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MODELING IN EXTRUSION COOKING OF BREAKFAST CEREALS

by

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A thesis submitted in conformity with the requirements for Degree of Master of Applied Science
Department of Chemical Engineering
and Applied Chemistry
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I wish to express my sincere gratitude to my supervisor, Prof. L. L. Diosady for his encouragement, advice, and financial support in conducting this research project.

A large part of the experimental work was conducted in the Weetabix' s plant, in Cobourg, Ontario. I am grateful to Susan Abel who made the arrangement for using the preconditioner and the twin screw extruder, and for her helpful suggestions and enthusiastic assistance through many long and colored runs.

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I would like to dedicate this thesis to my parents for their love and support that carried me through the difficult times in the past two years.
This project is part of a long-range research program to develop a thorough understanding of the effects of pretreatment and cooking extrusion on the structure of starch polymers.

Residence time distributions were measured using two different tracers in order to determine processing time characteristics, flow patterns as well as mixing efficiency in the preconditioner and in the preconditioner-extruder system.

The results indicate that there is poor mixing in the preconditioner which carries through the extruder.

An improved technique for obtaining RTD was developed. The technique clearly demonstrated that the pre-mixer provides poor mixing if two particle populations with distinctly different particle size are present.

The material flow inside the system can be described by two simultaneous mechanisms. Part of the material moves through an active volume, while the reminder, 10 to 30%, was held up in the dead space.
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Table 4-14 Parameter estimates for Wolf and Resnick model
Ready - to - eat (RTE) cereals have become the most popular breakfast products because of their convenience and acceptability. RTE cereals are generally manufactured from cereal grains as flakes, shreds or shaped materials. The traditional method of manufacturing a flaked cereal product begins by mixing grits, that are one-third to one-half the size of the original whole grain, with other ingredients and cooking these mixtures in rotating pressurized steam cooker with a live steam environment for 2 hours or longer. Variations of this process have been used to improve and develop new flaked cereal products. The primary difference between these processes and the traditional flaking process is the replacement of the steam-cooking step with extrusion processing. The first applications of extruders to the production of RTE cereals came in the late 1930's when they were used to form pellets from cooked cereal dough which were dried and eventually gun puffed. (Harper 1982) Extrusion is a continuous, flexible food processing operation capable of manufacturing a broad variety of final products reducing the over-all energy requirements. Extrudates are interaction products of food polymers such as starch, protein and hydrocolloids. The composition is even more complicated because of the presence of other functional components - emulsifiers, fat and lipids, salt and sugar, polyhydroxy alcohols such as sorbitol and propylene derivatives. This chemical complexity permits many interactions and by products that may be solids, liquids or gases. Extruded products thus vary in composition, texture, and shape (Shukla, 1998).
Unfortunately some variability of the product results because the extruders are extremely sensitive to variations in the raw materials: fluctuations in particle size, flour moisture, milling, starch damage and difference in protein, fat, or dietary fiber content. It has been postulated that pre-conditioning can reduce the effects of the raw material variability through extended residence times and reduced mechanical damage. Benefits of preconditioning include increased product capacity, because of the reduced mechanical energy requirement and the possibility of using larger particle size allowing extruded cereals to be close in flavour and texture to the traditionally cooked cereal.

Although they are often used, preconditioners and preconditioning-extrusion were developed and applied through empirical approaches, with no basic understanding of related processing functions. Thus this research program has the goal of developing a fundamental mechano-chemical model for the preconditioning and extrusion process.

The Food Engineering Group at University of Toronto has been working on the extrusion of starch since 1980. During this long-range research program a significant number of developments were achieved. Davidson et al. (1984a,b) investigated the physical and structural modifications that occur during extrusion cooking using gel permeation chromatography (GPC). They found that starch breaks down due to thermal and mechanical effects to lower molecular-weight macromolecules. A simple first-order model was proposed which defined the extent of mechanical degradation of the amylopectin component as a function of nominal shear stress and mean residence time in the extruder. The model, which was developed for a single screw extruder, was refined (Diosady et al.,1985) and then extended to the twin -screw extruder (Cai et al.,1992) including an improved description of starch gelatinization profiles.
Next a more detailed study of the molecular breakdown was carried out developing a rapid, accurate analytical technique for determination of molecular-weight distribution, using high-performance liquid-chromatography (HPLC) (Cai, 1992). Starch degradation occurred only in the cooking zone, reducing the fraction of the large amylopectin molecules and thus increasing the fraction of intermediate size molecules.

In this research study the techniques developed for the description of the processes occurring in the extruder were applied to the thermal pretreatment in order to develop a detailed understanding of the effects of preconditioning on the physical, chemical, functional properties of extruded products.

The first step in understanding the processes occurring in the preconditioner is the development of a model which describes the conditions experienced by the material as it is moved through the equipment.

The flow of material in paddle devices such as paddle mixers and preconditioners is not simple and intuitive because these devices are conveying solids (particulates) rather than liquids.

The residence time distribution is useful for determining processing time characteristics, flow patterns as well as mixing efficiency in the preconditioner.

The effects of several parameters such as feed rate, paddle configurations, screw speed and feed moisture were investigated. The results were compared with theoretical models derived for melt extrusion and other mixing unit operations. Also in order to describe the effects of the thermal pretreatment on the extrusion operation and to compare the difference in product characteristics we investigated the changes in physical, chemical and functional properties after the thermal pretreatment and extrusion and compared these with the effects of the traditional processes.
2. LITERATURE REVIEW

2.1 RTE Cereals

In this century, cereal grains have found significant uses as breakfast foods. Breakfast cereal technology has evolved from a simple procedure of milling grains for cereal products that require cooking to the manufacturing of highly sophisticated ready-to-eat (RTE) products that are convenient and are quickly prepared.

Ready-to-eat breakfast cereals are defined as “processed grain formulations suitable for human consumption without further cooking” (Fast, 1987)

RTE cereals fall into five broad categories: flaked, puffed, extruded, whole grain and granolas (Valentas et al., 1990).

The first form of RTE cereal products available to the consumer was flaked cereal grains and for many years, the most popular flaked cereal has been corn flakes (Lorenz and Kulp 1991). The processes for making the flakes are simple and result in products that are well cooked and have acceptable flavour. Two methods can be employed to produce these cereals: traditional and extrusion processing.

