MOISTURE MOVEMENT AND EQUILIBRIA IN THE DEHYDRATION OF
CASSAVA AND WHITE POTATO

DISSERTATION

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

By
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* * * * *

The Ohio State University
1976

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---
Co-Adviser
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Dedicated to

My father, Patrick O. Igbeka
My mother, Beatrice N. Igbeka
My late father-in-law, Paul C. Dibosa

whose belief in and love for education and knowledge has
made it possible for me to come as far.
ACKNOWLEDGMENTS

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The author wishes to express his deepest thanks to his wife, Josephine Umejei whose love, patience and encouragement were unending.
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<tr>
<td>A</td>
<td>Constant in Harkins-Jura, Chung and Pfost equations</td>
</tr>
<tr>
<td>a</td>
<td>Water activity or relative humidity in decimal</td>
</tr>
<tr>
<td>m</td>
<td>Coefficient of Noss transfers</td>
</tr>
<tr>
<td>B</td>
<td>Constant in Harkins-Jura, Chung and Pfost equations</td>
</tr>
<tr>
<td>b</td>
<td>Constant in Langmuir equation</td>
</tr>
<tr>
<td>C</td>
<td>Concentration</td>
</tr>
<tr>
<td>c</td>
<td>Constant in BET equation</td>
</tr>
<tr>
<td>D</td>
<td>Diffusivity, cm²/sec</td>
</tr>
<tr>
<td>E</td>
<td>Non-dimensional moisture</td>
</tr>
<tr>
<td>G</td>
<td>Gibbs free energy</td>
</tr>
<tr>
<td>H</td>
<td>Differential heat</td>
</tr>
<tr>
<td>J</td>
<td>Mass or heat flux</td>
</tr>
<tr>
<td>K</td>
<td>Constant in Henderson equation</td>
</tr>
<tr>
<td>K₁</td>
<td>Constant in equation</td>
</tr>
<tr>
<td>k</td>
<td>Constant in Langmuir equation</td>
</tr>
<tr>
<td>K₀</td>
<td>Boltzmann Constant, (1.38 \times 10^{-16})erg/°C</td>
</tr>
<tr>
<td>L</td>
<td>Product thickness, cm or weight, gm</td>
</tr>
<tr>
<td>M</td>
<td>Moisture content in gm of H₂O/gm d.b.</td>
</tr>
<tr>
<td>N</td>
<td>Constant in Henderson equation</td>
</tr>
<tr>
<td>P</td>
<td>Vapor pressure</td>
</tr>
<tr>
<td>P₀</td>
<td>Saturation vapor pressure</td>
</tr>
<tr>
<td>p</td>
<td>Constant in Roa's, Ngoddy's equation</td>
</tr>
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</table>
\( Q_s \)  Heat of desorption, cal/mole

\( q \)  Constant in Roa's equation

\( RH, rh \)  Relative humidity

\( R_g \)  Gas constant

\( r_k \)  Kelvin radius

\( S \)  Entropy

\( T \)  Temperature in °K

\( t \)  Time, sec

\( V \)  Volume adsorbed, gm/gm solid or cc/gm of solid

\( \bar{V} \)  Molar volume in Kelvin equation

\( v_m \)  Monolayer adsorbed value, the same unit as \( V \)

\( W \)  Moisture content in Smith equation

\( x \ & \ y \)  Constants in Smith equation

\( x \)  Distance, cm

\( \sigma \)  Surface tension

\( \gamma \)  Constant in equation

\( \eta \)  Boltzmann's transform

\( \rho \)  Density

\( \varepsilon \)  Constant in equation

\( \delta \)  Thermal gradient coefficient of transfer of vapor
I. INTRODUCTION

The importance of cassava (Manihot esculenta) and potato (Solanum tuberosum) as sources of carbohydrates in developing and developed countries can not be overemphasized. Much research is going on in many international research centers to improve the protein content of cassava and processing quality of potato. Cassava, used as human food, is also used as animal feed. Much data are available for the drying parameters of potato, while little data are available for cassava. The main product under investigation is cassava; potato was selected primarily for comparison.

CASSAVA:

Cassava is a root tuber, which is a major source of calories for 200 to 300 million people of the tropics (Nastel, 1973a).* The crop is known as Cassava in the English-speaking countries of Europe and Africa; as manioc in the French-speaking countries; as tapioca in the English-speaking countries of Southeast Asia and North America; as mandioca in Brazil and as Yuca in the Spanish-speaking countries of South America.

Cassava roots contain about 35-40% dry matter and 60-65% moisture (Hollenman and Aten, 1950). About 90% of this dry weight consists of

* Numbers in parenthesis following names refer to appended references.
carbohydrates in the form of starch and sugars. Cassava starch is classified as a starch of low-amylose and high-amylopectin content.

Much research is being carried out (CIAT, 1973) (IITA, 1974) to improve the protein content in the cassava products for human and animal consumption.

The most popular method of preserving cassava is drying. Chirife (1969) reported on the through-circulation drying of cassava. Roa (1974) reported extensively on the physical efficiencies of several natural drying systems.

Little information is available in the literature on the variation of rate constant with moisture or time during drying. This is an important information in understanding the mechanism of moisture movement during drying. In designing a drying system, the knowledge of the binding energy is of great importance. The data on equilibrium moisture content available in literature do not cover the extensive temperature range from storage to processing.

_Cassava as Human Food:_ Cassava and potato constitute the main source of starch food in the developing countries. Current FAO estimates (Nestel, 1974b) show that about 55 million tons, of an annual global production of 98 million tons, are consumed by humans. Phillips (1974) has projected that by 1980 consumption will increase to about 71 million tons. Nicol (1952) reported that 25–56% of dietary calories in Southern Nigeria came from cassava, whereas Bailey's survey (1961) in Java has indicated areas where the caloric intake from cassava exceeded 63%.

Normanha (1970) recorded Brazilian urban and rural per capita intakes
of 43kg and 200 kg respectively, to give a national average of 124kg in the mid-1960's.

The form in which cassava is consumed varies widely but usually involves drying, pelleting, or processing into chips, followed by grinding and cooking. If prepared from the raw state, it must be stored as raw tubers for some period. Some promising progress has been achieved in the storage field. Ingram and Humphries (1972) reviewed extensively the storage of cassava in its fresh and dried forms. They referred to works by Singh and Mathur (1953) in India and by Czyhrniciw and Jaffe (1951) in Venezuela under low-temperature storage conditions. Young, et al., (1971) claimed to have extended shelf life considerably by waxing. They concluded that the only assured methods for safely storing cassava for periods exceeding two months are: (a) freezing the root, (b) drying and maintaining a moisture content of 13%, wet basis at ambient temperatures, or (c) leaving the roots in the ground.

Cassava as an Animal Feed: Major current users of cassava as animal feed are feed compounders in the European Economic Community (EEC) who import much of their cassava from developing countries. Tables 1 and 2 show predicted supply and demand of cassava in 1980. The major potentials for using cassava as animal feeds appear to be in cassava-producing countries. Muller, et al., (1971) satisfactorily substituted cassava meal for corn up to levels of 60% in broiler rations. Hamid and Jalaiudin (1972) successfully replaced corn at levels of up to 60% for layers. Cassava root meal has been used in both grower and layer chicken diets in Hawaii, (Enriquez and Ross, 1972), where egg production, feed conversion, egg weight and shell thickness were related to levels of cassava
Table 1. Predicted Supply of Cassava for 1980 (1000 fresh metric tons) (Phillips 1973a)

<table>
<thead>
<tr>
<th>Region</th>
<th>Low Estimate</th>
<th>High Estimate</th>
</tr>
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<tbody>
<tr>
<td>Latin America</td>
<td>48,052</td>
<td>60,491</td>
</tr>
<tr>
<td>Africa</td>
<td>37,107</td>
<td>37,207</td>
</tr>
<tr>
<td>Far East</td>
<td>26,357</td>
<td>29,592</td>
</tr>
<tr>
<td>Total</td>
<td>111,516</td>
<td>127,290</td>
</tr>
</tbody>
</table>

Table 2. Predicted Demand for Cassava in the EEC for 1980 (1000 fresh metric tons) (Phillips 1973a)

<table>
<thead>
<tr>
<th>Country</th>
<th>Low Estimate</th>
<th>High Estimate</th>
</tr>
</thead>
<tbody>
<tr>
<td>Netherlands</td>
<td>1,632</td>
<td>3,808</td>
</tr>
<tr>
<td>France</td>
<td>251</td>
<td>3,120</td>
</tr>
<tr>
<td>Denmark</td>
<td>893</td>
<td>1,953</td>
</tr>
<tr>
<td>W. Germany</td>
<td>1,083</td>
<td>1,858</td>
</tr>
<tr>
<td>United Kingdom</td>
<td>755</td>
<td>1,515</td>
</tr>
<tr>
<td>Belgium</td>
<td>755</td>
<td>1,160</td>
</tr>
<tr>
<td>Italy</td>
<td>187</td>
<td>923</td>
</tr>
<tr>
<td>Total</td>
<td>5,557</td>
<td>14,347</td>
</tr>
</tbody>
</table>
intake. Maner (1972) included up to 60% cassava in a Swine ration, while Muller, et al, (1972) recommended cassava meal in pig rations when the price of an 85% cassava meal/15% soya meal was significantly less expensive than that of corn. They stressed the importance of pelleting the cassava to reduce bulk and dust problems. In Malagasy, Serres (1969) reported the use of cassava roots as a component of rations for intensive finishing of cattle. Serres and Tillon (1972) also reported on the production of cassava root silage for pigs (fed 3 kg/day) and cattle (fed 5 kg/day).

The most common forms of using cassava in the feed industry are pellets and chips, produced by cutting and drying for chips, followed by grinding and pressing for pellets.

POTATO:

Potato is one of the main sources of carbohydrate in the developed countries. This is consumed baked, cooked, fried, or in chips. It could be dried and made into flour or flakes to be used as "instant" potato.

Fresh potatoes may be stored under controlled environmental conditions for durations up to 10 months. During storage both temperature and relative humidity play major roles in determining the quality of potato chips obtained from a potato stored for a specific time period.

Potatoes are usually stored at low temperatures (between 4°C and 10°C) to diminish significant weight losses, sprouting, and fungal deterioration.
SCOPE AND LIMITATION OF STUDY

At equilibrium the corresponding relative humidity, otherwise known as water activity, has been used as a criterion for microbial growth and to characterize the water available for enzymatic reactions. Several investigators, including Isbuzza, et al, (1970), and Rockland (1969), attempted to divide the relationships between relative humidity and moisture into that contributed by water in its several states. They used this local isotherm to suggest a basis to delimiting the regions of particular spoilage mechanisms. The relationship between moisture sorption isotherms and temperature has provided measures of the heat and free energy of desorption in foods.

The moisture in excess of equilibrium, called the "free" water, is usually taken as the driving force for moisture transfer during processing or storage. The chemical potential or the free energy change would be a more thermodynamically acceptable driving force in products where moisture transfer can take place due to different mechanisms. The knowledge of the equilibrium moisture isotherms provides estimates of this free energy difference. The study of moisture isotherms will be limited to desorption. Sorption will not be considered, hence, hysteresis effect will not be investigated.

Rate constant study will be limited to diffusion coefficient. Some data are available for potato, so the main product under investigation is cassava, with potato used for comparison.
The effect of the temperature gradient within the material will be neglected, as long as the difference is less than 1.5°. Therefore, an isothermal condition within the material will be assumed during the analysis of the drying rate experiments.
II. REVIEW OF LITERATURE

Moisture Content and Sorption Phenomena in Food

Moisture is essentially the vaporizable constituent of a wet material, (Keey, 1972). The role of water in food materials depends very much on the chemical composition and physical structure. These govern the form and distribution of the water. Kuprianoff (1958) presented five possible forms:

(a) Water could be found in a pure form as a surface water; in that case, it is not part of the product, but comes from external sources like condensation or washing.

(b) Water could be chemically bound to some salts, either by its prime valence or as a hydrate. This chemically bound water is not expelled by common methods of food processing.

(c) Water could be adsorbed as a very thin mono- or poly-molecular layer on the internal or external surfaces of the product by molecular forces or in fine pores by capillary condensation--two different mechanisms.

(d) Water could be adsorbed by colloid substances and remain in a gel as water of swelling due to its dipolar character. This water is also called "water of hydration."

(e) Water could be present as a continuous phase, in which other substances may be dispersed molecularly, colloidal, or as an emulsion.
Keey (1972) distinguished five regimes of moisture-retention by analyzing the sharp changes in gradient in a thermogram. He asserted that these changes appeared at transitions in the way that the moisture was held, since the energy of bonding is characteristic of the bond's nature. A thermogram is obtained by plotting the mean difference between the ambient temperature and sample temperature, versus time, during drying in a controlled environment and given boundary conditions.

Determination of Moisture Content:

Moisture content determination will be the most frequent operation during the experimental phase of this research. Therefore the knowledge of the different available methods is important as a means of choosing the most appropriate one. The choice of any depends on the physical and chemical properties of the material, availability of instruments, the level of precision needed and the kinds of water to be detected. Some of the popular methods will be reviewed with their advantages and disadvantages. In general, there are two broad classifications of the methods: Direct and Indirect Methods.

The direct methods include those in which the moisture is measured directly as lost or gained. In the indirect methods, amount of moisture is determined by measuring either the chemical or electrical properties of water.

Direct Methods:

(a) Vacuum Oven: This is the most popular method of determining moisture content for foods (ASTM, 1962, Stitt, 1958).

A moist sample is weighed, dried and weighed again. The decrease in weight is taken to be the weight of water which was
present in the sample. Normally, the drying is done at 105°C for 8 hours; foodstuffs and other thermally sensitive materials are usually dried at 60°C for 30 hours at oven pressure not exceeding 700 Newtons/N². A suitable desiccant is interposed between the vacuum pump and the oven to stop back-diffusion of water-vapor and also to keep water out of the pump. The hot dried sample should be cooled in a desiccator before the final weighing.

(b) **Infra-red Moisture Balance:** This is commonly used for grains.

A sample of about 5 gm. is spread thinly over a pan and then dried under an infra-red lamp. The balance is zeroed against a vertical scale which is calibrated directly in percentage moisture content.

The above methods lend themselves to some sources of errors.

(i) For the infra-red (I-R), the dryness of the sample will depend on the ambient vapor pressure, when drying has ceased. This is because, as the sample dries, its vapor pressure decreases; drying will slow down as the difference between the vapor pressure of the sample and the ambient vapor pressure gets smaller and will until the two pressures are the same.

(ii) For both the I-R and oven, heating may bring about the liberation of adsorbed volatiles besides water. Also, oxidation of the material, if the heating is carried out in air, or hydrolysis of the material may occur.
(iii) Difficulty may be encountered in dislodging all the bound water if it is desired to bring this water into the determination.

(iv) A surface crust impervious to water may be produced by heating, so that some moisture becomes trapped within the sample.

(v) The sample may absorb moisture or gasses from the surrounding atmosphere during heating in the case of the I-R or during any time elapsing between its separation from bulk and initial weighing or between the end of the drying process and the subsequent weighing.

Ways of overcoming some of these difficulties have been suggested by Stitt (1958). These include grinding the material into granules or powder and drying at very low temperature to prevent liberation of adsorbed volatiles.

(c) Distillation: This method entails the oil-distillation of the water, Petzer (1951), Pande (1963), and Brown (1907). The distilled water is collected and measured. The popular apparatus for this is the Brown-Duvel, used mainly for grains. The removal of the moisture from the sample may be more rapid than by drying because the sample is penetrated by and dispersed and agitated within the distillation liquid. The temperature of the sample is only raised to the boiling point of the distillation liquid employed which can be below the boiling point of water. Four liquids commonly used are benzene, toluene, oil and xylene.
There are also some sources of errors. In addition to the moisture which is not removed from the sample, all that is removed, is not measured in the receiver. Diffusion of moisture within the material limits the rate of removal of water at low moisture levels. Thermal degradation is also encountered.

Indirect Methods:

(d) Karl-Fischer Method: It is based upon the reaction of iodine in the presence of water (Fischer, 1935). The method consists of titrating the mixture of pyridine, sulphur dioxide and iodine (the Karl-Fischer reagent) into a solution prepared from the sample by leaching with water-free methanol. The method is ideally suited to determining the moisture content of small samples. The reagent is light-sensitive, so freshly prepared solutions and a nitrogen blanket must be used for best results (BS 2511 and ASTM Standard E203). Some materials interfere with Karl-Fischer reaction; they are oxidants such as chromates, dicromates, cupric and ferric salts.

The time of determination is very short, less than 60 minutes for white potato without heating (Stitt, 1958). A mild, 40-second heat treatment reduced the time to 15 minutes. It is a suitable method for determining the water content of dehydrated foodstuffs.

(e) Infra-Red Absorption: Water absorbs infra-red radiation at certain wave lengths so that the degree of attenuation of infra-red radiation at one of these wave lengths when the radiation is either transmitted by or reflected from a moist material is a measure of the moisture content of the material. Transmission or
reflection measurement may be made directly on a moist solid. Alternatively, the moisture in a sample of a ground solid can be extracted with an extraction liquid on which a transmission measurement is made. Transmission measurements require a constant thickness of material, and reflection measurements require that the surface layers represent the bulk of the specimen. Wave lengths at which infrared absorption by water occurs are 0.76, 0.97, 1.18, 1.45, 1.93, 3.0 and 6.1 μm. Factors affecting the choice of wave length are: the need to avoid a wave length which will be absorbed by constituents of the moist material, other than water, and the desirability of having a conveniently measurable amount of attenuation of incident radiation. The attenuation varies according to the moisture content of the material and will depend also on the wave length selected, and in transmission measurements, on the thickness of material to be penetrated. The most commonly used wave length is 1.93 μm. This method of moisture determination is rapid, contactless and accurate and applicable to foodstuffs having moisture content from 2 to 99% (Gold, 1964).

