

EFFECT OF MICROWAVE DRYING ON PAPER PROPERTIES

by

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ABSTRACT

Effects of microwave drying on the mechanical and optical properties of handsheets made from kraft and chemi-thermomechanical pulps were studied experimentally. The quality of paper dried in a microwave field of 2450 MHz is compared with that of paper dried by conventional method under standard conditions. Key physical properties measured include burst index, density, tear index, breaking length, zero-span tensile strength, double fold, STFI compressibility and optical properties include brightness, opacity and scattering coefficient. All properties were found to be either enhanced or at the same level as those obtained under standard conditions. Furthermore, it is suggested that microwave drying could replace the conventional drying method in the standard testing of pulp and paper samples for quality control purposes.

RÉSUMÉ

Les effets du séchage par micro-ondes sur les propriétés mécaniques et optiques de feuilles de papier fabriquées à partir des pâtes Kraft et chimico-thermomécanique ont été étudiés expérimentalement. La qualité du papier séché dans un champ de micro-ondes de 2 450 MHz est comparée avec celle du papier séché par la méthode conventionnelle sous des conditions standard. Les propriétés physiques clés mesurées incluent, l'indice d'éclatement, la densité, l'indice de déchirure, la longueur de rupture, la résistance à la traction à mâchoires jointes, le pli double et la compressibilité STFI. Les propriétés optiques incluent, la blancheur, l'opacité et le coefficient de diffusion de la lumière. Toutes les propriétés ont été trouvées soit renforcées, soit au même niveau que celles obtenues sous les conditions standard. En plus, il a été suggéré que le séchage par micro-ondes peut remplacer la méthode de séchage conventionnel dans les essais standard sur des échantillons de pâte et papier pour fins de contrôle de la qualité.

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CHAPTER 1

INTRODUCTION

1. INTRODUCTION

1.1. REVIEW OF DEVELOPMENT OF MICROWAVE DRYING

Increasing interest is being shown in recent years in the application of electromagnetic drying techniques for paper/ paperboard drying. These drying techniques are being considered alternative drying methods because of their high drying rates, moisture-selective nature, energy efficiency, compactness and ease of control

Major constraints to today's papermaking technology requiring manufacturing of more uniform product are related to product moisture. The objectives are to achieve optimum quality at higher machine speeds and to improve the economics of paper production. Use of microwaves in selected areas of papermaking and converting has the potential of better control over the water removal process, resulting in improved moisture levelling, quality and profitability.

The electromagnetic methods of drying are not new and were first tried in the paper industry 20-30 years ago. Microwave drying is one of the most well known industrial applications of microwave energy since 1966 (92). Many patents have been issued since then on applicators of microwaves. Goerz and Jolly developed an energy balance and economics model in 1967 (30) based on a hypothetical 300 ton-per-day paper machine. The comparison was made by substituting conventional energy with microwave energy. This model concluded that, while the estimated capital investment is somewhat higher for microwave equipment, the overall operating costs are lower for microwave dryers than conventional steam dryers. In areas with less than 15-20% moisture microwave drying is more efficient than the conventional process and yields better moisture levelling. They also calculated a reduction in the total length of the paper machine of about 30% with microwave dryers.

Hankin, Leidith and Stephenson (32) tested a 30 kW microwave paper dryer on a 24-inch wide pilot paper machine as early as 1970. They concluded that moisture levelling depends on many factors such as the type of stock, basis weight as well as electrical properties of white water. Fig 1.1 gives a block diagram of experimental equipment used by Hankin et al. On the basis of a cost model they concluded that use of microwaves for paper drying is technically and economically feasible.

Andersson et al (3) in 1972 tried a one-sided microwave applicator on Swedish Forest Products Research Laboratory's (STFI) 1 meter wide paper machine while making newsprint, and concluded that the efficiency of conversion of microwave energy to thermal energy in paper web is a function of the moisture content of web e.g. the efficiency increased from about 40% at 8% moisture to 80% at 20% moisture. They noted that the distance between the applicator surface and the paper web must be small (< 0.5 mm) and recondensation must be prevented by use of suitable ventilation. Based on economic calculations they concluded that the use of microwave drying for profile levelling can be a profitable alternative.

Metaxas and Merendith (61) have discussed the advantages of electromagnetic energy, specifically noting that heat transport does not limit rate of drying as it does in conventional processes. It can also increase the internal water flow and filtration flow driven by the total pressure gradient.

Microwave heating technology has also been applied in other industries to take advantage of its various features. According to Pendergrass et al (72), use of microwave drying can enhance the uniformity of deposition of dyes and finishes in textile and other fibrous materials. The conventional hot air drying process leaves high dye concentration on the surface. Takahashi, Vasishth and Cote (87), measured resin distribution in overlay paper dried at various drying temperatures.

under convection heating and microwave heating and found that papers dried using convection drying were starved internally for polymers, most of the polymer having migrated to the surface presumably during the drying process. Thomas and Flink (89) found that microwave drying can be used to dry water soaked books without any noticeable changes in paper quality and rapid drying avoids mould growth. Minami and Preston (63) impregnated paper on a pilot coater with a water soluble phenol formaldehyde resin and dried it by conventional hot air drying as well as using microwaves. They concluded that the microwave-dried paper had a more uniform resin concentration distribution across the sheet thickness and were superior in internal bond strength and in surface abrasion resistance. There are a number of microwave drying systems for drying coatings on plastic and paper e.g. drying of silver halide on photographic film. Microwaves are also used to dry pasta products. Use of microwaves not only reduces drying time from eight hours to one hour but also gives a better product with lower bacterial count. Onion drying using microwaves leads to a 90 % reduction in bacterial count while saving 30 % of energy (80).

Recently Roussy, Thiebaut, Bennani and Mouhab developed a simple kinetic model for microwave drying (79). According to them during microwave drying of paper the mass balance is governed by a first-order linear differential equation the constant coefficient of which is linearly related to the square of the instantaneous intensity of the electrical field inside the paper web. They emphasize that during microwave drying of paper much cool air is needed to evacuate the water to avoid condensation. Accad & Schmidt (1) did model studies of evaporation under combined microwave and convection heating and concluded that a trade off is possible between the microwave power density and the air temperature to achieve comparable evaporation rates. High evaporation rates can be achieved at low input power levels with air at ambient temperatures. They also concluded that for a fixed level of incident microwave power, the instantaneous rate of energy absorption is dependent on the mass of water present and its temperature.

Although several studies reported in the literature deal with some theoretical aspects of such drying methods, little has been reported on the quality aspects. If the application of microwave drying is to expand or to become an integral part of papermaking, there is need for a better definition of the advantages and cost-effectiveness its application will introduce in papermaking. For a new application which requires high capital investment, high operating costs and high risk advantages of moisture levelling and increase in drying capacity are unfortunately of little impact in the paper industry. For one, customers accept a certain degree of moisture non-uniformity. Also the increased production due to the additional microwave drying capacity is more of a consolidation than justification since today many of the paper machines in existence can increase their production capacity by methods less expansive and lower risk than by means of microwave drying.

The application of microwave drying must be made specifically where it introduces benefits not achievable by conventional drying and in such a fashion as to increase the overall drying economy of paper machine rather than production and to improve product quality. So it is imperative that research efforts aimed at determining the commercial viability of new drying technologies investigate product quality and property development as well as energy efficiency and capital costs of such systems.

1.2 CONVENTIONAL DRYING

The multiplicity of cylinder dryers in conventional drying leads to several non-uniformities in paper and the use of other systems such as pocket-ventilation, felt dryers etc. Usually it is necessary to overdry the web down to 1.5-2% moisture content to achieve the desired minimum moisture level in the web and usually rewetting and drying back to 4 to 6% moisture content level follows the overdrying

step. Some studies indicate that higher moisture content is present in the centre of web thickness during a considerable drying period in the machine direction (20). This is an important characteristic inherent to hot surface drying since moisture levels in the center third of the web thickness remain unchanged over the first two-thirds of the dryer section length (28).

Another characteristic worth mentioning is the effect of poor contact between the web and surface. The web is not smooth which leads to reduction in contact area with the metal cylinder surface. As the web becomes drier, the contact area with hot surface decreases. Also the centrifugal force due to rotation lifts the web off the cylinder. Further a falling rate period of drying begins around the last third section of the dryer. Consequently the cost of evaporating 1 kg of moisture in this region is much higher than that in any one of the preceding sections.

In terms of mass transfer, the temperature across the web thickness remains below the boiling point corresponding to ambient pressure for much of drying time so mechanism for water removal from the web is mass transfer driven by the vapor partial pressure gradient.

These drawbacks and specific product requirements have led to development of other drying methods e.g. combined conduction and impingement drying in Yankee dryers, combined impingement and throughflow drying of newsprint, press drying etc. Some new techniques such as high intensity drying, superheated steam drying and impulse drying are under various stages of investigation; none has been commercialized yet.

1.3 MICROWAVE OVEN USABLE PRODUCT DEVELOPMENT

Paper is the second major packaging material in microwave usable food products and with the more and more attention to environment and recycling, use of paper is bound to grow. The trends for the US market show that paper represents second largest dollar share estimated at 37% of overall microwave packaging materials (95).

The primary driving force for growth in the use of microwave containers and the food market is convenience. According to a study by Tubridg "... traditional pressed paperboard containers, which have the appeal of low cost will capture the largest share of unit volume". (95) In addition to paper/paperboard as the base/support material, quality paper consumption will increase due to ease of attractive printing/handling and lower unit costs than plastics. The move towards recycling shall also lead to increased usage of paper/paperboard in microwave usable products packaging.

1.4 MAIN FEATURES OF MICROWAVE DRYING

1.4.1 ADVANTAGES:

Economic Savings:

- Reduction of personnel
- Faster processing
- Immediate warm up of product
- Simple plant layout and work scheduling
- Lower breakage losses
- Lower inventories

Product Quality:

- Less temperature degradation of product
- Product temperature self limiting
- Lower product surface temperature
- Stable product dimensions

- no fluff/lint on paper
- no high tension on web

Working Environment:

- Smaller space required
- Cooler surroundings in the plant

Process Automation:

- Immediate on-off process heat control
- Rapid response to automatic process control

Special Features:

- Selective heating
- Supplement conventional heating
- Cleaner process
- Less variation due to difference in products
(e.g. degree of refining of fibers, additives, basis-weight etc.)

1.4.2 DISADVANTAGES:

- Higher heat losses due to non availability of proper design of applicator.
- Higher cost per unit of energy
- Energy flow not selective to moisture content i.e. extra energy has

to be either recirculated or absorbed in dummy load.

1.4.3 GENERAL DESCRIPTION OF MICROWAVE DRYING

The process of microwave heating consists of dissipating part of the microwave energy flow in material which is generally a lossy dielectric. The microwave energy is generated from either magnetron or klystron which are devices causing electrons to be bunched and released at a set frequency. The energy is transmitted by means of waveguides, which are precisely made rectangular section tubes. The applicator may be of different types. Another type is the multimode cavity which can vary in size from the 500 watt domestic microwave oven to a hundred or more kilowatt devices used in industry.

Two basic advantages of microwave drying are speed and selectivity of drying and quality of final product. The in-depth heating equivalent to volumetric rather than a surface distribution of heat sources, results in uniform and high drying rates. The temperature distribution at a given time in a microwave heated product depends primarily on the dielectric properties of the solid and its specific heat. The thermal conductivities of the constituents may tend to equalize the local temperature variation but often the rate of heating with microwave energy is so high that internal conduction of heat can not keep up with the former. This also reduces the mass-transfer resistance considerably.

Most of the conventional drying processes involve three stages i.e. an initial constant rate or saturated surface drying period, a falling rate period which is mass transfer limited by capillary or diffusion flow and a final, very slow drying period in which both heat and mass transfer are limiting. Microwave energy helps to eliminate above constraints especially during the falling and final drying periods.

1.4.4 PARAMETERS AFFECTING MICROWAVE HEATING

The ability to dry paper with microwaves is affected by the type of equipment being used and the nature of paper. Some of the important parameters are listed below and are described in detail by Schiffmann (80) along with available correlations.

1. Frequency of microwaves
2. Microwave power or speed of heating
3. Basis weight of paper
4. Moisture content of paper
5. Density of paper
6. Temperature of paper
7. Conductivity and specific heat of paper

1.4.5 DIELECTRIC ANISOTROPY OF PAPER

Paper exhibits dielectric anisotropy with respect to the measured dielectric constant (21). In addition to depending on the orientation of the sample, the dielectric constant is a function of the frequency of the applied field, the temperature of the sample, the amount of moisture present in sheet and the apparent density of the sheet.

Although in general the dielectric properties of polymers are very complex, the combined effects of frequency and temperature are often discussed as being the result of actions occurring at a simplified molecular level. Permanent molecular dipoles in the material rotate with the applied field to contribute to the polarization of the material. The dipoles are described by a certain relaxation frequency, f_r , above which they can no longer keep up with the field and thus no longer contribute to the polarization of the material. By assuming that the orientation of dipoles can be represented by two states separated by a potential barrier, application of simple reaction rate theory leads to an equation of the form (21)

$$f_r = B \exp(-H / RT)$$

where H is an activation energy per mole, R is ideal gas constant, and T is the absolute temperature. B is assumed to be constant as it is actually a function of temperature and the activation energy, H .

Dry cellulose has been observed to exhibit four different states of relaxation. The highest frequency relaxation occurs around 10^7 Hz at 20 °C (46). The presence of water in the cellulosic material tends to increase the magnitude of the measured dielectric constant by two mechanisms. Water acts as a plasticizer and shifts the relaxation processes to higher frequencies. Also because water has a permanent dipole moment, it will contribute to the measured dielectric constant by rotating with field. Indeed free water has a very high dielectric constant, being

about 60 at 10^{10} Hz and 20°C . The dielectric constant of dry cellulose is about 4 at this frequency and temperature.

The dielectric anisotropy observed in paper could be associated with the anisotropy of the structure of paper on two different levels. On a molecular level, because of the arrangement of atoms and bonds, the cellulose molecule itself would be expected to exhibit anisotropy. The preferential orientation of fibers also exhibit anisotropy. On the other hand, because fibers are long and slender enough and because paper is an assemblage of fiber and air, anisotropy could be exhibited in paper even if the fibre themselves were isotropic.

1.5 OTHER ELECTROMAGNETIC DRYING PROCESSES

1.5.1 INFRARED DRYING

Infrared radiation occupies the part between the visible light and the radio-waves. Infrared emitters are divided in three groups

- a. Long wave emitters with a wavelength greater than $3\text{ }\mu\text{m}$, and an emitter surface temperature of 700°C or less.
- b. Medium wave emitters (electric or gas) with wavelength between $1.6\text{--}3\text{ }\mu\text{m}$, and surface temperature from 700 to 1500°C
- c. Short wave emitters (only electric) with wavelength of $1.2\text{ }\mu\text{m}$.

Electrical systems with short wave emitters (quartz tubes) have recently been developed for the paper industry. The main difference between these electrical IR dryers is the power density which varies from 60 kW/m^2 to 400 kW/m^2 . In the case of IR, the radiation is derived from a hot surface, the temperature of which is determined by its actual temperature in accordance with

1 the Stefan-Boltzman Law, and the emission factor of material. A higher temperature is known to shift the maximum value on the curve in the direction of shorter wavelengths, while at the same time a larger proportion of the radiated energy is concentrated around the wavelength giving maximum intensity. At higher temperature, the absolute intensity increases at all wavelengths.

Since all heat radiators emit in a specific band width, it is not possible to select radiation sources based on a particular wavelength at which the absorption is high. The governing factor is the interaction between the spectrum from the radiator and the material being heated or dried.

A lower temperature in the radiation source gives higher absorption, which results in better utilization of the radiation energy. The advantage of a higher temperature source, however, is partly that a higher intensity is easier to obtain (which reduces the space requirement) and also the element is more quickly regulated, in the case of thicker grades of paper, the energy penetrates more deeply into the paper and consequently gives more uniform heating. In thinner grades of paper, a reflector can be placed behind the paper to augment the amount of energy absorbed. As the heating of outer surface plays a major role in IR drying so rate of mass transfer is important. Often convective air is used to enhance the drying rates.

1.5.2 RADIO-FREQUENCY (RF) DRYING

Radio frequency drying is a process in which heat is generated within a dielectric material by application of high frequency electrical energy (10-100 MHz). RF generation usually employs a class C oscillator circuit, typically a modified Colpitts circuit based on triode valve, which may be air-cooled or water-cooled depending on the output power of the generator. The actual generator circuit

consists of a power supply transformer, stepping the main voltage up to typically 6000 - 10,000 volts. This is followed by a rectifier and then by an oscillator circuit. Energy is transferred from the generator to the heater by means of transmission lines which ideally are concentric conductors but for short distances may be flat busbars.

1.6 DRYING AND PAPER QUALITY

Kumar and Mujumdar (51) have presented a short bibliographic review on the effects of drying on paper properties. From the diversity of uses of paper, it is clear that the desired paper properties depend upon its end uses e.g. absorbanacy for tissue, diapers, sanitary napkins, table napkins, medical dressings etc; loss factor for capacitor tissue, transformer and cable papers; surface resistivity and stiffness for photocopier and laser printing paper; wet-strength for overlay and other conventional and new-generation wet-strength papers etc. Drying plays a key role in the development or control of most physical and optical properties as discussed by Koran (47).

1.6.1 STRUCTURAL DEVELOPMENT OF PAPER

The wet web which is dried in the dryer section is formed on top or in between moving wires, from a fiber suspension ranging from 0.3 to 1.5 percent concentration. The rheological conditions in the formation of the fiber mat create non-uniform fiber distribution and water drainage in the wire. The alignment and distribution of these fibers is a stochastic problem. Consequently, the optimization of the deposition process, in regard to basis weight and thickness, is a difficult problem to solve. Even if good dispersion exists at the headbox, the flocculant characteristics of the fibers and fines tend to compound the problem.

The sheet is a two dimensional array of fibers. During the formation process no bonding has occurs between fibers. Bonding takes place initially when the web is conveyed by means of felt through a press section. Application of external mechanical pressure to the web brings fibers into contact. For good fiber-fiber bonding a certain moisture content is necessary below which the absence of enough water around the swollen fibers will impair the interdispersing of fibrils. So before entering drying section paper web is loaded with these non-uniformities and any further parameters will accentuate its non-uniformity (9).

There are various kinds of forces which apparently lead to the development of the final strength levels in paper e.g. mechanical entanglement, hydrogen bonds, electrostatic forces and van der walls' forces although it is generally accepted that hydrogen bonds between hydroxyl groups of adjacent cellulose fibers are the most important forces holding fibers together. Hydrogen bonds also exist between cellulose-OH and water-OH groups (11,12).

The development of web structure during drying is accompanied by measurable changes in its physical properties, which have direct bearing on the mechanical, optical and other performance characteristics of the consolidated sheet. In a recent study by Nanko and Ohsawa (65) examined microscopically the drying process of a wet web. They divided the process into the following five stages.

<u>Stage</u>	<u>Solid Content %</u>	<u>Changes</u>
1	- 55	Evaporation of the free water, without any change in the web.
2	55 - 60	Evaporation of free water from lumen and pits. Fiber collapse begins.
3	65 - 70	All of the free water has evaporated, water begins to move out of the fiber wall. First sign of fiber shrinkage is noticed.

- | | | |
|---|---------|---|
| 4 | 70 - 75 | Dewatering of fiber wall continues resulting in the transverse shrinkage of fibers in the unbonded areas. Fiber collapse continues. Longitudinal wrinkles appear on the fiber surface. Formation of the inter-fiber bonding begins. |
| 5 | 75-95 | Dewatering of the fiber wall continues resulting in the transversal shrinkage at fiber crossings regions and in unbonded areas. Longitudinal wrinkles become more distinctive. As a result hydrogen bonding takes place in the fiber to fiber bond areas. When the fiber-wall loses most of the water, the fiber shrinks transversely even at the bonded regions. |

Note that the Nanko and Oshwa study was conducted at very low drying rates under ambient conditions and their observations may not necessarily apply to paper dried at higher temperatures and higher rates on conventional machines. For example, it is known that at higher dryness the tensile strength is higher for paper dried at lower temperatures. The relative bonded area of fibers in paper dried at higher temperatures is lower than that in paper dried under milder conditions. It has also been reported that high drying temperatures increase the overall pore volume, hydrogen bonds and electrostatic forces. Htun (36) found that the greatest strain-to-failure is achieved by permitting free drying between 35 and 60 % solids content, however the greatest loss in strain-to-failure also occurs if the sheet is under restraint in this same range of solids content.