2.1.1 Traditional method

First, corn is dry milled to remove germ and bran. In this process, the endosperm is usually split into two pieces. These large pieces are the starting material for corn flakes manufacture. The large grits retain their identity throughout the process, each producing a single flake.
The corn grits are pressure cooked with a solution containing sugar, malt (nonenzymatic), and salt. Typical cooking conditions are two hours at 230 kPa of steam pressure, but different lots of corn may vary considerably in cooking time. The end point of cooking can be determined by visual inspection. Uniform translucency is desirable because it indicates that the water has penetrated to the center of the piece. This process increases the moisture content of the grain to about 34%, which is 10-15% too high for acceptable flaking. So after cooking, the lumps are broken up and the cooked grits are partially dried. The drying is accomplished in a tower dryer in which the wet product falls countercurrent to a stream of hot air (~65°C). The height of the tower may be several floors. This process dries the outside of the particles so that they are no longer sticky and after about 10 min the moisture is reduced to 21%. Since at this point the grits are not of a uniform moisture content, dry on the outside and moist in the interior, they are placed into tempering tanks to allow for uniform moisture distribution. After approximately 2 hr tempering, the grits are ready for flaking and toasting. The same method of flaking and toasting are used in both the traditional and extrusion method (Hoseney, 1994).

The entire process takes 8 hr or more to complete (Midden, 1989).

### 2.2.2 Extrusion method

The ingredients, in this case corn meal or corn flour, malt, syrup, sugar, salt and minor ingredients are preheated in the preconditioner by mixing them with water and steam. In preconditioning, heat and water must be uniformly distributed to avoid temperature and moisture gradients before feeding and cooking in the extruder.
The extruder has a defined screw configuration, and operates at approximately 150 °C in the cooking section of the barrel and 25 °C in the cooling section.

To prepare the product for the subsequent flaking and toasting, the extrusion process produces a dense extrudate, which is pushed through the discharge die in continuous strands of cooked product. After a few seconds of cooling, these strands can be cut into pellets. By varying the speed of the cutter, the size of the pellet and thus the size and the shape of the final flake can be controlled. Once cut into approximately 5 mm lengths, the cooked pellets are sent forward for flaking and toasting according to the traditional method.

Flaking occurs between two large steel rolls weighting as much as a ton each and operating at a speed differential that reshapes the pellets into thin flakes. After coming from the flaking rolls, the flakes are toasted for approximately 50 sec at 300 °C.

Toasting can be carried out in a fluid bed or belt dryer using hot air at high velocities to achieve high heat transfer rates. Hot air is directed into several zones along the dryer so that drying conditions in each section can be accurately controlled.

When the flakes enter the toaster, they still have a moisture content of around 20 %. Toasting reduces this to 3-4%, and also provides the characteristic surface blister of corn flakes. The final flakes have a crisp texture and golden brown colour.

After cooling, the flakes may be sprayed with a solution of vitamins and minerals.

The complete process, from feeding the preconditioner to the finished flakes takes less than 30 min (Midden, 1989).

Extruded corn flakes can thus be processed faster than corn flakes made by conventional method - in 30 min as compared to 8 hr.
Despite numerous advances and improvements in extrusion processing, manufactures still have some difficulties making same quality final products as those produced by the traditional, batch-cooking process (Levine, 1994).

There are a multitude of reasons for this, some of which have been discussed in the literature (Fast and Caldwell, 1990). The most common explanation is that cooking with limited water availability in the presence of shear, as occurs in the extrusion process, results in the disruption of starch granules and the dextrinization of starch molecules (Levine 1994). The poorer quality is usually observable as reduced “bowl life”, that is, the increased affinity of the cereal for water.

Another difference is that the conventional process incorporates a tempering step where significant retrogradation of the starch occurs. It has been suggested that this has a significant effect on the flakes properties and the quality of the final product.

Corn flakes quality is largely determined by texture. Textural studies have mostly focused on starch, which constitutes the majority of the flake. However, proteins may also play a significant role in texture formation. Corn generally contains 8-9% protein of which 60% are zeins, the storage protein of corn.

Protein bodies in corn changed shape and $\alpha$-zeins are released during two types of processes. In the traditional process protein bodies appeared flattened or misshaped but mostly still intact and $\alpha$-zeins are only partly released. In the extruded corn flakes, the protein bodies are completely destroyed and $\alpha$-zeins are dispersed. Therefore, the extrusion process appeared to be much harsher than the conventional process.

Released zein may form a viscoelastic protein network or matrix that could influence texture and quality of the final product (Batterman-Azcona and Hamaker, 1998).
2.2 Extrusion Cooking

2.2.1 Extrusion cooking and its application

The extruder can be considered as a complex biochemical reactor operating at high temperatures, with short residence time, under high pressures, high shear forces, on food ingredients having a wide range of moisture contents and viscosities. Derived from the plastic extruder and from the screw press used for pasta production, the food extrusion cooker has developed from the production of pet foods and snack to that of textured vegetable protein, flat bread, breakfast cereals, biscuits and a number of other products (Cheftel, 1984). The impetus for these developments has come from modern food processing’s need for: (1) continuous processes having a high throughput; (2) energy efficiency; (3) modified textural and flavor characteristics of foods; (4) control of the thermal effects on food constituents; and, (5) new and unique food products (Harper 1984).

Extrusion cooking is generally applied to starch-rich or protein-rich food mixes. Although most present applications deal with low moisture food mixes, recent research also concerns products with higher moisture contents (40-80%). The variety of food products being produced on food extruders continues to expand. Recent applications have gone far beyond the forming, cooking, expansion and texturizing of cereal and vegetable protein ingredients. The newer applications often utilize the extruder’s ability to convey and heat viscous materials. An example would be the continuous production of candies and chewing gums.
To increase product variety, the co-extrusion of food products has been an area of increased interest. Filled snacks which contain an oil emulsion filling, RTE cereal with a fruit gel or fruit flavored component, and pet foods containing multicolored textured pieces to simulate bones or other meat products are examples. Such processes require the synchronization of two extruders producing the contrasting parts of the product which are combined in a special die.

Extrusion of oil seeds, such as soy, has been shown to improve the extraction process. During extrusion the cells are ruptured, exposing the oil while making the resulting meal more dense, so greater quantities of meal can be loaded into the extractor increasing its throughput (Hayakawa and Yuki, 1992).

Another recent example of the application of extruders to the vegetable oil industry is their use to stabilize rice bran. Extrusion at 140 °C inactivates the lipase, which is responsible for the hydrolysis of rice bran oil to free fatty acids in a matter of hours once the bran is removed (Sayre et al., 1982). This makes rice bran oil available as a food source and an edible oil source.

Because of its versatility extrusion has found increasing applications in a variety of food processes and it is not easy to list all these applications. Cheftel (1990) summarized most of the industrial applications, the published potential applications and research perspectives.

2.2.2 Preconditioners and food extruders
Preconditioning has long been used in pasta and pet food manufacturing areas and has been introduced to breakfast cereal manufacture in the last decade as a means to increase twin screw extruder throughput.

