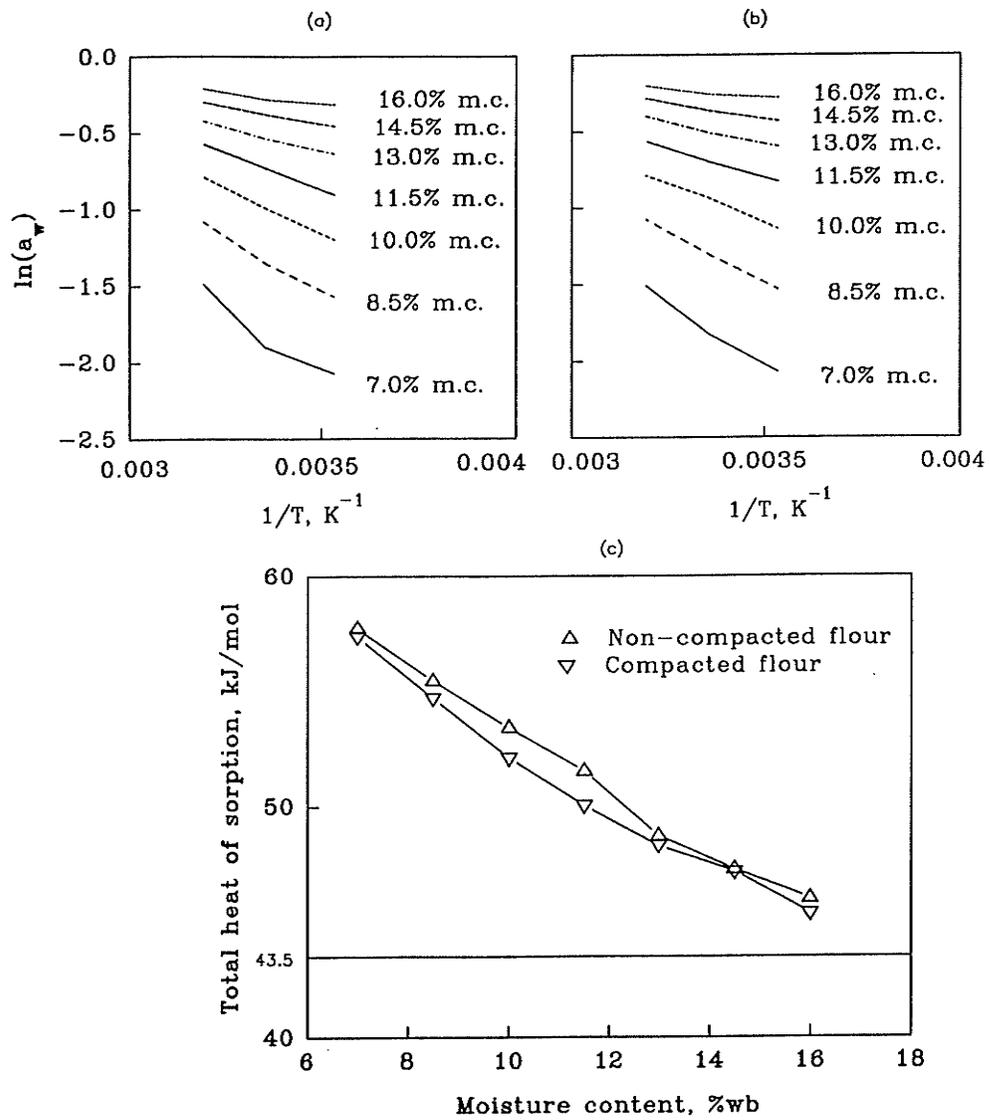


Fig. 5.18. Isosteric heat of sorption for (a) non-compacted flour and (b) compacted flour and (c) heat of sorption as a function of moisture content - for the adsorption isotherm of patent flour.