(f) Nuclear Magnetic Resonance: This is applicable to both static and flowing samples of moist materials (Stitt, 1958).

The sample is placed within or passes through a radio-frequency coil located between the poles of a field magnet. The magnitude of the resonance signal from hydrogen nuclei in the water present in the sample, measured by means of a radio-frequency spectrometer, indicates the moisture content of the material. The
$H_2$ nuclei in liquids in the sample produce a narrow and strong peak signal, whereas, the $H_2$ nuclei in the solid material yields a broad and weak signal.

The presence of other liquids like oils and fats in foodstuffs, makes the determination of water more difficult. Another factor affecting water determination by this method is the increase of signal produced by any substances dissolved in the water present, such as salt and sugar.

(g) Tritiated Water Method: This method has been used for quantitative determination of water flow distribution in materials or places like oil wells where it is difficult to reach (Kuyper, et al. 1962). It is also applied in the determination of moisture content in materials. The principle involves the injection of tritiated water into the material. The dilution of the tritiated water, determined by the tritium count in sample from the material, gives an estimate of the moisture content in the material. The determination of tritium is performed with the aid of a liquid scintillation counter or gas counter depending on the sample weight. The sample weight that can be accommodated in liquid scintillation counting is larger than that used in a gas counter. Tritium is chosen as a tracer because of its being cheap and relatively safe to handle. Also, it can easily be brought into a form nearly chemically identical with that of the material to be traced, e.g., tritiated hydrocarbons to trace oil and tritiated water to trace water. Moreover, tritium, which decays by a low-energy beta-ray emission, has a very favorable half-life (12.3 years).
This means that the decrease in radioactivity is only 5.5% in 1 year; so that even long-term experiments can be run without serious loss in accuracy.

Sorption Phenomena in Food: The concept of isotherm equations dates back to Langmuir (1918) whose equation to describe the polymolecular adsorption is based on the assumptions that adsorption is centered at definite points of attraction on the adsorbing surface and that the adsorption is limited to a single molecular layer. From a balance of the vaporization-condensation rate he obtained the equation:

\[
\frac{V}{V_m} = \frac{ba}{k + ba}
\]  

(1)

Where \( V \) = Volume adsorbed either in cc/gm solid or gm/gm solid

\( V_m \) = monolayer value (same units as \( V \))

\( k = \frac{k_o}{R T_o} \exp \left( \frac{Q_s}{R T_o} \right) \) = constant

\( b = \frac{1}{P_0} \) where \( P_0 \) is vapor pressure of water at \( T_o \)

\( a \) = water activity

\( T_o \) = absolute temperature °K at 0°C

\( Q_s \) = heat of adsorption

\( T \) = temperature °K

\( k_o \) = Boltzmann Constant, \( 1.38 \times 10^{-16} \) erg/°C

\( R \) = gas constant

The above equation gives an isotherm that is hyperbolic in form, rising rapidly at low pressures but becoming parallel to the pressure axis as ba becomes large compared with unity. The curve exhibits a saturation effect at high pressures, the adsorbed volume not increasing further with increase of pressure.
The first theory to meet with considerable success was the multi-
molecular adsorption theory of Brunauer, et al. (1938), now commonly
known as the BET theory. This model assumed that Vander Waal's forces
account for adsorption of gas molecules on the surface of the solid
adsorbent, that multi-layers of sorbate molecules may be present on the
surface, and that the energy of sorption is the same for all molecules
in the first layer. The equation for the BET isotherm:

$$\frac{V}{V_m} = \frac{ca}{(1 - a)} \frac{1}{1 + (c - 1)a}$$ (2)

Where $c = K \exp (QS/R \ T)$$

$K = \text{constant} = 1$

is generally written in the form

$$\frac{a}{(1 - a)V} = \frac{1}{V_m c} + \left[ \frac{a(c - 1)}{V_m c} \right]$$ (3)

for determination of the constants $V_m$ and $c$.

Many sigmoid isotherms are found to be fitted by the BET equation in
the range of relative humidities from 0.05 to 0.5 (Labuza, 1968).

As reviewed by Agrawal, Clary and Nelson (1969), Harkins and Jura
proposed an equation for sorption isotherms, valid only in the regions
in which the adsorbed molecules form a condensed film, and suggested
in the equation:

$$\ln a = B - A/V^2$$ (4)

Where $A$ and $B$ are constants

$V = \text{adsorbed volume}$

A plot of $\ln a$ vs $1/V^2$ should yield a linear curve with $B$ as the $y$-
intercept and $A$ as slope. The equation does not apply above a water
activity of 0.4 to 0.5, where multilayer adsorption and capillary condensation commence (Labuza, 1968).

Smith (1947) from his work with high polymers suggested a model of the form:

\[ W = X - Y \ln(1 - A) \]  \hspace{1cm} (5)

Where \( W \) = moisture content, wet basis

\( X, Y = \) constants; \( A \) is water activity.

This model fits the data on biological material beyond activities of 0.20 within an error of 2-3% as reported by Agrawal, et al. (1969).

None of the above models has been accurate throughout the entire range of water activity at all temperatures.

Henderson (1952) introduced an empirical equation to cover the whole isotherm which relates temperature, relative humidity and weight fraction of water adsorbate in biological materials. The form:

\[ 1 - a = \exp(-KT(M)^n) \]  \hspace{1cm} (6)

can be linearized:

\[ \log(-\ln(1 - a)) = n \log M + \log KT \]  \hspace{1cm} (7)

A log-log plot of \( M \) and \(-\ln(1 - a)\) for actual products often do not result in a single straight line as expected, instead there are two or three straight-line portions. Rockland (1957) has reported some success with this model for food materials. He obtained in most cases, three straight lines for walnut.

Since the Henderson equation was introduced, many modifications or combinations with other models have been made by many investigators to provide greater accuracy.
Day and Nelson (1965), in their work with wheat, expressed the constants in Henderson's equation as functions of temperature. This relationship has been found to be valid for relative humidities of 0 to 0.70.

Agrawal, et al. (1969) provided tables of constants in Henderson's equilibrium moisture content, (EMC), equation for rough rice in the ranges of 10-95% R.H., at 20°C and 30-75% for 43.89°C. They also reviewed the range of applicability of various desorption isotherm models and approximate groupings according to state of water present.

Chung and Pfost (1966b) developed a new isotherm equation by adapting the framework of potential theory of Polanyi (1910). Their equation was of the form:

$$\ln a = \frac{A}{R T} \exp (-B m)$$

(8)

Where A and B are constants and m is moisture content.

These constants were determined for corn, corn products, and wheat (Chung and Pfost, 1969). They found the equation to be applicable throughout the entire range of relative humidity except for corn germ.

Ngoddy and Bakker-Arkema (1970) derived an isotherm equation which defines the specific adsorbed volume due to molecular and capillary phenomena in the tissue of biological materials:

$$V_a = \frac{M_a}{\rho} = \frac{K}{\eta} \left( z^n - \lambda^n \right)$$

(9)

Where \( V_a \) = specific adsorbed volume

\( M_a \) = specific adsorbed mass
\[ z = 3.2 \left( \frac{1.75}{\ln x_1} \right)^4 + \frac{\sigma \bar{V}}{g T \ln x_1} \] (10)

\[ \lambda = 3.2 \left( \frac{1.75}{\ln x_2} \right)^4 + \frac{\sigma \bar{V}}{g T \ln x_2} \] (11)

\[ x_1 = \frac{(\bar{C} + \bar{m}}}{(P + \bar{m})} \] (12)

\[ x_2 = \frac{(P_0 + \bar{m})}{\bar{m}} \] (13)

Where \( \bar{V} \) = molal volume of sorbate in its bulk liquid condition

\( \sigma \) = surface tension of sorbate

\( P_0 \) = saturation pressure

\( P_m \) = vapor pressure corresponding to the adsorbed monolayer

\( R \) = universal gas constant

\( T \) = temperature, °K

This equation is very specific because the parameters \( \xi \) and \( \eta \) are respectively functions of the pore-size distribution parameters, \( k_1 \) and \( \gamma \) as shown below:

\[ \xi = \frac{4}{3} \pi k_1 \] (14)

\[ \eta = 4 + \gamma \] (15)

The parameters are constants obtained by assuming a power function distribution of pore radius of the form:

\[ \phi (R) = k_1 (R) \gamma \] (16)

They indicated that the parameters \( k_1 \) and \( \gamma \) are both product and temperature dependent. The equation was verified by experimental values of Chung and Pfost (1967) for whole corn kernels, and those of Saravavos (1965) for raw freeze dried beef.
The most recent work done in this field of isotherm equations for biological materials was by Roa, et al. (1974). They fitted experimentally-determined isotherms to the following equation by multiple linear regression techniques:

$$M_{eq} = \left[ p_0 + p_1 RH + p_2 RH^2 + p_3 RH^3 + \ldots \right] \exp[q_0 + (q_1 + q_2 RH + q_3 RH^2 + \ldots) T_{abs}]$$

(17)

Where $p_0$, $p_1$, $p_2$, $p_3$, $q_1$, $q_2$ & $q_3$ are constants.

They reported testing the generality of the equation for corn, rice, cassava and coffee and met with reasonable degree of success.

The values of the coefficients tabulated for cassava by the authors are questionable because when they were fitted to the equation resulted in a sinusoidal plot.

**Thermodynamic Functions:** Bull (1944) in his study of sorption isotherms for a number of proteins calculated the free energy. Dole and McLaren (1947) evaluated the free energy and heat of sorption of water vapor by protein and polymers. Others who have determined the heats of sorption of water vapor include Buesk and Winkler (1957) for wheat flour, starch and gluten; by graphical evaluation of Clausius-Clapeyron plots; Becker and Sallans (1956) for wheat. Rodriguez-Arias, et al. (1963) have determined the heats of vaporization for shelled corn. Chung and Pfost (1966a) calculated the isoteric heats (heat at constant mc), and free energy changes of adsorption and desorption for corn, corn starch, corn germ, corn hull and corn gluten at three different temperatures over a relative humidity range of 8.9 to 88.9%. They found that isoteric heats and free energy changes of adsorption and desorption
decreased continually with increasing moisture content, and isothermic
heats and free energy changes of desorption were consistently greater
than those of adsorption.

However, no data of thermodynamic functions for cassava are avail-
able, and only limited data are available for white potato and its
product. Görling (1958) presented a set of isotherms and used them to
estimate the characteristic pore sizes of potato and of the binding
energy of adsorbed moisture.

Fish (1958) obtained desorption data for the thermodynamic partial
quantities for water in potato starch gel.

![Graph showing thermodynamic quantities](image)

**Fig. 1 - Thermodynamic quantities calculated from moisture
sorption isotherm data for potato starch gel at 298°K.**
Fish (1958).

From Figure (1), dry potato starch gel is seen to have a differential
heat of wetting \(\Delta H_w\) of about 270 cal/g or 4.9 kcal/mole. This drops
rapidly as water is absorbed in the region where the isotherm straightens
out (Figure 1). This qualitatively indicates that sorbed water is
bound by energies characteristic of hydrogen bonding in the low-moisture region. The partial molal entropy, $T\Delta S_w$, of water in potato starch gel is also seen to have high negative values at low moisture contents and decreases in magnitude with increasing hydration in a manner similar to the differential heat of wetting. This decrease in entropy indicates an increased degree of orientation and rigidity of water molecules in the sorbed state over that in liquid water.

**Air Drying of Porous Solids:**

**General Concept:** Air drying of solids involves vaporization of water contained by the solid, and removal of the vapor in a stream of air. The heat requirement is large because of the latent heat of vaporization.

Water vaporizes from a very wet solid into the air, the same way as it evaporates from a free water surface.

The phenomenon is one of diffusion and mechanical mixing, the diffusional resistance being mainly in an air "film" just above the liquid surface. (Sherwood, 1936). This diffusional resistance is the sum of the resistance of a surface laminar layer through which moisture is transferred by molecular diffusion and the resistance of an eddy layer in which the transfer is by eddy motion or mixing.

**Constant Rate Period:** As long as the surface of solid remains wet with water, the rate of vaporization is constant, and the factors affecting the rate of drying are similar to those affecting the vaporization of $H_2O$ from a free liquid surface.

Because the rate of vaporizations is constant, this part of the drying process has been called "Constant rate period."
The end of constant rate period is marked by a "critical" moisture content and the rate of drying decreases thereafter. In porous solids, water is fed to the surface during the constant rate period by capillarity, being replaced by air which enters through a few larger openings, (Sherwood, 1936). The three important factors influencing the rate of drying in the constant rate period, for a given size of a product, are, air temperature, humidity, and velocity.

**Air Temperature and Humidity:** The rate of vaporization is proportioned to the pressure difference $P_s - P_a$, where $P_s$ is the vapor pressure of water at the surface temperature and $P_a$ is the partial pressure of $H_2O$ vapor in the air; and is inversely proportional to the average partial pressure of air in the surface film. At ordinary temperatures and humidities, the partial pressure is approximately equal to the total pressure and may be taken as constant. The vapor pressure $P_s$ increases with air temperature for a given relative humidity; $P_a$ increases with humidity at any fixed temperature.

**Air Velocity:** As long as the surface is wet, the rate of drying is greatly influenced by the air velocity over the surface. The effect is analogous to the influence of fluid velocity on the dissipation of heat from a hot surface placed in contact with a fluid stream. It will therefore be expected that the relationship will not be linear, but should follow the power function relation with an exponent somewhat less than unity. At very low imposed air velocities, however, the rate of vaporization is relatively high, because of free convection effects.
Sherwood (1939) represents the relation by this equation:

\[ W = 0.027 v^{0.8} (P_s - P_a) \]

(18)

Where \( W \) = rate of vaporization \( \text{kg/m}^2 \text{hr} \)

\( v \) = Velocity of parallel air stream over wet surface \( \text{m/sec} \)

\( P_s \) = V.P. of \( \text{H}_2\text{O} \) at temp of liquid surface, \( \text{mm H}_g \)

\( P_a \) = Partial pressure of \( \text{H}_2\text{O} \) in the air stream, \( \text{mm H}_g \)

**Hygroscopic Materials:** Most food substances are hygroscopic, that is, they absorb water to an extent depending on the temperature, pressure and humidity of air. Under drying conditions, drying rate falls toward zero as the moisture remaining in the body approaches vapor pressure in equilibrium with the air surrounding it.

In drying of porous hygroscopic solids by the evaporation of the liquid from the surfaces of the solids, using air currents to effect the evaporation, Van Arsdel in Van Arsdel & Copley (1973) has developed theories of moisture transfer showing four possible phases in the drying process:

1. As water evaporates from wet surface, the diameter of the superficial water-filled pores and capillaries diminish, solid structural elements pull closer under the influence of surface tension and the effect spreads into deeper layers of tissue and eventually all the way to the center. Volume shrinkage is equal to the volume of water evaporated and the drying rate per unit surface remains constant.

2. Structural elements of the body begin to deform by crumpling so as to occupy less space as additional water is removed by evaporation at the surface but increasing resistance
to the deformation is encountered. Water vapor also moves to the surface by molecular diffusion through the air in the open capillaries.

(3) The thick water layers which wedge apart long flexible molecular chains in the wet solid start to release the less tightly-held water molecules in form of diffusion to region of lower water concentration.

(4) In the final phase, water molecules adsorbed on the inside surface of the material move by process of activated diffusion in the direction of lower surface spreading potential, equivalent to lower pressure.

Capillary-porous Bodies: Luikov (1966) considered a body to be capillary-porous and the pores to be capillaries, if the capillary potential is significantly greater than the potential of gravity $\Psi_{\text{cap}} \gg \Psi_{f.g}$. In this case, the action of gravity on the liquid in the capillary could be neglected. He added that if the capillary potential was comparable with the potential of the field of gravity then the body was called simply a porous body.

Luikov (1960) likened the pore of a body to a system of cylindrical capillaries, connected among themselves and said that the finer capillaries were fed by liquid from the wider ones, so that the rate of motion of the liquid in every capillary depended on the presence of other capillaries.

Luikov (1966) derived an equation describing the coupled mass transfer of liquid and vapor in capillary porous body. He postulated that vapor moved inside a capillary-porous body by diffusion and it is
in thermo-dynamic equilibrium with the bound liquid. In the hygroscopic zone the partial pressure of the vapor is a function of the moisture content and temperature; therefore, the relative concentration of vapor, \( C_{10} \), in the capillaries of the body also depends on moisture content, \( u \); and temperature, \( T \).

\[
C_{10} = P_{10} \frac{M}{H} = f(u, T) \tag{19}
\]

The flux of the vapor diffusion \( J_1 \) in the capillary-porous body would be equal to:

\[
J_1 = -\varepsilon DC \Delta C_{10} = -Am_1 \gamma_0 [u + \delta_1 \Delta T] \tag{20}
\]

Where \( \varepsilon \) is a dimensionless coefficient, \( 1/\varepsilon \) represents the resistance to diffusion of vapor inside the porous body; \( Am_1 \) is the coefficient of mass transfer, \( \delta \) is the thermal gradient coefficient for transfer of vapor; \( P \) is pressure, \( D \) is diffusivity and \( \gamma_0 \) is density of body.

He further proposed that liquid transfer in a capillary-porous body took place by means of selective diffusion and in the form of capillary absorption under the influence of the capillary potential, which was also a function of moisture content and temperature. The liquid flux was therefore given as:

\[
J_2 = Am_2 \gamma_0 [\nu u + \delta_2 \Delta T] \tag{21}
\]

where \( Am_2 \) is the liquid diffusion coefficient in the body and \( \delta_2 \) is the thermal gradient coefficient of the liquid.