Paper is made from different types of fibers, including softwood, hardwood, grass, straw, bagasse etc. These fibers differ in structure and chemical composition. For example, hardwoods contain more cellulose, less extractives and lignin than softwood. There are various other basic differences in these components. For example the degree of crystallization of cellulose, hemicellulose and lignin are different. This leads to differences in individual fibers strength,

length/ width ratio as well as to different levels of development of hydrogen and other types of bondings and hence affecting the final paper strength (12).

Similarly the hemicelluloses also have strong influence on development of paper structure and properties. Generally higher amount of hemicelluloses lead to higher physical strength and internal bonding. While lignin has been shown to form bond with cellulose they are much weaker than those formed by hemicellulose (31). Although lignin does not participate as much as hemicellulose in bond formation, there is evidence that it still plays an important role by sealing the bonds formed by cellulose and hemicellulose against moisture if the sheet has been heated to a high enough temperature to initiate lignin flow within the sheet.

Several theories like molecular, structural and phenomenological are reported in literature to describe the rheology of paper. Molecular theories treat paper as an isotropic continuous network of hydrogen bonds in three dimensions. They have been successful in describing the effects of temperature and moisture content on Young's modulus (76).

Structure theories correlate the mechanical properties of paper with the geometry and properties of the fibers making up the paper. An important parameter in structural theory is the relative bonded area which has been found to correlate with many mechanical properties of paper. Structural theories have been unsuccessful in describing the effects of moisture and temperature on the mechanical properties of paper, although they are able to explain differences between machine and cross-machine direction moduli (76). On the other hand phenomenological theories pay little attention to the precise physical or molecular structure of the material by mechanistic models, such as springs and dashpots. Phenomenological theories are mainly used in the study of time dependent mechanical processes.

1.6.2 MAJOR CHANGES IN CELLULOSE DURING DRYING

Degree of polymerization (DP) is the basic parameter which defines the effect of physical or chemical changes in cellulose or other polymers. This is generally measured by CED viscosity or by decrease in tensile strength. Even though cellulose is quite a stable polymer, different processing parameters can have lasting influence on its properties. Cellulose is degraded for example, if it is heated for a prolonged period over 100 °C, as well as by the hydrolyzing effects of any contained or absorbed acids (12). Hydrolysis and oxidation are two of the main chemical reactions occurring during drying which can cause scission in cellulose and hemicellulose material hence reduction in DP (2,5).

Hydrolysis is a reaction where a compound is split into other compounds by taking up the elements of H₂O. The glycoside linkage in polysaccharides can be broken especially in acid media. The hydrolysis results in an increase in the reducing power of the degraded product (67). Linkages in the amorphous part of cellulose are much more readily accessible than in crystalline cellulose and are hydrolyzed at a much higher rate. The hydrolysis of carbohydrate structures is enhanced by the presence of oxidized groups such as aldehydes and carboxyls which result from oxidation reactions. The combination of oxidative and hydrolytic reactions thus have a synergistic effect. Increased temperature and pressure tend to accelerate the hydrolysis rate. Cellulose hydrolysis and oxidation can produce glycolic acid which can react with glucose to give reversion products. Degradation products of cellulose can cause colour reversion (77). Further, paper additives such as alum, acids may serve as initiators for the process of hydrolysis. Much research is needed in these areas.

The thermal instability of cellulose and carbohydrate structures can also lead to depolymerization. Thermal degradation usually occurs above 200°C and increases with increasing temperature, although extended exposure at lower

temperatures under certain conditions may also lead to degradation. If the thermal treatment is severe enough pyrolysis and charring begin with a number of breakdown products. These include acetic and formic acids, anhydro-sugars, tars, gases and low molecular weight products, water, char, CO_2 and CO . Heating to the point where the water of constitution is lost results in improved dimensional stability and wet strength at the expense of embrittlement and permanent loss of dry strength. The heating time required to achieve a certain degree of stabilization increases logarithmically with decrease in temperature (83). The combination of heat and moisture produces different effects than dry heat alone. Stamm (82) reported that heating at a relative humidity of 95% or in steam reduces the activation energy for degradation to half the value for dry heat, while increasing the rate of reaction. Crosslinking involves the formation of bonds between cellulose chains in and between fibers. Treatments with formaldehyde in the presence of an acid catalyst have been used commercially to impart dimensional stability to paper (50).

Physical processes lead to development of hydrogen bonding or other kinds of bonds between fiber surfaces and fibrils. Alignment of the fibrils in the plane of the sheet and their bonding together with those of adjacent fibers provide the web with considerable strength. These parameters are usually represented in terms of the relative bonded area (RBA) which is the ratio between the actually bonded surface of fibers in contact in a sheet of paper and their total external surface (12). The intrinsic cohesiveness of fibers depends on the area concentration of cellulose and hemi-cellulose as well as fibrils. This concentration is decreased by drying and increased by mechanical treatment of fibers (12). These physical processes in addition to bonding may also develop stresses in paper. In case of high yield pulps with more than 75% yield, lignin plays an active role in bonding development as it prevents adequate fibrillation and causes the fibers to be relatively inflexible (12). The action of restraining the sheet during drying has a profound effect on the mechanical properties and is thought to equalize the load born by each fibrous element, making the sheet more uniform. Sheets dried under tension generally

have higher tensile strength and elastic modulus but lower extensibility than that dried without restraint. Burst and strength in the thickness direction are generally decreased by drying under tension.

The glass transition temperature on the other hand plays a key role in property development especially in high-yield pulp. This becomes more important for raw-materials having higher percentage of amorphous cellulose or for high yield pulps. The ultimate effect of the glass transition temperature also depends on various parameters such as moisture content, temperature cycle and distribution of the amorphous substrates and their percentage. The modified amorphous materials such as kraft lignin have different glass transition temperatures e.g. kraft lignin may have glass transition temperature as low as 80°C when wet to 200°C when dry (5). Hemicelluloses have softening temperatures ranging from 180°C to about 50°C when wet (31). On the other hand the degree of crystallinity plays a key role in strength development. Changes in crystallinity of cellulose due to disordering regions, increased degree of perfection of ordered regions, or transformations to different polymers can be induced by changing the moisture content, heating, mechanical action, chain scission, and applied tension during drying. Moisture and chain scission permit the cellulose chains to become more mobile and realign themselves in a more orderly fashion, thus increasing crystallinity (31).

1.6.3 SHRINKAGE OF SHEET OF PAPER DURING DRYING

During drying, a sheet of paper shrinks mainly in the solids content range 60-85% (47). The extent of the shrinkage depends, amongst other factors, on the degree of swelling of the fibers, the orientation of the fibers and whether or not paper is allowed to shrink freely. The change in length is higher with chemical pulp than with mechanical pulp because of the different degree of swelling. There is more shrinkage on drying highly beaten pulps. If the pulp is beaten at a high concentration, further drying shrinkage is obtained through compression of the

fibers during processing. The shrinkage is more in cross direction of paper. Shrinkage can be prevented by tensioning a sheet of paper during drying. The tension that is required to avoid shrinkage increases as drying proceeds (33); it is defined as:

$$\text{Drying tension} = \text{drying force} / (\text{specimen width} \times \text{grammage})$$

The final drying tension depends on the degree of shrinkage that would occur if the sheet were dried freely. For example, an unbeaten bleached sulphate pulp develops a drying tension of approximately 5 kNm/kg, whilst the same pulp beaten to 275 °CSF develops almost three times this drying tension. Higher drying tensions occur in machine direction during restrained drying. The drying tension depends to a great extent on the drying history of paper, temperature, humidity and drying rate. By selecting a suitable drying strategy it is possible to control the drying tensions and paper properties. Light scattering is also affected by shrinkage during drying due to changes in available surface area for reflection.

1.7 OBJECTIVES OF PRESENT WORK

The basic objective of the present research was to determine the influence of microwave drying on the key physical and optical properties of handsheets of paper as compared to those due to conventional cylinder drying at the laboratory scale. The objectives included an attempt to develop an alternative method to pulp handsheets testing as well as to define a way to develop the standards for paper/paperboard products used in microwave ovens. The objective of this preliminary study is to identify possible product quality benefits of microwave drying of paper.

The kinetic aspects of the drying process were not studied. Since all the previous work on dielectric drying of paper web deals almost exclusively with the heat and mass transfer aspects this study was conducted to assess the quality aspects of the process.

CHAPTER 2

EXPERIMENTAL APPARATUS AND PROCEDURE

2. EXPERIMENTAL APPARATUS AND PROCEDURE

2.1 INTRODUCTION

To study the properties of paper dried in microwaves, the fundamental choices required are the type of equipment and conditions for drying, the choice of pulp for the paper to be dried and the physical as well as optical properties to be measured. The choice of equipment should permit the most favourable microwave field frequency i.e. 2450 MHz for drying as this is the major microwave frequency considered for industrial use. The Sharp domestic oven (model 8465) met these conditions well. A teflon ring was developed to hold the handsheet in a microwave-conformable form as well as for ease in use in conventional handsheet making and testing. Comparative physical and optical properties measurements were made for the same type of handsheets dried in the cylinder dryer of a dynamic sheet former, which is a very good laboratory-equivalent of the actual industrial cylinder dryers. Further, to provide a base for comparison the physical and optical properties of similar handsheets were dried in ambient air under the conditions of CPPA standard C4.

To assess the possible effect of the lignin content on the microwave-dried paper, handsheets made of bleached kraft pulp with negligible lignin content as well as bleached chemi-thermomechanical pulp with comparatively higher lignin content were used. Bleached pulps with high brightness (82%) were taken to be able to measure the optical properties with good resolution. This study is unique in terms of the range of properties measured. A wide range of useful physical and optical properties were measured to develop micro as well as macro perspective of the effect of the drying method on the final product quality. For example, the

physical measured properties included not only the commonly used properties like bulk, tensile, and tear but also such important properties like zero-span strength, compressibility, double fold etc. Optical properties studied were not limited to brightness but also included other important properties like print opacity, scattering coefficient and colour parameters.

The design and operation of the laboratory method of microwave drying, the design of a special ring holder for the handsheets placed in a microwave oven, the experimental program and the physical as well as optical tests are detailed below.

As described in Chapter 1, the mechanism of drying and its relation to inter-fibre bonding, bond strength, individual fibre strength is quite complicated. Several physical tests are required to actually be able to predict the effect. Some of the key properties measured in this work and their relevance for are described in this chapter.

The so-called burst test is one of the most widely used tests; it basically indicates the resistance of the sheet to rupture. This complex property is the outcome of several factors such as the relative bonded area, bond strength, individual fibre strength and fiber length. Any increase in these parameters will generally increase the bursting strength. These parameters are difficult to control individually during a given drying scheme. As the bursting strength depends on type, proportion, preparation and amount of fibres present, it typically increases based on increase in basis-weight, sheet densification and degree of pulp refining. Burst strength also depends on the sheet formation and shows two-sidedness in paper. This test is sensitive to moisture. So this test combined with density can be an important test to reveal the effect of drying on the relative bonded area, bond strength and individual fibre strength.

The tensile strength is expressed in the paper industry as stress, which is

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The tensile strength is expressed in the paper industry as stress, which is

measured as force per unit width. The most common way of representing this is in the form of breaking length which is usually defined as the length of the paper strip whose weight is equivalent to the force that would break it. Other associated tests with this measurement are Young's modulus, and Tensile Energy Absorption (TEA). TEA is represented by the area under the load-elongation curve. If TEA is divided by thickness, it yields energy per unit volume. Developed stresses during drying can have direct influence on this property, so this is a good parameter to measure the effect of stresses developed on paper properties during drying.

The zero-span tensile strength can be used as a measure of the individual fibre strength. This test implicitly assumes that the fibres fail together regardless of their state of stress (60). Stresses developed during drying or e.g. reduction in DP during drying may have direct implications on the results of zero-span strength.

The edgewise compression strength is one of the most useful properties to predict the performance of paper/ paperboard in packaging performance during shipping and stacking. This test explains the compressive failure which is the major failure phenomenon during such conditions. Paper may have high tensile strength but may fail in good packaging if its compressive strength is low. Compressive strength is also important from the point of view of folding, bending and creasing of paper/ paperboard. The STFI short span test was used to measure this property.

The tear strength of paper is one of the important properties from the point of view of machine runnability. The Elmendorf tearing test, which involves out-of-plane loading has been used to test the tearing strength as it is widely used. Double-fold is one of the most critical properties and is usually the best indicator of the effect of drying parameters on paper properties. This property takes into account the bonding level, individual fibre strength, fibre flexibility and others.

Optical properties measured in this work included brightness, opacity, scattering coefficient and colour parameters. Bleached sheets were taken to observe the effects of drying with more precision. The scattering coefficient was included as it is one of the key properties which gives information about bonding and is very useful when analyzing and comparing the sheets dried under different conditions. It also gives an inverse estimate of the internal bonding of the fibres in a sheet of paper, in which the fraction bonded is called " Relative Bonded Area (RBA) " with certain limitations

2.2 EXPERIMENTAL EQUIPMENT

2.2.1 MICROWAVE DRYING

As 2450 Mhz is the major frequency on which industrial scale microwave generators are available it was decided to look for microwave applicators at this frequency. Interestingly the domestic microwave oven fulfils this requirement very well. The uniformity of the microwave field in the oven is important for the purpose of this study. There are various types of control systems in modern microwave ovens e.g. sensing parameters like temperature of product, weight of product, moisture evaporated from load, temperature of exit air, aromatic gases given off by the load, colour change, weight loss, cavity field change and audible sounds in the cavity etc. As the wavelength of the microwave field is 12.24 cm, which is comparable to the oven cavity dimensions, standing wave patterns giving very nonuniform field distribution can occur unless some modifications are made. To ensure better uniformity, the following features were considered in selecting the microwave oven.

- a. Rotating mechanism at the point where the microwave energy enters the cavity, causing the mode pattern to occupy different positions with time and providing for more uniform heating of product.
- b. Rotating platform, which causes the product to be moved through regions of high and low microwave heating so it will be more uniformly heated over a period of time.
- c. Larger size of cavity. Because larger the cavity, the larger the number of mode patterns that can exist within the cavity and the easier it is to achieve uniform heating.

Selection of Microwave Oven

The following table lists some of the ways in which microwave ovens differ. In fact there are many more differences. For example, the pulse times used to vary power differ for different manufactures and/or for different modes. Some ovens use only a single power setting while others may have as many as 99 settings. Some of the parameters may not described by the manufacturer in the catalog.

-
- a. Power output : 400 -700 watts
 - b. Cavity Size : 0.4 to 1.8 cubic feet
 - c. Microwave feed system. mode stirrer, rotating antenna, rotating waveguide
 - d. Location of microwave input into the oven : at top of oven cavity or both top and bottom of the oven or the sides of the oven.
 - e. Cavity wall construction: stainless steel or painted cold rolled steel
 - f. Presence or absence of a turn table
 - g. Microwave only or microwave-convection or microwave-convection with a browning element.
-

Considering the available models in the market and the results of present study, the choice was limited to those made by Toshiba, Panasonic, and Sharp. The SHARP model 8465 domestic microwave oven was chosen as a compromise between oven specifications desired and cost. This oven has the following features.

- a. microwave , microwave-convection ,convection
- b. turntable
- c. cooking power 700 watt
- d. heater, 1500 watt
- e. timer
- f. cavity size 1.5 cubic feet
- g. microwave supply :top
- h. cavity wall : stainless steel

OVEN STANDARDIZATION

The microwave oven standardization procedure was divided into two parts:

- a. To select the parameters and settings to characterize and standardize the oven performance during the whole range of experimentation.
- b. To evaluate the uniformity of microwave heating especially in the area of experimentation where the handsheet is placed on the turntable.

Characterization of oven

Characterization of the microwave oven involved a series of measurements to determine its performance characteristics so as to enable selection of appropriate settings for the drying experiments. These measurements included:

- a) Rise in temperature vs. time at high, medium, medium-high levels of the oven
- b) Rise in temperature vs. volume
- c) Power output in watts vs load size in ml of water.
- d) Volume of water evaporated vs time
- e) Magnetron availability vs application level.

From the experiments done and results obtained several important points came up which were taken into consideration in drying experiments with paper. These characteristics were checked from time to time to test the reproducibility of the results. These experiments were repeated for 5 times.

Uniformity

A uniform microwave application over the handsheet is necessary to evaluate paper quality. The oven selected has a rotating table to make the microwave-heating uniform. A large cavity was selected to ensure better uniformity. Since the objects (loads) in the oven absorb and redistribute the incident microwave energy, the size of load can affect the uniformity of field in the oven. Following are some of the experiments conducted to access the uniformity of microwave oven in the desired region. The desired region was defined as the size of the paper handsheet, in a central circular area 20 cm in diameter. The experiments were designed to examine both macro and micro scale uniformities.

a. **Macro (overall) uniformity:** This was assessed by using one liter of distilled water in a glass-beaker of 2-litre capacity as the load. The temperature rise of this load was measured over a period of 10 minutes at one minute interval.

b. **Micro (field) uniformity:** Micro level uniformity was assessed by positioning a 100 ml glass beaker in a circle of 22.5 cm, with center of the beaker approximately on the periphery of circle. The temperature rise was measured at

every half minute interval over a period of 3 minutes. Furthermore, accuracy was assessed by measuring the volume of distilled water remained after 5 minutes of heating in the microwave oven.

The above tests were conducted at high, medium-high and medium microwave levels. The tests at medium-high microwave level were repeated at least four times and were found to be reproducible within 3%.

RING HOLDER DESIGN

Standard metal handsheet holding rings can not be used in a microwave oven while standard plastic rings are so light that during drying the sheet curled and induced excessive deformation. As the teflon plate was developed for pressing, similarly teflon plate was modified to use in the rings. The edges of the teflon plate were tapered and bevelled to give better and fast grip to the sheets.

"QuickGrip" rings were fabricated from polyethylene rings after testing them in the microwave oven. The receptive male and female edges of the QuickGrip rings were also modified to hold the sheet properly and uniformly. To provide uniform grip and to dry the sheets under tension, three QuickScrews per ringset were custom fabricated. These screws have many important features e.g. they are made of a microwave-transparent material; they maintain their threads for long time after repeated use in the microwave oven and they grip on the rings uniformly. Further, they were lubricated to provide better thread life and ease of opening and closing. The modular design of QuickGrip rings can be further extended to accommodate 6 rings per stack.

ASSESSMENT OF OPTIMUM TIME OF HANDSHEET DRYING IN MICROWAVE OVEN

To access the time required to dry handsheets of 60 g/m^2 , standard procedure of weight loss was used. Following sets of hand sheets were used to determine the optimum time to achieve around 95 % dryness after microwave drying:

- a. 60 g/m^2 handsheets made of repulped blotters : 10 sheets
- b. 60 g/m^2 handsheets made of 690 ml CSF kraft pulp: 5 sheets
- c. 60 g/m^2 handsheets made of 445 ml CSF kraft pulp: 5 sheets
- d. 60 g/m^2 handsheets made of 300 ml CSF kraft pulp: 5 sheets
- e. 60 g/m^2 handsheets made of 95 ml CSF kraft pulp : 5 sheets
- f. 60 g/m^2 handsheets made of 300 ml CSF CTMP : 6 sheets

In addition to the above 36 sheets, 120 g/m^2 sheets were tested separately. Testing was done with 4 - 6 dried sheets per batch.

2.2.2 CYLINDER DRYING

To simulate conventional cylinder drying, several alternatives such as oven drying under tension, hot air impingement, laboratory dryer and dynamic sheet former were considered. After considering the various parameters as well as previous studies, it was decided that the dryer of the dynamic sheet former is the optimum alternative because it not only simulates the temperature conditions encountered in practice but also the web compressive and tension forces normally encountered in the cylinder dryer under the felt. The results were found to be repeatable. Also the temperature and felt tension could be controlled well. A teflon sheet was used as the carrier for the hand-sheets. Usually 3 to 4 sheets were

dried simultaneously. To determine the time to dry the sheet to about 95 % the standard weight loss technique was used.

2.2.3 AMBIENT AIR (RING) DRYING

CPPA method C4 was employed to dry the handsheets to 6% w/w at room temperature. The press plates carrying the sheets were clamped in perforated drying rings and dried down to an equilibrium moisture content to room temperature in a conditioned room. In this drying process shrinkage takes place in the thickness direction only; these sheets were taken as the base cases. Some literature studies show that for the same stock, machine formed paper had a 24% lower burst due to poor formation and another 15% loss in burst strength because the mill sheet is dried under tension (12).