An analysis of the results using the paired t-test (SAS 1985) shows that the total heat of sorption as a function of moisture content for the non-compacted flour conditioned for desorption is significantly greater (95% level) than that of the compacted flour pellets in the moisture content range of 7 to 11% wb. Beyond this range the difference between the heat of sorption is statistically insignificant. This agrees well with the earlier findings that the water adheres more tightly at low moisture contents in the case of non-compacted flour conditioned for desorption (and hence it exhibits reduced water activities). However, this phenomenon was not observed to be statistically significant (95% level) for adsorption conditions. Here, the difference in heat of sorption between the non-compacted flour and compacted flour pellets at any moisture content was insignificant. Also, the heat of desorption was not significantly greater (95% level) than the heat of adsorption. This confirms the earlier finding that the hysteresis between the desorption and adsorption isotherms was not significant.

The hysteresis between the desorption and adsorption isotherms may be due to the initial conditioning of the samples. In desorption tests, the samples are brought up to a high initial moisture content first and this will account for the swelling of the particles. The amount of sorptive sites available for sorption of water molecules is considerably increased. In the case of adsorption tests, the samples are brought down to a low initial moisture content first and this accounts for the shrinkage of the particles. Thus, the amount of sorptive sites available for further adsorption of water molecules is considerably decreased. This phenomenon of swelling and shrinkage causes the hysteresis that occurs between the desorption and adsorption isotherms (Chung and Pfof 1967).

5.2.7 Calculation of Monolayer Values Using BET Equation

When $a_w/(1-a_w)M$ is plotted against a_w (Fig. 5.19), a straight line is obtained and from the slope and intercept of the straight line, a monolayer coverage value can be calculated. Here an attempt was made to calculate such values for the patent flour only (as enough data points could not be obtained in the case of straight grade flour). The monolayer coverage values include the determination of monolayer moisture content (M_m) which permits the calculation of the specific surface area of the adsorbent (S_0), and a characteristic constant related to the heat of adsorption of the monolayer. The calculation of S_0 from M_m is given in the earlier section (Eq. 3.2). The BET isotherm equation (Eq. 3.1) is applied only for the adsorption isotherm data.

Table 5.5. Monolayer moisture content (M_m) and specific surface area (S_0), calculated from BET equation (Eq. 3.1), for the patent flour.

Temperature, °C	Treatment	M_m g/100g	S_0 m ² /g
10	Non-compacted	7.26	256.5
	Compacted	6.90	243.8
25	Non-compacted	6.23	220.3
	Compacted	5.86	207.1
40	Non-compacted	5.31	187.6
	Compacted	5.16	182.2