Preconditioners are used extensively to preheat and prehumidify biopolymer-based raw materials such as flours and grits, by mixing them with steam and water. Benefits of preconditioning include increased product capacity by reducing the amount of mechanical energy required to cook the dough in the extruder, reduced extruder wear through reduced dough viscosity and improved product quality through more uniform cooking of dough. Larger particle size may be utilized with little change noted in finished product characteristics. Preconditioning can be considered part of the formulation and offers economical advantages. Practical experience has shown that for those products which can or ought to be extruded with high moisture content, the treatment of raw products with steam injection results not only in cost reductions, but also in quality improvement. So that, for example, the burnt taste of petfoods resulting from too high processing temperature can be avoided with preconditioning. Similarly, dextrinization in extruder-cooked cereal starches and flours can be significantly reduced, and as a result the cold paste viscosity of finished products approaches the quality of roll-dried products. (Wiedmann 1990)

Preconditioners can have two distinct designs: single paddled shaft and counter-rotating twin paddled shaft, inside an atmospheric chamber to which water and steam may be injected (Bouvier 1996). The two counter-rotating paddled shafts have considerably better mixing and forwarding capacity than single-paddled shaft versions. The location of the steam injection ports is typically along the bottom and in the front half of the chamber. Water injection occurs near the feed inlet. The paddle orientation on the shafts is usually adjustable and can be used to alter residence time and mixing.
The shafts have variable speed drives and when used in conjunction with appropriate paddle angle setting, good mixing can be achieved.

Extruders can be classified in a variety of ways. From a theoretical thermodynamic point of view, extruders are: 1) autogenous (nearly adiabatic), generating their own heat by conversion of mechanical energy in the flow process; 2) isothermal or constant temperature; or 3) polytropic, operating between the autogenous and the polytropic, with part of the energy from mechanical dissipation and part from heat transfer. These classifications become important only when modeling the behavior of the cooking extruder. This modeling is very complex because nearly all cooking extruders operate polytropically (Hauck et al. 1989).

Food extruders can also be classified by their method of pressure development: positive displacement or viscous drag. Single screw, corotating twin screw and nonintermeshing cooking extruders are viscous drag extruders. They depend on friction between the screw and barrel surfaces and the extrudate for conveying and pressure development. The intensity with which the extrudate is sheared is perhaps the most practical way to classify cooking extruders. This permits easy identification and cross reference of products to process variables and physical parameters of the extruder.

Low shear cooking extruders often have high compression and a grooved barrel to facilitate mixing but may have only moderate shear effect. Consequently they are suitable for relatively moist materials such as precooked doughs and many pet foods.

High shear cooking extruders have been classified as HTST cooking extruders because of their relatively high temperature processing at short residence time. (Linko et al. 1976). With high compression ratios and auxiliary heating and/or cooling mechanisms
they are suitable for producing a wide variety of precooked, pregelatinized, ready-to-eat cereal-based foods, textured plant proteins, snack, pet foods and animal feed.

The single screw extruder and co-rotating fully intermeshing twin-screw extruder are used most often in the food processing industry (Hauck and Huber 1989). Differences between these two kinds of extruders in design characteristics and operating principles were discussed by many authors (Fichtali and Van de Voort 1989, Hauk and Huber 1989 and Van Zuilichen et al., 1984).

Until recently, most food extruders were of the single-screw type, often modifications of the equipment originally designed for the plastics industry.

A single screw extruder is like an Archimedean friction pump. The transport results primarily from the differences in the frictional and viscous forces at the contact locations screw-product and barrel-product. For optimal pumping capacity, the fluid must adhere closely to the barrel wall and slip freely from the screw surface.

The mechanical efficiency is low, since a large part of the power supplied by the shaft is being dissipated as heat. Single screw extruders are thereby eminently suitable for processes in which the medium being transported must be heated.

Single screw extruders can generate high pressures, depending on the length $L$ of the screw, the channel depth $H$, the pitch and the apparent viscosity $\eta_{app}$ of the extruder product. (Van Zuilichen et al. 1984)

Screw design in single screw cooking extruders is quite varied. The helix of the screw can be of constant pitch and depth from inlet to discharge. Both the screw pitch and flight depth usually decrease from inlet to discharge. This is done to achieve complete
barrel fill at the varying extrudate density that is encountered in moving from the inlet to the die.

The barrel wall of the single screw extruder can be smooth, or can be constructed with longitudinal or helical grooves. These grooves force the extrudate to slip on the screw flight surface and thus be transferred from the inlet to discharge. Spiral grooves assist in pressure buildup by increasing the pumping action of the rotating screw (Hauck and Huber, 1989).

Twin-screw extruders are applied to an increasing range of cooking applications requiring better control and operating flexibility (Harper, 1989).

The screws in a twin screw extruder can be either corotating or counter-rotating (Fig. 2-1) and either fully intermeshing, partially intermeshing, and non-intermeshing. Each of these categories of extruders has distinct operating principles, functions and applications.

The non-intermeshing type operates basically on the same principle as the single screw extruder, with the frictional characteristics of the material being the main factor controlling the transport mechanism. Non intermeshing twin screw extruders are rarely used in food processing.

Mixing action is better with corotating screws. Closely intermeshing screws create a positive displacement pump action, and much less backmixing occurs than with non intermeshing screws. (Linko et al. 1976). In fact in extruders with intermeshing screws, where the screws meet and intermesh, the channels are not only restricted but also sometimes completely closed by the flights of the other screw, thereby totally impeding the rotation of the material around each screw. The material cannot rotate together with the screw, not even if it tends to stick to it.
With the rotation of the screws, the material contained in the channels is instead forced to proceed axially forward along the barrel. This action is positive and not dependent on the operating conditions (type of material, temperature, pressure, etc.), but due only to the geometrical characteristics of this type of machine (Martelli 1983)

The basic difference between single-screw and twin screw extruders is the conveying and the mixing mechanism. In a single screw -screw extruder mixing is caused by the pressure difference between the front and the rear of each flight. This type of motion can mix particles adjacent to one another but not mix particles a few flights apart unless there is a large backflow at the land or tip of the screw flights.

One of main limitation with a single screw-extruder is its limited ability to maintain process stability. Process instability is manifested in product surging (nonsteady-state flow at the extruder die), density changes, final product size variation and colour changes.

The twin screw extrusion process is more responsive to changes in screw speed than is the cooking process carried out in the single screw extruder. This is due to its feeding characteristics. By varying the speed of the screw in a twin screw extruder, it is possible to maintain more precise limits on product quality over the wear life of the barrel components.
Fig. 2-1 (a) Counter-rotating twin screw. (b) Co-rotating twin screw
(Benbow and Bridgwater, 1993)
Furthermore, certain variations in raw material characteristics can be compensated, final product colour may be more uniform, and burnt or overcooked product is reduced. The screw system is the central part of a food extruder. The screw normally has three distinct sections. The material first enters the feed or conveying section, which is an efficient pump having deep or high-pitch that transport material. Harper (1979) related many extrusion problems to the poor entry of feed into the extruder. As the material advances along the screw, the screw channels eventually fill up, and the material is compressed and worked into a uniform dough like mass in a transition or compression section, where the depth of the screw is decreased to initiate compression and processing of the product. The material finally enters a cooking (metering) section of relatively shallow flights and a reduced helix angle that provides a steady flow to the die. (fig 2-2) Other design parameters include the spacing between flights. Large spacing between flights is used to provide conveyance in a cooking section, while reduced spacing is used to pressurize the mass prior the die.