2.3 TEMPERATURE MEASUREMENT

The temperature measurements were made using a fibre optic probe and insulated probes described below. Thick sheets (600 g/m^2) were used for temperature measurement with probes inserted at various locations in the sheet or in between two sheets. Thermocouples (48) were passed through backwall of the oven, for continuous temperature measurements of handsheets being dried. Fig (2.3), shows the schematic of the grounded junction thermocouple (48). This is made from teflon coated chromel-alumel wires (no. 30) terminating inside a aluminium tube. The tip of the tube is shaped to obtain a spherical form, leaving a small orifice for the thermocouple junction. This orifice is filled with aluminium solder to seal the tube tip, prevent microwave energy from interacting with junction and ensure good electrical and thermal contact. The thermocouple wires were further segregated from the aluminium tube wall by a teflon sleeve which also served as a thermal insulator.

Beyond the rigid aluminium shielded tube, the thermocouple wires were contained within a copper-nickel braid aluminium soldered to the tube and passed out of the cavity via a microwave tight swagelock fitting which also served to obtain an electrically reliable ground for the thermocouple. The time-temperature response of this thermocouple was similar to that of a common fluoroptic sensor (Luxtron 1000 A), a more conventional but expensive method for measuring temperature in a microwave environment (48).

Fibre optic techniques are good for microwave use because all materials in these probes are by design good electrical insulators and the probes are transparent to microwave radiation. These probes are constructed from optical fibres that carry sensors at their tips and operate on optical principles. They do not alter the microwave field patterns, and the measurements are not perturbed by microwave fields. The temperatures were monitored at a regular intervals of 10 to 20 second during drying of handsheets of paperboard.

2.4 PHYSICAL AND OPTICAL PROPERTIES

2.4.1 BULK/ DENSITY

The specific volume is the inverse of density which is usually referred to as Sheet Bulk, expressed in cm^3/g . The density is represented in g/cm^3 . Sheet calliper is simply the measure of paper thickness in mm while the basis weight expresses the weight of paper per unit surface, g/m^2 . All of these parameters are inter-related. The density and bulk depends not only on the physical factors like pressure to which the sheet is subjected during pressing but also on the extent and strength of bonding in the paper amongst fibres.

Increase in bonding due to any reason e.g. increase in fibre flexibility that may occur because of lumen collapse is often accompanied by increase in density

or reduction in bulk. Usually bulk has an inverse relationship with most of the physical strength properties. The caliper or thickness of paper was measured according to CPPA method D4, at least 5 locations (usually 10 locations) per sheet using a 16.5 mm diameter micrometer. The results are reported as density which is the basis weight divided by the average caliper.

2.4.2 PHYSICAL STRENGTH PROPERTIES

The physical strength measurements involved several properties. A short description of the major ones is given below.

Bursting Strength

This was determined according to CPPA Standard C8. Two values of bursting strength were determined for a 3.25 cm diameter section of each alternate handsheet. The results are reported as "Burst Index", i.e. bursting strength divided by the basis weight of the sheet.

Tear Strength

The tear strength was determined on two-ply samples, 63 mm x 50 mm, obtained from the same alternate handsheet using CPPA Standard D9. Elmendorf tear tester (made by Thwing Albert company) was used for the test and the results are reported as "Tear Index", i.e. tear strength normalized by the paper basis weight. One value of the tear index was determined for each alternate handsheet.

Zero-Span Tensile Strength

The zero-span tensile test was based on a CPPA standard, using PULMAC standard tester. 15 mm wide strips with a clamp pressure of 80 psi were used. At least two tests were made every second handsheet. The results are reported

as "Zero-Span Breaking Length", which is obtained by the normalizing measured zero-span tensile strength to sheet basis weight.

Table 2.1 lists the methods used to measure other properties studied in this work.

2.4.3 OPTICAL PROPERTIES

The optical property measurements were carried out using the Technibrite TB-1C. Each set of measurements included optical properties such as Standard Brightness, Print and Tappi Opacity, Scattering and Absorption coefficient, dominant wave length and color parameters such as L^* , a^* , b^* , X, Y, Z as well as $R(X)$, $R(Y)$ and $R(Z)$. The Technibrite instrument was standardized using standard samples from the Institute of Paper Science & Technology and employing TAPPI standard method.

The standard brightness is measured at 457 nanometre wavelength and is defined as the ratio of the reflectance of an opaque pad of test sheets compared to reflectance of a thick pure white magnesium oxide surface under the standard conditions. The ability of the interior of the test sheet to scatter light is measured in terms of the scattering coefficient, while its ability to absorb the light energy is measured in terms of the absorption coefficient. In order to ensure the opaqueness of pads at least 8-12 sheets were used in measurements.

2.4.5 MISCELLANEOUS PROPERTIES

In addition to the above properties, the porosity of the handsheets was measured with a Bendsten porosity tester according to TAPPI standard RC-303 at 150 mm water pressure difference in cm^3/min and by standard Parker-print surf porosity tester. Both of these methods are based on air flow rate in a constant

area of paper under standard pressure difference. Parker print-surf results are useful in defining the printing characteristics of paper.

Free shrinkage of sheets at different freeness levels was measured by the marking technique. At least 25 readings (usually 40 readings) per test were employed to obtain the desired level of accuracy.

2.5 PREPARATION OF HANDSHEETS

Handsheets for drying were made according to CPPA Standard C4. As the kraft pulp was obtained directly from a mill (Kruger Inc., Quebec) at high consistency, it was disintegrated in ordinary water in a laboratory disintegrator. The CTMP pulp, which was in the form of dry sheets, was disintegrated with water at 80-90°C and then immediately diluted to a concentration of about 0.3% in normal water.

Further the pulps were diluted to about 0.15% concentration to permit an accurate measurement of stock for individual sheets. In case of PFI treated pulps, the same procedure was followed after refining at 30% consistency. The drainage time accuracy was checked during sheet formation to obtain as uniform a sheet as possible.

A standard British Sheet Former was used to make handsheets with 60 grams per square meter basis weight. Standard couching was done while making handsheets by covering wet handsheet with two dry blotters, then a brass couch plate and give the combination five rolls with the roller and lift off the combination of sheet blotters and plate in a way similar to that of opening the cover of a book. In the case of highly refined stock, however, a single blotter was used to ease the

lifting of the sheet. The two stage pressing was carried out in a standard machine under standard conditions and timings. Teflon plates were used for ease of removal of the handsheets after pressing without causing any stretch, deformation or fibre-pickup. This idea was developed in present study and will be of practical interest in similar studies.

The average moisture content of more than 40 handsheets after pressing was found to be more uniform than that of those formed with stainless steel. The sheets to be dried under standard conditions were transferred immediately to steel plates and placed in a controlled atmosphere room while the sheets to be dried under the laboratory cylinder were transferred to the teflon sheet and dried in the standard cycle of the dynamic sheet former. The sheets to be dried in the microwave oven were immediately packed in 100% barrier thick plastic envelopes and placed in a cold room to be dried within one week.

2.6 EXPERIMENTAL CONDITIONS

2.6.1 RAW MATERIALS

Two pulps were chosen for the experimental study (Table 2.2). Conventional bleached softwood kraft pulp and a bleached high yield pulp made of hardwoods by a chemi-thermomechanical process.

The bleached kraft pulp was made up mostly of spruce wood and was supplied by Kruger Inc. Trois-Rivieres, Quebec. The pulp was refined in a PFI mill by the standard method. The pulp had initial CSF of 690 ml and was refined further to three CSF levels i.e. 445 ml (8000 rev), 315 ml (13.5 K rev) and 95 ml (36 K rev). High yield pulp from Aspen with an initial CSF of 510 ml was refined to 315 ml CSF at 8500 PFI mill revolutions.

2.6.2 DRYING CONDITIONS

The following experimental conditions were employed for the three drying methods used in this study.

Microwave drying

Microwave application mode: Medium high

Drying time: 5 minutes for 60 g/m², 7 minutes for 120 g/m²

Cylinder drying

Drying temperature: 100°C

Drying time : 5.5 minutes for 60 g/m², 10 minutes for 120 g/m²

Ring Drying

24 hours at 25°C, 50% RH

Conditioning

Conditioning time for microwave and cylinder dried sheets:

24 hours at 25°C, 50% RH

2.7 PREPARATION OF SAMPLES FOR TESTING

The handsheets after drying by various methods were conditioned according to CPPA standard C4 at 25°C and 50% relative humidity. The non-destructive tests like basis weight, caliper, bulk/density, porosity and optical properties were conducted first. The destructive tests like burst, tensile, zero-span, fold, tear etc followed on the basis of the scheme given in fig 2.4.

All sets of sheets were measured for moisture content gravimetrically. Such a division enables measurement of burst-strength at two locations for each handsheet while the two sections used for two-ply tear testing gives one value of tear measurement per handsheet. One sheet was used to measure other properties like double-fold, STFI compressibility, zero-span tensile etc. Every

sheet yielded at least two set of values for each of these tests.

At least 5 readings of the thickness (or caliper) were taken per handsheet, and two readings of optical properties were taken per handsheet. Thus with replicates on each sheet varying between 1 and 5 combined with 8-12 replicate handsheets, nominally identical, dried under nominally identical drying conditions, give between 8 and 60 replicate measurements of the physical and optical properties per set.

TABLE 2.1: STANDARD CPPA METHODS

PROPERTY	METHOD NUMBER
Basis Weight	D.3
Caliper/Bulk	D.4
Tensile Strength	D . 6
Stretch	D.7
Bursting strength	D.8
Tearing Resistance	D.9
Folding Endurance	D.17
Brightness	E.1
Opacity	E.2

TABLE 2.2 : FURNISH VARIABLES

Softwood Kraft Pulp		Chemi-Thermo- Mechanical Pulp	
Freeness, ml, CSF	PFI Rev.	Freeness, ml, CSF	PFI Rev.
690	0	510	0
445	8000		
315	13500	315	8500
95	36000		

CHAPTER 3

RESULTS AND DISCUSSION

3. RESULTS AND DISCUSSION

3.1 INTRODUCTION

This chapter presents the results of the characterization of microwave oven, the physical and optical property tests done on handsheets of 60 g/m² and 120 g/m² made from bleached kraft pulp and chemi-thermomechanical pulp.

Figs 3.1 - 3.4 show the measured characteristics of the Sharp model 8465 microwave oven. These present the data for three application levels of the oven i.e. high, medium-high and medium. The temperature rise in distilled water and paperboard are also plotted.

The measured physical and optical properties of handsheets are listed in tables 3.1 - 3.12 and shown graphically in figs 3.5 - 3.24. These tables and graphs show the properties of the handsheets dried in the microwave oven, the cylinder dryer and the ambient air (ring) dryer at different freeness levels as well as densities.

Every point plotted in fig 3.5 - 3.24 is a mean of between 10 and 70 replicate measurements. The average, standard-deviation and 95% confidence limit are presented in tables 3.1 to 3.12. The 95% confidence limit is calculated as follows:

If $(x-d) < x < (x+d)$ represents the confidence interval, the value of the half interval 'd' was calculated from

$$d = c. s / (n)^{0.5}$$

where,

d = interval width

c = constant which depends on confidence level ($c = 1.96$ for 95% confidence)
 The value of c depends on the size of the set and follows a Student law. For a sample size of 10, the confidence interval is about 15% larger than for a size of 30. Also the correct value of c for confidence level of 95% with large set is 1.96.
 n = sample size
 \bar{x} = sample mean
 s = standard deviation

3.2 MICROWAVE OVEN

3.2.1 MICROWAVE OVEN CHARACTERIZATION

The microwave oven characterization results are shown in figs 3.1 - 3.4. Rise in temperature of 100 ml of distilled water under medium, medium-high and high levels of the microwave oven is presented in Fig 3.2. Fig 3.1 shows the magnetron availability (or the time for which the magnetron is generating microwaves for application in oven cavity) at the three operating power modes of the oven. Fig 3.3 presents the power output in watts with distilled water as the variable load. This is based on the output rating method proposed by the International Electrotechnical Committee (IEC) as mentioned by Schiffmann (80). The power output is measured using one liter distilled water load in a glass beaker. The power absorbed is calculated by the following formula:

$$P = 69.8 \times (t_2 - t_1)$$

where,

P = Power output in watts

t_1 = initial temperature of distilled water, $^{\circ}\text{C}$

t_2 = temperature of distilled water after 60 seconds in oven, $^{\circ}\text{C}$

These measurements were also made for different amounts of distilled water loads. As the size of the load gets smaller, it absorbs less microwave energy. The wattage was calculated using the following formulas (80):

2000 ml sample:

$$\text{Watts} = 140 \times \text{temperature rise in } ^{\circ}\text{C}$$

1000 ml sample.

$$\text{Watts} = 69.8 \times \text{temperature rise in } ^{\circ}\text{C}$$

500 ml sample:
Watts = 34.9 x temperature rise in °C

250 ml sample:
Watts = 17.4 x temperature rise in °C

Fig 3.1 shows that microwave power is available almost 100% of application time for high level, while it is available for 80% and 59% of the time respectively for medium-high and medium level settings of the oven.

Fig 3.2 shows the temperature rise for 100 ml of distilled water at the three available levels of microwave power application. As shown in fig 3.1 microwaves are available for 100% of time in high mode, so water starts boiling in about 180 sec, while it boils in 240 sec under medium high and about 360 sec in medium microwave power

Fig 3.3 which represents the effect of varying load size on the power output, conforms to the standards (80) and shows that the power output is proportional to the load-size. Fig 3.3 was verified 5 times during the experimental phase. These results on the microwave oven suggest, that the oven behaves in a reproducible way

3.2.2 MICROWAVE FIELD UNIFORMITY

A uniform microwave field is necessary for the drying experiments carried out in this study. The results presented in figs 3.2 and 3.3 were repeated by placing 100 ml distilled water in the center and on the periphery of a 180-200 mm diameter circle. The temperature rise was measured at these locations. In addition to these measurements water loss measurements were made again using 100 ml distilled water. From these experiments the following conclusions were drawn

- * The rate of heating and hence the microwave intensity is independent of the position of the beaker within the 200 mm circular area.
- * In the circle of 180 mm diameter, the maximum temperature difference noted is 2°C and in the circle of 220 mm maximum temperature difference noted is 3°C.

* Volumetric water loss data show that the location of beaker does not affect the amount of water evaporated at different times and application levels.

3.2.3 TEMPERATURE RISE MEASUREMENTS IN HANDSHEETS

The temperature rise in handmade paperboard was measured using a standard fiber-optic probe (Fluoroptic Thermometer, model 1000A with fluoroptic temperature probe model LIC) and a grounded junction thermocouple (40). Fig 3.4 shows results a series of measurements. Probes were inserted in 600 - 2000 g/m² sheets by placing them in between two handsheets.

During microwave drying, the temperature rise is very fast in the first few seconds and then stabilizes around 96°C (fig 3.4) at high mode of the oven. It further shows that the microwave drying of handsheets takes place at relatively low temperatures. Placing the fiber-optic probe in different positions as well as changing the load by varying the basis-weight showed similar trends. The results in fig 3.4 are at high level of microwave field and those at medium-high levels also showed similar patterns.

3.3 PROPERTIES OF KRAFT PULP HANDSHEETS

The following sections present results for 60 g/m² handsheets made from bleached kraft at different freeness levels. Possible factors influencing the sheet properties are discussed.

3.3.1 DENSITY/ BULK/ CALIPER

Table 3.1 presents the caliper and density of kraft pulp handsheets dried in the microwave oven, and by the cylinder and ring drying. These data are plotted in fig 3.6. The standard Tappi methods T205 and T220 (CPPA method D.3, D.4), using an area of

200mm² and pressure of 0.50 kg/cm², is used for this measurement. Standard bulk/density is one of the best indicators of the relative bonded area (RBA) (12).

As indicated by fig 3.6, sheet density increases with beating. As reported in literature (12), the rate of decrease of bulk for PFI mill is around 0.12 to 0.15; these data are within this range. The data in table 3.3 show that there is a small increase in bulk (or reduction in density) for handsheets dried in microwaves over the entire range of experiments. This may be attributed to less thickness direction shrinkage in microwave drying.

3.3.2 BURST INDEX

The burst strength is presented as burst-index or burst-factor in table 3.2. Fig 3.5 indicates that burst index increases with beating and levels off as beating proceeds further. The burst strength is an outcome of several parameters such as relative bonded area, internal bond strength and individual fiber strength. Any increase in one or more of these parameters will increase the burst strength.

As indicated by fig 3.5, the burst-index is practically identical for the three drying methods at a given freeness. In contrast, microwave dried sheets show higher burst factor at the constant density as indicated by fig 3.17. This may be attributed to increased bonding and the increased stretch as indicated by the instron tensile test. This is also confirmed by fig 3.16 which indicates that the microwave dried sheets show comparable burst factor at a constant tear index with ring dried sheets.

3.3.3 TEAR INDEX

The tear resistance, measured according to Tappi method T220 (CPPA method D.9), is presented as tear-index or tear factor in table 3.3 and fig 3.8. Fig 3.8 indicates that the tear index goes down with the increase in refining, indicating that force needed

to tear is diminished as the density of paper increased. Because the work needed to sever a fiber lying across the path of the tear is considerably less than that needed to pull it out from the sides of the sheet, so as the density is increased, it becomes more difficult to pull out fibers. This leads to increased severing of fibers and the reduced work. The tear factor also goes down with reduced stretch.

Fig 3.8 shows that the microwave dried sheets show improved tear index at a given freeness. However, fig 3.19 shows that there is no change in the tear factor at the a given density. This shows that the microwave dried sheets maintain the same tear index despite decreased density. This phenomenon may be attributed to the increased cohesion (12) of fibers to co-operate at the apex of the tear. Increased cohesion may be the result of less reduction in thickness (fig 3.6), while keeping the same burst factor (fig 3.16).

3.3.4 BREAKING LENGTH

The tensile strength, measured according to Tappi method T220 (CPPA method D.6), is presented as breaking length in fig 3.9. With increased refining the breaking length increases before it levels off. In tensile failure, the applied load stretches the fibers lying in its direction and as the stress is increased, bonds between the fibers begin to break as the fibers, or the elements of the fibers, lying in the non-aligned directions begin to turn toward the stress. In doing so they separate in part from their neighbours. This loosening of the structure continues as the load increases, and some of the fibers lying in the direction of the applied load have to break or else have one end pulled free. The load is now borne by fewer and fewer fibers until the strip suddenly parts (12).

As indicated by fig 3.9, that the breaking length is unaffected by the drying method at any level of freeness, though the values for the cylinder dried sheets are towards the lower side. On the other hand, fig 3.19 shows that at a given density, microwave dried sheets show improved breaking length. Same is evidenced in fig 3.15 i.e. microwave

dried sheets show better breaking length at a given tear factor.

3.3.5 DOUBLE FOLD

The folding endurance, measured according to Tappi method T511, is presented as MIT double folds at 1.5 kg load in table 3.5 and fig 3.7. Fiber length and coarseness have marked influence on the folding strength. According to Clark (12), folding endurance varies inversely as approximately the 3.5th power of the tension applied to the strip undergoing testing.

As indicated by fig 3.7, the microwave dried handsheets maintain the doublefold obtained in ring drying, while it goes down in cylinder drying. The microwave dried handsheets show higher double fold at constant density (fig 3.18). As described before, it appears that the microwave drying increases cohesion among fibers. Higher cohesiveness is able to join the fibers firmly enough to prevent separation for a long time during the folding process (12).

3.3.6 ZERO-SPAN BREAKING LENGTH

The intrinsic fiber strength is measured as zero-span breaking length by the Pulmac apparatus. The z-span strength can be useful as a measure to see if drying affects individual fiber strength. The stresses developed during drying or reduction in DP during drying have direct impact on z-span strength. The zero-span strength is presented in the table 3.7 and the fig 3.10. It is very much affected by the conditions the fibers have gone through in the process of sheet making. It is also a further indicator of fiber cohesiveness. With increased beating, the zero span strength goes up and reaches a plateau.

Fig 3.10 shows that the drying methods do not have significant differences between

them; the cylinder drying method shows a little decline in zero-span strength. Microwave dried handsheets show increased intrinsic strength as evidenced by fig 3.21. This further supports the increased double-fold value and the possible increase in cohesion among fibers.

3.3.7 STFI COMPRESSIBILITY

The edgewise compression strength, measured as STFI compressibility is presented in table 3.6 and figs 3.11 & 3.22. This property shows the structural behaviour of paper. Compression failure of paper may be as a result of an unstable loading and yielding at different structural levels. It is affected by the density, drying restraints, pulp yield, as well as by sheet forming variables. Increased refining leads to better utilization of fiber strength both in tension and compression (9). According to the data in table 3.6, difference between the three drying methods is not significant.

As is clear from, the compressibility increases with increase in refining but the rise is much higher for microwave and ring dried handsheets. Similar trends are noticed by plotting the data taking sheet density as base. According to Fellers (9), there is a transition from a buckling type of failure to a yielding type of failure in compression as density increases.