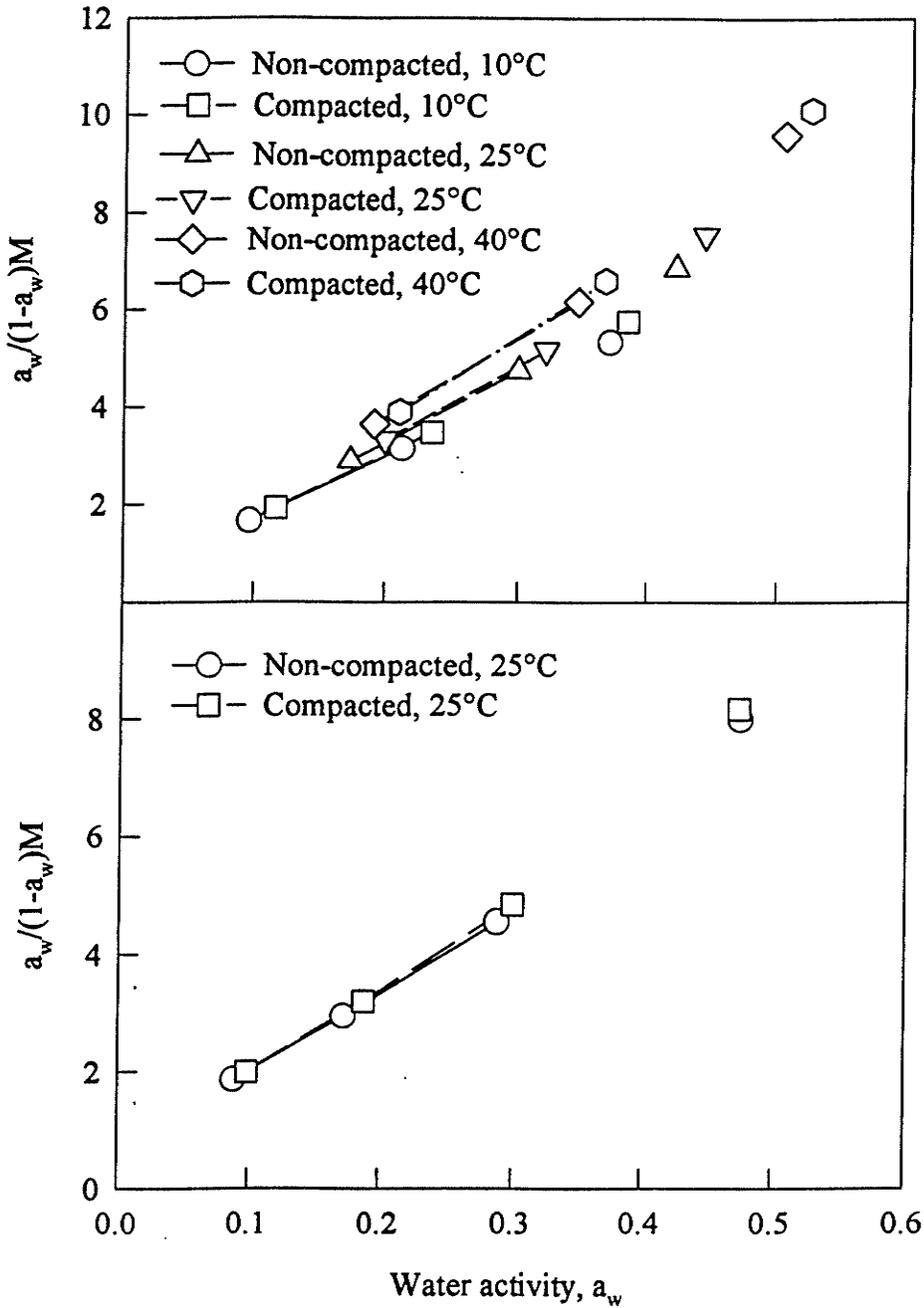


Fig. 5.19. BET monolayer plot (Brunauer et al. 1938) for patent flour (above) and straight grade flour (below).

Table 5.5 shows that the monolayer water content is temperature dependent. As would be expected, the compacted flour had a lower monolayer moisture content than did the non-compacted flour at all temperatures. This results in a decreased specific surface area of the adsorbent for compacted flour pellets as compared to the non-compacted flour. Lack of sufficient data points below 0.5 a_w range obviated the possibility of finding such values for the straight grade flour at 10 and 40°C. However, at 25°C, the straight grade flour had a monolayer moisture content of 7.04 g/100g solid corresponding to a specific surface area of 248.6 m²/g in the case of non-compacted flour as compared to 6.76 g/100g solid and 239 m²/g, respectively for the compacted flour pellets. This agrees well with the findings of Mok and Dick (1991), considering that compaction effected some starch damage in the flour, that the damaged starch had a lower equilibrium moisture content than the undamaged starch in the monolayer moisture region. A comparison of the data of straight grade flour with patent flour at 25°C suggests that the M_m (and hence S_0) of straight grade flour is higher than that of patent flour.

In general, it was found that the differences in moisture sorption behaviour of flour due to compaction were not significant. This seems to nullify the first hypothesis (section 1.3) where, it was stated that compaction of flour to granulates alters the sorptive capacity of flour and hence its sorption hysteresis. Information on particle size distribution of flour reconstituted after compaction is of no relevance as moisture sorption by flour is not dependent on the particle size (Bushuk and Winkler 1957).