Extrusion die geometry is also important for final product quality. The shape and total area of the dies has effect on the pressure inside the extruder and the final product shape, texture and density. It is the pressure drop across the die face which causes the water in the dough to vaporize and expand the surrounding dough matrix. According to Molina et al. (1978), decreasing the die diameter increased extrusion temperature, degree of expansion, water absorption and soluble nitrogen of an extruded blend of corn and soybean. As can be expected, an increase in die diameter also decreases starch gelatinization (Chiang and Johnson 1977).
Fig. 2.2 Functional zones along the extruder channel
Pressure at the extruder die is dependent on the design, processing conditions and the formulation. Detrimental characteristics are often observed in final products with excessive and fluctuating die pressure. (Fichtali and van de Voort)

Co-rotating twin screw cooking extruders have less fluctuation in die pressure because of the more positive transport provided by the two intermeshing screws.

2.3 Modelling, optimizing and scale-up of the extrusion cooking process

When an extrusion cooking process is scaled up or transferred from one extruder geometry to another, it is difficult to compare the mass flows (Jager et al., 1992).

Food extrusion cooking has been frequently considered as difficult to scale up. The most difficult problem is in obtaining the same product qualities on a large scale extruder as achieved on a pilot plant or laboratory extruder.

This problem is even more serious for the thermal pretreatment of extruder feed materials where the design and the operating conditions applied are still empirical.

In fact, with the exception of Bouvier (1996), relevant studies about preconditioning do not exist. The study of residence time distribution (RTD) is useful for determining processing time characteristics, flow patterns as well as mixing efficiency in the preconditioner system and also product quality variations.

The determination of the RTD provides the information required to scale up preconditioners and optimize downstream unit operation such as extrusion cookers.
2.3.1 Residence time distribution theory

The measurement of residence time distribution has been extensively used to evaluate reactor performance. The residence time distribution (RTD) is a measure of the length of time process material has spent in the reactor. To obtain experimental data on the residence time in the equipment an inert tracer is added to the feed material as an "instantaneous" plug.

Mathematically, Danckwerts has defined RTD function $E(t)$ such that $E(t)dt$ is the fraction of the mass at the exit which has spent a time between $t$ and $(t+dt)$ in the system.

$$E(t) = \frac{C(t)}{\int_0^t C(t)dt} = \frac{C(t)}{\sum_0^t C(t)\Delta t} \tag{2-1}$$

Where $C(t)$ is the tracer concentration appearing at the outlet at time, $t$.

By integrating $E(t)$, the cumulative residence time distribution function $F(t)$ can readily be obtained.

$$F(t) = \int_0^t E(t)dt = \frac{\sum_0^t C(t)\Delta t}{\sum_0^t C(t)\Delta t} \tag{2-2}$$

The two most important measures to characterize a distribution are the mean residence time $\bar{t}$.
and the spread of the distribution, measured by the variance $\sigma^2$. 

$$\sigma^2 = \int_0^\infty t^2 E(t) \, dt - \bar{t}^2 \equiv \frac{\sum_0^\infty t^2 C(t)}{\sum_0^\infty C(t)} - \bar{t}^2 \quad (2-4)$$

The width of the $E(t)$ distribution curve can be described by the variance $\sigma^2$ or by the Peclet number ($Pe$) because they are related (Levenspiel, 1972):

$$(\sigma / \bar{t})^2 = 2 \left( \frac{Pe^{-1} + e^{-Pe}}{Pe^2} \right) \quad (2-5)$$

$Pe$ is normally written as

$$Pe = \frac{\bar{v} L}{D_e} \quad (2-6)$$

in which $L$ is the barrel length and $\bar{v}$ the average axial velocity, and $D_e$ the axial dispersion coefficient. The Peclet number is the parameter that measures the extent of axial dispersion. The higher the $Pe$, the narrower the RTD curve. In fact, if we consider the inverse of $Pe$, $\left( \frac{D_e}{\bar{v} L} \right)$, called the vessel dispersion number:
When \( \frac{D_L}{\bar{v}L} \to 0 \) the system has negligible dispersion, hence plug flow

and when \( \frac{D_L}{\bar{v}L} \to \infty \) the system is widely dispersed, hence mixed flow

Denbigh and Turner (1971) defined the plug flow as an idealized state of flow such that over any cross section normal to the fluid motion the mass flow rate and the fluid properties (pressure, temperature and composition) are uniform. All elements of fluid spend an equal time in passing through the reactor and pass through the same sequence of pressure, temperature and concentrations changes.

While the fluid in a vessel is said to be perfectly mixed if its properties are uniform and identical with those of the outgoing stream.

Real flow systems do not usually reflect either of these extreme assumptions.

2.3.1.1 Flow Characteristics

Although the geometry of the extruder channel and the material properties complicate the fluid-dynamic analysis, there have been several attempts to construct conceptual mass flow models to describe the flow behavior inside the extruder. In this case the residence time distribution is derived from the theoretical velocity profiles.

The flow characteristics of Newtonian fluids were analysed by Pinto and Tadmor (1970). They predicted that the average residence time is determined only by geometric
factors and by screw speed and is independent of all other operating conditions.

While Bigg and Middleman (1974) developed a mathematical model for the two dimensional flow field of non-Newtonian fluids in a screw extruder of uniform channel geometry, having assumed that the flow is isothermal and the end effects and leakage flow can be neglected. At steady state they proposed these dynamic equations:

$$\frac{\partial p}{\partial x} = - \frac{\partial \tau_{xx}}{\partial y} \quad (2-8)$$

$$\frac{\partial p}{\partial z} = - \frac{\partial \tau_{zz}}{\partial y} \quad (2-9)$$

and the viscosity function for a power-law fluid:

$$\mu = m \left[ \left( \frac{\partial v_x}{\partial y} \right)^2 + \left( \frac{\partial v_z}{\partial y} \right)^2 \right]^{\frac{n-1}{2}} \quad (2-10)$$

The shear stress components are then related to the velocity field by

$$\tau_{xz} = - \mu \frac{\partial v_x}{\partial y} ; \quad \tau_{yz} = - \mu \frac{\partial v_y}{\partial y} \quad (2-11)$$

The velocity components $v_x$ and $v_y$ can be obtained by numerical solution of the nonlinear equation above.
Bigg and Middleman discussed in detail the numerical method used to calculate the velocity field from eq. (2-8) - (2-11) and derived the residence time distribution from the theoretical velocity profiles.