Consequently, the transfers of liquid and vapor was combined and expressed by one mass transfer law:

\[
J_m = J_1 + J_2 = -Am \gamma_0 (\nu u + \delta \Delta T) \tag{22}
\]
where \( A_m = A_{m1} + A_{m2} \)

\[
\delta = \frac{A_{m1}\delta_1 + A_{m2}\delta_2}{A_{m1} + A_{m2}}
\]  \( (23) \)

The Luikov (1966) approach was used by Husain et al. (1972) to analyze the simultaneous mass and heat diffusion in biological materials.

Harmathy (1969) derived a set of partial equations for the simultaneous transfer of moisture and heat in a porous organic material using evaporation-condensation theory. Husain et al. (1972) note that this theory does not apply to food products.

Jackson et al. (1975) calculated total water flux, \( J_{gb} \), by using a coupling-effect relation of both mass and heat diffusion.

\[
J_{gb} \text{ (cm/sec)} = -D_{\Theta l} \nabla \Theta - D_{\Theta v} \nabla \Theta - D_{Tl} \nabla T - D_{Tv} \nabla T - Kk
\]  \( (24) \)

where \( D_{\Theta l} = \text{isothermal liquid diffusivity, cm}^2/\text{sec} \)

\( D_{\Theta v} = \text{isothermal vapor diffusivity, cm}^2/\text{sec} \)

\( \Theta = \text{volumetric moisture content cm}^3/\text{cm}^3 \)

\( D_{Tl} = \text{thermal liquid diffusivity, cm}^2/\text{sec} \)

\( D_{Tv} = \text{thermal vapor diffusivity, cm}^2/\text{sec} \)

\( T = \text{temperature, } ^\circ\text{C} \)

\( K = \text{hydraulic conductivity, cm/sec} \)

\( k = \text{unit vector in vertical direction} \)

They took the temperature dependents of liquid diffusivity, \( D_{\Theta l} \), as the ratio of surface tension to viscosity of water. They found that the hydraulic conductivity, \( K \), was negligible for the water content range (0-.25 cm\(^3\)/cm\(^3\)) considered in their experiment.

For isothermal soil-water flux, \( D_{Tc} \) and \( D_{Tv} \) are zero. They found that liquid diffusion predominated, noting that the term, \( J_{\Theta 1} \), liquid
flux, was the predominant component of the various water fluxes calculated. Their results at 25°C showed that isothermal vapor diffusivity, $D_{sv}$, increased with moisture content, reaching a maximum at $m - c = 0.05 \text{ cm}^3/\text{cm}^3$, and decreased linearly with $m - c$. But, on the other hand, the liquid diffusivity, $D_{sl}$, increased continuously with moisture content in what seemed to be an exponential or logarithmic relation.

Changes in Tissues During Drying of Hygroscopic Materials:

Shrinkage Effect: When drying is going on, and water is removed from animal or vegetable tissue, there is a sort of plastic deformation. This is because these materials exhibit "turgor" property and hence results in some sort of shrinkage. (Van Arsdale & Copely, 1963).

If the unit tensile stress is increased beyond a modest value, the structure yields, in part irreversibly. If the stress is removed, the stretched material does not retract to its original no-load dimension. Volume shrinkage may be accompanied by various kinds of damage such as cracking.

Migration of Soluble Constituents: During the drying of food materials, some of the dissolved substances migrate and change their location, i.e., with the movement of moisture to the surface of the piece. As the solution nears surface, pure water evaporates leaving behind an increasingly concentrated solution in the outer layers. At the same time there is migration of sugar or solutes toward the inner layer where there is more water. Both of these physical activities are going on simultaneously in actual systems but one predominates according to conditions.
Diffusion Process During Drying

Sherwood (1931) noted that in certain slow-drying solids such as soap no constant rate period appeared, and internal liquid diffusion controlled from the start. He therefore described the relation between drying time, \( t \), and average moisture content (dry), \( M \), for an infinite slab by solving the diffusion equation for the boundary conditions of a step change in environmental moisture from initial to equilibrium, and a uniform control moisture ratio.

The solution was

\[
M = \frac{8}{n^2} \frac{\phi}{n^2} \frac{1}{(2n-1)^2} \exp \left( -\frac{\pi^2}{2} \frac{Kt}{(2n-1)^2 L^2} \right)
\]

where

\( K = \) drying constant  \\
\( L = \) half the slab thickness

Hall and Rodriguez-Arias (1958) suggested that moisture moves through cereal grains by the mechanism of diffusion during the following rate period.

Hougen, et al. (1940) postulated that other mechanisms such as capillarity, gravity, external pressure (shrinkage), convection and a sequence of condensation and evaporation may exist. They, therefore, suggested that caution should be exercised when characterizing moisture movement by the diffusion equation.

Crank (1975) defined diffusion as the "process by which matter is transported from one point of a system to another as a result of random molecular motion". We derived the fundamental equation of
diffusion in an isotropic medium from Fick's first law,

$$\frac{\partial C}{\partial t} = \frac{\partial}{\partial x} (D \frac{\partial C}{\partial x}) + \frac{\partial}{\partial y} (D \frac{\partial C}{\partial y}) + \frac{\partial}{\partial z} (D \frac{\partial C}{\partial z})$$  (26)

This is popularly known as Fick's second law.

Many different mathematical equations have been used by researchers to describe moisture movement from a fully exposed object. Lewis (1921) suggested that the rate of drying is proportional to the difference between the moisture in the material, per mass of solids, \( M \), and the equilibrium moisture content \( M_e \).

$$\frac{dM}{dt} = -k (M - M_e)$$  (27)

where \( k \) is a constant.

Page (1949) developed another related equation

$$\frac{dM}{dt} = -kn (M - M_e) t^{n-1}$$  (28)

where \( n \) depends on the relative humidity of the drying air.

**Drying of Agricultural Products**

Henderson and Perry (1955) assumed that the drying rate is also a function of the air velocity, \( v \), and the saturation vapor pressure, \( P_s \), corresponding to the dry bulb temperature of the drying air, and developed an equation which took care of these variables.

$$\frac{dM}{dt} = k v^w P_s^{w-1} (M - M_e)$$  (29)
Equations (28) and (29) are modifications of the basic Lewis equation. These equations are analogous to Newton's law of cooling. This model assumes that all the resistance to moisture flow is concentrated in a layer at the surface of the material. On the other hand, if it is assumed that the resistance to moisture flow is uniformly distributed throughout the interior of the material, then the fundamental diffusion equation would be an appropriate model.

Sherwood (1929, a and b), (1930), (1931) and (1936) and Newman (1931, a and b) in developing theories concerning the movement of moisture in fully exposed porous media used the diffusion equation. They assumed that the diffusion coefficient was constant and that the potential causing the flow was the difference in moisture concentration.

Van Arsdale (1947) suggested that the potential could be either concentration or vapor pressure difference, and both methods of expressing the diffusion equation are valid. The appropriate choice is determined by the experimental procedure. If vapor pressure were the potential, temperature effects would be reflected in the vapor pressure, while if concentration were used, temperature effects would be reflected in the diffusion coefficient.

Wang and Hall (1961) stated that if the temperature distribution within the medium is uniform, the diffusion equation with concentration as the driving force is adequate in describing moisture movement from the medium. But, if the temperature distribution
within the medium is non-uniform, then the simultaneous equations of moisture and heat diffusion will be used to describe moisture movement within the medium.

Chen and Johnson (1967) considered liquid flow by diffusion as representative of drying above the maximum hygroscopic point and derived the equation below

\[
\frac{\partial M}{\partial t} = D \nabla^2 M
\]  \hspace{1cm} (30)

in which \( M \) equals moisture content dry basis and \( D \) is the diffusion coefficient. They considered \( D \) to be independent of \( M \). For moisture contents below the maximum hygroscopic point, Chen and Johnson (1967) theorized that both vapor and liquid flows within the material. The resulting equation was

\[
\frac{\partial M}{\partial t} = D \nabla^2 M + \frac{\partial D}{\partial M} \left( \nabla M \right)^2
\]  \hspace{1cm} (31)

Where \( D \) here is a composite diffusion coefficient for both liquid vapor flows. In this case, \( D \), was considered dependent upon \( M \) and expressed the nonlinearity observed during the latter stages of drying. They later suggested an empirical equation by analogy to chemical kinetics

\[
\frac{\partial M}{\partial t} = -K \left( \bar{M} - M_e \right)^n
\]  \hspace{1cm} (32)

Where \( K \) and \( n \) are empirical constants. The above equation reduces to Equation (27) when \( n \) is equal to 1.
Henderson and Perry (1961), Hustrulid (1959) and Becker and Sailans (1955) described moisture flow in agricultural products using the diffusion equation in spherical coordinates

\[
\frac{\partial M}{\partial t} = \frac{1}{r^2} \left( \frac{3}{3} \left( r^2 D \frac{\partial M}{\partial r} \right) + \frac{1}{\sin^2 \theta} \frac{\partial}{\partial \phi} \left( D \frac{\partial M}{\partial \phi} \right) + \frac{1}{\sin \theta} \frac{\partial}{\partial \theta} \right) (\sin \theta D \frac{\partial M}{\partial \theta}) \right)
\]

(33)

Assuming symmetry with respect to the origin, the equation reduces to

\[
\frac{\partial M}{\partial t} = \frac{1}{x^2} \frac{\partial}{\partial r} \left( r^2 D \frac{\partial M}{\partial r} \right)
\]

(34)

Crank (1975) gave solution to Equation (10) assuming a constant diffusion coefficient, uniform initial concentration, a step change in constant surface concentration, no surface resistance and a homogeneous and isotropic sphere.

\[
\frac{M - M_0}{M_e - M_0} = 1 + 2R \frac{\theta}{n+1} \sum_{n=1}^{\infty} \frac{(-1)^n}{n} \sin \frac{n \pi \theta}{R} e^{-D n^2 \pi^2 t/R^2}
\]

(35)

The average moisture ratio is

\[
\bar{M} - M_e = \frac{6}{\pi^2} \frac{\theta}{n+1} \frac{1}{2} e^{-n^2 \pi^2 \theta t/2R^2}
\]

(35a)

Hustrulid (1959) used the average concentration based on Equation (35a) to describe the drying rate of individual corn kernels.
In a later study, he found that the diffusion coefficient varied with the initial moisture content of the corn kernel and concluded that it must apply at every point within the kernel.

Many other researchers, Sherwood (1929b), Hougen, et al., (1940), King (1945) and Van Arsdel (1947) also noted that the diffusion coefficient is a function of concentration but did not try to obtain a solution of the diffusion equation under these conditions.

Crank (1975) showed that the unidirectional diffusion equation,

$$\frac{\partial C}{\partial t} = \frac{\partial}{\partial x} \left( D(C) \frac{\partial C}{\partial x} \right)$$  \hspace{1cm} (26)

could be transformed into an ordinary differential equation by using the Boltzman transform, $\eta = \frac{x}{4t}$ for infinite and semi-infinite solid. It is necessary, however, that the initial and boundary conditions be expressed in $\eta$ alone.

Crank and Henry (1949) obtained numerical solutions for Equation (26) for a number of cases where the diffusion coefficient varies in particular ways with the concentration. Crank and Henry (1950) also published a method for determining empirically the variation of $D$ with concentration by observing the curve of average moisture ratio as a function of time.

Randy and Johnson (1966) used the method of finite differences to solve the unidirectional diffusion equation on a Pace RT-48 analog computer for a coefficient that was an arbitrary function of both position and concentration.
Whitaker, et al, (1968) applied the above procedure to solve the diffusion equation describing radial moisture diffusion in a sphere whose diffusion coefficient is an arbitrary function of both position and concentration.

**Drying of Cassava and White Potato:**

Cassava pellets and chips are the common forms of using Cassava in the feed industry. (Holleman and Atten, 1956). Chips are produced by mechanical slicing of Cassava followed by natural drying of the product in layers on concrete floors or wooden trays. In good sunshine the drying period is usually 2 to 5 days (Grace, 1971). No critical desorption rates related to the quality deterioration have been reported.

Gill (1972) dried Cassava chips on a tray by forcing artificially heated air through the layer by a fan. The data obtained were discussed with respect to average drying of different bed depths.

Roa et al. (1974) studied the natural drying of Cassava chips and bars and recommended ways of increasing drying efficiency. They recommended vertical layers of rectangular Cassava bars in trays.

Chirife and Cachero (1970) and Chirife (1971) analyzed the forced, high temperature air drying of Cassava layers of different depths. The data were analyzed by using moisture diffusion equations valid only for thin layers of Cassava fully exposed to the air. Chirife and Cachero (1970) deduced that a diffusional mechanism was controlling the drying rate by analyzing the semilog plots of nondimensional
moisture content against time.

Although Chirife (1971) agreed that the diffusion coefficient could be dependent on moisture content, he analyzed his data using constant diffusion coefficients as approximations.

Chirife and Cachero (1970) reported on the effect of air temperature on drying time. They noted that, although the increase in temperature reduced the drying time, tapioca slices were scorched at temperatures of 80 degrees C or higher and humidities of less than 3%.

Therefore optimum working temperature must be chosen not to affect the physico-chemical characteristics of the dried product.

Satavacos and Charm (1962) studied the effect of the air-drying conditions and the size of the material on the drying rate of potatoes. A small increase in drying rate during the constant-rate period was effected by increasing air velocity or decreasing air humidity. But these variables did not affect the falling rate period.

They found that the drying time was proportional to the square of the thickness of the potato slabs. This is evidence for a diffusional mechanism of drying.

Bleaching potatoes before dehydration had no effect on drying rate.

They assumed that the moisture diffusivity was constant with respect to moisture content. They found the "diffusivity" to increase with temperature, following the Arrhenius equation. Fish (1958) also found this behavior.
Gorling (1958) reported sorption isotherms of potatoes at different temperatures.

He also analyzed the drying-rate curve for one-side drying of potato slices; and identified three different stages of drying based on distinct breaks on the drying rate curve.

He determined the liquid conductivity as the function of the moisture content.

Ede (1958) reported some drying data on potato strips. He proposed that there is no constant rate drying by analyzing the drying rate curve.

He also reported the effect of size of strips, wet-bulb depression and air speed on the rate of drying individual strips of potato.
III. RESEARCH OBJECTIVES

Based on the introduction, literature review and time limitations, this research was undertaken to fulfill the following specific objectives:

A. To obtain the moisture isotherms of Cassava and White potato from which the following will be determined:
   1. Isotherm equation which will give the relation of moisture concentration in the product to its partial pressure.
   2. Energy of desorption, which is an estimate of the amount of heat required to remove the moisture from the products.

B. To determine experimentally the variation of the rate constant with respect to concentration and temperature, from moisture profiles.

C. To examine the mechanisms of moisture movement at different stages of drying.
IV. THEORETICAL ANALYSIS

Moisture Isotherms and Different Approaches

Definition, Curves and Their Meaning: The sorption isotherm of a biological material can be described by a plot of the amount of water adsorbed as a function of the relative humidity, or activity of the vapor space surrounding the material. This is the amount of water held in the material after equilibrium has been reached at a constant temperature. The moisture-vapor equilibrium humidity of food materials can be described by a curve shown below.

![Figure 2: Typical Moisture Isotherm](image)

Rockland (1969) has shown that the isotherm can be divided into three regions depending on the state of the water present, and stability of food. The region A corresponds to the adsorption of a monomolecular
film of water; region B to adsorption of additional layers over
the monolayer and microcapillaries, and region C to the condensation
of water in the pores of material followed by dissolution of the
soluble material present (Labuza, 1968).

**Thermodynamics of Sorption:** At equilibrium, the Gibbs chemical
potential (partial Gibbs free energy) for water must have the same
value throughout the system, (Stitt, 1958). This quantity must
then be the same in the solid material as in the vapor phase with which
the solid is in equilibrium. Taking pure liquid water at 760mm Hg
pressure and 15 degrees C, as a standard state, the change in partial
Gibbs function per gm of water for transfer of an infinitesimal quantity
from the pure liquid (vapor pressure $P_0$) to equilibrated solid with
vapor pressure $P$ is

$$\Delta G_w = \frac{RT}{18} \ln \left( \frac{P}{P_0} \right)$$  \hspace{1cm} (36)

The corresponding change per gm of sorbent referred to dry
material as a standard state is found by application of the Gibbs-
Wuhen relation.

$$Wd(\Delta G_w) + (1-w) d(\Delta G_s) = 0$$  \hspace{1cm} (37)

Where $W$ is the weight fraction of water in the equilibrated sample
and subscript $s$ denoted sorbent (Stitt 1958). Hence

$$\Delta G_s = - \int_{P}^{P_0} \left( \frac{W}{1-W} \right) d(\Delta G_w)$$  \hspace{1cm} (38)
The overall change in Gibbs function for the entire process of dry sorbent combining with water to form a gm of equilibrated material with vapor pressure, p, is

$$\Delta G = \Delta G_w + (1-W) \Delta G_s$$  \hspace{1cm} (39)

For an equilibrated sample of fixed moisture content, the dependence of the vapor pressure on temperature is related to the differential heat of wetting by

$$\frac{\partial \ln \left(\frac{p}{p_0}\right)}{\partial T} \bigg|_w = \frac{18 \Delta H_w}{R \pi^2}$$  \hspace{1cm} (40)

Where $\Delta H_w$ is the change in partial heat function per gm of water for sorption of an infinitesimal quantity from the pure liquid to equilibrated material of water content W.

It is the difference between the heats of vaporization of water from the sorption sample and from pure water.

In the same way as analyzed above, $\Delta H_s$ and $\Delta H$ may be calculated for the corresponding Gibbs free energy quantities. The entropy functions are given by

$$T \Delta S = \Delta H - \Delta G$$  \hspace{1cm} (41)

For each component for the corresponding equation for the overall process.
The above thermodynamic relations assume that the state of the system is defined by the temperature and the composition.