5.3 Rheological Behaviour of a Flour-Water Dough

5.3.1 Empirical and Imitative Rheological Tests

Initially, flour granulates prepared using straight grade flour of 13.1% wb m.c. (enough to make at least 100g of flour) were reconstituted using different milling devices namely, a juicer, hammer mill, burr mill, and bran finisher (section 4.6). The Farinograph absorption was determined, in duplicate, for each of the flours reconstituted using different devices. The purpose of this was to compare the water absorption of the untreated, control flour with that of the flour reconstituted by various methods after compaction. The Farinograph absorption is an excellent and a rapid indicator of starch damage for a given flour. The higher the starch damage, the greater the water absorption by flour (Farrand 1972).

The Farinograph absorption for the untreated flour was 61.3%, and for the reconstituted flour using juicer, hammer mill, burr mill, and bran finisher were 62.2, 62.2, 62.4, and 62% on a 14% moisture basis, respectively. Thus, the difference in water absorption by flour as influenced by different reconstitution techniques was not significant. Hence, the remaining flour granulates were reconstituted using the juicer in batches (considering the relative ease with which the granulates were reconstituted) for the rest of the empirical and flow behaviour tests of a flour-water dough. All the empirical determinations were done in duplicates.

(i) Farinograph

The Farinogram is characterized by Farinograph absorption, dough development time, stability, and mixing tolerance index. The Farinograph absorption is the amount of water to be added to 100 g of flour (on a 14% m.c. wet basis) to make a dough of uniform consistency. The

dough development time is the time required to develop a dough to optimum development (500 BU). The stability and mixing tolerance index of a dough are a measure of the resistance of the dough to overmixing. The Farinograph properties for both grades of flour are given in Table 5.6.

Table 5.6. Farinograph properties of control and treated dough of two grades of flour.

Flour Grade	Type	Farinograph Absorption (%)	Development time (min)	Stability (min)	M.T.I* (BU)
Patent flour	Control	60.3	4.25	15.0	20
	Treated	61.0	3.75	12.75	20
straight grade flour	Control	61.5	6.0	10.0	10
	Treated	63.2	5.5	13	15

* M.T.I (BU) = Mixing tolerance index, Brabender units

Brabender Farinograph water absorption, development time, stability, and mixing tolerance index were not significantly altered by compaction. A small increase in water absorption (0.7% for patent flour and 1.7% for straight grade flour) in the treated flour could be due to increased starch damage. Dexter et al. (1994) reported the increase in water absorption by durum wheat flour with increased starch damage, as influenced by different milling techniques. The decreased stability by 2.25 min in the treated patent flour may reflect the inability of the damaged starch to hold the water. However, this behaviour was not observed in the case of straight grade flour. This could be because that the water released by damaged starch was absorbed by gluten proteins.

(ii) Mixograph

The mixograph properties, namely peak time and peak height, were determined in a 35 g mixograph for a constant water addition of 65% (on a 14% moisture basis). The mixogram is characterized by peak time and peak height. The peak time is the time required for the peak to reach maximum height which is similar to the dough development time in a Farinogram. The peak height is the height from the base line to the centre of the curve at maximum plasticity. It is an indication of flour strength.

Table 5.7. Mixograph properties of control and treated dough of two grades of flour.

Flour Grade	Type	Peak time (min)	Peak height (MU)*
Patent flour	Control	5	450
	Treated	4.75	420
straight grade flour	Control	4	410
	Treated	4	440

* MU = Mixograph units

The mixograph peak time did not show any difference as influenced by compaction. The peak height is an indication of the resistance of the dough to mixing. The treatment in straight grade flour caused an increase (by 30 MU) in peak height (Table 5.7). The mixograph properties were evaluated at constant water addition. But, the treated flour with slightly increased starch damage could absorb more water than the untreated control flour to form a dough of uniform consistency. Hence, the dough made using the treated flour had an increased stiffness (as indicated by the increase in peak height).

(iii) Alveograph

The standard ICC (International Association for Cereal Science and Technology) procedure for Alveograph recommends 50% water addition on a 15% flour moisture content basis. Dexter et al. (1994) reported that the usefulness of this procedure, as an indicator of inherent dough properties, for hard wheats is limited because the flour starch damage, and hence flour water absorption, is highly sensitive to milling technique.