3.3.8 POROSITY

The porosity, measured as Parker Print Surf porosity, is presented in the table 3.8. The Parker print-surf apparatus provides surface void volume in ml/min but also gives a value for the mean separation G in μm of the surface from the reference plane, calculated according to relationship derived by Parker (71). The results in the table 3.8 show that by refining, the porosity of the handsheets decreases significantly. There is no practically significant difference in porosity among the handsheets dried by the three different methods.

3.3.9 SCATTERING COEFFICIENT

The scattering coefficient data, measured according to the Tappi testing method T425 by Technibrite model TB 1C, are presented in fig 3.14. This test indicates the extent to which the area of component fibers are bonded i.e. degree of bonding (68). This test also measures the scatter of light in the interior of the sheet.

As shown in fig 3.14, the scattering coefficient decreases significantly by refining while there is no major difference among three drying methods. The values for the cylinder dried sheets are however somewhat lower.

3.3.10 BRIGHTNESS AND OPACITY

The optical properties measured in this work included brightness, opacity and the color parameters. The scattering coefficient as well as the absorption coefficient were measured.

Brightness is the most important optical property and is presented as percentage brightness, according to Tappi Standard T217 (CPPA method E 1) using Technibrite TB 1C model shown in fig 3.12. Opacity test is expressed as print opacity as well as Tappi Opacity. Fig 3.13 is plotted using the print opacity data. The optical measurements show no specific dependence on the drying method except for minor variations. It is clear from fig 3.13, that the brightness and opacity decrease due to refining.

3.4 PROPERTIES OF HANDSHEETS MADE FROM MECHANICAL PULP

All the measurements and the tests mentioned in section 3.3 for kraft pulp were also carried out for chemi-thermomechanical pulp. The results of the various tests for 60

grams per square meter handsheets at 315 ml CSF are presented in fig 3.23 and table 3.12. Bar chart in fig 3.23 identifies the variation in the physical and optical properties among the three drying methods. As observed with kraft handsheets, there is slight increase in bulk or reduction in the density of the handsheets dried in microwaves.

Microwave dried sheets maintain the same trend for most properties. There is noticeable increase in the double fold for the microwave dried CTMP sheets, better zero-span breaking length and STFI compressibility. Other properties like paker print-surf porosity, tensile strength are well maintained. Brightness and opacity were also unchanged, while the scattering coefficient values were higher for microwave-dried handsheets. Evaluation was done only at one freeness level of 315 ml CSF.

3.5 PROPERTIES OF 120 g/m² KRAFT HANDSHEETS

To further confirm the effects of the drying methods, 120 g/m² handsheets prepared from kraft pulp (315 ml CSF) were also tested for most properties. These results are presented in fig 3.24. The pattern follow the same trend as was found for 60 g/m² meter handsheets but the effects on properties like double fold and STFI compressibility are more noticeable.

3.6 DISCUSSION

ROLE OF MICROWAVES IN DEVELOPMENT OF FIBER & SHEET PROPERTIES

This study on the effect of drying methods on the physical and optical properties leads to several interesting results and conclusions. To understand these effects and possible mechanisms one requires an in-depth analysis of the development of fiber

bonding process and development of paper strength. It is necessary to present the basic factors on which the interpretation of results is based. The following gives a brief account of these phenomena.

Drying of paper may be described as two stage process (it may be said three stage process if warm-up is considered), the constant drying phase and falling rate phase. During constant rate phase all vaporization takes place at the surface. Moisture leaving in surface is replenished by moisture migration from within the sample to surface in liquid form. The shape of drying curve during the constant rate period is characterized by a constant slope. While in the falling rate period, it becomes impossible for liquid water to migrate to the surface as fast as this water may be evaporated at the surface. Consequently, as drying proceeds a moisture front recedes into the material leaving a dry outer layer. In order to vaporize the moisture within the material, the heat of vaporization must be added to the moisture vaporization front which is accomplished by a temperature gradient that is established between the surface of the material and the vaporization front. The shape of the drying curve during the falling rate period is noted by a progressively declining slope with the drying rate decreasing as the drying insulating layer thickens.

In drying rate experiments on drying of cotton fabrics, Pendrgrass (72) noted that microwave drying curve differ significantly from those for drying using surface heating

1. Where the drying rate progressively decreases throughout the majority of the drying process. This extended constant rate period is a result of the energy being supplied to the sample at a constant rate drying the majority of the process with microwave heating. This is in contrast with conventional heating where the energy supplied to the vaporization front decreases progressively as the dry insulating outer layer thickens.

2. They found that the dye concentration was uniform throughout the sample. This indicates that drying using microwave heating results in the no movement in liquid state. Fluid movement that does occur in the vapor state and thus does not carry dyestuff.

Lyons and Vollers (57) also found that with internal heating there is much less moisture migration than with surface heating. Takahashi et al (87) found that microwave drying gives uniform distribution of resins in the cross section of paperboard.

In conventional drying moisture is first removed from the surface of handsheet producing an internal moisture gradient across the paper sheet. This is necessary for outward diffusional moisture flow. A microwave generated thermal gradient produces a completely different moisture distribution in the dried body. Because of the exponential dependence of the diffusivity on temperature, the diffusional flow rate for a given moisture gradient will be much higher in the center of the sheet than near its surface. As a result, a strong moisture levelling process exists as moisture gradients decrease with increasing depth, to compensate for the rapidly increasing diffusivity. Moisture content will decrease more uniformly throughout the bulk and thus eliminates part of the disadvantages of conventional drying process. In conventional drying two important parameters are the temperature gradient and vapor pressure gradient. But in microwave drying, heat is absorbed volumetrically i.e. a temperature gradient is not necessary for the interior to receive energy for vaporization.

Paper is a dielectric material. The value of the dielectric parameters depend greatly on the mode and quantity of the water content of the pulp. Free water and bound water have completely different characteristics. The tendency of the water to attach itself to hydroxyl groups of cellulose by a single hydrogen bond restricts the rotational energy absorption of the water molecule and decreases its loss factor value. As drying progresses along the paper machine more hydrogen bonds are formed and fiber wall collapse occurs. The latter is complete at about 70 % dryness (66). The dielectric constant also depends on the crystallinity of cellulose. Further, percentage of available hydroxyl groups will depend greatly on the volume fraction of crystallinity of cellulose.

The bonding of water is important from microwave point of view. Because of bonding, the dipole rotational absorption is restricted, the increase of energy absorption

with frequency is dampened and high frequency levels are of less importance (28). Water molecule is the major contributor to the microwave heating mechanism, since it has a permanent dipole whereas most substrates do not. The loss factor increases with temperature and since drying takes place at an elevated temperature, this may be regarded as being favourable for microwave drying. However as temperature rises the sheet moisture content is reduced as water is lost by evaporation; there is thus a self-limiting control.

Paper is a network structure composed of a multitude of discrete particles mainly of a fibrous nature. These cellulose fibers are basically composed of (1-4)-beta-D-glucopyranose. Cellulose exists in different phases with different degrees of order with irregular regions being interspersed between regular crystalline phases. The high content of hydroxyl groups in cellulose provides many possibilities for hydrogen bonding; these bonds play a crucial role in strength development.

Hemicellulose rings are residues of pentoses and hexoses, while lignin is dominated by aromatic rings. Both of them are quite amorphous. Hemicelluloses are low molecular weight hetero-polysaccharide conglomerates with much lower degree of polymerization than cellulose. Saccharide rings are joined together with beta-glycoside bonds and hydroxyl groups which are present and able to form intra-molecular or intermolecular hydrogen bonds. Ionic groups, mainly as uronic acids, are also present and play important role in microwave activity.

The lignin structure, with phenylpropane as basic structural unit, consists of a network of aromatic rings linked to each other mainly through alkyl-aryl-ether bonds and bearing an abundance of substituted groups. Kraft pulp lignin has carboxyl groups. Such ionizable groups give the lignin a polyelectrolyte character which is probably important for the softening mechanism (93). Typically softwood xylan and glucomannan has softening temperature of 20°C at 30% moisture content. Spruce wood lignin softening temperature drops from 151°C in dry state to 76°C at 33% moisture (45).

Several electromagnetic and thermal parameters of paper are involved in microwave drying process. Microwave radiation penetrates the bulk of the handsheet. Microwaves are electromagnetic waves. Any disturbance of either the electric or magnetic field induces fields in the surrounding region and a wave of electromagnetic energy propagates. The process of microwave drying consists of dissipating part of the microwave energy flow is a lossy dielectric material. The temperature rise is directly related to dissipated power, the heat capacity and density of the material. The complex permittivity of paper is the electrical parameter which defines the interaction of the paper with microwaves. The complex permittivity has two parts i.e. dielectric constant and loss factor. The dielectric constant represents the ability of paper to store the electrical energy, while the loss factor represents the loss of the electrical field energy in paper.

The two basic phenomena that contribute to the large values of the loss factor and are responsible for the heating effect at microwave frequencies are ionic conduction and dipole rotation. When a microwave field is applied to a wet handsheet and a wet material in general contain some number of ions; since about one molecule in 10^7 spontaneously breaks down at room temperature - the ions move in the direction of the field. The ions collide with other molecules and their kinetic energy is converted into heat through those collisions. The microwave heating process is not dependent on the microwave frequency to any degree and only to a small degree on temperature due to the increase of number of ions with temperature.

Water is polar molecule i.e. the electrical charges within the molecule are nonuniformly distributed in space. Such molecules, when placed in an electric field, try to align themselves with the field. In handsheet, the dipolar molecule alignment will also depend on the thermal/ Brownian motion. The degree of orientation will also depend on frequency of microwave, structure of molecule and placement of water molecule in it, effective viscosity, temperature and nature of bonds between water and fiber.

The energy of the electric field is converted into potential energy of oriented dipoles

e.g. water, which is then converted into kinetic energy of dipoles and to heat as the reinforcing dipoles interact with the surrounding molecules by friction. The absorbed power due to the relaxation phenomenon increases with frequency. The energy absorbed per cycle shows a resonant behaviour versus frequency, with a maximum when the applied field frequency is equal to the relaxation frequency. The relaxation frequency is temperature dependent, the higher the temperature, the faster the dipoles can reorient themselves and the higher the relaxation frequency. This temperature dependence of the relaxation frequency provides a very useful practical feature of temperature self regulation in microwave heating process.

Molecule relaxation due to dipole rotation occurs not only for water but also for several other molecules e.g. uronic acids, amino acids, proteins, polymers and alcohols. The relaxation frequency of these are lower than that of water. At microwave frequency the phenomenon that plays an important role in power absorption is the relaxation of bound water. The relaxation frequency of bound water depends on binding forces and the solvent viscosity. According to Stuchly (86), this frequency for most materials is between 100-1000 Mhz.

Attenuation occurs as the waves pass through paper sheet. However, the surface, even though it receives the most energy, is generally cooler than the interior of the material. This is due to evaporative cooling and the rate of energy input to interior, which is usually greater than the rate of heat transfer to the outside surface for dissipation. As a result, there is a positive vapor-pressure gradient from interior to the surface which accelerates the moisture transfer.

Drying leads to hornification (9) i.e. irreversible reduction in swelling ability of cellulose. This is due to the formation of new junction zones between microfibrils by means of trains of hydrogen bonds, which are stable toward rewetting because of their co-operative bonding effects. Irreversible changes during drying seem to be a general feature of many hydrogen bonded materials, including kraft lignin (9) and hemicellulose (34). During drying microfibrils associate themselves laterally to form sheets by formation

of tangential bonds between microfibrils (85). During drying larger pores close and contribute to the higher cell wall collapse for beaten fibers (34). The extent of cell wall collapse is increased by delignification. It may also be expected that higher internal stresses can be built into a stiffer cell wall matrix in which viscous flow during drying is limited. If the stresses are allowed to relax by increasing the time and temperature of drying the degree of irreversible cell wall collapse increases (78).

There are several factors which affect the bonding process e.g. treatment and state of swelling of fibers, time available for bonding, the temperature, the moisture content, the external and internal fibrillation of fibers, closeness of bonding surfaces and the fiber flexibility and conformability. For fibers with higher lignin content, the conditions which makes them more flexible and makes more bonding sites available, help improve paper properties.

Fiber bonding occurs when fibers are drawn together by surface tensional forces during water removal process in drying. As surface tension forces are proportional to the liquid-gas interfacial area, the most important part of the drying cycle is towards the end of the constant drying rate period when the gas-liquid interface at the surface of the paper breaks up and enters the interstices of the paper. Thus, it is during this phase of drying that fiber flexibility controls bonding and hence the bonding-dependent properties. Autocrosslinking is more important in paper containing resinous materials, which act as crosslinking agents. Autocrosslinking is therefore favored in paper containing incompletely delignified fibers and parenchyma cells, such as paper made from CTMP or paper made from hardwoods.

The physical properties of paper are determined by the strength of individual fibers and of inter-fiber bonding, but are also affected by the density of fibrous network of the sheet. Aside from the effect of pressing during drying - not a factor in the present study - the density of the dried sheet is determined primarily by the extent of fiber collapse and the magnitude of surface tension forces. An opposing factor may be present under conditions which produce very rapid evolution of water vapor. The force exerted on the

fibers by a high velocity flow of water vapor from the interior of the sheet, acting in the opposite direction to the forces of surface tension and fiber bonding, may act to reduce the density of sheet (17).

These results which relate primarily to the strength of individual fibers are considered prior to those which are dependent on interfiber bonding or on a combination of individual fiber strength and strength from interfiber bonding. Zero-span breaking length is primarily a measure of the strength of individual fibers, although lesser contributions due to fiber length, orientation and bonding have been reported (13). Thus the strength of individual fibers may be inferred from zero-span measurements.

In this study there is no significant difference in z-span but the values for cylinder dried sheets are always on lower side than ring/ microwave dried (fig 3.10). When the measurements are reduced to the density scale, they show that at a given density microwave dried sheets have noticeably higher z-span strength than the cylinder dried handsheets. These measurements at 100°C and those at higher temperature (17) show a very significant loss (7-10%) in zero-span strength. In microwave drying, the degradation of individual fiber strength, which is attributed to depolymerization of the cellulose chains in the higher temperature or oxidizing due to hot air (17) totally disappears. Similar advantages are noticed in drying of CTMP sheets. It appears that in this case the polar character of lignin, hemicelluloses and other extractives plays a key role. Microwave also does precise control of drying i.e. it removes moisture rather than 'overdrying', which quickly causes severe reduction of individual fiber strength in high temperature drying (17). It causes no reduction whatever in the microwave drying. The lack of any degradation of individual fiber strength in microwaves is attributable to the selective action of microwave in the wet regions and self-limiting temperature control in the dry areas.

The increase in strength for chemi-thermomechanical pulp handsheets may be attributed to polar autocrosslinking or a "welding effect" of cellulose within fibers, to a redistribution of resinous compounds inside the fibers resulting in less brittle, stronger

fibers and as noted earlier, to the small effect that improved bonding has on individual fiber strength. Auto-crosslinking and redistribution of resins are mechanisms primarily applicable to CTMP handsheets while improved bonding effect could apply equally to kraft and CTMP paper. Caliper and basis weight interpreted as bulk or density are very important paper properties. These affect most properties.

For all measurements at different freeness the sheet bulk for kraft as well as CTMP handsheets is higher in microwave drying than in ring or cylinder drying. This reduction in density is a measurable difference but is not very significant considering the standard deviation range. This may be attributable to very rapid evolution of water vapor from the interior of sheet due to very fast drying especially during the falling rate period in microwave field as compared to ring and cylinder drying. Other reason may be, as mentioned earlier, during microwave drying there is very low migration of water in liquid form so the structure does not collapse as much as it does in hot air or cylinder drying.

The properties which are a result of fiber strength as well as interfiber bonding are shown in fig 3.5 -3.24. Each property has been discussed in detail in previous sections. Microwave dried sheets show measurable enhancement of many of the physical strength properties. On the other hand, cylinder drying shows a slight reduction in most properties. This reduction is comparatively much lower than that reported in an earlier study (17) done at comparatively high temperatures of up to 400 °C. A reduction of up to 25 % in the burst index and the tear index for the air dried sheets as compared to the ring dried sheets was reported by David (17).

The increase in strength properties of microwave-dried sheets may be attributed to a possible increase in the individual fiber strength over that obtained in cylinder drying and in part from better inter-fiber bonding. The fast, selective and uniform temperature rise may be attributed for such increase, while surface hornification and slow drying in the center of web may be attributed to reduction in strength in cylinder drying. These effects have been noticed in a whole spectrum of handsheets made of stock refined to different freeness range from 695 ml CSF to 95 ml CSF.

In this study no significant differences were noted in optical properties for the three drying methods. However an earlier study done at high temperature noted a reduction of up to 5% in brightness due to oxidation at higher temperatures (17).

3.7 PROPOSED " WELDING EFFECT " OF MICROWAVES

Results displayed in Figs 3.5 - 3.24 indicate that microwave drying slightly increases fiber bonding, as well as individual fiber strength as compared to cylinder drying. This may be attributed to an increased utilization of the available bonding surface or to the "welding effect" of microwaves as postulated in this thesis.

Conventional drying can not modify hydrogen bonding while microwaves may increase the possibility of such bonding by vibrating water and other polar molecules at the applied frequency of 2450 Mhz, this is believed to make more bonding sites available. In contact drying heat has to pass through the non-conductive dry fibers across the thickness of paper web and the moisture must follow the same path. So previously dried fibers undergo a phenomenon of stress and strain which also leads to differential heating of the paper sheet in thickness direction. Fibers in the surface layer get hornified fast and get stiffened while those inside the sheet are still flexible. This can lead to strength loss and cause problems such as linting, picking and low surface strength. In contrast, in microwave drying there is no possibility of linting, picking or loss of surface strength because no differential heating takes place. No surface picking is there because the sheet does not come in direct contact with hot surface. Here the moisture removal process reinforces and redistributes the hydrogen bonds so that increased bonding levels can be achieved per unit fiber surface. In addition conventional drying causes differential stresses in the paper which are frozen after drying so that paper displays dimensionally unstable behaviour.

In cylinder drying it appears that the phenomenon of macro-shrinkage takes place i.e. the whole matrix is involved in the shrinkage process. Microwave drying probably involves micro-shrinkage which works as reinforcing fibers/springs in the composite matrix resulting in increased strength while maintaining higher bulk as evidenced by figs 3.5-3.24. Fast evaporation of water due to localized heat generation inside fibers leads to faster escape of moisture which may lead to opening up of more fiber surface for bonding or making otherwise "dead" areas available for bonding. This might also lead to greater fiber flexibility as evidenced in figs 3.7 and 3.18 (increased folding strength) at a higher scattering coefficient

In addition to above mechanism, increased influence of microwaves in the case of mechanical pulps can be attributed to, increased microwave activity due to the presence of more "microwave-groomable" molecules. Several extractives in wood are highly polar in nature which might help in the development of additional bonding sites. The welding effect might be more evident in the case of mechanical pulps because of localized heating and redistribution of lignin to attain increased bonding levels. In general the polyelectrolyte characteristics of lignin and extractives, and steric factors (due to their random coil motion) act to keep lamellae apart. Microwaves may help remove this or reorient this barrier of micro-stearic effects leading to effective bonding.

3.7.1 CLOSURE

This study concludes that above proposed "welding effect" during microwave drying leads to enhancing or maintaining the measured physical and optical properties. As the physical properties results of microwave drying closely follow the ambient air drying, it is also proposed to look into feasibility of incorporating this as standard test method of handsheet testing for pulp evaluation. This will help reduce the time of testing.

CHAPTER 4

SUMMARY AND CONCLUSIONS

4. SUMMARY AND CONCLUSIONS

The effect of microwave drying on key physical and optical properties of the handsheets have been determined. Development of properties in the microwave drying have been compared with cylinder and ambient air drying. These comparisons are made for handsheets made from kraft pulp and a high lignin chemi-thermomechanical pulp. The following conclusions apply for drying of handsheets in microwave field obtained in a microwave oven:

4.1 FIBER PROPERTIES

Microwave drying provides improved fiber strength at comparable density than cylinder drying. This increase amounts to 5 to 7% at a given density. Expressed relative to ring drying there is no difference in both drying methods. Similar increase is noticed for the handsheets made from chemi-thermomechanical pulp. It shows that drying paper in microwaves reduces any fiber degradation. Increased fiber strength in microwave drying is further evidenced by increase in properties like compressibility strength and double-fold.

4.2 PHYSICAL AND OPTICAL PROPERTIES

1. Microwave dried kraft handsheets show a decrease of density of about 2 to 4% as compared to ring or cylinder drying. This decrease is almost constant over the entire freeness range for both chemi-thermomechanical and kraft pulp handsheets. This decrease in density is marginal at 95% confidence limit.