The residence time for a power law fluid depends on the power law index, n, and the volumetric flow rate. The lower the power-law index n, the wider is the RTD.

These results are important to the operation of cooking extruders since food doughs are pseudoplastic materials, but the use of these results for quantitative analysis is still limited because of the assumptions and simplifications made in order to calculate the velocity profile.

The area of powder flow in paddle mixers and preconditioners is still far from being completely understood and with the exception of Levine (1995) and Bouvier (1996), engineering studies of the flow patterns inside these devices do not exist.

The problem in understanding occurs because preconditioners are conveying solids rather than liquids. Particulates (powders) behave in a different way, methods which are successful in mixing liquids are often unsuitable for the mixing of solids.

Three different mechanisms of mixing can be defined (Williams, 1990):

1. diffusive mixing, which occurs when particles roll down a sloping surface;
2. shear mixing, which occurs when slip zones are established in a powder;
3. convective mixing, which occurs when circulation patterns are set up inside a bulk powder mass.

Generally more than one of these three effects will be present in a mixer but usually it is possible to identify which of the mechanisms predominates in a given type. Paddle mixers are mainly using shear mixing. Slip zones are established by the paddles, mixing takes place by the interchange of particles between layers.
2.3.1.2 Theoretical models

An alternative method to characterize the residence time distribution is the use of theoretical models which represent the physical flow inside the preconditioner-extruder system.

Two models suggested by Levenspiel (1972) one being the tanks in series (2-12) model

$$E(\theta) = \frac{N(N\theta)^{N-1}}{(N-1)!} \cdot e^{-N\theta}$$  \hspace{1cm} (2-12)

where $N$ is the number of perfectly mixed tanks in series and $\theta$ is the reduced time

$$\theta = \frac{t}{\tau}$$

and the other being the dispersion model (2-13) were considered by Bouvier (1996) to model experimental RTD curves.

$$E(\theta) = \frac{1}{2\sqrt{\pi \left( \frac{D_c}{uL} \right)}} \exp \left( -\frac{(1-\theta)^2}{4\left( \frac{D_c}{uL} \right)} \right)$$  \hspace{1cm} (2-13)

When one-parameter models are unable to satisfactorily describe the flow systems then more complicated models must be attempted. These usually consider the real system to consist of different regions (plug, dispersed plug, mixed, deadwater) interconnected in various ways (bypass, recycle or crossflow) (Levenspiel, 1972).
For a combination of plug flow and perfect mixing a RTD model has been developed by Wolf and Resnick (1963):

\[ F(t) = 1 - e^{-b(\theta - \frac{n\varepsilon}{i})} \left[ 1 + b \left( \frac{\theta - \frac{n\varepsilon}{i}}{i} \right) + \ldots + \frac{b}{(n-1)!} \left( \frac{\theta - \frac{n\varepsilon}{i}}{i} \right)^{n-1} \right] \]  

(2-14)

where \( n \) is the number of stages in series and \( b \) can be considered to be a measure of the efficiency of mixing. For the case of perfect mixing, \( b \) is equal to unity, whereas for plug flow, \( b \) tends to infinity.

The term \( \varepsilon \) is a measure of the phase shift in the system. A positive value indicates that the system response lags behind that expected for perfect mixing, a negative value indicates an anticipatory response and may occur as a result of short circuiting.

The plug-flow/perfect mixing model given by eq. (2-14) has been shown to be adequate in describing RTD in a twin screw extruder (Altomare and Ghossi, 1986; Balke, 1985; Agur, 1986, Fichtali et al. 1995). Antila et al. (1984) found that a model which combines one plug flow and two CSTR units in series described the extruder behaviour. A further increase in the number of CSTR units did not significantly improve the model.

Fichtali et al. (1995) observed that the deviations in the data from the theoretical plug flow/perfect mixing model curves indicate that other phenomena, such as recirculation and dead space, might occur in the extruder. To take these into account, the multistage Wolf and Resnick model (2-14) was applied. The deviation of the parameter \( b \) from unity indicated that a state of perfect mixing has not been achieved due to plug flow and
dead space and the fact that $\varepsilon > 0$ indicated that the system response lags behind that expected for perfect mixing due to plug flow.

The experimental residence time distributions reported in the literature have shown that the RTD curves have long tails due to material held in dead spaces within the extruder. According to Davidson et al. (1983) the Wolf and Resnick multistage model represents the tail portion of the distribution better than the early section. The peak of distribution can be better approximated by using the Levich model (Levich et al, 1967). This model considers the dead space not to be completely stagnant but allows a slow interchange or crossflow with the active fluid.

Specific studies of residence time distribution in a food extruder have been reported by Fichtali et al. (1995), Altomare and Ghossi (1986), Davidson (1984) and van Zuilichem (1973).

Altomare and Ghossi observed that, in a twin-screw extruder of all the variables investigated screw speed, moisture content, throughput, die size, barrel temperature and screw profile, only throughput, screw speed and screw profile had strong influence on the mean residence time.

Fichtali et al. (1995) studied the performance of an extrusion process to convert acid casein to sodium caseinate and found that the mean residence time decreases with increasing screw speed, temperature, or feed rate. Feed rate and temperature also affect the shape of the distribution, indicated by the variance $\sigma$.

Davidson et al. (1983) reported that RTD is primarily affected by screw speed and moisture. A change in the screw speed shifts the distribution. Barrel temperature has only a small effect on the mean of the residence time distribution.
The aim of this study was to investigate the effect of several parameters such as feed rate, paddles configurations and shaft speed on the residence time distribution for a preconditioner and to understand the influence of the pretreatment on the flow distribution inside the extruder. The experimental results were compared with theoretical models derived for melt extrusion and other mixing unit operations for characterizing the observed residence time distributions in the system preconditioner-extruder system.

2.4 Starch modification during extrusion cooking

2.4.1 Starch Chemistry

Starch is found in plants in the form of granules. In cereal and other higher plants, granules are formed in plastids. Those plastids that form starch are called amyloplasts. In the cereal with simple starch granules (wheat, corn, rye, barley, sorghum and millets) each plastid contains one granule. In rice and oats, which contain compound starch granules, many granules are found in each amyloplast (Hoseney, 1994).

Starch granules vary in size from 2 to 100 \( \mu m \) and may be round, oval, or irregular in shape (Greenwood, 1976).

Starch granules are birefringent. Birefringence refers to the characteristic cross appearing on the ungelatinized granule in polarized light and the loss of birefringence is taken to indicate the irreversible loss of orderly molecular orientation. X-ray diffraction studies show that the molecular arrangement in a starch granule is such that there are crystalline regions imbedded in an amorphous matrix.
Because starch is synthesized in plastids, those structures must possess all the enzymes necessary for granule formation. The starch grows by apposition. The new layer deposited on the outside of the granule varies in thickness, depending upon the amount of carbohydrate available at the time.