The Alveogram is mainly characterized by three parameters, namely the height of the curve, P, (an indication of the resistance of the dough to extension), the length of the curve, L, (an indication of the extensibility of the dough), and the total work needed to cause the dough to rupture, W, (an acknowledged indicator of the overall dough strength) (Rasper et al. 1985).

Table 5.8 Alveograph properties of control and treated dough of two grades of flour.

Flour	Treatment	P mm	L mm	W ($\times 10^{-4}$ J)
Patent flour	Control	88.2	111.0	363
	Treated	92.4	105.0	366
Straight grade flour	Control	71.1	119.2	265
	Treated	88.4	105.8	304

For dough made from compacted flour, the height of curves increases and the length of the curves decreases - the effect being much more pronounced in case of the straight grade flour (Table 5.8). This could be attributed to the fact that the reconstitution of the compacted flour granulate causes a slight increase in starch damage and as the alveograph was performed at

constant water addition, the amount of water added to the treated flour was much less than what it could actually absorb [damaged starch molecules could absorb twice as much water as the undamaged starch molecules (Farrand 1972)]. This perhaps might have resulted in a stiffer dough being produced using the treated flour. The above observation was confirmed by the amount of work, W , (represented by the area under the curve) needed to cause the dough to rupture. The treated flour had a 15% higher W value than the untreated flour, again the effect being predominant in straight grade flour.

(iv) Visco-Amylograph

The visco-amylograph is mainly characterized by peak viscosity. The peak (maximum) viscosity, irrespective of the temperature at which the peak is reached, indicates the highest viscosity yielded by the starch during the gelatinization process under the conditions of the test (Rasper 1980).

As starch gelatinization depends on the amount of starch granules present initially and their ability to swell and increase in viscosity during heating, it is reasonable to expect a low peak viscosity from samples with very high starch damage. The peak viscosities obtained for the control and treated flour, in the case of patent flour, were exactly the same (760 BU). In case of straight grade flour, the treated flour had a peak viscosity of 710 BU as against 730 BU obtained for the control flour, which is not significant.

(v) Analytical Tests

To confirm the findings of the above rheological tests, analytical tests were made on wet gluten content and starch damage according to the standard procedures (sections 4.4.6 and 4.4.7). The results obtained are given in Table 5.9.

Table 5.9. Results of wet gluten and starch damage tests for control and treated flour of two grades.

Flour grade	Type	Wet gluten %	Starch damage %
Patent flour	Control	27.1	7.7
	Treated	27.7	7.8
Straight grade flour	Control	28.2	7.7
	Treated	29.6	8.4

The results of the analytical tests confirm the findings of Weiland (1977) that the starch contained in the treated flour is damaged to a considerably greater degree because of the compression and subsequent conversion to flour than due to the initial milling process. This increases the ability of the flour to absorb more water. In straight grade, the treated flour had more wet gluten than the control flour. This also agrees with the findings of Weiland (1977) that the heat generated during compaction resulted in the modification of gluten characteristics and hence in an increase in the wet gluten content.

(vi) Canadian Short Process Baking (CSP)

Dexter et al. (1994) suggested that the absence of a long fermentation period in CSP would minimize the negative effects of high starch damage. The baking test was performed only

for the straight grade flour because of the shortage of material in patent flour. The results obtained in the baking test are given in Table 5.10.

Table 5.10. Results of the Canadian Short Process bake test for the control and treated straight grade flour.

	Control	Treated
Baking absorption, %	62	63
Remix time, min	1.7	1.6
Loaf volume, cm ³	730	745
Bread appearance, units	6.2	6.5
Crumb structure	6.5-open	6.0-open
Crumb colour, units	5.0-gray	5.0-gray

Both flours baked exactly as would be expected for a No.1 grade Canadian Western Red Spring wheat. The differences between the two flours are very minor and statistically insignificant. This is in contrast with the findings of Weiland (1977) who reported that flour from freshly harvested wheat, when evaluated with Rapid Mix Test, produced baked goods with a volume of 5400 cm³ / 1000g flour, while the same flour after compression and reconversion to flour produced baked goods with a volume of approximately 6100 cm³ / 1000g flour.