**Analysis of Isotherm Curves:** Four essential types of approaches could be said to be available in the theoretical analysis of isotherms (Labuza, 1969) they include:

1. The "equilibrium" approach or thermanalysis
2. Kinetic approach
3. Potential approach
4. Capillary condensation approach

(a) **Kinetic Approach:** The classical kinetic approach to isotherms as proposed by Langmuir (1918) describes the adsorption of a monolayer of vapor on the surface of the solid material. Labuza (1969) noted that this model does not hold for most food materials because of the following reasons:

   i) Interaction between absorbed molecules probably occurs, whereas the model assumes no interaction.

   ii) \( Q \), the heat of adsorption, is not constant in food materials, as the model assumes. The surface is composed of many differing sites with different attractions for water vapor.

   iii) The maximum adsorption possible is much larger than just a monolayer as the model assumes.
The BET isotherm after the work of Brunauer et. al. (1938), is the most popular in all fields. This model makes the following basic assumptions:

i) $Q_1$, the heat of sorption for first layer, is constant and equal to the total $\Delta H_v$ (heat of vaporization) plus a constant heat due to site interaction $Q_s$.

ii) $Q_1$ for all layers above a monolayer is equal to $\Delta H_v$ the heat vaporization.

iii) Sorption occurs only on specific sites.

The general equation for the BET isotherm derived kinetically (Adamson, 1963) is given as follows

$$\frac{V}{V_m} = \frac{C}{(1-a)(1 + (C-1)a)} a$$

(42)

Where $C = k \exp \left( \frac{Q_s}{RT} \right); \ K > 1$

This can be rearranged into

$$\frac{a}{(1-a)V} = \frac{1}{V_mC} + \frac{a(C-1)}{V_mC}$$

(43)

Where $V =$ volume adsorbed gm/gm solid

$V_m =$ monolayer value

$a =$ activity

A plot of $a/(1-a)V$ vs $a$ gives a straight line (Fig. 3). From the slope and intercept of this line the monolayer coverage value can
be calculated. The intercept is \( \frac{1}{CV_m} \), and slope = \( \frac{C-1}{CV_m} \).

The BET isotherm usually holds only between activities from .1 to .5. This model also helps to measure the water surface area, assuming the area of water molecule to be 10.6 Å\(^2\). The surface area \( S_0 \) in \( \text{meter}^2/\text{gm} \) of solid is given by

\[
S_0 = v_m \cdot \frac{1}{H_{\text{H}_2\text{O}}} \cdot N_0 \cdot \lambda_{\text{H}_2\text{O}} = 3.5 \times 10^3 \text{Vm} \tag{44}
\]

![Graph](image)

**Fig. (3)**

**BET model**

Halsey (1948) modified the BET theory by employing the concept of cooperative adsorption. This implies that adsorbate molecules influence each other by horizontal interaction during the adsorption process. This modification helped to derive an equation for predicting the thickness of adsorbed multilayer. In the specialized form, the Halsey equation is given as

\[
t(A^0) = 3.2 \left[ \frac{1.75}{\ln \left( \frac{P_0}{P} \right)} \right] \tag{45}
\]
Where \( t = \text{multilayer thickness} \)

\[
\frac{P_0}{P} = \frac{1}{a}
\]

(b) **Potential Approach:** The two most important models are by Polanyi (1928) and Harkins-Jura (1944) and are based on the force field caused by the surface of the solid material.

Polanyi (1928) proposed that the total work necessary to adsorb a molecule is equal to the work to overcome the field strength in bringing a molecule from a distance \( X \) to just above the surface and the work of condensation.

The usefulness of this model is that once one isotherm has been determined, similar curves can be predicted because a plot of \( V \), the amount adsorbed, vs. \( (\ln a)^{-1/3} \) yields a straight line at constant temperature. The major disadvantage is that it can not be used to predict the monolayer value which is of prime importance to the food field.

The Harkins-Jura (1944) model considers the surface force distribution to act by causing the adsorbed film to behave as a liquid in a two-dimensional state, according to the following equation

\[
\ln a = B - \frac{A}{V^2}
\]  (46)

Where \( A \) and \( B \) are constants.

A semilog plot of a VS \( \frac{1}{V^2} \) should be a straight line with slope equal to \( A \).
\[ A = \frac{a_1 S_0^2 V_0^2}{2RT} \quad (47) \]

Where \( a_1 \) = gas compressibility
\( S_0 \) = surface area \( \text{m}^2/\text{g} \)
\( V_0 \) = molar volume of film in \( \text{cc/mole} \)

A plot by Labuza (1968) for Salmon indicated nonconformity and hence he concluded that the model was not adequate for food materials. Also the equation does not hold above water activity of .4 where multi-layer adsorption and capillary condensation take place.

(c) Capillary Condensation Approach: The capillary condensation mechanism as originally developed by Kelvin applies to equilibrium vapor pressure in a liquid in a cylindrical capillary which is reduced below its saturation value by negative hydrostatic pressure arising from tensile components of curved surfaces of tension of the liquid.

The reduction in pressure is related to the radius of curvature of the meniscus by the Kelvin equation

\[ a = \frac{\pi}{4P_0} = \exp \left( \frac{-2\sigma \cos \theta V_0}{\gamma RT} \right) \quad (48) \]

Where \( \sigma \) = liquid surface tension \( \text{dynes/cm} \)
\( \theta \) = contact angle
\( V_0 \) = molar volume
\( \gamma \) = radius of capillary
Zsigmondy (1911) applied the above equation to the relationship between adsorption and pore size in microporous adsorbents. The principal assumption was that the adsorbate exists as a condensate liquid in the capillaries of the sorbent and has liquid phase. This approach is applied to the high moisture region of the isotherm.

**Henderson Approach**

Henderson (1952) derived an empirical equation to cover the whole isotherm.

\[ 1 - a = \exp \left( CV^n \right) \]  \hspace{1cm} (49)

Where \( C = \) constant  
\( n = \) constant  
\( V = \) amount adsorbed

A plot of \( \ln \left( \frac{-\ln (-a)}{1-a} \right) \) vs amount adsorbed should give a straight line. But in some cases, two or three straight line portions result as shown below.

**Fig. 4**

Henderson Isotherm  
Labuza (1968)
Labuza (1968) suggest that these portions may be related to the state of water. The breaks were found by Rockland (1969) to correspond to those indicative of the three types of bound water from studies using nuclear magnetic resonance (NMR) and electron spin resonance (ESR) signal.

**Heat of Sorption**

If isotherms are made at several temperatures, they show a decrease in the amount adsorbed with an increase in temperature at constant relative humidity. When the activities are plotted vs the reciprocal of absolute temperature (at constant m.c.) a straight line is gotten. The slope of this line is $-Q_s/R$. From here the heat of sorption $Q_s$ can be calculated.

![Diagram showing the method of determination of $Q_s$.](image)

Fig. 5. Method of Determination of $Q_s$.

This is the application of the Clausius-Clapeyron equation

$$\log P = -\frac{\Delta H_v}{RT}$$

(50)
Pore Size Distribution Function:

In developing a distribution function for these products, the power law type function (Ngoody 1971 III) is used. This was tested by Ngoody et al. (1971 III) for some food products and found to be valid. The number of pores is obtained by dividing the total average volume of pores in a given range by the average volume of one pore in that range. The total average volume of pores in the range of \( \bar{R} \) is given by

\[
V_{12} = r_{12} (V_{12} - K_{12}) \sum_{R_2}^{R_{\text{max}}} \left( \frac{R_{\text{max}} - t_{12}}{2R_2} \right) R_{\text{max}}^2 \left( V_{R} \cdot A_{R} \right)
\]  

(51)

where

\[
r_{12} = \frac{1}{\xi_1 \eta} \frac{1}{(\bar{R} - t_1)/\bar{R}}
\]  

(52)

\[
K_{12} = 2\xi_2 (t_2 - t_1)
\]  

(53)

\[
\Delta R = R_2 - R_1
\]  

(54)

where \( \xi_1, \xi_2 \) and \( \eta \) are shape parameters.

The number of pores is given by \( N(R) = \frac{1}{V_{12}} 4 \frac{\pi}{3} R^3 \)  

(55)

Here a spheroidal pore shape was assumed. The distribution function

\[
\Psi(R) = \frac{dN(R)}{dR}
\]  

(56)

To verify the assumption of the power law, a log-log plot of \( \Psi(R) \) and \( R \) is made. If the law is valid for the products, a straight line will be obtained, resulting in the following relation.

\[
\Psi(R) = K(R)^\gamma
\]  

(57)
Where \( \gamma \) is the slope and \( \log K \) is the \( \gamma \) intercept.

**Diffusion and Moisture Transfer**

**Definitions and Application:** Houghen et al. (1939) defined diffusion as the distribution of molecules within a single phase brought about by molecular motion of translation and mutual bombardment. This they distinguished from another similar type of flow - capillarity.

Capillarity they said, referred to the flow of a liquid through the interstices and over the surface of a solid due to molecular attraction between the liquid and the solid. The force which acts between the molecules of the solid and the liquid produces wetting and is called adhesion tension.

Besides diffusion and capillarity, water is also distributed or moved in a solid during drying by the action of gravity, by external pressure, by convection and by a sequence of vaporizations and condensations due to temperature differences.

The force of gravity tends to pull water to the bottom of the solid. This distribution is effective where the interstices are large and interconnecting. Gravity acts only vertically whereas diffusion acts in the direction of decreasing concentration, and capillarity in the direction of decreasing capillarity.

Temperature differences within a solid effect a change in the rate of flow of a liquid by diffusion, capillarity or gravity, but in addition may cause movement by convection. Temperature differences also cause vapor pressure gradients which result in evaporation of the liquid and its subsequent condensation on a colder surface.
Hougen et al. (1939) suggested that in drying, the term diffusion should be restricted to the molecular movement of vapors where convection is negligible and to the motion of water molecules in homogeneous solids. They noted that there were cases in drying where diffusion takes place but the supporting experimental evidence available is extremely meagre.

The relationship between diffusivity and moisture content in wet solid is complex. Luikov 1950, (in Keey 1972) suggests that the relationship may be explained by the way in which moisture is bound to the solid skeleton. He postulated four regions as illustrated below.

![Graph](image)

**Moisture content \( k_g/k_g \)**

*Fig. 6. Variation of Moisture diffusivity with Moisture content*

(After Luikov, 1950) FROM Keey 1972
Region I AB. Here moisture exerts its full vapor pressure and the migration of moisture is primarily by capillary movement and is independent of the moisture content. This can be termed the constant rate period, and the moisture is unbound.

Region II BC. The moisture is held in the microcapillaries and the migration of moisture becomes a little bit more difficult than in Region I. Here \(0.8 < X < 1.0\) where \(X\) is relative humidity.

Region III CD. The moisture is adsorbed in the multimolecular layers; moisture movement is in the liquid phase and there is a corresponding increase in diffusivity with decrease in M. C. \((0.2 < X < 0.8)\).

Region IV DE. Monomolecular adsorption takes place and moisture moves by vapor phase diffusion \((0 < X < 0.2)\).

The explanation for these behaviors at different stages has been suggested by the results of different authors.

Some of the results contradict Luiykov's postulate in some aspects.

Krischer and Mahler (1959) found that roofing tiles \((\rho = 1880\) \(k_g/m^3)\) and foamed concrete \((\rho = 650\) \(k_g/m^3)\) showed the relationship AB CD. Schauss (1940) also found that pine wood followed the general shape B C D E but these data give a min. DE at a moisture content of 1.0 \(k_g/k_g\) and diffusivity plateau about a moisture content of 0.3 \(k_g/k_g\) db.

DeVries (1958) had confirmed Luiykov's relationship with liquid movement through a sand medium. He suggested that the vapor-phase diffusivity passes through a maximum at low moisture content, eventually becoming the larger conductance, because the total conductance then varies in a way that is superficially similar to the curve B C D E.
That this relationship applies to a complex cellular-capillary materials such as cassava or potato is yet to be proved or disproved.

Stamm (1946) described the movement of moisture through softwoods by considering a simplified model of the complex combinations of series and parallel paths. Besides the continuous path through the cell walls, there are more tenuous passages through the fibre cavities, pit membranes and their communicating cell-wall capillaries as illustrated in Figure 7.

Fig. 7. Longitudinal moisture movement through wood fibre

(Simplified) (From Keey, 1972)
Diffusion Equation and Methods of Solution: Various cases of one-dimensional diffusion in a medium bounded by two parallel planes, e.g. the planes at $X = 0$, $S = L$, will be considered because this will be the situation that will be encountered in this research. These will also apply in practice to diffusion into a plane sheet of material so thin that effectively all the diffusing substance enters through the plane faces and a negligible amount through the edges.

The basic differential diffusion equation for non-steady state is,

$$\frac{d c}{d t} = D \frac{d^2 c}{d x^2}$$

(58)

for the case where the diffusion coefficient is constant. But if the diffusion coefficient is a function of concentration, then equation is

$$\frac{d c}{d t} = \left( \frac{d}{d x} \right) \left( D \frac{d c}{d x} \right)$$

(59)

Clank (1975), Barrer (1951), Jost (1952) and Caslaw and Jaeger (1959) have solved the constant diffusivity equation under different boundary conditions. Some of the BC's pertinent to drying and their solutions are presented here.

(a) Surface Concentrations Constant Initial Distribution $f(x)$: If

- $C = C_1$  \quad $X = 0$  \quad $t \geq 0$
- $C = C_2$  \quad $X = L$  \quad $t \geq 0$
- $C = f(x)$  \quad $0 < X < L$  \quad $t = 0$

The Fourier Series solution in the form of a trigonometrical series is
\[ C - C_1 = (C_2 - C_1) \frac{X}{L} + \frac{2}{\pi} \sum_{n=1}^{\infty} \frac{C_2 \cos \frac{n\pi}{L} - C_1 \sin \frac{n\pi x}{L}}{L^n} \exp \left( -D \frac{n^2 \pi^2}{L^2} t \right) \]

\[ \int_0^L f(x') \sin \frac{n\pi x}{L} \, dx' \]

Crank (1975) noted that in most cases \( f(x) \) is either zero or constant so that the integral is readily evaluated.

(b) Uniform Initial Distribution-Surface Concentrations Constant:

This is the case encountered in sorption and desorption by a membrane.

If the region \(-L < X < L\) is initially at a uniform concentration \(C_o\), and the surfaces are kept at a constant concentration \(C_1\), then the Fourier series solution becomes

\[ \frac{C - C_o}{C_1 - C_o} = 1 - \frac{4}{\pi} \sum_{n=0}^{\infty} \frac{(-1)^n}{n+1} \exp \left[ -D \frac{(2n+1)^2 \pi^2 t}{4L^2} \right] \]

\[ \cos \frac{(2n+1) \pi x}{2L} \]

If \(M_t\) denotes the total amount of diffusing substance which has entered the sheet at time \(t\), and \(M_\infty\) the corresponding quantity after infinite time, then:

\[ \frac{M_t}{M_\infty} = 1 - \frac{8}{\pi} \sum_{n=0}^{\infty} \frac{1}{(2n+1)^2 \pi^2} \exp \left[ -D \frac{(2n+1)^2 \pi^2 t}{4L^2} \right] \]

The corresponding solution useful for small time is:
\[
\frac{M_t}{M_\infty} = 2 \left( \frac{D_t}{L^2} \right)^{1/2} \left[ \sum \frac{1}{2} + 2 \sum_{n=1}^{\infty} (-1)^n \text{ie fo} \frac{nL}{V(Dt)} \right] (63)
\]

Crank (1975) did not mention the range or magnitude of time equation (63) is applicable. The time may be relative depending on percentage of moisture lost.

Equation (63) can be solved graphically for \( D_t/L^2 \) and hence \( D \) obtained from the measured sorption or desorption curves, by plotting \( M_t/M_\infty \) against \( t/L^2 \).

(c) Uniform Initial Distribution, Surface Concentrations Different:

This is the case of flow through a membrane. The example, if one face \( X = 0 \) of a membrane is kept at a constant concentration \( C_1 \) and the other \( X = L \) at \( C_2 \), and the membrane is initially at a uniform concentration \( C_0 \), there is a finite interval of time during which the steady-state condition is set up. During this time the concentration changes according to

\[
C - C_1 = (C_2 - C_1) \frac{X}{L} + \frac{2}{\vartheta} \sum_{n=1}^{\infty} \frac{C_2 \cos n\vartheta - C_1}{n} \sin \frac{fnx}{L}
\]

\[
\exp \left(-Dn^2\frac{\vartheta^2t}{L^2}\right) + \frac{4}{\vartheta^2} \sum_{m=0}^{\infty} \frac{1}{2m+1} \sin \frac{(2m+1)n\vartheta x}{L}
\]

\[
\exp \left[-D (2m+1)^2 \frac{\vartheta^2t}{L^2} \right]
\]

(64)

For moisture ratio

\[
\frac{M_t}{M_\infty} = 1 - \frac{8}{\vartheta^2} \sum_{n=0}^{\infty} \frac{1}{(2n+1)^2} \exp \left[-D (2n+1)^2 \frac{\vartheta^2t}{L^2} \right] (65)
\]
(d) Concentration-Dependent Diffusion: In cases, such as the diffusion of vapors in high-polymer substances and food materials, the concentration dependence is a very marked, characteristic feature.

Crank (1975) notes that some of the methods used to obtain numerical solutions, are applicable to any type of concentration-dependent diffusion coefficient, and others restricted to particular types.