2.4.1.1 Chemical and physical properties

Starch is composed essentially of polymerized glucose. Cereal starches contain low levels of fats. The lipids associated with starch are generally polar lipids, which require polar solvents such as methanol-water for their extraction. Generally, the level of lipids in cereal starch is between 0.5 and 1%. Besides low levels of other minerals, starches contain phosphorus and nitrogen. In the cereals, most of the phosphorus is in the form of phospholipids. All starches also contain low levels of nitrogen (<0.05%); part of this is from the lipids, and the remainder may be proteinaceous, perhaps remnants of enzymes involved in starch synthesis.

Starch is basically a polymer of α-D-glucose. Chemically, at least two types of polymers are distinguishable: amylose, an essentially linear polymer, and amylopectin, which is highly branched.

Amylose is generally assumed to be a linear polymer of α-D-glucose linked α-1,4 (Fig. 2-3). The molecular weight of amylose is around 250,000 but varies quite widely, not only between species of plants but also within a species and depends upon the plant's stage of maturity.

Like amylose, amylopectin is composed of α-D-glucose linked primarily by α-1,4 bonds. Amylopectin is branched to a much greater extent than is amylose, with 4-5% of the
glycosidic bonds being α-1,6 bonds. This level of branching means that, on the average, the unit chain in amylopectin is only 20-25 glucose units long. The molecular weight of amylopectin has been reported to be as high as \(10^8\). Its large size and highly branched structure are responsible for the high viscosity of amylopectin dispersions.

How the amylose and amylopectin molecules are arranged in the starch granule is not known with great certainty. It appears that most, if not all, of the molecules in a starch granule are oriented at a right angle to the granule's surface. The amylopectin molecules in the granule appear to be the ones that are partially crystalline.

The starches from different cereals vary widely in their size, shape, and gelatinization properties. The ratio of amylose to amylopectin is relatively constant for a given species. Corn and wheat have a higher amylose content (about 28%), compared with potato and tapioca (about 20%). The waxy starches contain no amylose fraction. However, mutants that have greatly changed ratios are known in a number of cereals.
Fig.2-3 (a) Glucose unit. (b) Linear - chain structure of amylose molecules.
(c) Structure of amylopectin branching points (Swinkels, 1985)
2.4.1.2 Starch gelatinization

Starch granules are insoluble in cold water but swell in warm water. When aqueous suspensions of granules are heated, a temperature is reached at which the hydrogen-bonding forces holding together the constituent molecules of the granules are weakened to the point that the granules can absorb far more water. This temperature is called initial gelatinization temperature (Dengate, 1976).

Gelatinization or partial gelatinization of starch is responsible for large changes in food properties that occur during food preparation or processing. The definition for gelatinization and the terminology associated with starch phenomena was reviewed by Atwell et al. (1988).

Starch gelatinization is the collapse (disruption) of molecular order within the starch granule manifested in irreversible changes in properties such as granular swelling, native crystalline melting, loss of birefringence and starch solubilization. The point of initial gelatinization and the range over which it occurs is governed by starch concentration, method of observation, granule type and heterogeneities within the granule population under observation.

Pasting is the phenomenon following gelatinization. It involves granular swelling, exudation of molecular components from the granule and eventually total disruption of the granules.

Gelatinization phenomena start at the center of the granule and swell rapidly to the periphery. Gelatinization occurs initially in the amorphous as opposed to the crystalline areas of the granule, because the hydrogen bonding is weakest in those areas.

Starches from different cereals have widely different gelatinization properties.
Typical gelatinization values for wheat starch are initial gelatinization temperature 58 °C, midpoint 61 °C and end point 64 °C. The last point is often referred to as the birefringence end point temperature.

In systems with limited water, the gelatinization range appears to broaden with increasing starch concentration. (Ghiasi et al. 1986).

According to de Willingen (1976) corn and wheat granules may swell up to 30 times their original volume and potato starch granules up to 100 times their original volume, without disintegration.

The factors governing gelatinization are many e.g., amylose content and size of the granules, but these are not fully understood.

Since gelatinized starch plays an important role in determining the structural and textural properties of many food products, numerous methods were developed for its estimation. Gelatinization can be followed by the increased susceptibility of the granule to enzymatic degradation (Shetty et al. 1974) while changes in the state of organization of the water in the granule enable the phenomenon to be studied by magnetic resonance techniques (Lelievre and Mitchell 1975). The colorimetric measurement of the starch-iodine complex is probably the most rapid and simple technique available.

### 2.4.2 Starch gelatinization during extrusion

Many research studies have adopted degree of gelatinization as a means of following the degree of thermal and mechanical processing during cooking extrusion of starch-based products.
Lawton et al. (1972) investigated the effects of fifteen extrusion variables on the gelatinization of corn starch. They found that the important variables are: moisture content, temperature, screw speed and screw geometry.

The interaction of temperature and moisture significantly affects starch gelatinization. Increasing extrusion temperatures increases starch gelatinization when moisture contents are between 18 and 27%. (Chiang and Johnson, 1977).

Moisture contents do not significantly affect starch gelatinization at low temperature (65 - 80 °C) but affect gelatinization at high temperature (95-110 °C).

Bhattadharya and Hanna (1987) indicated that the degree of gelatinization decreased with increasing moisture. This effect might be due to the high shear or high mechanical energy input at low moisture content.

Owusu-Ansah et al. (1983) reported for corn starch a maximum gelatinization at 100 °C when the feed had a moisture content of 23%. It decreased slightly with further increase in barrel temperature. This may be due to the formation of resistant starch and starch-lipid complexes (Mercier et al. 1980) at high temperature.

Also according to Chiang and Johnson increasing shear rate (screw speed) decreases starch gelatinization. The effect of screw speed is also dependent on screw geometry; therefore, it is not easy to compare the results from different extruders.

The profiles of starch gelatinization along a twin-screw extruder channel was determined by Cai et al. (1992). They found that the extruder channel could be divided into two major functional zones: a solid conveying zone and a cooking zone. The physical transition from solid to melt occurred in a relatively short section at the beginning of the cooking zone.

The gelatinization of starch during extrusion cooking took place only in the cooking zone.
There are very few studies of the kinetics of starch gelatinization at low moisture content, i.e. between 15% and 40 %, at which extrusion cooking processes are mostly done. Bhattacharya and Hanna (1987) published the results of an investigation of the kinetics of starch gelatinization during extrusion with a single screw-extruder, but their results do not describe the actual kinetics of gelatinization inside the extruder channel, since they used the data only from the end products.