5.3.2 Fundamental Rheological Test

The flow characteristics of a flour-water dough namely, flow behaviour index and the consistency coefficient, were determined for the control and treated dough of both grades of flour using a capillary rheometer, similar to the one employed by Sharma et al. (1993).

The optimum water absorption by flour and optimum mixing time to produce a dough of uniform consistency were determined using a Farinograph and a Mixograph, respectively. Subsequently, larger quantities of dough for capillary rheometer tests were prepared by mixing suitable amounts of flour and water for a known time in a pin mixer (Hlynka and Anderson 1955).

Table 5.11. Force and flow rate values obtained at different crosshead speeds with a capillary of 3.15mm diameter and 101.2mm length for the control and treated flour dough in patent and straight grade flour.

Crosshead speed (mm/min)	Flow rate Q $10^{-8} \text{ m}^3/\text{s}$	Patent flour		Straight grade flour	
		Control	Treated	Control	Treated
		Force, N	Force, N	Force, N	Force, N
5	2.31	136	135	110	109
10	4.63	194	212	171	164
20	9.26	291	302	280	234
50	23.1	524	530	424	444
100	46.3	716	685	632	651
200	92.6	909	922	824	839
350	162	1067	1038	928	930
500	231	1213	1142	1050	1036

Table 5.11 gives the volumetric flow rate of the dough, Q , and force required to extrude the dough through a capillary of known dimensions, for the control and treated flour dough for patent and straight grade flour, at different crosshead speeds. The force values were obtained by subtracting the force when the dough was about to enter into the capillary from the force when

the dough was about to be extruded out of the capillary. Each reading is an average of four replicates.

Table 5.12 gives the pressure drop, shear stress (Eq. 3.7), and shear rate (Eq. 3.8) values calculated using the equations given in section 3.2. The pressure drop values were obtained by dividing the force values (Table 5.11) by the cross-sectional area of the plunger.

Table 5.12. Pressure drop (ΔP), shear rate ($\dot{\gamma}$), and shear stress (τ) values for the treated and control flour dough.

Cross head speed (mm/min)	Shear rate $\dot{\gamma}$ (1/s)	Patent flour				Straight grade flour			
		Control		Treated		Control		Treated	
		ΔP kPa	τ kPa	ΔP kPa	τ kPa	ΔP kPa	τ kPa	ΔP kPa	τ kPa
5	7.5	490	3.8	486	3.8	396	3.1	393	3.1
10	15.1	699	5.4	763	5.9	616	4.8	591	4.6
20	30.2	1048	8.2	1087	8.5	1009	7.9	843	6.6
50	75.4	1887	14.7	1909	14.9	1527	11.9	1600	12
100	150.8	2578	20.1	2467	19.2	2277	17.7	2345	18
200	301.5	3273	25.5	3320	25.8	2968	23.1	3022	24
350	527.7	3842	29.9	3738	29.1	3343	26.0	3350	26
500	753.9	4368	34.0	4112	32.0	3783	29.4	3732	29

It can be noticed that increasing crosshead speed increases both the shear rate and the pressure drop. It is because an increase in crosshead speed increases the volumetric flow rate which in turn increases the shear rate. In case of a non-Newtonian material, the pressure drop is non-linearly proportional to the volumetric flow rate and thus the pressure drop increases with

increasing shear rate (Sharma et al. 1993).

A linear regression was performed using the values of log shear stress as the dependent variable and log shear rate as the independent variable (Toledo 1991). The slope of the regression was the flow behaviour index, n , and the Rabinowitsch-Mooney correction factor, CF , was evaluated according to Eq. 3.9 in section 3.2. The values are given in Table 5.13. The coefficient of determination for the plot of $\log \tau$ vs. $\log \dot{\gamma}$ is given by R^2 .

Table 5.13. Rabinowitsch-Mooney correction factor (CF) for the treated and control dough of both flour grades.