2. Burst index is practically unchanged for the three drying methods at a given freeness. However, microwave dried kraft handsheets show an increased burst-

index at constant density in the range of 8 -12%. This difference holds at all freeness levels studied. The increase in burst-index for CTMP handsheets is comparatively lower viz 4-6%.

3. The tear index shows no difference for any of the drying methods at constant density. This implies that the microwave dried handsheets show no change of tear-index, in spite of a marginal decrease in density.

4. Microwave drying shows comparable breaking length with ring drying, which are higher than cylinder drying at the same density. However, ring drying shows higher breaking length at increased refining.

5. Folding endurance was found to be higher for handsheets dried in microwaves compared to those dried in cylinder drying. This increase is 20 to 30% for kraft handsheets and 10 to 15% for CTMP hand sheets. This property shows increase at constant freeness as well as constant density. The values for ring and microwave drying are comparable.

6. Scattering coefficient, brightness and opacity show no significant difference among drying methods. It is clear from these figures that the brightness and the opacity go down by refining, due to decrease in the scattering power of sheet with increase in refining.

4.3 THE MECHANISM OF STRENGTH IMPROVEMENT

Results of this work indicate that microwave drying improves fiber bonding and fiber strength as compared to cylinder drying leading to overall better quality. This is attributed the efficient utilization of available bonding surfaces or to the "welding effect" as postulated in this thesis.

TABLE 3.1: EFFECT OF DRYING METHOD ON HANDSHEET DENSITY
(BLEACHED KRAFT PULP)

FREENESS ml CSF	RING DRYING				CYLINDER DRYING				MICROWAVE DRYING			
	Thickness			Density	Thickness			Density	Thickness			Density
	x	s d	d	kg/m ³	x	s d	d	kg/m ³	x	s d	d	kg/m ³
690	115 65	2 38	0 56	538	112 70	10 07	2 36	537	118 10	2 88	0 68	519
445	107 00	3 74	0 88	673	105 80	7 42	1 74	670	105 30	6 88	1 61	658
315	84 90	1 45	0 34	712	85 07	3 89	0 91	696	86 79	3 34	0 78	692
95	85 30	5 40	1 26	750	81 02	2 20	0 52	742	85 17	3 37	0 79	735

TABLE 3.2: EFFECT OF DRYING METHOD ON BURST INDEX
(BLEACHED KRAFT PULP)

FREENESS ml CSF	RING DRYING				CYLINDER DRYING				MICROWAVE DRYING			
	Bursting Strength			Burst-Index	Bursting Strength			Burst-Index	Bursting Strength			Burst-Index
	x	s d	d	kPam ² /g	x	s d	d	kPam ² /g	x	s d	d	kPam ² /g
690	148 75	12 58	5 03	2 39	139 23	5 19	2 08	2 58	137 25	6 99	2 80	2 11
315	561 26	22 79	9 12	9 35	539 83	17 93	7 17	9 25	555 24	11 52	4 61	9 13
95	630 50	58 08	23 23	10 48	576 23	31 91	12 76	9 85	599 00	24 77	9 91	10 20

TABLE 3.3: EFFECT OF DRYING METHOD ON TEAR INDEX
(BLEACHED KRAFT PULP)

FREENESS ml CSF	RING DRYING				CYLINDER DRYING				MICROWAVE DRYING			
	Tearing Strength			Tear-Index	Tearing Strength			Tear-Index	Tearing Strength			Tear-Index
	x	s d	d	mNm ² /g	x	s d	d	mNm ² /g	x	s d	d	mNm ² /g
690	13 27	0 75	0 42	10 56	13 55	3 16	1 79	11.35	14 60	2 60	1 47	11 19
315	13 20	0 98	0 56	11 26	13 10	0 96	0 54	11 74	14 55	0 62	0 35	12.04
95	13 03	0 99	0 56	10 38	11 58	0 49	0 28	11 45	13 40	0 88	0 50	11.94

TABLE 3.4: EFFECT OF DRYING METHOD ON BREAKING LENGTH
(BLEACHED KRAFT PULP)

FREENESS ml CSF	RING DRYING			CYLINDER DRYING			MICROWAVE DRYING		
	Breaking Length, km			Breaking Length, km			Breaking Length, km		
	x	s d	d	x	s d	d	x	s d	d
690	3 22	0 470	0 16	3 03	0 39	0 130	3 1	0 32	0 110
315	10 39	0 950	0 33	10 35	0 78	0 270	10 61	0 85	0 300
95	12 2	0 870	0 31	11 25	0 92	0 360	11 47	0 77	0 270

TABLE 3.5: EFFECT OF DRYING METHOD ON FOLDING STRENGTH
(BLEACHED KRAFT PULP)

FREENESS ml CSF	RING DRYING			CYLINDER DRYING			MICROWAVE DRYING		
	Double Folds #			Double Folds #			Double Folds #		
	x	s d	d	x	s d	d	x	s d	d
690	6 80	0 910	0 36	6 50	0 84	0 330	7 20	0 41	0 160
315	896 80	32 350	12 90	751 70	47 28	18 900	880 30	51 52	20 570
95	1103.70	62 310	24 93	1062 70	56 15	22 460	1118 17	29 78	11 910

TABLE 3.6: EFFECT OF DRYING METHOD ON STFI COMPRESSIBILITY
(BLEACHED KRAFT PULP)

FREENESS ml CSF	RING DRYING			CYLINDER DRYING			MICROWAVE DRYING		
	STFI Compressibility, kN/m			STFI Compressibility, kN/m			STFI Compressibility, kN/m		
	x	s d	d	x	s d	d	x	s d	d
690	6 61	0 880	0 27	6 54	0 84	0 250	6 54	0 82	0 250
315	6 79	0 580	0 17	6 70	0 55	0 160	6 77	0 73	0 220
95	6 97	0 880	0 27	6 69	0 53	0 160	6 92	0 80	0 240

TABLE 3.7: EFFECT OF DRYING METHOD ON ZERO-SPAN TENSILE STRENGTH
(BLEACHED KRAFT PULP)

FREENESS ml CSF	RING DRYING				CYLINDER DRYING				MICROWAVE DRYING			
	LR σ			Z-SPAN B length	LR σ			Z-SPAN B length	LR σ			Z-SPAN B length
	x	s d	d	km	x	s d	d	km	x	s d	d	km
690	38.77	2.25	0.900	15.45	37.75	2.46	0.953	15.18	47.14	1.75	0.699	15.75
445	39.63	3.83	1.531	16.90	38.41	3.02	1.207	16.35	46.35	4.35	1.739	16.77
315	47.73	2.68	1.071	19.30	45.25	2.19	0.876	19.23	37.12	2.70	1.079	19.42
95	51.35	4.35	1.739	19.81	45.22	3.35	1.340	19.27	47.14	2.28	0.911	19.85

TABLE 3.8: EFFECT OF DRYING METHOD ON PARKER-PRINT-SURF POROSITY
(BLEACHED KRAFT PULP)

FREENESS ml CSF	RING DRYING			CYLINDER DRYING			MICROWAVE DRYING		
	PPS Porosity, ml/min			PPS Porosity, ml/min			PPS Porosity, ml/min		
	x	s d	d	x	s d	d	x	s d	d
690	2348.80	79.95	20.220	2266.60	75.25	19.037	2380.20	19.88	5.030
315	463.80	41.96	10.617	414.20	35.09	8.878	478.60	34.50	8.730
95	23.60	5.50	1.391	22.00	3.17	0.802	23.25	2.16	0.546

TABLE 3.9: EFFECT OF DRYING METHOD ON BRIGHTNESS
(BLEACHED KRAFT PULP)

FREENESS ml CSF	RING DRYING			CYLINDER DRYING			MICROWAVE DRYING		
	Brightness (%)			Brightness (%)			Brightness (%)		
	x	s d	d	x	s d	d	x	s d	d
690	86.50	1 71	0 529	85 62	2 21	0 684	87 00	1 65	0 511
315	82 19	0 95	0 294	81 72	1 97	0 609	82 77	1 99	0 294
95	77 92	1 01	0 312	77 75	1 85	0 573	78 44	1 09	0 337

TABLE 3.10: EFFECT OF DRYING METHOD ON OPACITY
(BLEACHED KRAFT PULP)

FREENESS ml CSF	RING DRYING			CYLINDER DRYING			MICROWAVE DRYING		
	Opacity (%)			Opacity (%)			Opacity (%)		
	x	s d	d	x	s d	d	x	s d	d
690	77 28	1 23	0 381	77 12	0 95	0 294	77 12	0 89	0 275
315	67 21	1 11	0 343	67 19	0 90	0 281	67 72	0 49	0 151
95	64 24	0 65	0 263	64 17	0 47	0 146	63 93	0 64	0 198

TABLE 3.11: EFFECT OF DRYING METHOD ON SCATTERING COEFFICIENT
(BLEACHED KRAFT PULP)

FREENESS ml CSF	RING DRYING			CYLINDER DRYING			MICROWAVE DRYING		
	Scattering Coefficient, cm ² /g			Scattering Coefficient, cm ² /g			Scattering Coefficient, cm ² /g		
	x	s d	d	x	s d	d	x	s d	d
690	39 68	0 55	0 171	38 68	0 94	0 291	39 45	0 65	0 201
315	22 32	0 92	0 285	22 27	0 95	0 294	23 16	0 24	0 074
95	20 49	0 44	0 136	18 89	0 78	0 242	19 58	0 54	0 165

**TABLE 12: EFFECT OF DRYING METHOD ON
PROPERTIES OF CTMP HANDSHEETS**
(CHEMI-THERMO-MECHANICAL PULP)

	RING DRYING	CYLINDER DRYING	MICROWAVE DRYING
Density, kg/m ³	535	538	496
Double Folds, #	97	134	149
STFI Compressibility, kN/m	6.59	6.43	6.89
Z-Span Breaking Length, km	12.72	14.31	14.86
PPS Porosity, ml/min	1075	1097	1062
Breaking Length, km	5.06	5.66	5.58
Elastic Modulus, MPa	3330	3237	3387
Stress, kgf/cm ²	2.82	3.09	2.96
Strain, %	1.87	1.69	2.08
Brightness, %	79.69	80.01	79.94
Opacity, %	74.88	75.39	75.91
Scattering Coeff., cm ² /g	303.0	303.8	315.4