The solution of equation (66) for one-dimensional diffusion when $D$ is a function of concentration $C$ is obtained by applying Boltzmann transformation (Crank, 1975)

$$\frac{\partial C}{\partial t} = -\frac{\partial}{\partial x} \left( D \frac{\partial C}{\partial x} \right) \quad (66)$$

Introducing a new variable $\eta$, where

$$\eta = \frac{1}{2} \frac{x}{t^2} \quad (67)$$

Thus we have

$$\frac{\partial C}{\partial x} = \frac{1}{2t^2} \frac{dc}{d\eta} \quad (68)$$

And

$$\frac{\partial C}{\partial t} = -\frac{x}{4t^3} \frac{dc}{d\eta} \quad (69)$$

And hence
\[
\frac{\partial}{\partial x} (D \frac{\partial C}{\partial x}) = \frac{\partial}{\partial x} \left( \frac{D}{2t^{1/2}} \frac{dC}{d\eta} \right) = \frac{1}{4t} \frac{d}{d\eta} \left( D \frac{dC}{d\eta} \right) \quad (70)
\]

Therefore

\[-2 \frac{d}{d\eta} \left( D \frac{dC}{d\eta} \right) = \frac{d}{d\eta} \left( D \frac{dC}{d\eta} \right) \quad (71)\]

Equation (66) has now been reduced to an ordinary differential equation in \( C \) and \( \eta \) (71).

The Boltzmann's transformation (eq. 67) can be used provided the transformed boundary conditions are independent of \( x \) and \( t \) such as diffusion in an infinite or semi-infinite medium with a uniform initial concentration.

For an infinite medium, the boundary conditions

\[
C = C_1 \quad x < 0 \quad t = 0
\]
\[
C = C_2 \quad x > 0 \quad t = 0
\]

are transformed to

\[
C = C_1 \quad \eta = -\infty
\]
\[
C = C_2 \quad \eta = +\infty
\]

Also for a semi-infinite medium, the boundary conditions

\[
C = C_0 \quad x = 0 \quad t > 0
\]
\[
C = C_1 \quad x > 0 \quad t = 0
\]

are transformed to

\[
C = C_0 \quad \eta = 0
\]
\[
C = C_1 \quad \eta = \infty
\]
Crank and Park (1949, a, b.) gave a method of solving the transformed equation subject to semi-infinite medium based on iterative quadrature, which is illustrated below.

Equation (71) can be written as

\[
\frac{-2\eta}{D} \frac{d}{d\eta} \left( \frac{d}{d\eta} \right) = \frac{d}{\eta} \left( \frac{d}{d\eta} \right)
\]

(72)

Where \( c = C/C_0 \) and \( c = 1 \) on \( \eta = 0, c = 0, \eta = \infty \).

Integrating equation (72) twice we obtain

\[
c = 1 - A \int_0^\eta \exp \left( -\int_0^\eta \frac{2\eta}{D} \frac{d}{d\eta} \right) \frac{1}{d\eta} \frac{d}{d\eta} \frac{1}{d\eta}
\]

(73)

Where \( A \) is a constant of integration to be chosen such that

\[
A \int_0^\eta \frac{1}{D} \exp \left( -\int_0^\eta \frac{2\eta}{D} \frac{1}{d\eta} \right) \frac{1}{d\eta} \frac{1}{d\eta} \frac{1}{d\eta} = 1
\]

(74)

The diffusion coefficient \( D \) is a known function of \( c \), eg, \( D = \exp (kc) \). If a first approximation to the function \( C (\eta) \) is known, the function \( D (\eta) \) can be deduced using \( D = f (c) \) and carry out the integrations in (73) numerically using (74) to obtain a second approximation, and so on.

Measurement of Diffusivity: Several methods of determining moisture diffusivity that is independent of concentration are available (Crank, 1975; Crank and Park, 1978; Barrer, 1951, Jost, 1952 and Luikov, 1966). In some of these methods, the difficulties lie in the experimental techniques rather than in the subsequent mathematics. Both steady-state and transient methods are used, sometimes in combination. Various methods based on the analysis of concentration-distance
curves are reviewed by Crank (1975) and Barrer (1951). These methods are basically for constant diffusivity.

Another method for determining constant diffusivity is to observe the over-all rate of uptake or loss of diffusing substance by a specimen of known size and shape, and to compare this with the calculated rate of uptake expressed as a function of $Dt/L^2$. In all these measurements it is usually assumed that the total volume of the system remains constant as diffusion proceeds. This method was used by Chirife (1971) to determine $D$ for cassava chips.

Crank and Park, (1968) reviewed the more useful experimental techniques for measuring diffusion coefficients and their concentration dependence. Two of these techniques will be analyzed. They are the sorption method and the concentration-distance curve analysis method.

(a) Sorption Method: When a plane sheet of known thickness $2L$ is hung in an atmosphere of known vapor pressure ratio and temperature, there is either increase or decrease in weight. If the increase in weight of the sheet for a known time, $t$, is observed, the appropriate solution of the diffusion equation may be written as in equation (75).

$$\frac{M_t}{M_\infty} = 1 - \sum_{n=0}^{\infty} \frac{1}{(2n+1)^2} \exp\left[-D\left(\frac{2n+1}{2}\right)^2 \frac{t}{L^2}\right]$$

(75)

for a constant $D$ and no swelling. This solution assumes that immediately after the sheet is placed in the vapor, the concentration at each surface attains a value corresponding to the equilibrium uptake for the vapor pressure existing and remains constant afterwards. The value of $t/L^2$ for which $M_t/M_\infty = 1/2$ conveniently written $(t/L^2)_{1/2}$ is given
by

\[
\left( \frac{t}{L^2} \right)_{1/2} = -\frac{1}{\gamma D} \ln \left\{ \left[ \frac{\gamma^2}{16} - \frac{1}{9} \left( \frac{\gamma^2}{16} \right)^9 \right] \right\}
\]

(76)

approximately, the error being about 0.001%.

Thus we have

\[ D = \frac{0.049}{(t/L^2)_{1/2}} \]  

(77)

If the half-time of a sorption process is observed experimentally for a system in which the diffusion coefficient is constant, the value of \( D \) can be determined from (77). If this is done at the same temperature for different humidities, the values of \( D \) at the corresponding \( M \) could be determined and the relation plotted.

Crank and Henry (1949a) have shown that for any one experiment, \( D \), which is an average value of \( D \) over the range of concentration, provides a reasonable approximation to

\[ \bar{D} = \frac{1}{C_0} \int_0^{C_0} D dC \]  

(78)

Therefore a graph showing \( \bar{D} C_0 \) as a function of \( C_0 \) can be drawn and numerical or graphical differentiation of the curve with respect to \( C_0 \) gives an approximate relationship between \( D \) and \( C \).

(b) Concentration-distance Curve Analysis Method: If two semi-infinite media are brought together at \( t = 0 \), or a semi-infinite media is desorbed from one face, the diffusion coefficient and its concentration-dependence can easily be deduced from concentration distribution observed at some known subsequent time. For a semi-infinite media,
the conditions of the experiment are

\[
\begin{align*}
C &= C_e & x &= 0 & t > 0 \\
C &= C_0 & x &= 0 & t = 0
\end{align*}
\]

Assuming there is no overall change of volume, we may use the Boltzman variable \( \eta = x/2t^{1/2} \) and obtain the ordinary differential equation

\[
-2\eta \frac{dc}{d\eta} = \frac{d}{dn} \left( \frac{dc}{d\eta} \right)
\]

(79)

On integration with respect to \( \eta \) we have

\[
-2\int_{C_i}^{C_0} \eta dc = \left[ \frac{d}{dn} \frac{dc}{d\eta} \right]_{C=C_0}^{C=C_i} = \left( \frac{dc}{d\eta} \right)_{C=C_i}
\]

(80)

Since \( \frac{dc}{d\eta} = 0 \) when \( C = 0 \). Finally, by rearrangement and introducing \( x \) and \( t \), we have

\[
\frac{d}{dc} = \frac{1}{2t} \frac{dx}{dc} \int_{C_i}^{C_0} x dc
\]

(81)

The choice of any of the two methods depends on the availability of experimental set up. Some moisture might be lost in trying to slice the product for moisture analysis in the case of the second method.

The second method, however, will be used in this investigation to determine the moisture diffusivity because of the ease in controlling the experimental conditions. Also it does not lend itself to average values.
The diffusivity, $D$ at various moisture contents $C_1$ is evaluated from equation (22). The integrals are obtained by using a planimeter or counting squares and the gradients $\frac{dx}{dc}$ obtained by drawing tangents lends itself to personal errors, the gradients will be found by numerical method after obtaining the integrals or areas as follows:

Let $C = f(x)$

Then

$$A = x \, dc = f(x) \, dx$$  \hspace{1cm} (82a)

And

$$\frac{dA}{dx} = \frac{df(x) \, dx}{dx} = df(x)$$  \hspace{1cm} (82b)

Also

$$\frac{d^2A}{dx^2} = \frac{df(x)}{dx}$$  \hspace{1cm} (82c)
Therefore

\[ \frac{dx}{dc} = \frac{dx}{df(x)} = \frac{dx^2}{d^2 A} \] (82)

In Figure 8, the area representing \( \int_{c_i}^{c_f} x \, dc \) is shown shaded.
MATHMATICAL MODELING OF MOISTURE TRANSFER IN A SLAB

If moisture diffuses as a vapor or liquid, the diffusion equation is represented by (18) using moisture content

$$\frac{\partial m}{\partial t} = \frac{\partial}{\partial x} (D(m) \frac{\partial m}{\partial x})$$

where $Dm = f(m)$

The boundary conditions are

\[
\begin{align*}
  m &= m_e & x &= 0 & t > 0 \\
  m &= m_0 & x > 0 & t = 0 \\
  m &= m_0 & x = L & t > 0
\end{align*}
\]

The model assumes an isothermal condition within the product. The product initial moisture concentration is assumed uniform and also the moisture content at the bottom is equal to the initial moisture content at all times. It was further assumed that there is no film layer just above the surface of the product to impede moisture flow from the product surface, so internal resistance is controlling.

Because the above model cannot be solved by any classical method, an IBM 360 digital computer which utilized the statement oriented 360 CSMP (Continuous System Modeling Program) was used. This system with its limitation has only one independent variable, time, and cannot solve problems with two independent variables like this model.

The space coordinate was eliminated as a variable (Hamdy and Barre, 1969) by dividing the slab into 10 equal slices and assuming the moisture diffusivity in each slice is constant.
The sets of equations used are similar to those of Agrawal et al (1975) for bologna slab, but the diffusivity $D$ was made a function of moisture content.

![Diagram](image)

**Fig. 9.** Illustrative nodes in the 10 slices of the sample.

It is important to note that the nodes start from the bottom to the top for the purpose of this computer analysis. This is to conform with the direction of moisture movement and eliminate sign problems. Also, non-dimensional moisture, $C$, and thickness, $\bar{d}$, are used.

$C = \frac{M - M_e}{M_0 - M_e}$ and $\bar{d} = \frac{x}{L}$

Sample equations are:

$$C_1 = \frac{100}{2} \int_0^T Dm_2 (C_i - C_i^1) \, dt$$

$$C_1^{(2-9)} = \frac{100}{2} \int_0^T Dm_i \left( C_{i+1} - 2C_i + C_{i-1} \right) \, dt$$

(34)
\[ C_{10} = \frac{400}{31^2} \int_0^T f_0 \quad D_{m10} \left(-3c_{10} + c_9\right) \, dt. \]

where

\[ D_m = f(C). \]  

(85)
V. EXPERIMENTAL

Instrumentation:

Three different sets of experiments were carried out. These were, the equilibrium moisture content, the moisture diffusivity and the drying rate experiments.

a. Equilibrium Moisture: In general there are two techniques for obtaining water vapor isotherms for food products. In one the material is placed in desiccators containing salt solutions which give a temperature-dependent equilibrium relative humidity, Hall (1957). The second method as described by Taylor (1961) involves the measurement, by a sensitive manometric system, of the vapor pressure of water in equilibrium with a food at a given moisture content.

These two approaches were employed in this research but a more sophisticated equipment was used for the second. The apparatus included constant relative humidity desiccators, (Wink, 1946) used for the low and low-intermediate moisture ranges, agitated constant relative humidity desiccators (dynamic) (Bosin and Easthouse, 1970) for the intermediate and low-high ranges, and the Beckman hygrometer probe for the high moisture ranges.

The apparatus for the agitated method consisted of three main parts -- the magnetic stirrer unit, the weighing balance and the equilibration chamber, Fig. 10. The magnetic stirrer complex was of the Lab-line Instruments, Inc., six unit, "Multi-Magnestir", Catalog No. 1278A. The rotating speed of the magnets was variable.
Figure 10. Sketch of Apparatus for Agitated Desiccator
Fig. 11. Temperature Control Setup in Agitated Desiccators.

Fig. 12. Beckman Hygrometer Sensor (From Beckman Instrument)
Bosin and Easthouse (1970) recommended a top loading balance that had an under-pan capability, to enable insitu weighing. A Volant 220D model balance, of the knife edge suspension type, with two weighing pans and chain-slider mechanism for milligram measurements was used. It could measure up to .0001 gm. This balance was modified for an under-pan capability by attaching a V-bar on the left pan, (Fig. 10). A nylon thread with a hook attached hung from the bar. With this arrangement, it was possible to weigh the sample intermittently without disturbing the setup. A sensitivity test was performed and the balance calibrated.

The equilibrium chambers, (six in all) were made of wide-mouth "Kerr" bottles, Fig. 10. In each chamber stood a stainless steel tripod which supported a magnetically driven aluminum fan. The shaft to which the fan and the teflon-coated spinbar magnet were attached passes through a teflon bushing.

The sample dish, made from 6.35 cm diameter copper tubing and copper mesh screen is suspended above the fan.

The equilibration chambers were placed in the center section of a 3-sectional water bath. The walls dividing the section are perforated with 1.3 cm holes. The bath was kept at a uniform constant temperature by means of an immersion unit, Thermomix II Model. The Thermomix consisted of a thermostatic contact thermometer with a range of 0-110°C, tubular heater, a stirrer which rotates at about 1800 rpm and a pump which can deliver water at a maximum of 12 liters per minute. There was a reference thermometer. The Thermomix was immersed in the left section and a tube connected to the outlet of the pump led to the right
section. This setup ensured uniform temperature throughout the bath, to within \( \pm 0.5^\circ C \).

The Beckman hygrometer probe, Model SMT-B was used for the high moisture levels, has a measuring range of 5-100% RH. It is a self-balancing measuring bridge which is coupled to a sensor for measuring humidity. The moisture sensor consists basically of two resistors. One of them varies according to the temperature and is called a reference resistor \( R_T \). The other one varies in accordance with the moisture and temperature and is the true measuring resistor \( R_m \). Both resistors have a negative temperature coefficient (NTC). The measuring resistor decreases its value as the moisture increases. When switching both resistors, \( R_T \) and \( R_m \) together in a measuring bridge, only the moisture influence on \( R_m \) results in a variation of the ratio \( R_m \) and \( R_T \), as a variation in the temperature has the same effect on both resistors and is thus ineffective.

It is known that any material containing water develops on its surface a water vapor pressure which corresponds to its water content. The sensor (Fig. 12) is arranged in such a way that there is a very thin air layer between it and the sensor element. The material conditions this air layer and consequently the sensor element, that is, the water vapor pressure of the material either moistens or dries the air layer and, in its turn, the element, until all three are in equilibrium.

b. Moisture Diffusivity and Drying Rate Experiments:

A wind tunnel, Fig. 13, comprised of a fan, a heating section, steam injection tower, drying column and a test section, was used for the drying experiments. The fan blows air through the heating section,
which contains three 3 KVA heating elements. The heaters could be used separately or together. Temperatures (dry-bulb and wet bulb) were controlled with thermostatic recorder controllers to within 1.5°C. The humidity of the air could be varied by injecting steam into the dryer. The test section, Fig. 3, is 523 sq. cm. in cross-section and 41 cm. in length. Flow straighteners were inserted at the bends to reduce transverse flow and flow mixers in the form of screens ensured a uniform distribution of air velocity across the test section. The tunnel was usable with continuous recirculation, direct flow through or proportions of the two by means of shutters at appropriate places. The air velocity was measured by Almor anemometer to within ± 0.01 m/sec. and could be varied with the range of 0 to 2.71 m/sec. (0-9750 m/hr.). The wet bulb thermometer was made by sleeving a thermocouple end with a linen wick, one end of which dipped into a constant-head water container.

A microtome was used for slicing the product for determination of moisture profile. The instrument could produce slices within the range of 0.001 cm. to 0.05 cm.

Procedure and Test Conditions:

a. Equilibrium Moisture Content: For the low and low-intermediate moisture range experiments, wide-mouth desiccators were half-filled with the various saturated salt solutions. Samples (0.5 to 1.3 gm) were placed in screen dishes, supported on dish holders standing in the desiccators. The desiccator covers were sealed using vacuum grease, and held in a constant-temperature cabinet controlled to within ± 10°C. The samples were weighed at 1 to 2 hour intervals initially. Two replications
were made. Upon reaching equilibrium (ΔW < .001 gm per 5 hours, 19-48 hours total), the samples were removed and dried in a vacuum oven to determine their bone-dry weights. From this the equilibrium moisture content was calculated.

In the agitated method, the tare weights of the empty sample dishes and their suspension hangers were determined by a precision Volland 2200 balance adapted for the experiment. Samples (0.5 - 1.5 gm) were placed in the sample dishes and weighed as quickly as possible. Each chamber in a constant temperature water bath, was placed on its magnetic stirrer to provide rotation to the fan. The samples were occasionally weighed, in-situ, without removing the chamber from the stirrer, until no further weight loss was noted. The samples were then removed and dried in a vacuum oven at 60°C for 18 hours to determine bone dry weights.

Initial moisture contents in the above two sets of experiments were determined as an error check and for calculation of moisture contents at each weighing. Two replications were made.

In the high moisture range, the samples were predried in a constant low relative humidity (11%) chamber and weighed. In order to assure a uniform moisture distribution in the samples, they were put in plastic bags and refrigerated for 48 hours. They were then equilibrated with the calibrated Beckman humidity probe at a known temperature, and reweighed. They were then dried in a vacuum oven to determine their bone dry weights. Because the samples lost weight during equilibration, the weights after equilibrations were assumed to be correct. Equilibration
time ranged from \( \frac{1}{2} \) to 4 hours. The experiments were replicated twice.