	Patent flour		Straight grade flour	
	Control	Treated	Control	Treated
n	0.411	0.390	0.409	0.410
CF	1.36	1.39	1.36	1.36
R^2	0.985	0.981	0.981	0.979

The corrected shear rate ($\dot{\gamma}_w$) values were obtained by multiplying the apparent shear rate values by the correction factor (Eq. 3.10). The shear stress and the corrected shear rate values (after applying Rabinowitsch-Mooney correction factor, CF) were adequately described by the power law model (Eq. 3.11). A non-linear regression, PROC NLIN (SAS 1985), was performed to estimate the constants namely n , the flow behaviour index and K , the consistency coefficient. The values along with standard error and the 95% confidence limits are given in Table 5.14 for the treated and control flour.

The values of K and n indicated that the flow parameters for the treated and control flour were not significantly different (95% level). This confirms with the earlier findings, using the empirical rheological tests, that the treated flour behaved the same as control flour. This is in contrast to the findings of Ramanathan and Cenkowski (1994a) who observed changes in the flow parameters of dough due to flour compaction using all-purpose flour.

Table 5.14. Flow behaviour index (n) and consistency coefficient (K) values, with their standard error and confidence intervals, for the treated and control dough of both grades of flour.

Flour	Constant	Estimate	Std. error	95% Confidence Intervals	
				Lower	Upper
Control, patent flour	n	0.411	0.028	0.342	0.481
	K	2090	369	1187	2993
Treated, patent flour	n	0.390	0.031	0.315	0.466
	K	2296	439	1222	3371
Control, straight grade flour	n	0.409	0.032	0.331	0.486
	K	1861	366	966	2755
Treated, straight grade flour	n	0.410	0.040	0.311	0.509
	K	1858	466	717	2999

This suggests that the accuracy of the fundamental tests should be improved. The possible sources of error may be in filling the dough in the cylinder without air gaps and in measuring the force when the dough is about to extrude out of the capillary. A plot of the shear stress versus corrected shear rate for patent flour and straight grade flour (Fig. 5.20) revealed that dough exhibits a pseudo-plastic nature (shear thinning material) and that the apparent viscosity