FIG 3.1 MAGNETRON AVAILABILITY

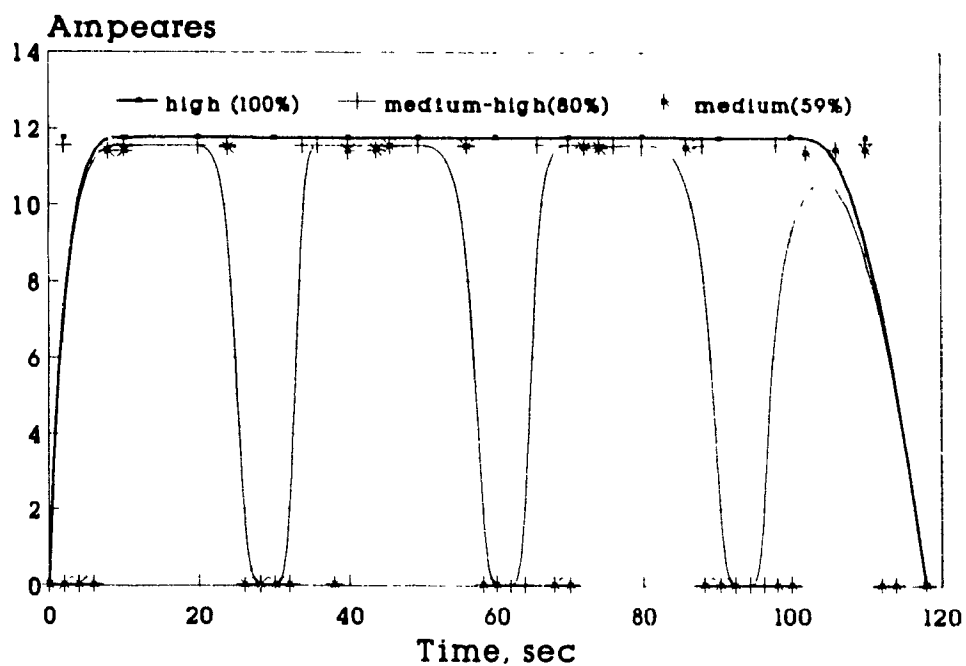


FIG 3.2 RISE IN TEMPERATURE vs. TIME

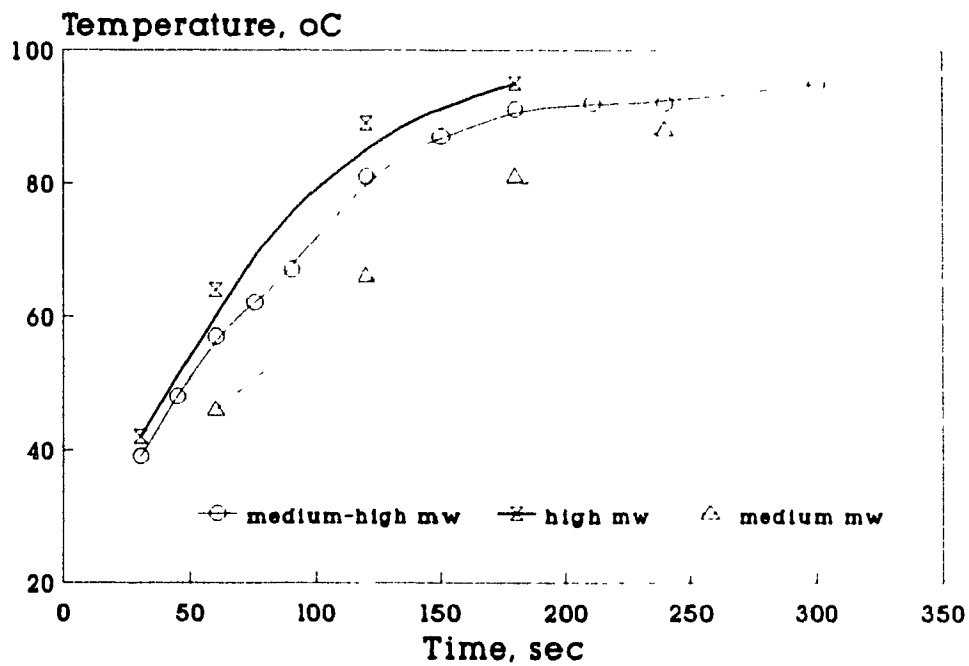


FIG 3.1 MAGNETRON AVAILABILITY

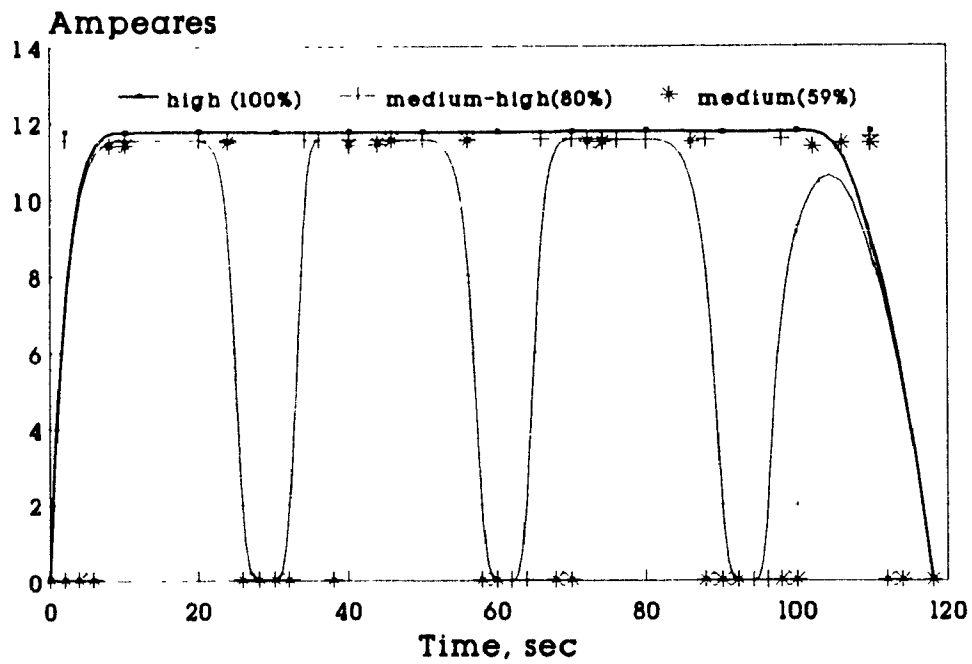


FIG 3.2 RISE IN TEMPERATURE vs. TIME

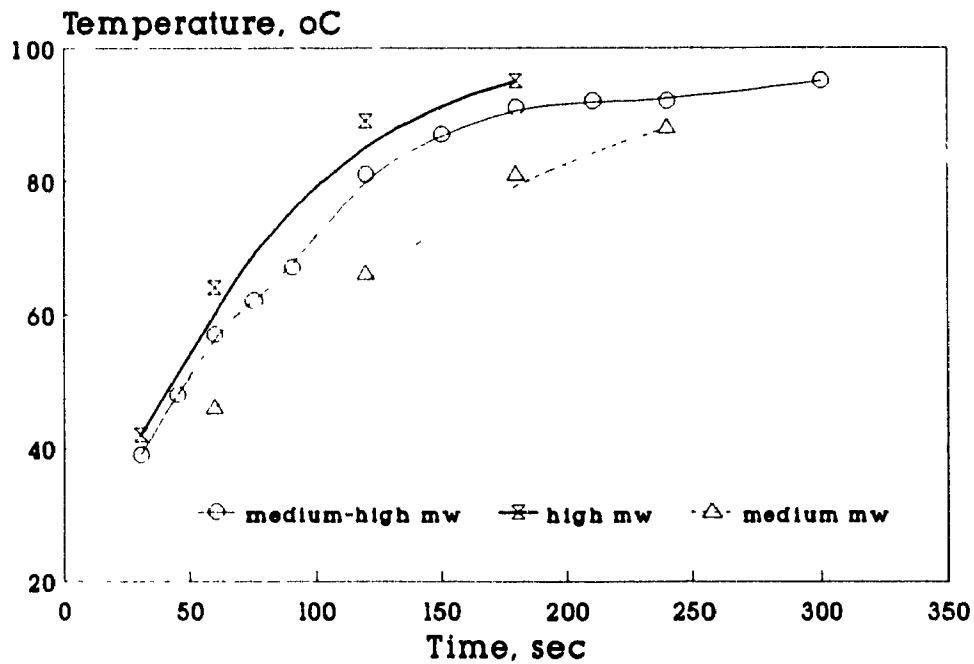


FIG 3.3 LOAD SIZE vs. POWER OUTPUT

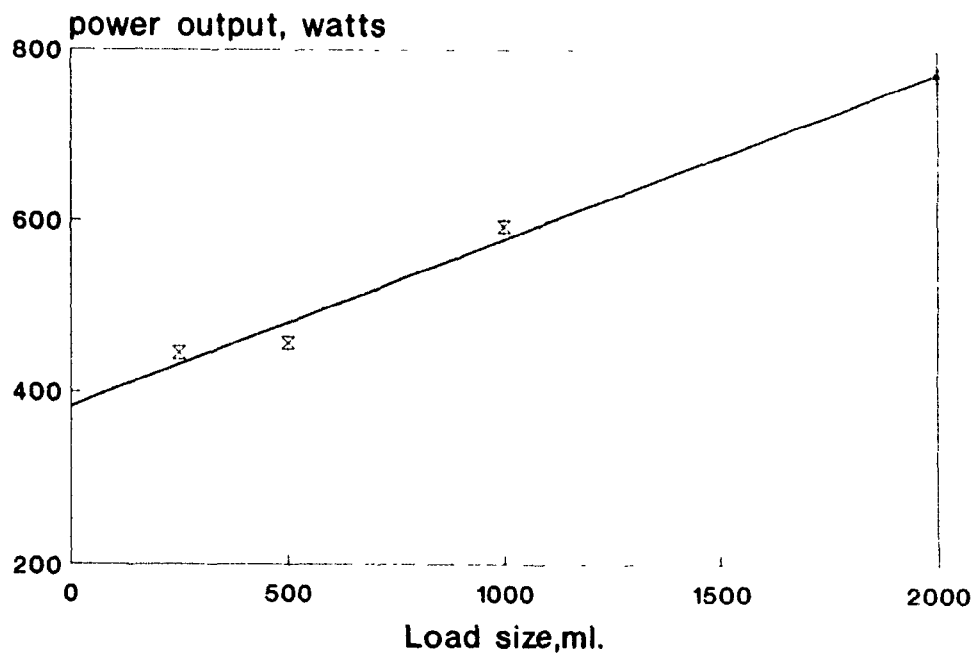


FIG 3.4 RISE OF TEMPERATURE IN HANDSHEET

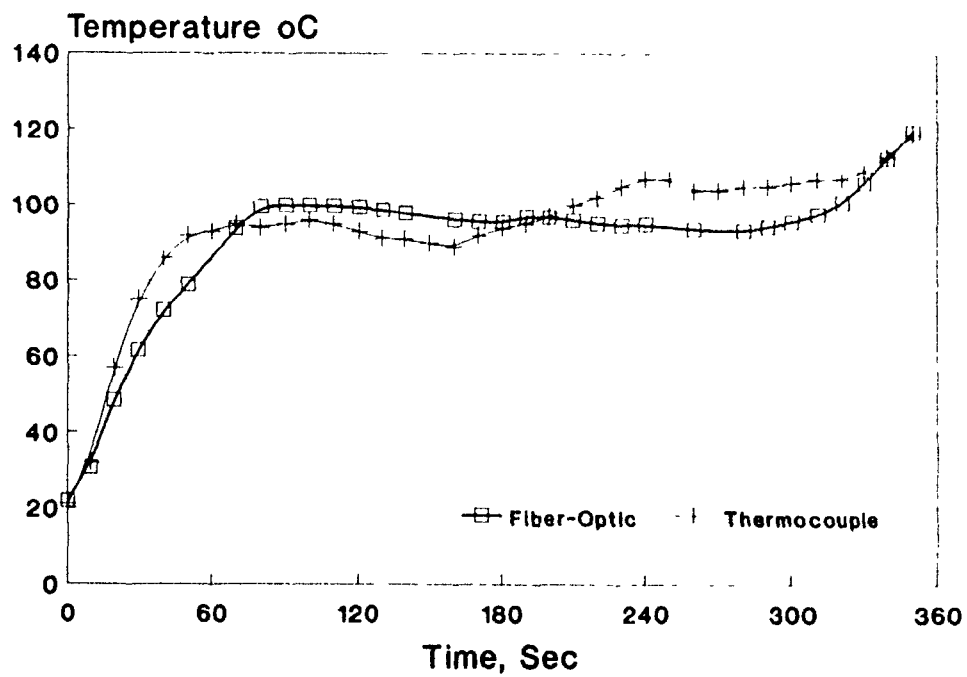


FIG 3.5 EFFECT OF DRYING METHOD ON BURST INDEX

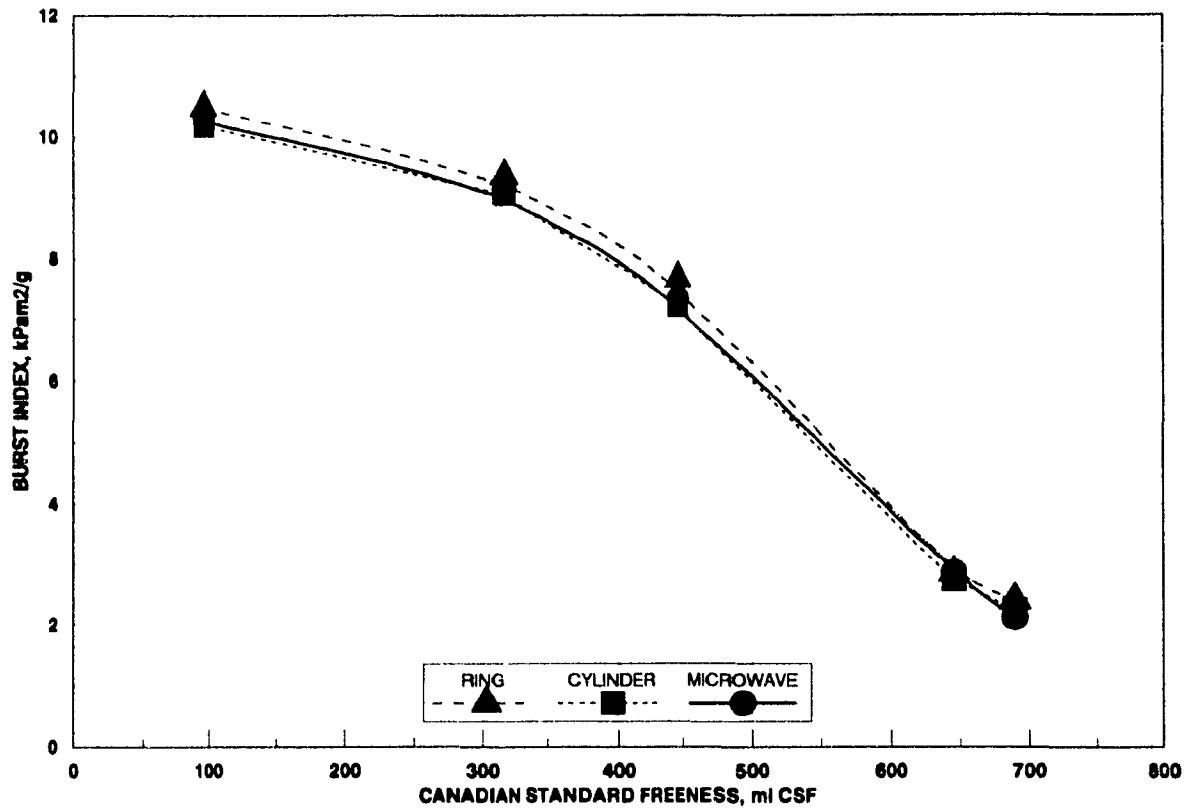


FIG 3.6 EFFECT OF DRYING METHOD ON DENSITY

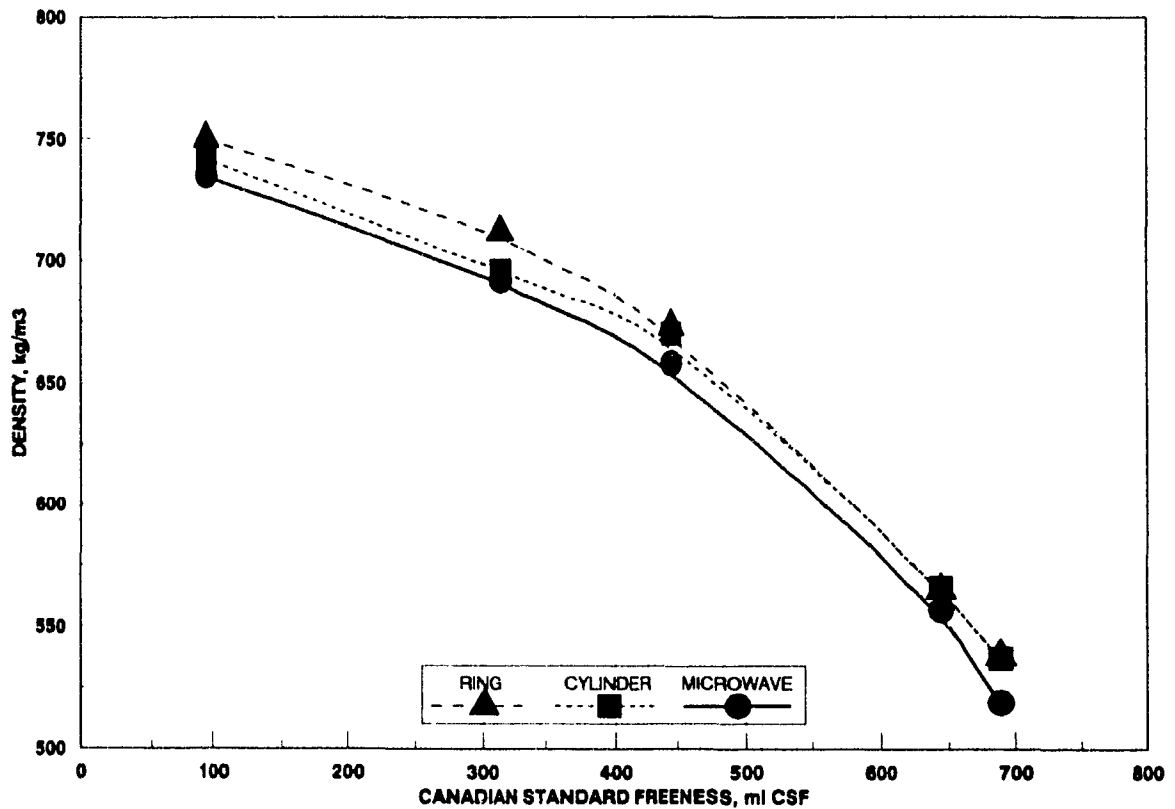


FIG 3.7 EFFECT OF DRYING METHOD ON DOUBLE FOLD

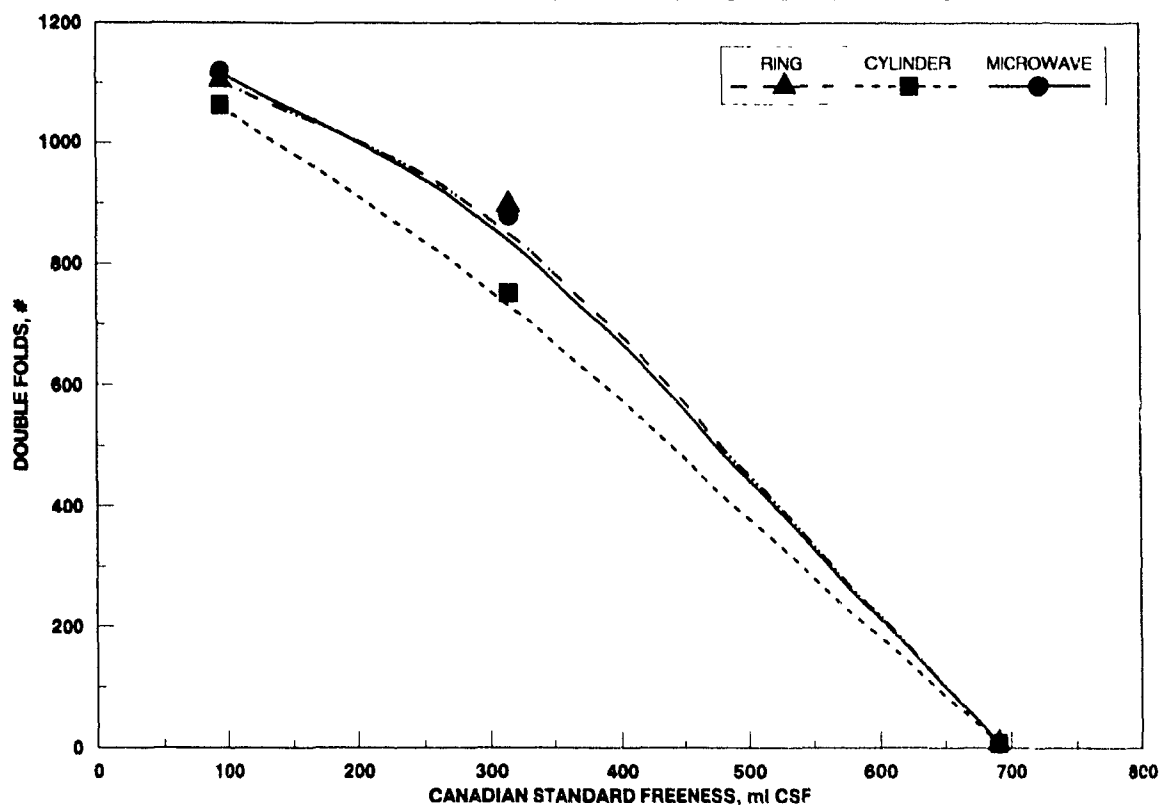


FIG 3.8 EFFECT OF DRYING ON TEAR INDEX

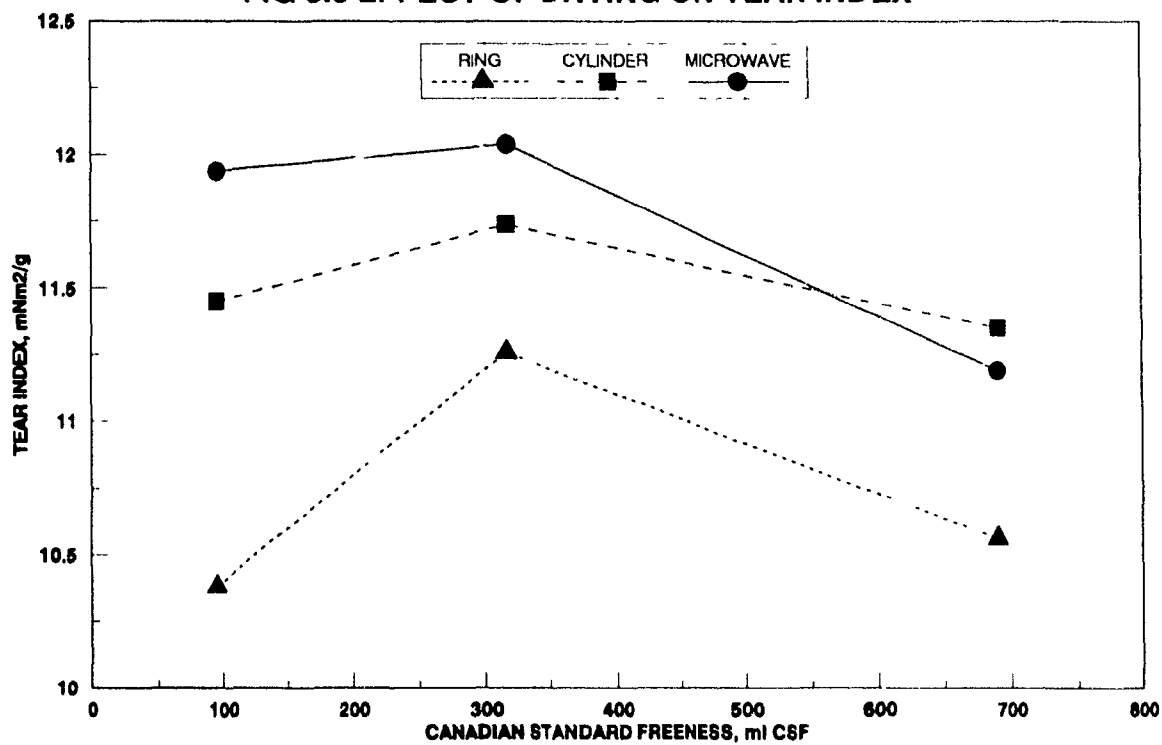


FIG 3.9 EFFECT OF DRYING ON BREAKING LENGTH