A modification in the sample cluster was made in order to monitor the sample temperature by attaching a surface thermocouple at the bottom of the sample chamber. To assure that the samples attained a uniform constant temperature before equilibration, they were preheated, wrapped in aluminum foil, in plastic bags.

All the experiments were conducted at five temperatures \(-5^\circ C\), storage temperature of the crops; \(32^\circ C\), the prevailing average ambient temperature in the tropics; \(55^\circ C\), the gelatinization temperature; \(65^\circ C\) and \(75^\circ C\), the processing temperatures.

b. **Diffusivity and Drying Rate.**

Slabs, 5.8 cm in diameter and 1\(\frac{1}{2}\) cm. thick of the products were used. Sample, 28 \(\pm\) 1.5 gm., supported on an aluminum plate was suspended in the test section and attached to a sensible balance (smallest reading 10 mg.) placed above the section.

Air of known relative humidity, and at a considerable high velocity 2.26 meters/sec. was blown past the surface of the sample to effect moisture removal from the surface. The high air velocity was used to minimize the effect of film resistance at the surface (Whitaker, et. al., 1967, Chirife and Cachero 1970); therefore the film resistance was assumed to be zero.

The sides of the sample were coated with a special high vacuum grease, of vapor pressure \(10^{-3}\) mm Hg at 300\(^\circ\)C and \(10^{-10}\) to \(10^{-11}\) mm Hg at room temperature, to eliminate moisture loss from the sides and
assure that the moisture loss was purely unidirectional from the upper surface of the sample.

The weight was recorded at intervals of ten minutes and drying was stopped when the product had lost 15% to 25% of its original weight. This was to maintain the second boundary condition of \( C = C_0 \) at \( x = \infty \).

A core was removed from the center and cut into slices of approximately 0.1 cm. thickness using the microtome. The moisture content of the slices was determined by an air oven. The slice thickness was also measured by a micrometer screw gage.

An identical sample of the same size and weight placed also in the test section was used to monitor the temperatures at three places (0.2 cm., 0.5 cm., and 0.8 cm. from the top). This was to verify the assumption that there was no significant \( (\Delta T < 2^\circ C) \) temperature gradient within the product during the experiment. The surface temperature was also measured during the drying period for use in the analysis of moisture movement mechanism.

Experiments were conducted at 55, 65, and 75°C at 10% and 60% RH. Two replications of each experiment were made.

**Materials:** Six salt solutions were used to produce the following relative humidities at 20°C: lithium chloride, 11.2%; potassium acetate, 23.2%; magnesium chloride, 33.6%; potassium carbonate, 43.9%; sodium nitrate, 65.3%; sodium chloride, 75.5%. Variations of humidities with temperature are given in Table B1. Salts used as checks and for calibration were: ammonium sulphate, 90.6%; potassium nitrate, 93.2%; and potassium sulphate, 97.2%. 
The white potatoes were of the White Rose variety, grown in Bakersfield, California. The average maturity time was ninety days. It was estimated that they were purchased ten days after harvest. They were refrigerated after purchase at 40°F (5°C).

The cassava tubers, of the sweet variety, were imported frozen from Costa Rica and distributed by Productors Ticos of Miami, Florida.
VI. RESULTS

The results are presented under three major headings—Equilibrium Moisture Content, Diffusivity, and Drying Rate. The major graphs and tables are presented here while the supplementary tables are given in the appendices. Also, the computer program and other results are included in the appendices.

Equilibrium Moisture Content:

The moisture isotherms for cassava and potato are presented in Figs. 14 and 15. In developing an isotherm equation, the Henderson-Rockland approach was applied. Figs. 16 and 17 show plots of Henderson equation at different temperatures. The plots were analysed by fitting one, two and three straight lines statistically and comparing correlation coefficients.

Two or three sections of straight lines were obtained instead of one, as Henderson (1952) proposed. This finding was in agreement with Rockland's (1957) findings with walnut and other food products. Therefore, the plots were analyzed using Rockland's local isotherm concept. The Henderson constants for each sectional straight line were determined from the linear regression analysis, and are presented in Tables 3 and 4. The moisture content at the breaks occur in the Henderson plots and are presented in Table 5.

The isothermic heat of desorption of cassava and potato at any moisture content, m, was evaluated graphically by plotting the
Figure 14. Moisture Isotherms of Cassava
Figure 15. Moisture Isotherms of Potato
desorption isotherm, i.e., the variation of equilibrium pressure ratio with temperature corresponding to a constant amount of vapor or gas absorbed, as ln a vs. 1/T, (Fig. 18 and 19). The isosteric heat, $Q_s$, was calculated from the slope of the plot by fitting straight lines statistically. The correlation coefficient values ranged from .98 to .99.

It is seen from the figures that the slope decreased with increase in moisture and that at low moisture contents ($m \leq 1$ gm/gm db), two intersecting straight lines were obtained instead of one. The two lines intersect at a temperature of 55°C for cassava and at 59°C for potato with the first part of the lines having a greater slope than the second part.

The value of the isosteric heat of desorption ranged from 12.85 k cal/mole to 10.55 k cal/mole for potato and from 16.61 k cal/mole to 10.41 k cal/mole for cassava, (Table B13 of appendix).

Plots of the BET equation, Figs. 21 and 22, show two distinct regions: a linear region at low relative humidity and a non-linear region at high relative humidity. The values of constant, $C$, and the monolayer, $V_m$, calculated from the slopes and intercepts of the linear portions are tabulated in Tables 6 and 7 for both products. The monolayer value at different temperatures is almost constant at an average value of 0.075 gm of $H_2O$/gm b.d. at high temperatures and .09 gm/gm at low temperatures for potato and ranges between .05 and .10 for cassava.

For both products, the monolayer values decrease with increase in temperature but there is not any marked trend of variation of constant $C$ with temperature. The $V_m$ value for potato is generally higher than
Figure 16. Henderson Plots for Cassava
Figure 17. Henderson Plots for Potato
TABLE 3. Constants n and K in Henderson plot for cassava

<table>
<thead>
<tr>
<th>Temp °C</th>
<th>Part I</th>
<th>Part II</th>
<th>Part III</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>$K_1 \times 10^3$</td>
<td>$n_1$</td>
<td>$K_2 \times 10^3$</td>
</tr>
<tr>
<td>5</td>
<td>3.3</td>
<td>1.9</td>
<td>0.7</td>
</tr>
<tr>
<td>32</td>
<td>2.1</td>
<td>2.3</td>
<td>48.0</td>
</tr>
<tr>
<td>55</td>
<td>5.0</td>
<td>2.3</td>
<td>72.0</td>
</tr>
<tr>
<td>65</td>
<td>5.6</td>
<td>2.1</td>
<td>380.0</td>
</tr>
<tr>
<td>75</td>
<td>5.1</td>
<td>1.2</td>
<td>11.0</td>
</tr>
</tbody>
</table>

TABLE 4. Constants n and K in Henderson plot for potato

<table>
<thead>
<tr>
<th>Temp °C</th>
<th>Part I</th>
<th>Part II</th>
<th>Part III</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>$K_1 \times 10^3$</td>
<td>$n_1$</td>
<td>$K_2 \times 10^3$</td>
</tr>
<tr>
<td>5</td>
<td>1.6</td>
<td>2.1</td>
<td>210.0</td>
</tr>
<tr>
<td>32</td>
<td>5.5</td>
<td>1.7</td>
<td>260.0</td>
</tr>
<tr>
<td>55</td>
<td>0.9</td>
<td>2.6</td>
<td>420.0</td>
</tr>
<tr>
<td>65</td>
<td>6.0</td>
<td>1.9</td>
<td>460.0</td>
</tr>
<tr>
<td>75</td>
<td>31.0</td>
<td>1.1</td>
<td>4.0</td>
</tr>
</tbody>
</table>

TABLE 5. Moisture contents, % d.b., at which L.I. breaks occur in Henderson plot

<table>
<thead>
<tr>
<th>Temp °C</th>
<th>Cassava</th>
<th>Potato</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>1st Break</td>
<td>2nd Break</td>
</tr>
<tr>
<td>5</td>
<td>19.5</td>
<td>21.9</td>
</tr>
<tr>
<td>32</td>
<td>15.2</td>
<td></td>
</tr>
<tr>
<td>55</td>
<td>12.0</td>
<td></td>
</tr>
<tr>
<td>65</td>
<td>13.9</td>
<td></td>
</tr>
<tr>
<td>75</td>
<td>6.3</td>
<td>12.0</td>
</tr>
</tbody>
</table>
that of cassava at any temperature except for 50°C where they are the same.

The pore-size distribution of the products were calculated using both the Halsey and Kelvin equations. The pore-size, at a given temperature increases with moisture content as seen in Table 8.

The pore size distribution function was evaluated using the equations presented in the theoretical analysis sector. The computer program is given in the Appendix. Fig. 23 shows log-log plots of the distribution function versus pore radius at 32°C. The plots give approximately straight lines. The plots at other temperatures are presented in the Appendix.

The plots were analyzed statistically to obtain the values of $k_1$ and $\gamma$ of equation (57). The values of $k_1$ and $\gamma$ together with the correlation coefficients are presented in Table 9. From the R-square values, cassava shows a better correlation than potato at all temperatures. Table 9 suggests that $k_1$ and $\gamma$ are characteristic parameters of the pore structure because they are unique for each product. Both of these parameters show a definite temperature dependence.
Figure 10. Semilog Plots of Activity vs $1/T$ for Cassava
Figure 19. Semilog Plot for Potato
Figure 20. Variation of $Q_s$ with Moisture Content
Figure 21. BET Plots for Cassava
Figure 22. BET Plots for Potato
### TABLE 6. BET monolayer values for cassava

<table>
<thead>
<tr>
<th>Temp °C</th>
<th>C</th>
<th>( V_m ) gm/gm db</th>
<th>Correlation Coefficient</th>
</tr>
</thead>
<tbody>
<tr>
<td>5</td>
<td>8.1</td>
<td>0.09</td>
<td>0.97</td>
</tr>
<tr>
<td>32</td>
<td>16.7</td>
<td>0.07</td>
<td>0.98</td>
</tr>
<tr>
<td>55</td>
<td>16.0</td>
<td>0.06</td>
<td>0.98</td>
</tr>
<tr>
<td>65</td>
<td>19.6</td>
<td>0.05</td>
<td>0.94</td>
</tr>
<tr>
<td>75</td>
<td>3.7</td>
<td>0.06</td>
<td>0.95</td>
</tr>
</tbody>
</table>

### TABLE 7. BET monolayer values for potato

<table>
<thead>
<tr>
<th>Temp °C</th>
<th>C</th>
<th>( V_m ) gm/gm db</th>
<th>Correlation Coefficient</th>
</tr>
</thead>
<tbody>
<tr>
<td>5</td>
<td>19.5</td>
<td>0.09</td>
<td>0.98</td>
</tr>
<tr>
<td>32</td>
<td>12.7</td>
<td>0.09</td>
<td>0.96</td>
</tr>
<tr>
<td>55</td>
<td>25.4</td>
<td>0.08</td>
<td>0.97</td>
</tr>
<tr>
<td>65</td>
<td>7.3</td>
<td>0.08</td>
<td>0.95</td>
</tr>
<tr>
<td>75</td>
<td>5.7</td>
<td>0.07</td>
<td>0.94</td>
</tr>
</tbody>
</table>
Figure 23. Pore-Size Distribution Function Plot For Cassava and Potato at 32°C
TABLE 8: Pore Size Distribution at 32°C.

<table>
<thead>
<tr>
<th>Moisture Content ( \text{gm H}_2\text{O/gm d.b.} )</th>
<th>Cassava</th>
<th>Potato</th>
</tr>
</thead>
<tbody>
<tr>
<td>0.04</td>
<td>2.79</td>
<td>0.05</td>
</tr>
<tr>
<td>0.07</td>
<td>3.34</td>
<td>0.08</td>
</tr>
<tr>
<td>0.09</td>
<td>3.86</td>
<td>0.11</td>
</tr>
<tr>
<td>0.11</td>
<td>5.55</td>
<td>1.13</td>
</tr>
<tr>
<td>0.13</td>
<td>6.57</td>
<td>0.15</td>
</tr>
<tr>
<td>0.14</td>
<td>7.95</td>
<td>0.18</td>
</tr>
<tr>
<td>0.16</td>
<td>9.99</td>
<td>0.22</td>
</tr>
<tr>
<td>0.19</td>
<td>13.59</td>
<td>0.30</td>
</tr>
<tr>
<td>0.24</td>
<td>22.06</td>
<td>0.48</td>
</tr>
</tbody>
</table>

TABLE 9. Characteristic Parameters \( k_1 \) and \( \gamma \), of pore size distribution plots for cassava and potato.

<table>
<thead>
<tr>
<th>Temp ( ^\circ\text{C} )</th>
<th>Cassava</th>
<th>Potato</th>
</tr>
</thead>
<tbody>
<tr>
<td>( k_1 \times 10^{-17} )</td>
<td>( \gamma )</td>
<td>R-square</td>
</tr>
<tr>
<td>5</td>
<td>7.23</td>
<td>-1.48</td>
</tr>
<tr>
<td>32</td>
<td>7.68</td>
<td>-1.79</td>
</tr>
<tr>
<td>55</td>
<td>7.81</td>
<td>-1.95</td>
</tr>
<tr>
<td>65</td>
<td>7.98</td>
<td>-2.33</td>
</tr>
<tr>
<td>75</td>
<td>8.22</td>
<td>-2.51</td>
</tr>
</tbody>
</table>
**Drying Tests:** The two products were dried at two relative humidities (10% and 60%) and three temperatures: 55°C, 65°C, and 75°C.

**Observation:** During drying at 60% RH, there was condensation initially because the temperature of the product was below the wet bulb temperature. During this period there existed a reasonable temperature gradient (3-5°C) between the top and lower parts of the product. This gradient disappeared as the product temperature approached the wet bulb temperature and the condensate dried out. Therefore, the data at 60% RH were analyzed from the time the product attained the original weight and the temperature gradient within the product was negligible.

The thickness of the sample was measured before and after drying. The reduction in thickness (.02 - .05 cm, or 2 to 5%) was insignificant, and therefore did not affect the data analysis. The drying data are given in Tables B15 to B18 of the Appendix.

The moisture profile plots given in non-dimensional moisture ratio and distance for the two products are presented in Figures 24 to 27.

Plots of gross weight versus time are shown in Figures 28 to 31. Also included in the plots is the mean temperature of the product. The product mean temperature never reached the ambient temperature because of duration of drying. If the product had been dried to equilibrium moisture content, its temperature could have been very close of the air temperature. However, in analyzing the effect of temperature, the air temperature was used as this could be controlled.
Figure 24. Non-dimensional Moisture Profiles for Cassava at 10 % RH
Figure 25. Non-dimensional Moisture Profile for Cassava at 60% RH
Figure 26. Non-dimensional Moisture Profile for Potato at 10% RH
Figure 27. Non-dimensional Moisture Profile for Potato at 60% RH
Figure 28. Average Weight and Mean Temperature Histories of Cassava at 10% RH
Figure 29. Average Weight and Mean Temperature Histories of Cassava at 60% RH
Figure 30. Average Weight and Mean Temperature Histories of Potato at 10% RH
Figure 31. Average Weight and Mean Temperature Histories of Potato at 60% RH
Evaluation of Diffusivity: From the moisture profile, the diffusivity at a given moisture content was determined by the procedure analyzed earlier. Figures 32 and 33 show the plots of diffusivity vs. moisture content.

The shape of the plot shows that a linear relationship cannot be fitted; instead it appears to be an exponential function or a combination of linear and exponential or a power function. The shape is similar to that reported to Matano (1932-33) for Cu-Ni, Mehl and Rhines for Cu-Si, Cu-Zn and Cu-Al, and discussed by Jost (1952 pp. 210-262). These investigators also used Boltzmann's method for evaluating the diffusion coefficient.

For both products, the plot shifts up with increase in temperature. At the same temperature, the values at 10% RH are higher than at 60% RH for low moisture contents, but both approach the same value as the moisture content increases. This means that the effect of relative humidity on diffusivity was more recognizable at low moisture content than at high.

To verify if relative humidity had a significant effect on diffusivity as observed at 10% and 60% RH levels, additional tests were conducted at 65°C, 30% RH for cassava. Also, the effect of time of drying was investigated by drying the sample at 65°C, 60% RH for 60, 80, and 106 minutes. The results shown in Figures 34 and 35 indicate that the plots shift up with decrease in relative humidity and shifts down with increase in time of drying. This could be explained by the effect of structural changes (shrinkage) during drying. But these changes
Figure 32. Plots of Moisture Diffusivity vs Moisture Content for Cassava
Figure 33. Plots of Moisture Diffusivity vs Moisture Content for Potato
Figure 34. Plots Showing the Effect of RH on Diffusivity for Cassava at 65°C and 106 min. drying time
Figure 35. Plots Showing the Effect of Time of Drying on Diffusivity for Cassava at 65°C, 10% RH
would be lateral (reduction in surface drying area) rather than vertical because from experimental results, the vertical or axial changes were less than 0.3%. The effect of relative humidity on diffusivity was apparent in the data reported by Husain, et. al., 1972, but no attempt was made by the investigators to discuss reasons for this.

The diffusivity values determined range from $6.0 \times 10^{-7}$ cm$^2$/sec. at 55°C, low moisture, to $1.6 \times 10^{-5}$ cm$^2$/sec. at 75°C, high moisture for cassava. The range for potato is from $2.9 \times 10^{-7}$ cm$^2$/sec. at 55°C to $9.1 \times 10^{-6}$ cm$^2$/sec at 75°C.