According to Cai (1992) the gelatinization of starch initially followed a pseudo-second-order rate law, but the rate soon reverted to pseudo-first-order. Thus the overall process could be approximated by a first order model, since the second-order region was short. As indicated by Wang et al. (1989), starch gelatization is not an elementary reaction; a series of reactions may be involved in the process so that only global reaction kinetics can be obtained as long as the reaction mechanism of gelatinization is not fully understood.

The rate constant was a function of both shear stress and temperature. Since starch gelatinization takes place in a shearing environment and the results proved that shearing has a significant effect on starch gelatinization.

### 2.4.3 Rheological changes

The effects of extrusion on starch structure have been examined by a number of authors using a variety of methods. These methods include the measurement of: past viscosity over a heating and cooling cycle, water absorption index (WAI), water solubility index (WSI), X-ray diffraction and differential scanning calorimetry (DSC).
Changes in viscosity of extrudate powders, produced under various operating conditions have been studied to determine the factors affecting pasting properties (Anderson et al. 1969; Mason and Hoseney, 1986; Lawton et al. 1972).

The parameters that are usually measured to characterize pasting properties included:

- Initial Viscosity which is equal to the viscosity obtained at the beginning of heating period.
- Peak Viscosity which is the highest viscosity attained by the paste during the heating period.
- Hot Paste Viscosity which is equal to the viscosity of the paste at the end of the heating cycle.
- Cold Paste Viscosity which is obtained as the viscosity of the paste at the end of the cooling cycle.

Mason and Hoseney (1986) investigated operating variables affecting cold and hot paste viscosity of extrusion cooked wheat starch by using the AmyloViscograph. They concluded that hot paste viscosity was effected by die temperature and by an interaction between screw speed and barrel temperature. Davidson et al. (1984) observed a shift in the molecular weight distribution of extruded starch toward larger molecular fragments (less degradation) when lower screw speed and higher temperatures were used. They explained this as an increased importance of thermal degradation as the dough temperature increased. At higher temperatures, mechanical degradation was less pronounced as the dough viscosity decreased, while the kinetics of thermal degradation became more favorable.

Paton and Spratt (1978) examined the changes in the rheological behaviour of extruded starch and wheat flour. Analysis of their data for initial cold viscosity and hot viscosity
indicated that the moisture level of the feed and the barrel temperature were important variables which determined the functional properties of the starch extrudate.

According to Hoseney et al. (1992) cold paste viscosity was affected by an interaction between moisture content and production rate.

Kim (1984) studied the effect of moisture level and shear rate on solubility and viscosograms of the extrudates. He found that cold viscosity increases gradually with increasing moisture content of the raw material and high shear rate. These results indicated that starch is not only gelatinized by heat and moisture but also degraded to soluble dextrins by friction between starch granules. The relative importance of the two reactions depends on the extrusion parameters, especially shear in the barrel and moisture content of the feed product.

Other factors affecting final product paste properties include particle size and composition, starch-lipid interaction, starch quality, soluble fiber and the effects of other ingredients such as sugars, syrups, malt and salts. (Whalen et al. 1997).

Ryu et al. (1993) determined the effects of 6 conventional baking ingredients (sucrose, nonfat dry milk, dry egg, shortening, glyceryl monostearate and sodium bicarbonate) on pasting properties of extruded wheat flour using the Rapid Visco Analyser.

They found that sucrose lowered water activity in a starch-water system and interacted with the starch chain, delaying starch gelatinization in starch-sucrose systems. Shortening powder (lipids) acting as a lubricant, can modify some starches during extrusion-cooking. Lipids reduce the mechanical degradation of starch, leading to a higher peak viscosity.

2.4.4 Changes in functional properties
Biopolymer transformation introduced by thermal and mechanical treatment markedly affect water absorption index (WAI) and water solubility index (WSI), consequently, such functional properties in the product are indicators of the phenomena that occur during cooking extrusion. The WAI and WSI are complementary measures of damage to the granule structure and starch solubilization which are obtained from the same test.

WAI is the weight of gel obtained per gram of insolubles. WSI expresses the percentage of dry matter recovered by evaporating the supernatant from the WAI determination and it is related to the quantity of soluble molecules (Anderson et al. 1969 a,b).

When plotted as a function of extrusion temperature, WAI of corn and sorghum grits reaches a maximum at 180 °C- 200 °C (Anderson et al., 1969b). WSI, however, increases progressively with increasing severity of thermal treatment.

A rapid increase in WSI coincides with the decrease in WAI that occurs after peak value.

The fact that certain product characteristics reach maximum with increasing severity of processing and others do not, makes modelling of the overall process difficult. (Linko et al. 1976).

Mercier and Feillet (1975) and Anderson et al. (1969 a) indicated a decrease in WSI with increase in moisture content of corn grits before extrusion. On the other end WAI and final cooked paste viscosity (50 °C) increase with increasing moisture content. This phenomenon is believed to be caused by greater shear degradation of starch during extrusion at low moisture levels.

According to Anderson et al. (1969b) particle size and feed rate are also important. Grits coarser than about 14 mesh received inadequate treatment during extrusion and at very
high feed rate some material was underprocessed. Using grits of 16-20 mesh and operating the extruder with a retention time of about 30 sec. reduced the variations due to these factors.

Charboniere et al. (1973) studied the changes in functional properties in various starches during extrusion. Their results indicated that WSI decreased with increasing amylose content. WAI increased with amylose content.

This review indicates that there has been considerable work on the effects of extrusion conditions on functional properties but a clear explanation of the changes of WAI and WSI along an extruder channel has not been reported before Cai and Diosady (1993).

The results showed that the changes in WSI during extrusion cooking occurred only in the cooking zone. WSI changed rapidly at the beginning of this zone and then more gradually till the die. This trend was the result of the dependence of WSI on starch gelatinization in the extruder. A correlation with degree of gelatinization was found. WSI is an exponential function of the degree of gelatinization: $\text{WSI} = 4.876 \, e^{2.05 f}$ where $f$ is degree of gelatinization.
3. EXPERIMENTAL PROCEDURES

3.1 Materials

Experiments were conducted using as feed material yellow corn meal (U.S.35), yellow corn flour (U.S.100) and yellow corn grits (U.S. 3.5) packed by J.R. Short Milling Company (Kankakee, Illinois, USA).

HexaKleen FD&C Red 40 supplied by Pointing Canada Ltd.(Kingston, Ontario, Canada) was used as tracer. FD & C Red 40 principally the disodium salt of 6-hydroxy-5-[(2-methoxy-5-methyl-4-sulfophenyl)azo]-2-naphthalenesulfonic acid (fig 3-1), is a water soluble sulfonated dye.(Marmion, 1984)

Manganese dioxide, 80% pure, was obtained from Quadra chemicals Ltd.(Burlington, Ontario, Canada).

The other chemicals and reagents were analytical grade.