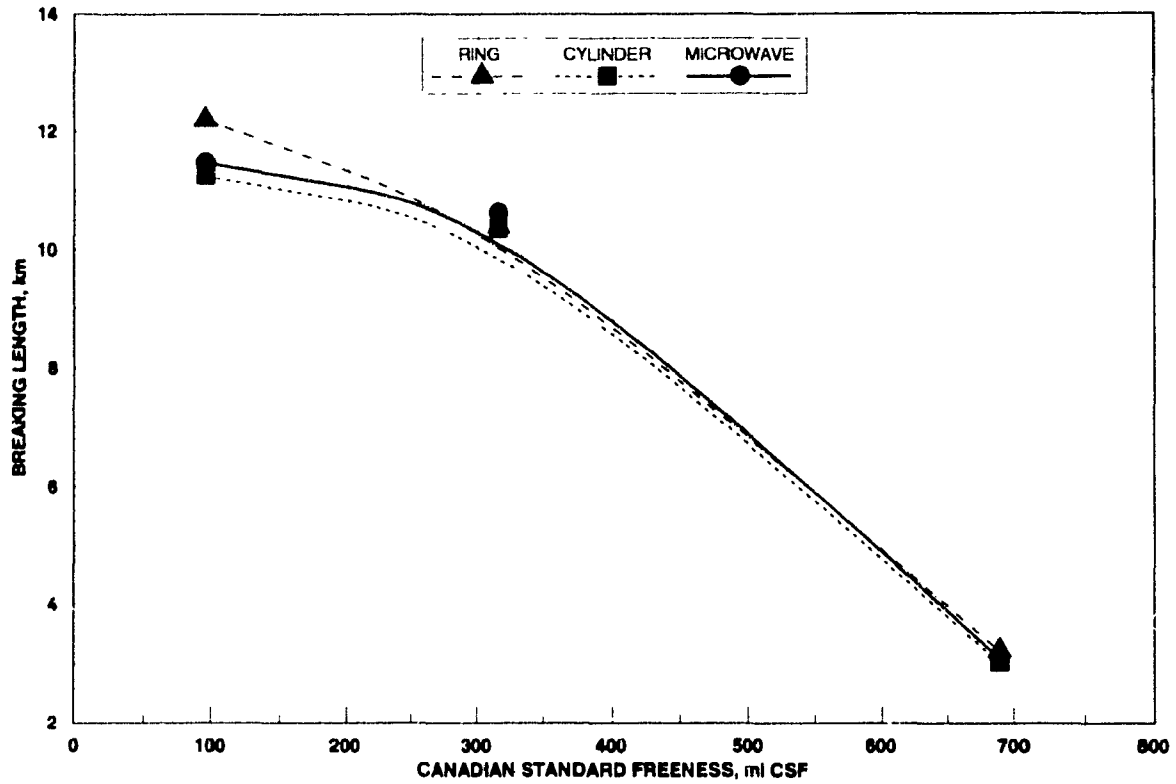


FIG 3.10 EFFECT OF DRYING ON ZERO-SPAN STRENGTH

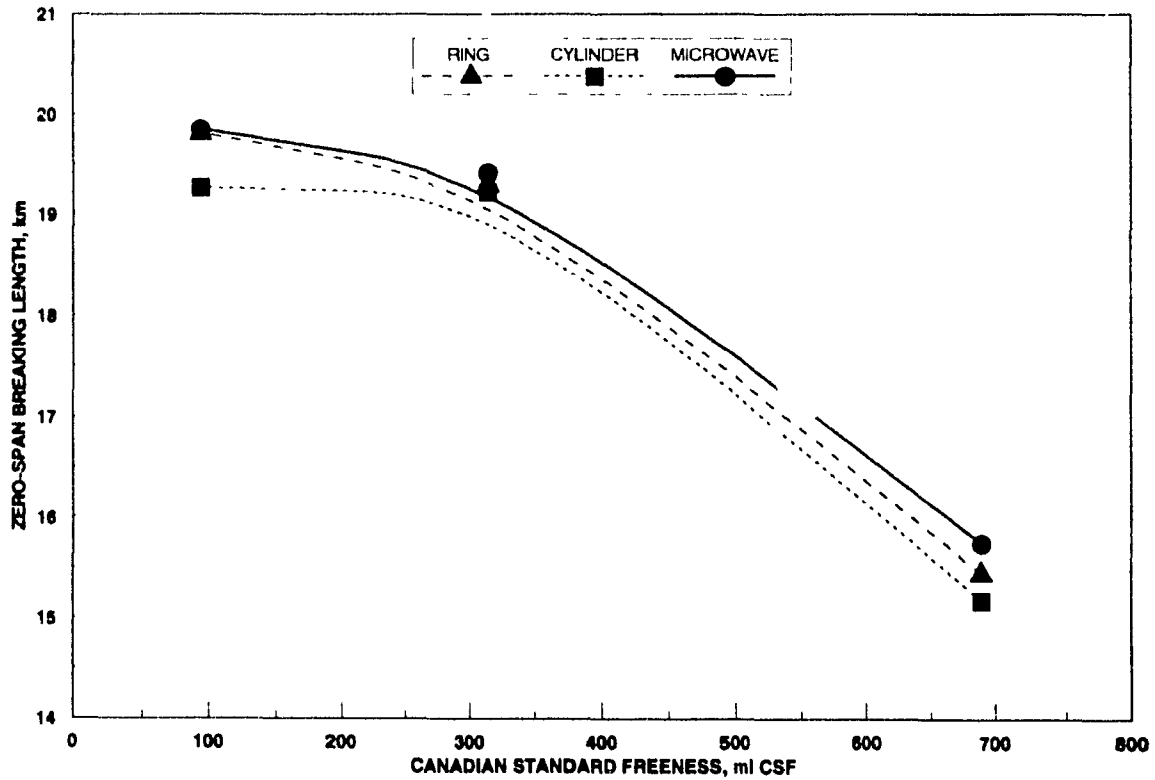


FIG 3.11 EFFECT OF DRYING ON STFI-COMPRESSIBILITY

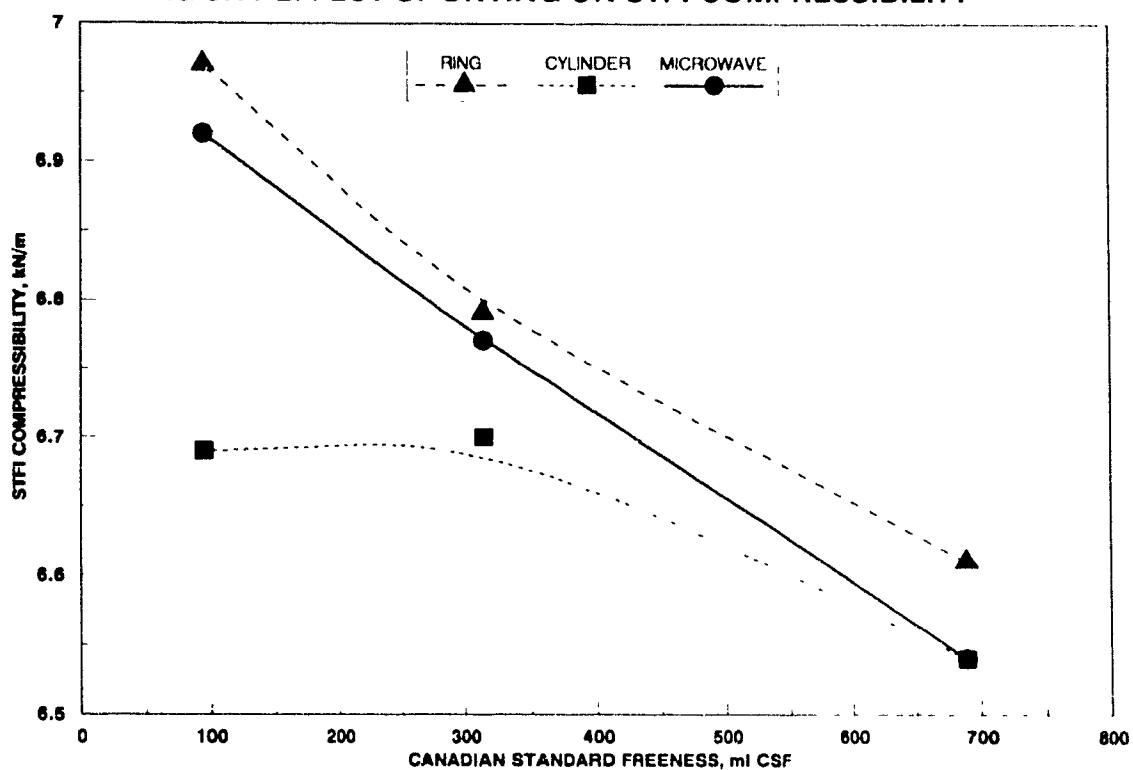


FIG 3.12 EFFECT OF DRYING ON BRIGHTNESS

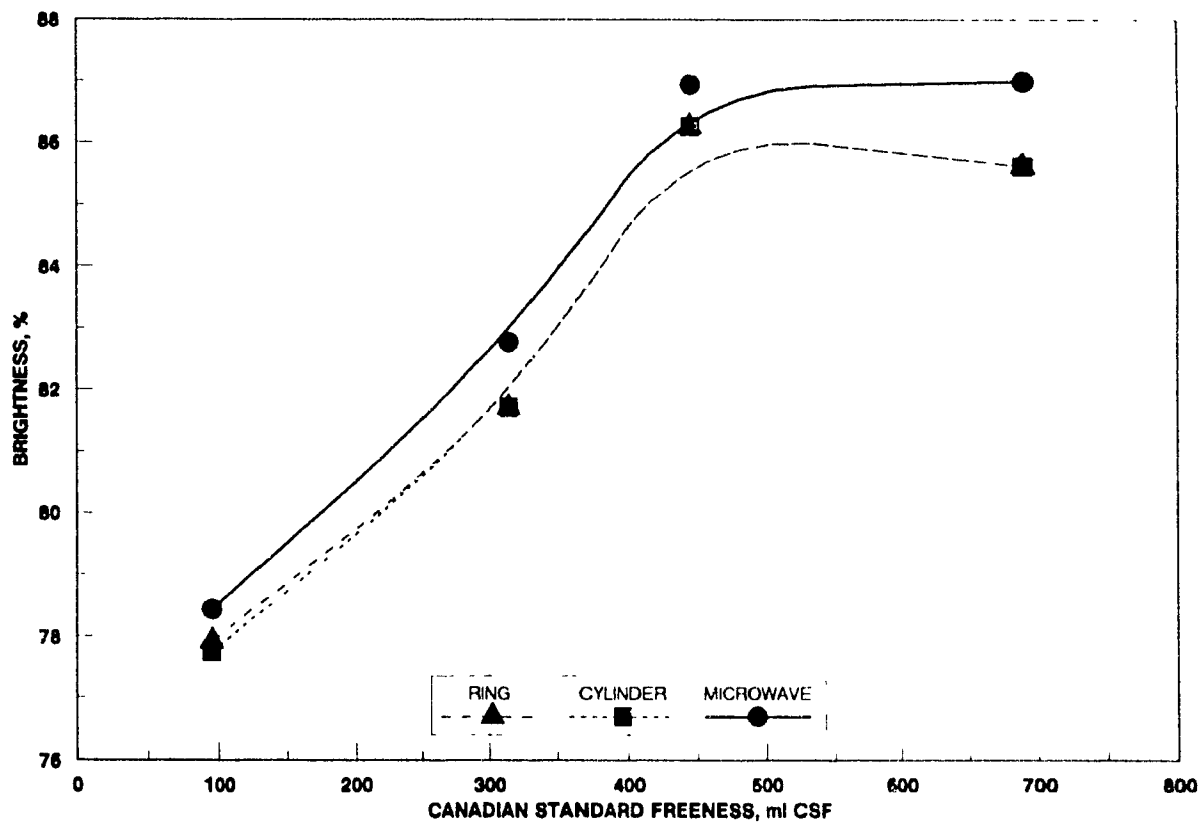


FIG 3.13 EFFECT OF DRYING ON OPACITY

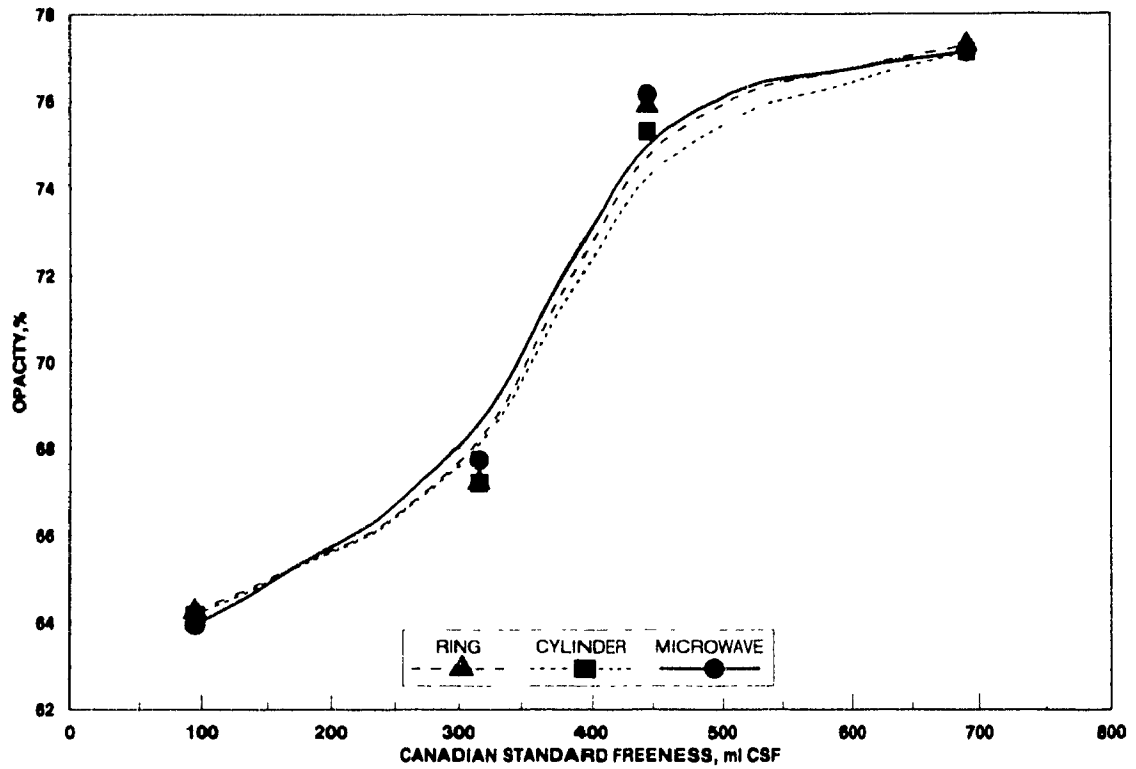


FIG 3.14 EFFECT OF DRYING ON SCATTERING COEFFICIENT

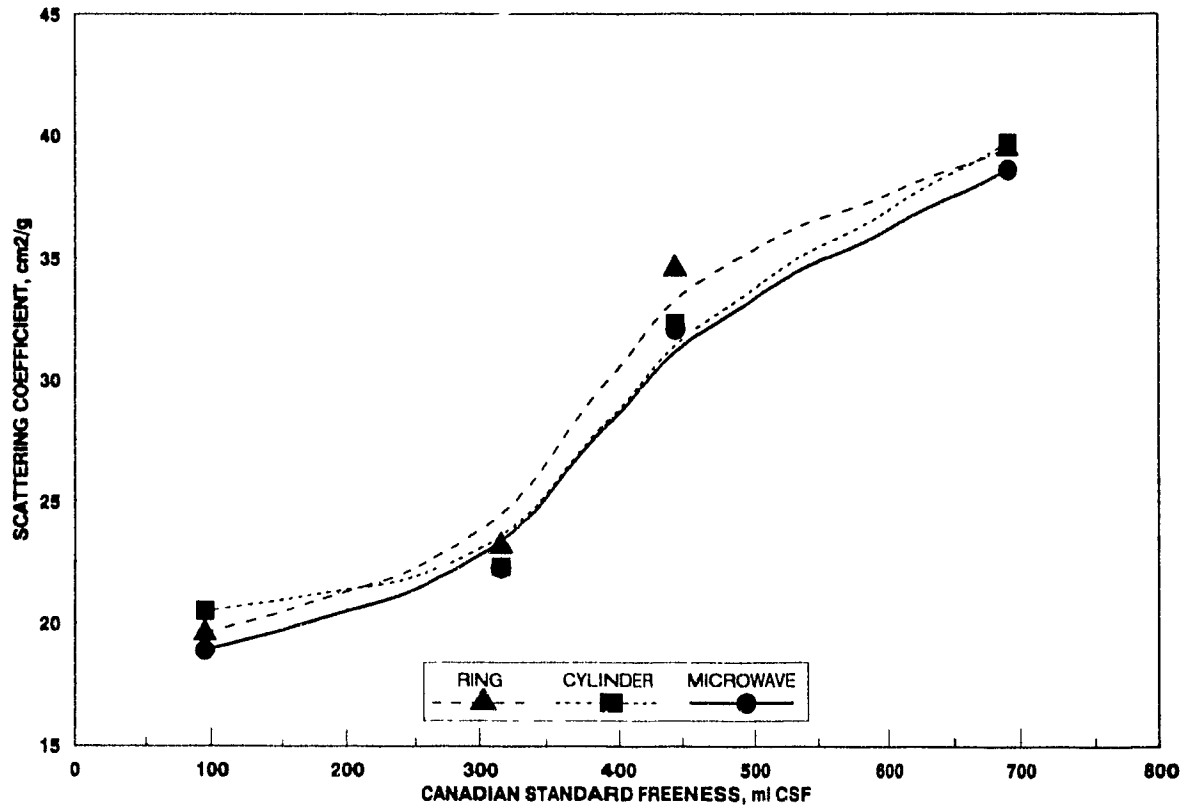


FIG 3.15 EFFECT OF DRYING ON TEAR vs BREAKING LENGTH RELATIONSHIP

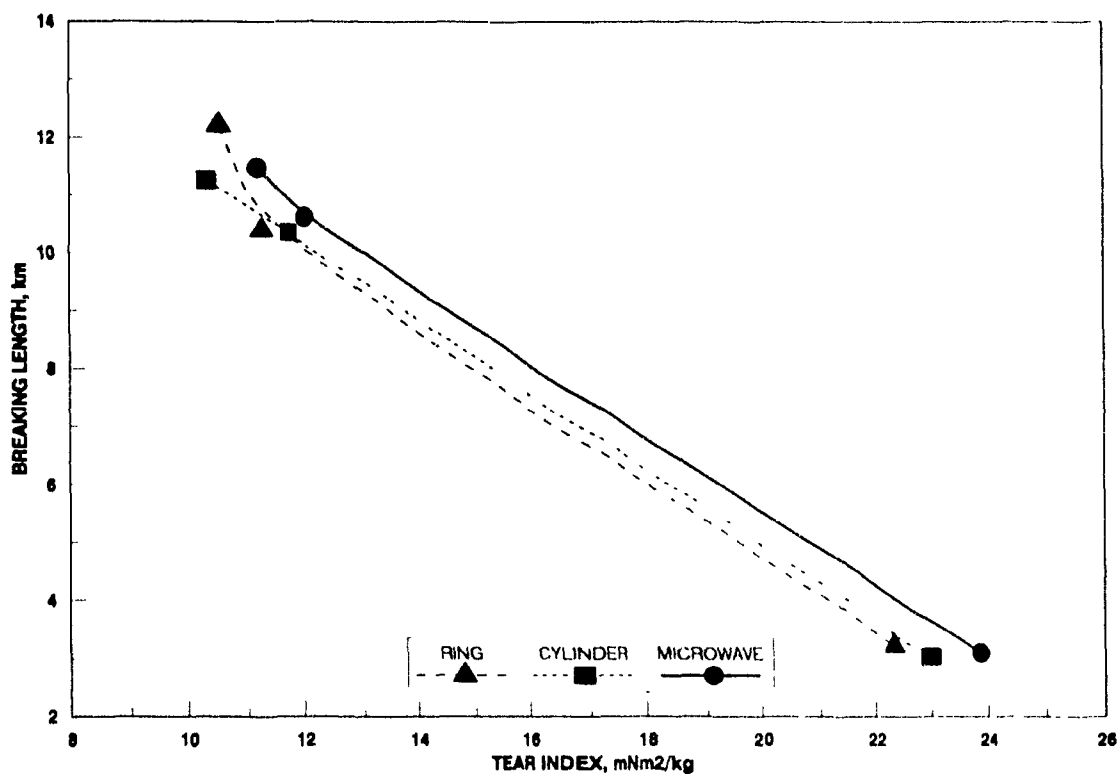


FIG 3.16 EFFECT OF DRYING ON TEAR vs BURST INDEX RELATIONSHIP

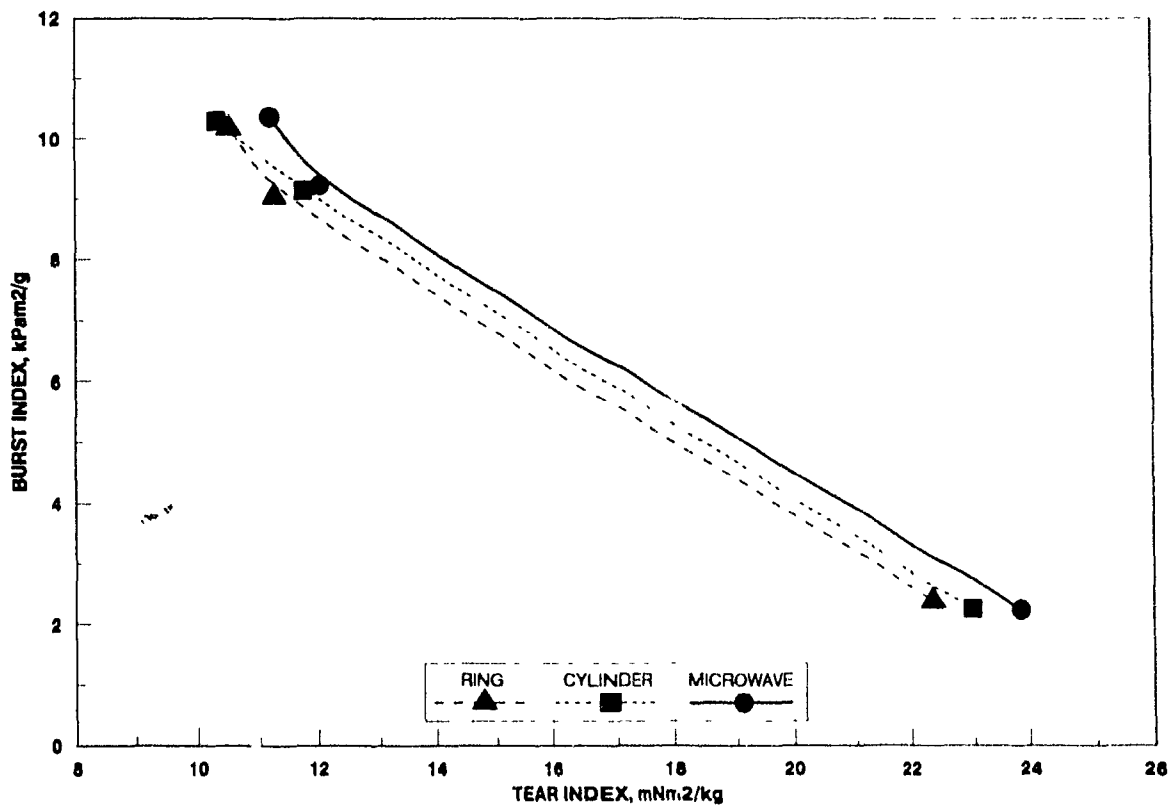


FIG 3.17 EFFECT OF DRYING ON DENSITY vs BURST INDEX RELATIONSHIP

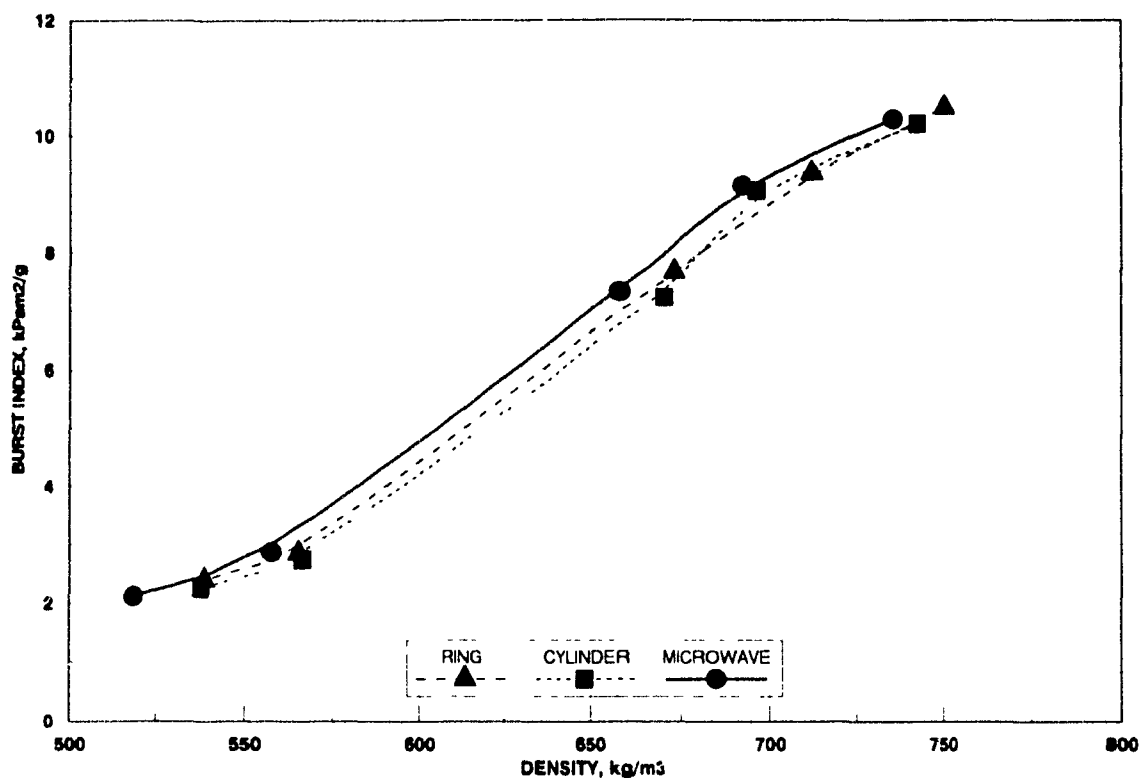


FIG 3.18 EFFECT OF DRYING ON DENSITY vs DOUBLE FOLD RELATIONSHIP