To see the effect of temperature on diffusivity, the diffusivities at a moisture content of 1.0 gm/gm db and were plotted on a semilog with 1/T. Figures 36 and 37 show the plots for the two products at 10% and 60% RH, and two mc levels. A straight line was received in each case, confirming an Arrhenius relation. This is in agreement with the results of Chirife (1971) for Cassava, Fish (1958), Saravacos and Charm (1962) and Husain, et. al., (1972) for potato.
Figure 36. Effect of Temperature on Diffusivity at Three Moisture Contents for Cassava
Figure 37. Effect Of Temperature on Diffusivity at Three Moisture Contents for Potato
VII DISCUSSION OF RESULTS

Isotherms and Isotherm Equations:

The moisture isotherms of cassava and potato are sigmoidal in shape and can be said to be of the Type 2 as classified by Rockland (1969). The shape for potato is similar to that of Corling (1958) Fig. 40 and Stitt (1958) and the values of the equilibrium moisture correspond almost to those of Corling at intermediate and high temperatures but differ slightly (Corling's being slightly on the low side) at low temperatures.

The equilibrium moisture values for Cassava are in agreement with those found by Rao, et. al. (1974), Fig. 39, but their isotherm equation resulted in a sinusoidal plot when tested.

From the Henderson constants of the local isotherms, an equation relating the moisture content with the relative humidity at a given temperature was formulated.

\[(1-a) = \exp - k_z m^n_z \quad (86)\]

where \(k_z\), \(n_z\) are constants at \(z\) moisture range.

For the purpose of illustration the isotherm predicted using Equation 86 for both products at 32°C are presented in Figs. 38 and 39.

The plots in comparison with the experimental data are within limits of variability. The two sections of the isotherm plots intercept at moisture contents approximately equal to those at which the local isotherm (LI) breaks occur in the Henderson plot.
Figure 38. Comparison of Experimental and Predicted Isotherm using Modified Henderson Equation for Cassava at 32°C
Figure 39. Comparison of Experimental and Predicted Isotherm using Modified Henderson Equation for Potato at 32°C.
Heat of Sorption:

Nonlinear plots of log a vs. 1/T for both products at very low moisture content (.05 gm/gm) at temperatures approximately equal to the gelatinization temperature of 56°C could be interpreted to indicate some change in the product properties. This change, therefore, has resulted in more energy being apparently required to desorb moisture from these products at temperatures greater than 56°C.

For both products, the heats of desorption are highest at low moisture and decrease continually with increasing moisture content until they approach constant values (Fig. 20). This agrees with the findings of Labuza (1968) and Chung and Pfost (1967,1), and means that quite a substantial energy must be applied to desorb the last traces of water while comparatively little energy is needed to desorb the moisture from high moisture stage. Because the heats of sorption indicate the intermolecular attraction forces between sorptive sites and water vapor, it can be concluded that the binding energy is highest at very low moisture content. Comparing the values of cassava and potato, the energy of desorption for cassava is less than that of potato at high moisture until to a moisture level of 0.15 gm H₂O/gm b.d., when it becomes more difficult to desorb moisture from cassava than from potato.

BET Monolayer Coverage and Pore-Size Distribution:

The result of two distinct regions—linear and non-linear, indicates the limited application of the BET equation. The linear region, including relative humidities less than 50%, indicates the equation can only be applied to RH less than 50%. This is in agreement with the findings
of various other researchers such as Buschuk and Winkler (1957), Dole and McLaren (1947) and Hall, et. al. (1963). One of the uses of the monolayer values is that it is possible to estimate the water surface area by assuming the area of a water molecule to be $10.6 \text{Å}^2$. The surface area in m$^2$/gm of solid is given by:

$$S_o = V_m \cdot \frac{1}{(M\cdot W)_{H_2O}} \cdot N_o \cdot A_{H_2O}$$

where

$V_m$ = monolayer value in gm/gm solid.

$(M\cdot W)_{H_2O}$ = molecular wt. of H$_2$O = 18 g/mole

$N_o$ = Avogadro's Number, $6 \times 10^{23}$ molecules/mole

$A_{H_2O}$ = Area of H$_2$O molecule = $16.6 \times 10^{-20}$ m$^2$

Another use of the monolayer value is its application in defining a moisture range for optimum stability of stored dehydrated foods. The amount of water which represents a monolayer according to the BET theory may be regarded as a protective film which protects the particles of food from attack by oxygen (Salwin, 1959). The monolayer corresponds to the number of available reactive absorption sites in the protein carbohydrates and fat components of the food. When the amount of water is adequate for combining with these functional groups, they are protected from reaction with oxygen (Salwin, 1959). In other terms, the relative humidity at this point represents a partial pressure of water vapor which is competitive with the oxygen partial pressure to the extent of being protective.

**Pore-Size Distribution Function**

The parameters $k_1$ and $\gamma$ increase with temperature as seen from Table 9. This agrees with the findings of Ngoddy et. al. (1972 III).
The logical implication of this dependence is that the pore structure of the sorbent changes with isotherm temperature. Although the exact nature of this change cannot be determined from a priori considerations, it appears reasonable, in view of the effect of temperature on swelling forces, that pores could undergo a certain amount of deformation with changing isotherm temperature.
OVERVIEW

From the tremendous data and analysis done, it might be reasonable to go a step beyond Rockland's (1957) hypothesis and state the following hypothesis.

The state of water in a biological material like cassava and potato could be divided into three regions:

1. The BET monolayer value which has a maximum value of about 0.08 gm/gm db at an RH of 45%.

2. Water of solution or multilayer region, following Henry's Law. This region will account for the stability of the product. This region would extend between 35% RH to 75% RH, overlapping sometimes with the BET region, depending on the product.

3. The third region could be termed the Kelvin region, representing the condensation region. This might extend between 75% RH and saturation.

In some cases, the three regions may not be distinctively obtained and two regions result. In such cases, the water of solution region would extend to the Kelvin region.

Therefore, an isotherm plot can be said to be an additive plot of these three or two regional plots, Fig. 40.
Figure 40. Proposed Moisture States in Biological Material
Moisture Diffusivity: To effectively utilize the diffusivity data for design of drying process, it was necessary to establish a mathematical relationship between diffusivity and moisture content. Therefore, different types of equations were fitted to the curves by Statistical Analysis System (SAS) program using the IBM 370 computer. The program was designed to give the analysis of variance, regression coefficients, and statistics of fit for dependent variables. Also, the program was ordered to plot the residuals versus the variable and also versus the predicted.

A detailed procedure of the analysis is given in the appendix.

From the analysis it is seen that two different equations were used to characterize the variable diffusivity of moisture in cassava and potato.

The relationship for cassava was:

\[
D = B_0 + B_1 e^m + B_2 e^m + B_3 e^m + B_4 X_R \tag{88}
\]

where,

\( B_0, B_1, B_2, B_3, \) and \( B_4 \) are constants and \( X_R \) is relative humidity indicator as given in the appendix.

For potato the relationship was as follows:

\[
D = f_0 + f_1 e^m + f_2 X_R + f_3 X_6 + f_4 X_1 e^m \tag{89}
\]

where,

\( f_0, f_1, f_2, f_3 \) and \( f_4 \) are constants, \( X_1 \) and \( X_6 \) are temperature
indicators, \( X_r \) is relative humidity indicator.

The term \( X_l e^m \) signified that there was interaction, and was also noticed in the preliminary plots. The values of the coefficients are given in Table 10.

These relationships are similar to that proposed by Husain, et al. (1972) for potato, which was also exponential with respect to moisture. The function for cassava shows a sum of linear and exponential, which is quite apparent from the plots.

To ascertain the validity of this relationship, the equation was tested in the mathematical model at 65°C, 10% RH using the CSMP program described earlier in the theoretical analysis section. The predicted moisture profile was compared to the experimental data. Figure 41 shows that the predicted profile compares well with the experimental data.

The activation energies were found from the Arrhenius plots at three moisture levels for both products. The values 4.90 kcal/mole for cassava at 1.0 mc, db and 6.54 kcal/mole for potato at 1.5 mc, db, (Tables 10 and 11) are near those reported by Chirife (1971), 5.40 kcal/mole for cassava, the value of \( Q = 6.3 \) kcal/mole computed by Fish (1958) for diffusion of water in starch gel, and the value of \( Q = 7.19 \) kcal/mole as reported by Jason (1958) for the first falling rate period of drying of fish muscle. The value reported by Saravacos and Charm (1962) which is 12.50 kcal/mole, for potato is almost the same as those found for cassava at 0.1 mc, db (12.72) and for potato at mc of 0.25 db (12.94 kcal/mole).

The average diffusivity value for cassava at 75°C, 10% RH is lower than that of Chirife (1971) at 80°C, 3% RH, but within a comparable
difference range for the temperature and RH difference. The average value for potato at 55°C and 65°C are in good agreement with those of Yish (1958) for scalded potato slice and comparable to those of potato starch gel.

Table 10. Coefficients in the Diffusivity Function for Cassava and Potato

<table>
<thead>
<tr>
<th></th>
<th>Cassava, ( r^2 = .95 )</th>
<th>Potato, ( r^2 = .91 )</th>
</tr>
</thead>
<tbody>
<tr>
<td>( B_0 )</td>
<td>-0.0274</td>
<td>( f_0 ) 1.56 ( \times 10^{-6} )</td>
</tr>
<tr>
<td>( B_1 )</td>
<td>-5.74 ( \times 10^{-6} )</td>
<td>( f_1 ) 0.07 ( \times 10^{-6} )</td>
</tr>
<tr>
<td>( B_2 )</td>
<td>5.98 ( \times 10^{-6} )</td>
<td>( f_2 ) -46 ( \times 10^{-6} )</td>
</tr>
<tr>
<td>( B_3 )</td>
<td>0.2275</td>
<td>( f_3 ) 2.97 ( \times 10^{-6} )</td>
</tr>
<tr>
<td>( B_4 )</td>
<td>3.23 ( \times 10^{-6} )</td>
<td>( f_4 ) .01 ( \times 10^{-6} )</td>
</tr>
</tbody>
</table>

The results have shown that this technique can successfully be adapted to biological materials. But some limitations are imposed on the technique by a semi-infinite boundary condition and isothermal condition requirements. The drying time is therefore short and hence, makes the evaluation of diffusivity at very low moisture (0-0.02 gm/gm) content difficult and inaccurate. But, generally, foods are not dehydrated to as low moisture content range, and therefore, might not be of importance to drying processes.

**Drying Mechanism:** The drying mechanism was analyzed at 65°C, 55°C and the two relative humidities. The analysis of the moisture
Table 11. Activation energies at three moisture contents for cassava.

<table>
<thead>
<tr>
<th>mc, db</th>
<th>r-square</th>
<th>Q, kcal/mole</th>
<th>r-square</th>
<th>Q, kcal/mole</th>
</tr>
</thead>
<tbody>
<tr>
<td>.1</td>
<td>.99</td>
<td>12.722</td>
<td>.98</td>
<td>9.222</td>
</tr>
<tr>
<td>1.0</td>
<td>.99</td>
<td>4.90</td>
<td>.97</td>
<td>4.595</td>
</tr>
<tr>
<td>1.4</td>
<td>.99</td>
<td>4.166</td>
<td>.99</td>
<td>4.222</td>
</tr>
</tbody>
</table>

Table 12. Activation energies at three moisture contents for potato.

<table>
<thead>
<tr>
<th>mc, db</th>
<th>r-square</th>
<th>Q, kcal/mole</th>
<th>r-square</th>
<th>Q, kcal/mole</th>
</tr>
</thead>
<tbody>
<tr>
<td>.25</td>
<td>.991</td>
<td>12.944</td>
<td>.985</td>
<td>15.288</td>
</tr>
<tr>
<td>1.5</td>
<td>.999</td>
<td>6.54</td>
<td>.999</td>
<td>6.855</td>
</tr>
<tr>
<td>3.0</td>
<td>.995</td>
<td>5.222</td>
<td>.971</td>
<td>4.727</td>
</tr>
</tbody>
</table>
Figure 41. Comparisons of Experimental and Predicted Moisture Profiles Using Equations for Cassava and Potato, respectively, at 65°C, 10% RH
content - time curves shows that no constant drying rate exists. This is in agreement with the findings of Chirife (1970) for cassava, Chen (1971), Jason (1958) and Gorling (1958) but is in contrast with the results of Saravacos and Charm (1962). However, the use of those curves is not sufficient for this purpose because of the decrease of transfer area caused by shrinkage of the material (Chirife, 1970; Fish, 1958).

To confirm the conclusion that there is no constant rate drying, a thin thermocouple probe was inserted just below the surface of the product to monitor the surface temperature. The experimental result is shown in Figure 44. It shows that the surface temperature rises continuously and approaches the dry bulb temperature. This allows the deduction that there is no constant drying period, since the surface temperature is never constant. Therefore, the internal water movement is the controlling mechanism from the beginning of drying process.

Figures 42 to 43 show the non-dimensional moisture content, \((m - m_e)/m_o - m_e\) against time plotted in a semilog for two temperatures and two relative humidities. Analysis of these plots allows one to deduce that: 1) in all cases, straight lines are obtained which indicates that a diffusional mechanism is controlling. 2) The falling rate period has one stage within the range of moisture dried. This corresponds to the first falling rate period, (Chirife, 1971 for cassava; Husain et al., 1972 for potato).

It might be possible that mechanisms other than liquid diffusion could be responsible for moisture movement. These mechanisms are analyzed to see how well they fit the experimental results.
Figure 42. Effects of Temperature and Relative Humidity on the Drying Rate of Cassava
Figure 43. Effects of Temperature and Relative Humidity on the Drying Rate of Potato
Capillarity Flow: The flow of gases in a capillarity system is similar to that encountered in diffusion the rough a solid and can be characterized by a permeability coefficient, \( P \). According to Poiseulle's law, in a capillary of diameter \( d \) and Length \( L \)

\[
N_A = \frac{d^2 g_c}{32 \mu L R T} \quad \text{Pave} \ (P_1 - P_2)
\]

(90)

where \( N_A \) = rate of flow \( g/(sec) \) \( \text{cm}^2 \)

\( P_1, \ P_2 = \) pressure \( \text{dynes/cm}^2 \)

\( R_g = \) universal gas constant \( 1,98 \text{ cal/(gm mole) K} \)

\( T = \) absolute temperature \( K \)

\( \mu = \) viscosity, \( g/(cm)(sec) \)

If the rate of flow is measured in terms of a gas or liquid volume \( V \), at the average pressure flowing per unit time per unit cross-section of the solid, then Equation (90) becomes

\[
\text{Pave } V = \frac{P \text{Pave} \ (P_1 - P_2)}{h}
\]

(91)

where \( h \) is the solid thickness and \( P \) is the permeability, which has the same units as diffusion coefficient. Equation (90) shows that \( P \) varies inversely as the gas viscosity, which in turn increases with temperature; \( P \) then varies with \( 1/T \) and not with \( \exp (-1/T) \) as found experimentally. Therefore, the possibility of Poiseuille flow in cassava and potato must be excluded.

Vapor Diffusion: Simple kinetic theory of gaseous diffusion gives
the relationship between diffusion coefficient, \( D \), the mean thermal velocity \( \bar{u} \) and the mean free path, \( \lambda \), as

\[
D = \frac{1}{3} \bar{u} \lambda \quad (92)
\]

But since \( \bar{u} \) is proportional to \( T^{1/2} \), (Geankoplis 1972 pp. 23-25), the temperature dependence of \( D \) will be \( T^{3/2} \), which is again different from the experimental value.

**Knudsen Flow:** If the capillary diameters in cassava and potato are small in comparison with the mean free path of the gas molecules, flow will be Knudsen type. For a single straight capillary of radius, \( r \), the Knudsen diffusivity is given by

\[
D_k = \frac{2}{3} \bar{u} \quad (93)
\]

where \( \bar{u} = \) mean velocity, cm/sec

From the above equation, \( D_k \) varies with \( T^{1/2} \), which is not the temperature dependence found experimentally.

**Thermal Diffusion:** The hypothesis of thermal diffusion is rejected because for the small temperature gradients observed experimentally during the drying of cassava and potato makes the effect of thermal diffusion negligible.

On the basis of these discussions and analysis, it may be concluded that liquid water diffusion is the most appropriate mechanism to describe the drying behavior of cassava and potato. This agrees with the findings of Chirife (1970) for cassava.
Figure 44. Surface Temperature History of Products at 65°C  
Air Temperature and 10% RH
VIII. SUMMARY

This study was carried out to determine the equilibrium moisture content of cassava and potato and to investigate the variation of moisture diffusivity with moisture content and temperature in the two products and examine the moisture movement mechanism in the products during drying.

These data are important in the design of drying processes.

Equilibrium moisture contents were determined by three methods - static desiccator, agitated chamber and equilibrium relative humidity probe methods. The data were analyzed using Henderson plots to obtain local isotherms, Clausius Clapeyron plots to obtain the heat of desorption, BET approach to obtain the monolayer coverage and the power law function to characterize the pore-size distribution. The heats of desorption ranged from 16.6 K cal/mole to 10.45 K cal/mole for cassava and from 12.9 K cal/mole to 10.58 K cal/mole for potato. For both products, the heat of desorption was found to decrease with moisture content and approached a constant value at high m.c., equal to the heat of vaporization of liquid water. The BET approach was valid for both products up to a R.H. of 50%.

The moisture diffusivity was obtained by analyzing moisture profiles plotted from drying data. The method described by Crank and Park (1949) and used by Mateno (1933) for Cu-Al alloy was employed. The final expression used was

\[ D_{M_1} = \frac{1}{2t} \frac{dx}{dm} \left( \frac{M_0}{M_1} \right) x \text{ d m} \]
The data received at different temperatures and relative humidities were fitted statistically to different functions and the one with the best fit was chosen.

Two functions, one for each product, were found to best fit the data. For cassava, it was:

\[
D = B_0 + B_1 e^m + B_2 e + B_3 e^{\frac{1}{T}} + B_4 XR
\]

where,

\( B_0, B_1, B_2, B_3 \) and \( B_4 \) are constants and XR is relative humidity indicator. The r-squared value for this was 0.95.