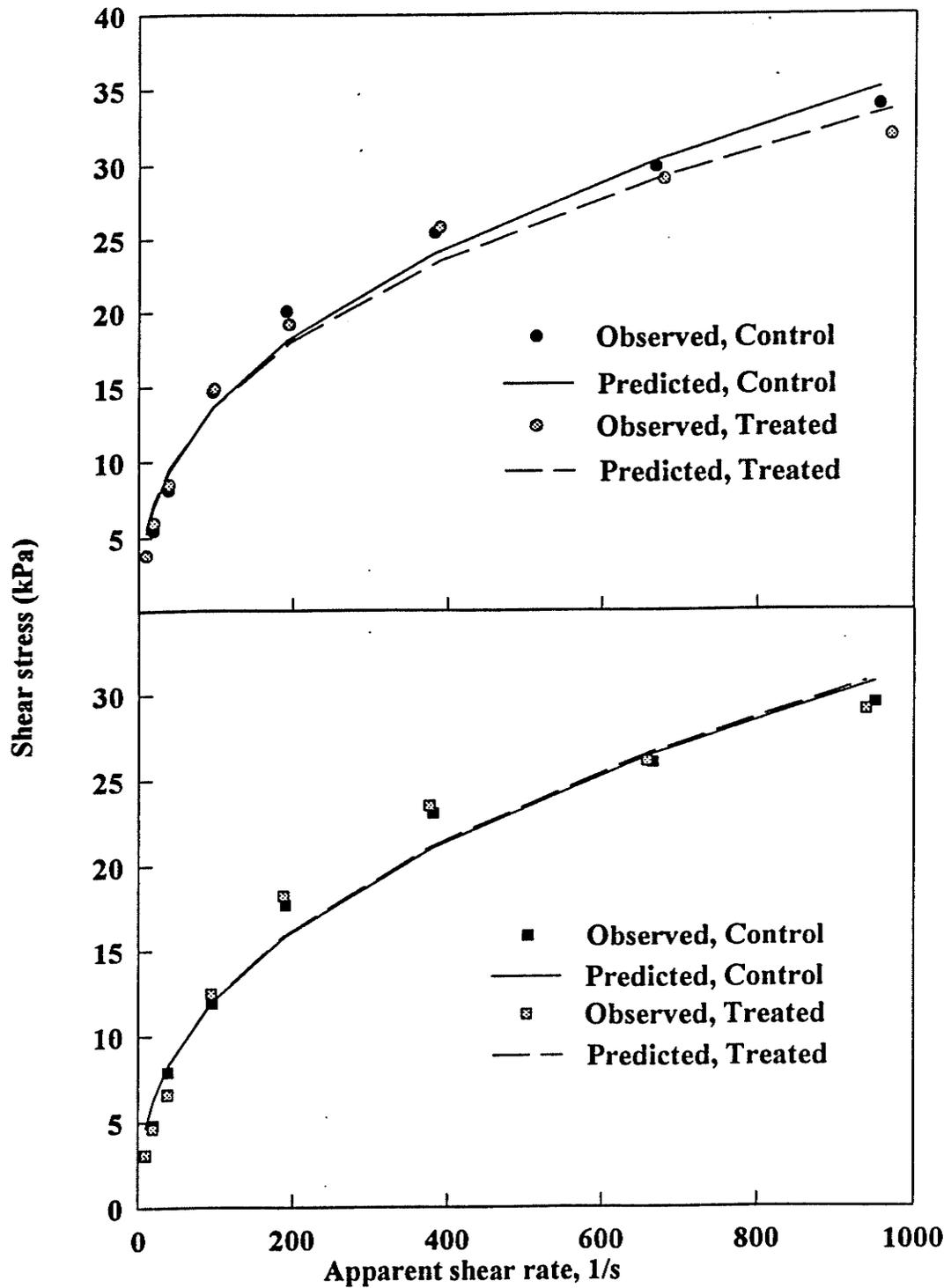


Fig. 5.20. Flow curves for the dough prepared using the treated and control flour in patent flour (above) and straight grade flour (below).

of dough decreased with increasing shear rate.

In general, the flour reconstituted after compaction had no adverse influence on the rheological behaviour of a dough made using that flour. This seems to nullify the second hypothesis (section 1.3) where it was stated that flour reconstituted after compaction might change in its functional properties and hence in rheological behaviour of a dough made using that flour.

6. CONCLUSIONS AND RECOMMENDATIONS

The compaction behaviour of two grades of flour, a low extraction (64% extraction) patent flour and a high extraction (79%) straight grade flour, was not influenced by flour moisture content (13.1, 13.9, and 14.6% wet basis) or loading rates (5, 50, and 100 mm/min).

The compaction of flour had little influence on its moisture sorption hysteresis patterns at 10, 25, and 40°C. However, the temperature had a significant influence (95% level) on the moisture sorption isotherms of both non-compacted flour and compacted flour pellets. The hysteresis between the adsorption and desorption isotherms was not significant (95% level), at any of the temperatures, for either non-compacted flour or compacted flour pellets.

Modified Halsey equation predicted the sorption isotherms of flour with a reasonably good accuracy and the estimates of parameters were in good agreement with the published data. Thermodynamic changes, such as Gibb's free energy change of sorption and isosteric heat of sorption using Clausius-Clapeyron equation were not influenced by compaction of flour. Monolayer coverage values such as, monolayer moisture content and hence the surface area available for water sorption were little influenced by compaction.

The fundamental and empirical rheological properties of a flour-water dough, prepared using the non-compacted flour and the flour reconstituted after compaction, were not significantly different. Both flours baked exactly as would be expected for a No.1 Canadian Western Red

Spring wheat.

It is recommended that the future aspect of this research be focused on the large scale compaction of flour which would simulate the practical export conditions. Much work needs to be done on the quality aspects of the flour reconstituted after compaction to ensure that the flour maintains its quality which is in turn very important for high quality baked foods. The particle size distribution of the flour reconstituted after compaction needs to be studied as it has been shown to influence dough development (Farrand 1972). It is also recommended that the compacted flour granulates be stored at various temperatures for increasing periods of time, diverse set of flours be used and different degrees of compaction be achieved.

7. REFERENCES

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