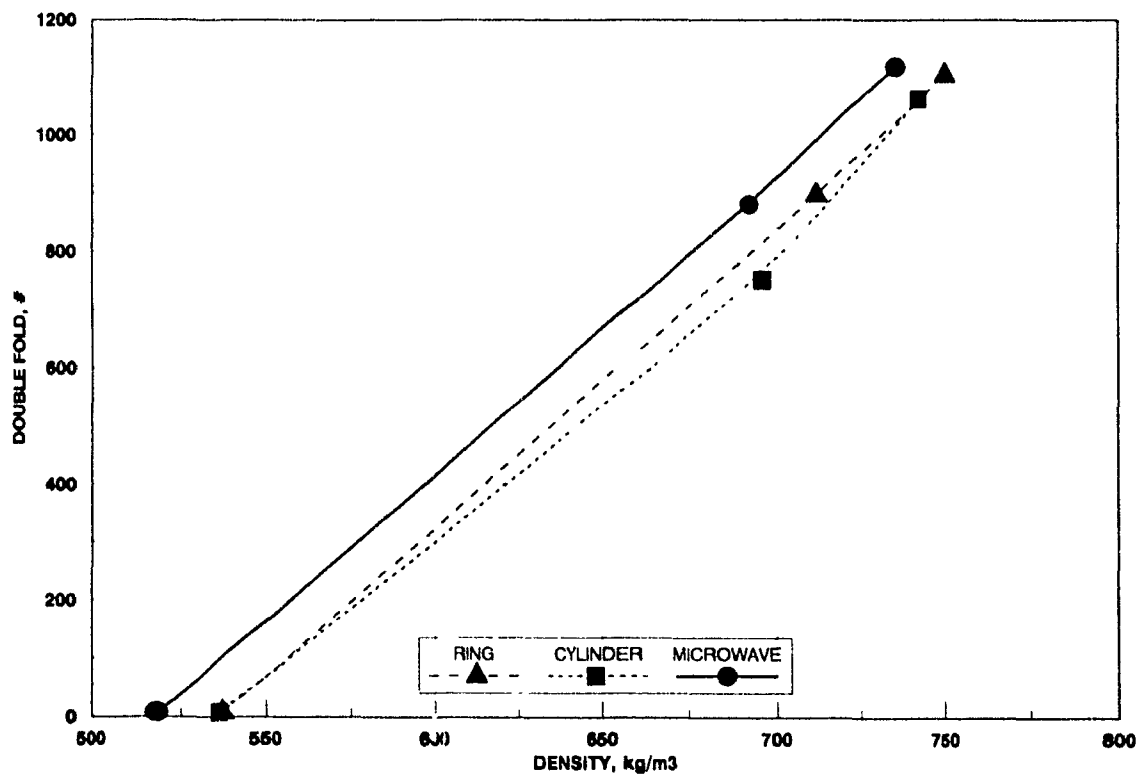


FIG 3.19 EFFECT OF DRYING ON DENSITY vs TEAR INDEX RELATIONSHIP

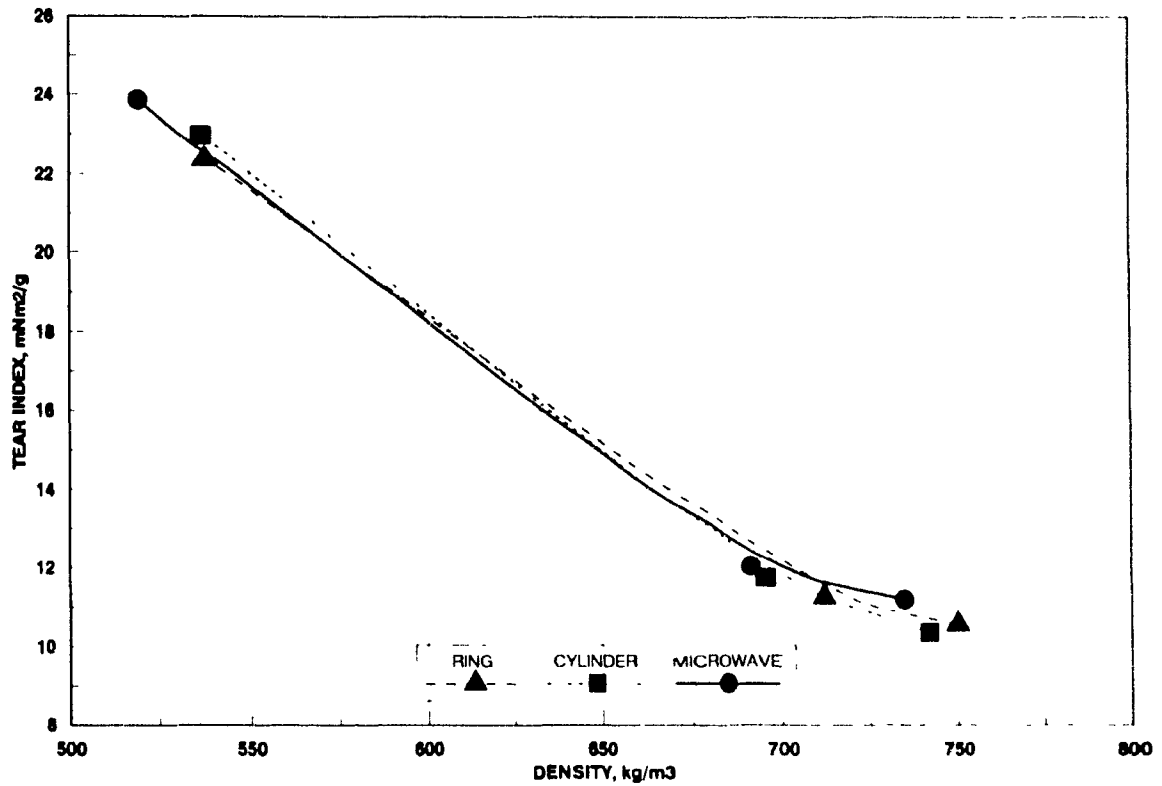


FIG 3.20 EFFECT OF DRYING ON DENSITY vs BREAKING LENGTH RELATIONSHIP

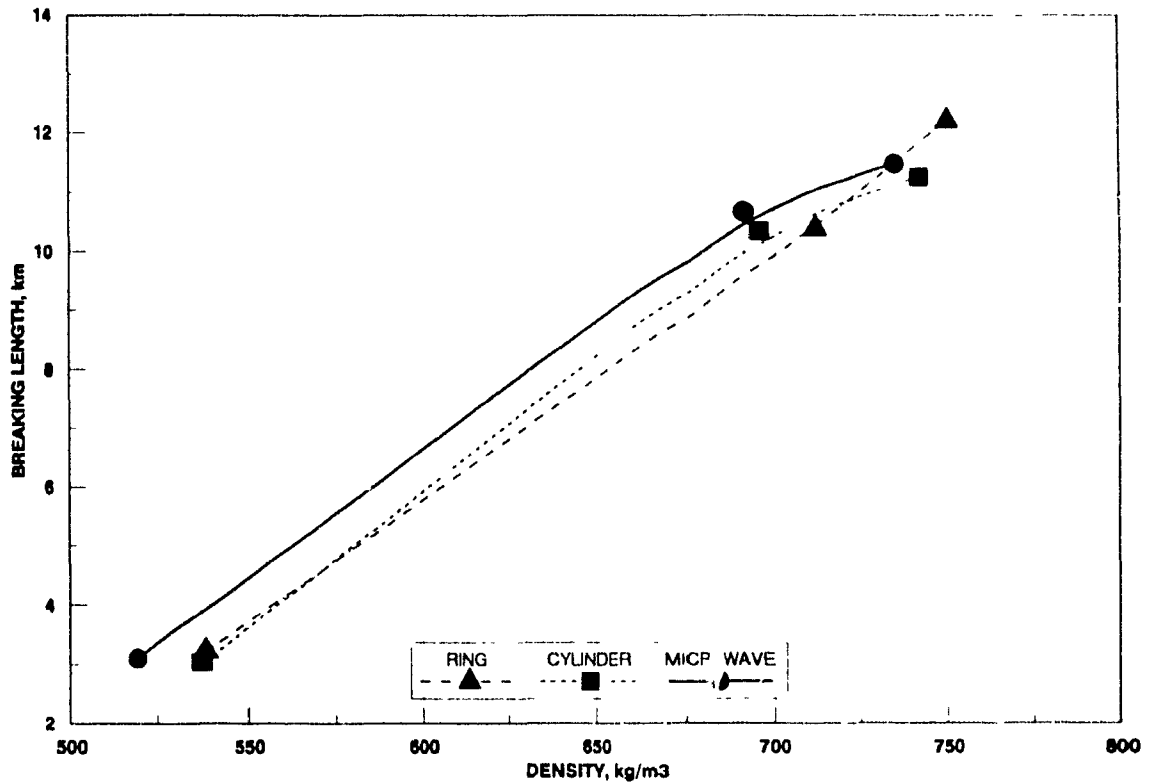


FIG 3.21 EFFECT OF DRYING ON DENSITY vs ZERO-SPAN BREAKING LENGTH RELATIONSHIP

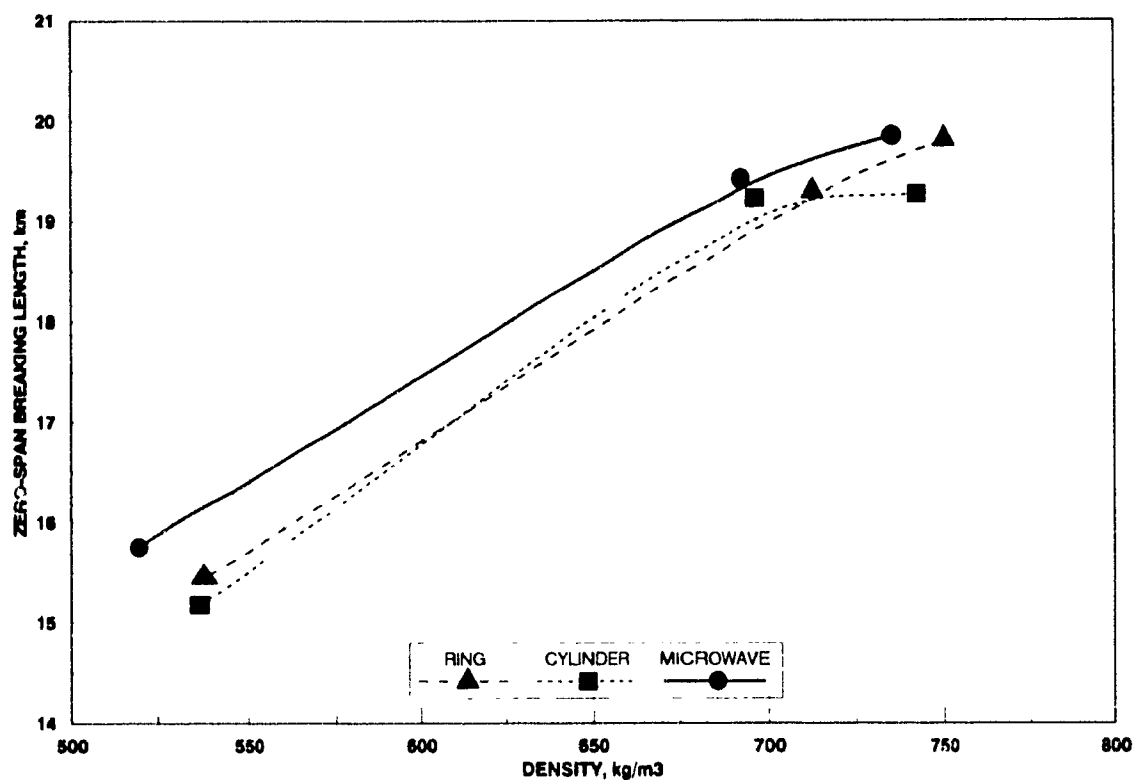
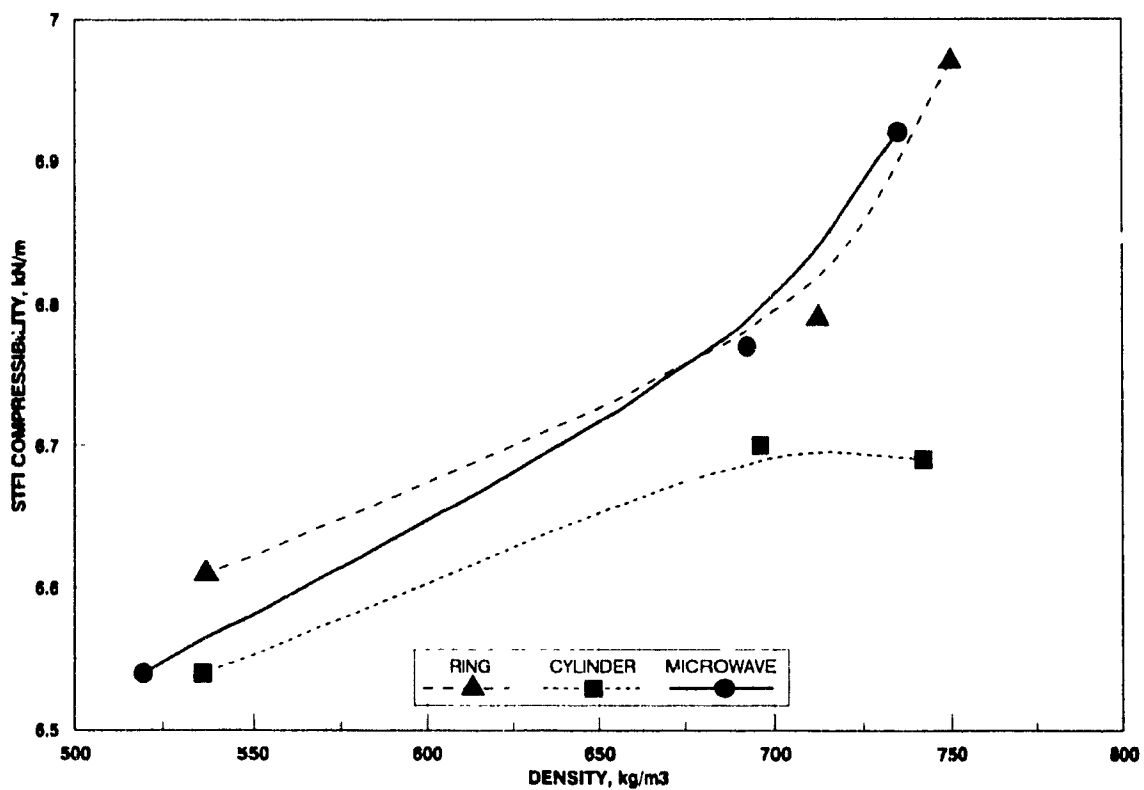
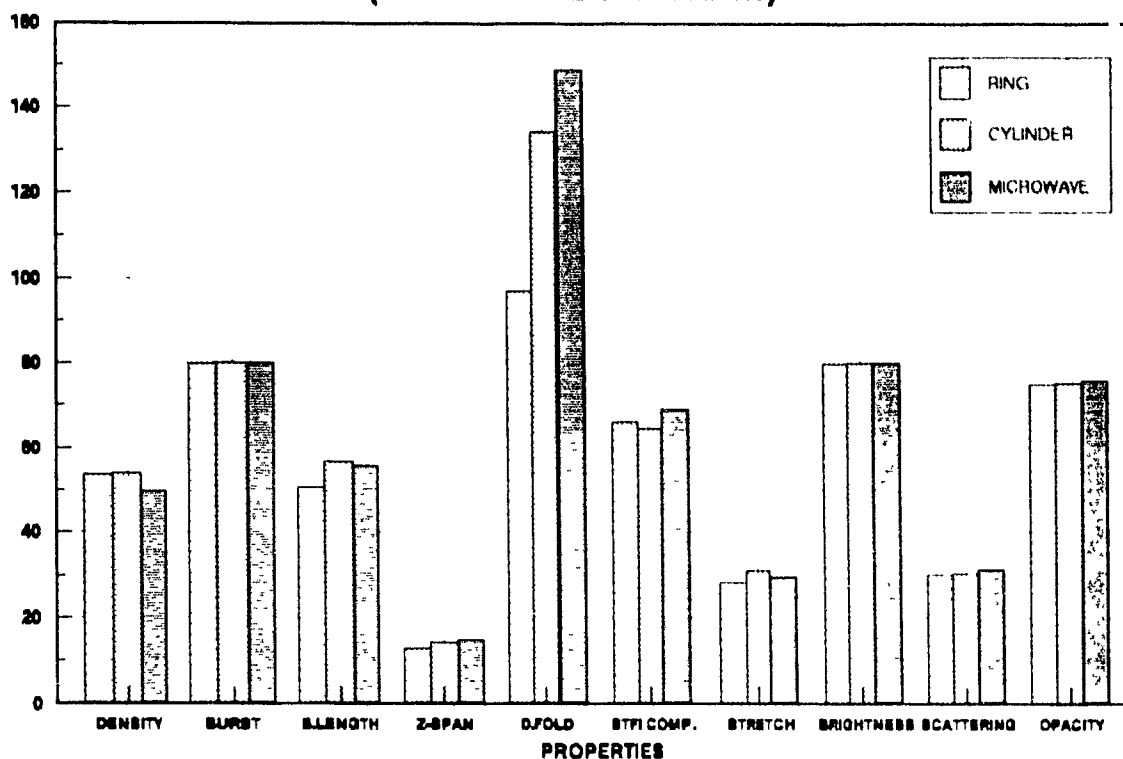


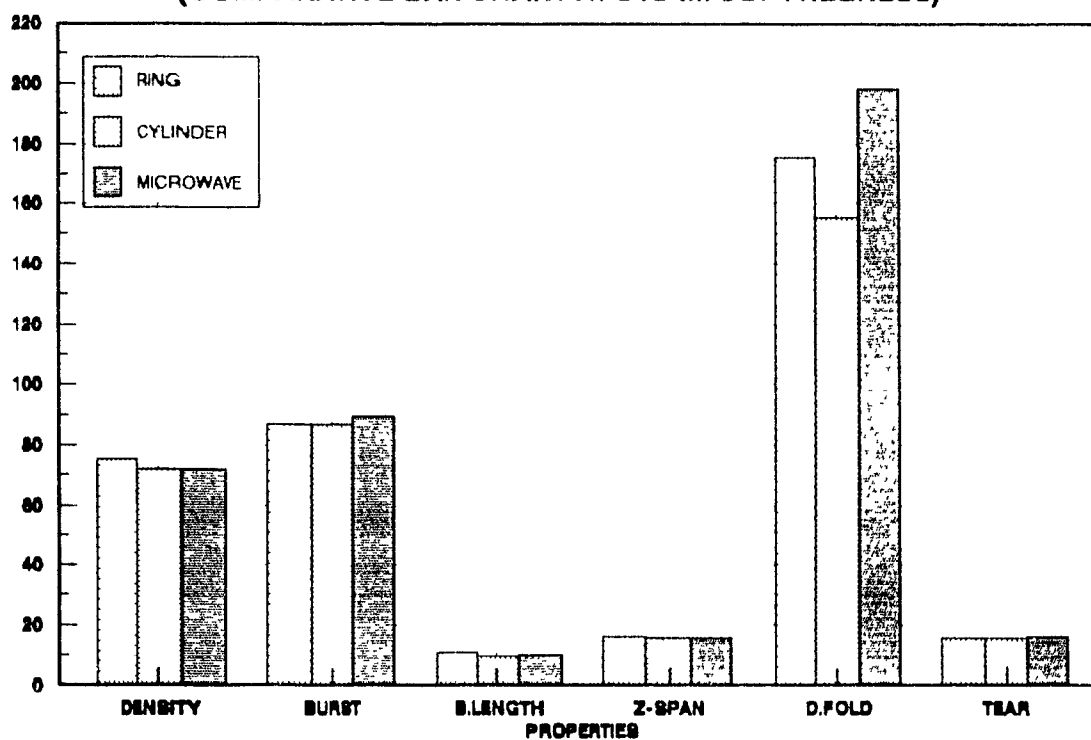
FIG 3.22 EFFECT OF DRYING ON DENSITY vs STFI COMPRESSIBILITY RELATIONSHIP



**FIG 3.23 EFFECT OF DRYING ON CTMP HANDSHEET PROPERTIES
(COMPARATIVE BAR-CHART)**



**FIG 3.24 EFFECT OF DRYING ON 120 g/m2 HANDSHEETS
(COMPARATIVE BAR-CHART AT 315 ml CSF FREENESS)**



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