For potato it was:

\[
D = f_0 + f_1 e^m + f_2 XR + f_3 X6 + f_4 XL e^m
\]

where,

\( f_0, f_1, f_2, f_3 \) and \( f_4 \) are constants and XL and X6 are temperature indicators. XR is relative humidity indicator. The R-squared value was 0.91.

The functions were used to predict moisture profile at 65\(^\circ\)C, 10% RH and compared with the experimental data.

The drying rate data were analyzed by plotting non-dimensional moisture vs. time in a semilog. It was found that there was no constant rate of drying and that the mechanism of moisture movement in the falling rate was by liquid diffusion due to concentration gradient. This was confirmed by analyzing the surface temperature of the products during drying.
IX. CONCLUSIONS

The following conclusions may be drawn from this research:

1. Moisture isotherms of fresh cassava and white potato are sigmoid in shape and are described by the Henderson equation with acceptable precision; the correlation coefficients ranging from 0.98 to 0.99.

2. The heats of desorption of both products decrease with increase in moisture content and approach a constant value, the heat of vaporization of pure water, at high moisture contents. More energy is needed to desorb moisture from cassava than from potato at moisture content below .15 gm H₂O/gm dry matter, (16.60 K cal/mole for cassava and 12.85 K cal/mole for potato at 0.05 gm/gm md.), while the reverse is the case at moisture contents greater than 0.15 gm/gm, (10.86 K cal/mole for cassava and 11.1 K cal/mole for potato at 0.2 gm/gm).

3. The power law function describes the pore size distribution in cassava better than in potato statistically.

4. The technique of moisture profile analysis can be successfully adapted to biological materials in determining moisture diffusivity.

5. The variable diffusivity function with respect to moisture content for cassava is a sum of a linear and exponential function while it is only exponential for potato.

6. The moisture diffusivity in both products follows an Arrhenius relation with respect to temperature.

7. No constant drying rate is observed for cassava and potato and the
entire drying takes place in the falling rate.

3. Liquid water diffusion is the most appropriate mechanism to describe the drying behavior of cassava and potato within the drying range of the experiment, but the possibility of capillary flow at the initial stage cannot be discounted.
RECOMMENDATIONS FOR FURTHER RESEARCH

Based on this investigation, the following possible areas are suggested for further research:

1. Development of a shrinkage model for cassava and potato should be investigated.

2. The effect of relative humidity on diffusion coefficient should be further studied to give an idea of how this affects the activation energy.

3. The effect of boundary film layer on the rate of drying cassava and potato by varying the air velocity.
APPENDICES
<table>
<thead>
<tr>
<th>Column 1</th>
<th>Column 2</th>
<th>Column 3</th>
</tr>
</thead>
<tbody>
<tr>
<td>Data 1</td>
<td>Data 2</td>
<td>Data 3</td>
</tr>
<tr>
<td>Data 4</td>
<td>Data 5</td>
<td>Data 6</td>
</tr>
<tr>
<td>Data 7</td>
<td>Data 8</td>
<td>Data 9</td>
</tr>
</tbody>
</table>

**APPENDIX A-1**

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Appendix A-2

Computer fitting of equation to variable moisture diffusivity data for cassava and potato known relationships: For agricultural products no known relationships exist between diffusivity and moisture content, temperature and relative humidity jointly. However, it is known that temperature has exponential relationship (King, 1968, Saravacos and Charn, 1962, Husain et al., 1972, and Agrawal, et al., 1975) and moisture content could have linear relationship (Whitaker, et al., 1969) or exponential relationship (Husain, et al., 1972).

Expected Relationship: Whereas the statistical analysis will indicate the true relationship, based on past experience and simple plots of data, it can be hypothesized that a relationship of the following nature may come up:

\[ D : T \text{ (temp)} - \text{exponential} \]
\[ D : \text{RH (rel. humidity)} - \text{linear} \]
\[ D : \text{moisture content} - \text{exponential or logarithmic} \]

Variables: The dependent variable was diffusivity. 

1) There were three independent variables--moisture content, 
\[ M, \text{gm/gm db} \]
Temperature, \( T, ^{\circ}\text{C} \)
Relative humidity, \( \text{RH} \% \)

Suspected Relationships: The diffusivity was plotted against moisture
content, relative humidity and temperature. From the preliminary plots, it was concluded that for the same temperatures, diffusivity was higher at 10% RH than at 60% RH.

**Models fitted to data:** Based on the conclusions from the preliminary analysis of graphical display, the following independent variables were created.

\[
X_1 = \text{temp. indicator variable} = \begin{cases} 
0 & \text{if } T = 55 \text{ or } 75 \text{ C} \\
1 & \text{if } T = 65 
\end{cases}
\]

\[
X_6 = \text{temp. indicator variable} = \begin{cases} 
0 & \text{if } T = 55 \text{ or } 65 \\
1 & \text{if } T = 75 
\end{cases}
\]

\[
X_R = \text{RH indicator or variable} = \begin{cases} 
0 & \text{if } RH = 60\% \\
1 & \text{if } RH = 10\% 
\end{cases}
\]

\[
X = \frac{1}{T} 
\]

\[
XXP = e^{-X}
\]

\[
XX6 = XXP \times X6
\]

\[
EXM = e^M
\]

\[
XREM = XR \times e^M
\]

\[
LNM = \log_e M
\]

\[
LM = \log_{10} M
\]

\[
XX1 = X \times X1
\]

A number of variables transformed from \(X\)'s were also defined as is seen in the programs.

Analysis of covariance models were fitted to the data using CLASSES and Dummy B features of Statistical Analysis Systems (SAS). Then all possible regressions—stepwise regression, forward selection and backward
elimination procedures were employed to select a suitable model (Tables A2 - 1 to 3).

From the model selection procedure, two models were chosen, one for cassava and the other for potato. The following was chosen for cassava:

\[ D = B_0 + B_1 M + B_2 e^M + B_3 e^{\frac{T}{10}} + B_4 XR \]

And for potato the following was chosen:

\[ D = f_0 + f_1 e^M + f_2 XR + f_3 X^6 + f_4 X^6 e^M \]

A number of other models as suggested from stepwise, forward selection and backward elimination procedures were also analyzed for the best fit. Residual plots were obtained for all these models against \( m \), \( X \) and predicted diffusivity \( \hat{\gamma} \). Also \( \hat{\gamma} \) was plotted against \( M \) to check the predictive ability of these models. The above two models were selected as the best for the products.

The model for cassava is given as

\[ D = -.0274 - 5.74 \times 10^{-6} M + 5.98 \times 10^{-6} e^M + .0275 e^{\frac{1}{T^{0.0}} K} + 3.23 \times 10^{-6} XR. \]

This means that for RH = 10% the model is

\[ D = 27398 \times 10^{-6} - 5.74 \times 10^{-6} M + 5.98 \times 10^{-6} e^M + .0275 e^{\frac{1}{T^{0.0}} K} \]

for RH = 60%:

\[ D = -0.0274 - 5.74 \times 10^{-6} M + 0.98 \times 10^{-6} e^M + 0.0275 e^{\frac{1}{T^{0.0}} K} \]
The model for potato is given as

\[ D = 1.56 \times 10^{-6} + 7 \times 10^{-8} e^M - 4.6 \times 10^{-7} XR + 2.97 \times 10^{-6} X6 + 10^{-8} M \]

Therefore this means that

At 55°C, 10% RH then
\[ D = 1.10 \times 10^{-6} + 7 \times 10^{-8} e^M \]

At 55°C, 60% RH then
\[ D = 1.56 \times 10^{-6} + 7 \times 10^{-8} e^M \]

At 65°C, 10% RH
\[ D = 1.10 \times 10^{-6} + 8 \times 10^{-8} e^M \]

At 65°C, 60% RH
\[ D = 1.56 \times 10^{-6} + 8 \times 10^{-8} e^M \]

At 75°C, 10% RH
\[ D = 4.07 \times 10^{-6} + 7 \times 10^{-8} e^M \]

At 75°C, 60% RH
\[ D = 4.53 \times 10^{-6} + 7 \times 10^{-9} e^M \]

Adequacy of model fit: The models fitted had r-square of 0.95 and 0.91 for cassava and potato, respectively, which are considered good. The residual plots obtained for these models were fairly scattered, which supported the adequacy of the model. Predicted values of diffusivity, D, plotted against M for different levels of temperature and relative humidity, compared quite well with the observed values.
INIT
K=1/T
PARAM LE=9.93,N=10,T=330
PARAM EI=1.00,E2=0.714,E3=5.947,E4=.924,E5=881,E6=833,E7=771,
   E8=6.757,E9=2.456,E10=3.234
INCX IC1=1.0,IC2=1.0,IC3=1.0,IC4=1.0,IC5=1.0,IC6=1.0,IC7=1.0,
   IC8=1.0,IC9=1.0,IC10=1.0
DYN
DX1=-0.057391-5.774-6.06C1*5.947-6.0E0(1)+0.007556*EXP(K)
DX2=-0.057391-5.774-6.06C2*5.947-6.0E0(2)+0.007556*EXP(K)
DX3=-0.057391-5.774-6.06C3*5.947-6.0E0(3)+0.007556*EXP(K)
DX4=-0.057391-5.774-6.06C4*5.947-6.0E0(4)+0.007556*EXP(K)
DX5=-0.057391-5.774-6.06C5*5.947-6.0E0(5)+0.007556*EXP(K)
DX6=-0.057391-5.774-6.06C6*5.947-6.0E0(6)+0.007556*EXP(K)
DX7=-0.057391-5.774-6.06C7*5.947-6.0E0(7)+0.007556*EXP(K)
DX8=-0.057391-5.774-6.06C8*5.947-6.0E0(8)+0.007556*EXP(K)
DX9=-0.057391-5.774-6.06C9*5.947-6.0E0(9)+0.007556*EXP(K)
DX10=-0.057391-5.774-6.06C10*5.947-6.0E0(10)+0.007556*EXP(K)
X1=100.0,40.0M2/L,T=60/1,0<=C2<=C1
X2=100.0,40.0M2/L,T=60/1,C3=C1+0.2,C2+C1
X3=100.0,40.0M2/L,T=60/1,C4=C3+C1
X4=100.0,40.0M2/L,T=60/1,C5=C4+C1
X5=100.0,40.0M2/L,T=60/1,C6=C5+C1
X6=100.0,40.0M2/L,T=60/1,C7=C6+C1
X7=100.0,40.0M2/L,T=60/1,C8=C7+C1
X8=100.0,40.0M2/L,T=60/1,C9=C8+C1
X9=100.0,40.0M2/L,T=60/1,C10=C9+C1
C1=INTEGRATIC1,X1
C2=INTEGRATIC2,X2
C3=INTEGRATIC3,X3
C4=INTEGRATIC4,X4
C5=INTEGRATIC5,X5
C6=INTEGRATIC6,X6
C7=INTEGRATIC7,X7
C8=INTEGRATIC8,X8
C9=INTEGRATIC9,X9
C10=INTEGRATIC10,X10
TERMINAL
RMS=SQRT(1+(E1-C1)^2+(E2-C2)^2+(E3-C3)^2+(E4-C4)^2)
   RMS=1+(E5-C5)^2+(E6-C6)^2+(E7-C7)^2+(E8-C8)^2+(E9-C9)^2+(E10-C10)^2)

END
PRINT 10RMS
20 FORMAT (1X,5F15.4)
TIME DEL=0.0001, P=11.01, DELT=0.05, T0=0.0, D=12.0, D1=5.0, D2=15.0
PRIPLET C1,X1,C2,X2,C3,X3,C4,X4,C5,X5,C6,X6,C7,X7,C8,X8,C9,X9,C10,X10
PRTPLT DMB(C8), DNY(C9), DML(C10)
END STOP
END DATA
//
### TABLE B1 Variation of R.H. of Saturated Salts Solution With Temperature.

<table>
<thead>
<tr>
<th>Salt</th>
<th>Temperature °C</th>
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<tbody>
<tr>
<td></td>
<td>5</td>
</tr>
<tr>
<td>Lithium Chloride</td>
<td>11.2</td>
</tr>
<tr>
<td>Potassium Acetate</td>
<td>26.0*</td>
</tr>
<tr>
<td>Magnesium Chloride</td>
<td>35.0*</td>
</tr>
<tr>
<td>Potassium Acetate</td>
<td>44.0</td>
</tr>
<tr>
<td>Sodium Nitrite</td>
<td>66.0</td>
</tr>
<tr>
<td>Sodium Chloride</td>
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</tr>
<tr>
<td>Ammonium Sulphate</td>
<td>82.0</td>
</tr>
<tr>
<td>Potassium Nitrite</td>
<td>96.0</td>
</tr>
<tr>
<td>Potassium Sulphate</td>
<td>99.0*</td>
</tr>
</tbody>
</table>

Source: Hall, 1957

*Measured by YSI hygrometer
Table B12 Comparison of the Correlation Coefficients for fitting 1, 2 and 3 straight lines to Henderson Plots for Cassava.

<table>
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<th>2 st. lines</th>
<th>3 st. lines</th>
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<td>55</td>
<td>.943</td>
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<td>65</td>
<td>.954</td>
<td>.979</td>
<td>.994</td>
</tr>
<tr>
<td>75</td>
<td>.942</td>
<td>.983</td>
<td>.998</td>
</tr>
</tbody>
</table>

Table B13 Comparison of the Correlation Coefficients for fitting 1, 2 and 3 straight lines to Henderson plots for Potato

<table>
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<th>Temp</th>
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<tr>
<td>Relative Humidity (°)</td>
<td>Equilibrium Moisture</td>
<td>Equation</td>
<td>Equation</td>
</tr>
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<td>----------------------</td>
<td>-----------</td>
<td>-----------</td>
</tr>
<tr>
<td>11.2</td>
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</tr>
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<td>---------------------</td>
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<td>--------------------------</td>
</tr>
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<td>0.1989</td>
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<tr>
<td>Relative Humidity %</td>
<td>Equilibrium Moisture</td>
<td>Equilibrium Moisture</td>
<td>Equilibrium Moisture</td>
</tr>
<tr>
<td>---------------------</td>
<td>----------------------</td>
<td>----------------------</td>
<td>----------------------</td>
</tr>
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Table B6  Equilibrium Moisture Content of Cassava at 75°C

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Table B7  Equilibrium Moisture Content of Potato at 5°C

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Table B10 Equilibrium Moisture Content of Potato at 65°C

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Table 811 Equilibrium Moisture Content of Potato at 75°C

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Table B15 Moisture Profiles of Cassava at 10% RH

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Table 818 Moisture Profile of Cassava at 65°C, 60% RH for 3 Different Drying Times With Condensation

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Table B19 Moisture Diffusivities, D of Cassava at 10% RH

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Table B20 Moisture Diffusivities, D of Cassava at 60% RH

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Table B21: Moisture Diffusivity, D of Potato at 10% RH

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Table B22: Moisture Diffusivities, D of Potato at 60% RH

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Table B23  Moisture Diffusivities, D
of Cassava at 65°C, 60% RH for Three Drying Times

| Moisture Content gm./gm. d.b. | Diffusivity x 10^6, cm²/sec. |
|                             | 60 min. |         | 80 min. |         | 106 min. |
|                             | I   | II  | I   | II  | I   | II  |
| 0.1                         | 3.12 | 3.20 | 2.01 | 1.91 | 0.76 | 0.85 |
| 0.2                         | 3.78 | 3.61 | 2.18 | 2.12 | 1.06 | 1.10 |
| 0.4                         | 4.10 | 4.02 | 2.76 | 2.89 | 1.92 | 2.02 |
| 0.6                         | 4.50 | 4.55 | 3.95 | 4.06 | 3.18 | 3.23 |
| 0.8                         | 5.08 | 5.23 | 5.72 | 5.75 | 4.42 | 4.31 |
| 1.0                         | 6.00 | 5.96 | 7.93 | 8.10 | 6.58 | 6.55 |
| 1.2                         | 6.49 | 6.44 | 10.22| 10.31| 8.28 | 8.62 |
| 1.4                         | 7.42 | 7.39 | 12.54| 12.73| 12.72| 12.81|

Table B24  Moisture Diffusivities, D
of Cassava at 65°C, for 106 min. Drying Time at Three RHs

| Moisture Content gm./gm. d.b. | Diffusivity x 10^6, cm²/sec. |
|                             | 60% RH |         | 30% RH |         | 10% RH |
|                             | I   | II  | I   | II  | I   | II  |
| 0.1                         | 0.95 | 0.85 | 2.98 | 3.05 | 4.71 | 4.60 |
| 0.2                         | 1.15 | 1.10 | 3.33 | 3.28 | 4.92 | 4.93 |
| 0.4                         | 2.10 | 2.02 | 3.72 | 3.62 | 5.29 | 5.72 |
| 0.6                         | 3.21 | 3.23 | 4.69 | 4.74 | 6.24 | 6.92 |
| 0.8                         | 4.65 | 4.31 | 6.30 | 6.35 | 8.73 | 8.84 |
| 1.0                         | 6.42 | 6.55 | 9.22 | 9.10 | 11.80| 11.31|
| 1.4                         | 11.97| 12.81| 15.28| 15.42|       |       |
Table B25  Temperature Profile - History of Cassava at 55°C

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* distance from surface
  Point 1 - 0.2 cm
  Point 2 - 0.5 cm
  Point 3 - 0.8 cm

| 100 | 47 | 47 | 47 |
| 120 | 47 | 47.5 | 47.5 |
| 140 | 47 | 47.5 | 47.6 |
| 160 | 47 | 47.5 | 47.6 |
| 180 | 47.2 | 47.5 | 47.6 |
Table B26 Temperature Profile - History of Cassava at 65°C

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Table B27: Temperature Profile - History of Cassava at 75°C
Table B28  Temperature Profile - History for Potato at 55°C

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LIST OF REFERENCES