3.2 Equipment specification

Experiments were carried out in a counter-rotating twin shaft preconditioner (Extru-tech Inc.,Kansas,USA) and in a BC-92 (the number reflects the interaxis distance in mm) co-
rotating twin-screw extruder, with 2 m barrel length and 114 mm inside diameter (Clextral Inc., Tampa, Florida, USA).

The barrel had five temperature controlled sections, which were heated by magnetic induction and cooled by a refrigeration system using glycol as the heat transfer medium. Two dies, each of them having eight holes, with a diameter of 5.5 mm and a length of 150 mm, were fitted in the die plate for every experiment.

The preconditioner paddles configuration is shown in fig. 3-1. While the extruder screw configuration used throughout the program is described in table 3-1.

### 3.3 Operating conditions

The traditional corn flakes were made by the Weetabix's plant in Clinton (Massachusetts, USA). Detailed operating conditions are reported in Appendix 2.

The preconditioner and extruder operating conditions are shown in table 3-2. A complete description of the extrusion process and equipment characteristics are discussed in Appendix 2.
Fig.3-1 Chemical Structure of FD&C Red No.40
Fig. 3-2. Paddle configuration used in the Residence Time Distribution experiments.
Table 3-1. Screw configuration

<table>
<thead>
<tr>
<th>Screw configuration</th>
<th>% of shaft length</th>
</tr>
</thead>
<tbody>
<tr>
<td>Infeed section</td>
<td>15%</td>
</tr>
<tr>
<td>Mixing section</td>
<td>15%</td>
</tr>
<tr>
<td>Forward conveying</td>
<td>15%</td>
</tr>
<tr>
<td>Mixing</td>
<td>15%</td>
</tr>
<tr>
<td>Forward conveying</td>
<td>15%</td>
</tr>
<tr>
<td>Mixing</td>
<td>15%</td>
</tr>
<tr>
<td>Forward conveying</td>
<td>8%</td>
</tr>
<tr>
<td>Reverse pitch</td>
<td>2%</td>
</tr>
</tbody>
</table>

Table 3-2. Operating Conditions

<table>
<thead>
<tr>
<th></th>
<th>0% Steam</th>
<th>1% Steam</th>
<th>3% Steam</th>
<th>5% Steam</th>
<th>8% Steam</th>
</tr>
</thead>
<tbody>
<tr>
<td>Screw Speed (rpm)</td>
<td>100</td>
<td>100</td>
<td>100</td>
<td>100</td>
<td>100</td>
</tr>
<tr>
<td>Extruder Amps</td>
<td>120</td>
<td>127</td>
<td>121</td>
<td>122</td>
<td>129</td>
</tr>
<tr>
<td>Precond. Speed (Hz)</td>
<td>60</td>
<td>60</td>
<td>60</td>
<td>60</td>
<td>60</td>
</tr>
<tr>
<td>Feed rate (kg/h)</td>
<td>450</td>
<td>450</td>
<td>450</td>
<td>450</td>
<td>450</td>
</tr>
<tr>
<td>Steam ratio*</td>
<td>0%</td>
<td>1%</td>
<td>3%</td>
<td>5%</td>
<td>6%</td>
</tr>
<tr>
<td>Liquor pump (water)*</td>
<td>18%</td>
<td>16%</td>
<td>16%</td>
<td>16%</td>
<td>16%</td>
</tr>
<tr>
<td>Water pump (kg/h)</td>
<td>42</td>
<td>35</td>
<td>22</td>
<td>16</td>
<td>22</td>
</tr>
</tbody>
</table>

*Percent of feed rate
3.4 Analytical techniques

3.4.1 Determination of RTD

The residence time distribution (RTD) is a measure of the length of time process material spends in the system. The experimental procedure for the determination of the residence time distribution functions involves an impulse stimulus tracer in the feed stream. This is also known as a Dirac pulse or delta function in the input.

After the preconditioner and/or the extruder was operated at steady state with regard to the flow rates, pressure and temperature a known quantity of tracer was added instantaneously into the feed stream and the time of injection was recorded. The output was collected every 20 seconds over a period of 4-8 minutes.

In some experiments, for the preconditioner, at the end of the run the barrel was quickly opened and some samples were collected inside and analysed for tracer content.

Some runs with “dead stop” operation were also conducted. During these runs the preconditioner was stopped every minute and after opening, samples were collected in different positions along the barrel.
3.4.1.1 Neutron Activation Analysis

Manganese dioxide was used as tracer for the evaluation of the RTD as described by Wolf, D. & White, D. (1976).

The reasons for choosing this particular tracer is that it is inert in this system and it is readily detected by neutron activation analysis with little or no interference from food components and its relatively short half-life, which avoids longtime radiation contamination problems. The half-life for the γ emitting Mn$^{56}$ isotope is 2.576 hr. The Mn$^{56}$ is obtained by radiation of the stable isotope Mn$^{55}$ in a nuclear reactor (Knoll, 1989). The reaction is:

\[ \text{Mn}^{55} \xrightarrow{(n,\gamma)} \text{Mn}^{56} \]

Samples of product (< 1 g) were placed in polyethylene vials and irradiated at the SLOWPOKE reactor at the University of Toronto according to the method of Davidson et al. (1983).

An irradiation time of 180 sec was used at a neutron flux of $5 \times 10^{11}$ neutrons cm$^{-2}$ sec$^{-1}$. The amount of Mn$^{56}$ isotope produced was determined by counting the γ ray emittance of each sample for 200 seconds with a Canberra 8180 Multichannel Analyzer equipped with a Ge detector after a delay time (time between end of irradiation and starting of counting) of 20 minutes. Emission energies at 847 and 1811 keV were measured.

3.4.1.2 Colorimetric method
The food color FD&C No.40 was used as tracer for the evaluation of RTD. For each run 200 g of feed material were coated with the red dye. An amount of 10 g of color was first dissolved in 50 ml of water (solubility at 25 °C is 22g/100mL) and then mixed with 200 g of feed material. This mixture was oven dried at 110 °C for 2 hours so at the end the moisture was (12% - 16 %). This tracer was injected and the samples were collected as previously described.

For the determination of the red dye concentration 1g of sample was dispersed in 40 mL of 1% ammonia solution, shook for 1 hour and then centrifuged at 7000 r.p.m. for 15 min. The supernatant was filtered through a membrane filter with 0.45 microns pore size to separate all the fine particles suspended. The absorbance of the clear solution was read at 510 in a Beckman DU-7 spectrophotometer against a reagent blank. For each run a sample with no color was used as reagent blank.

3.4.2 Paste viscosity

The viscosity curves of the sample pastes were measured over a heating and cooling cycle in an HAAKE rotovisco RV12 viscometer, using the M-150 measuring -drive -unit. The MVI sensor system was used but the rotor was replaced with a special one: 4-bladed anchor -type. The gap between the anchor blade and the cup was 1.28 mm, while the diameter of the cup was 42.00 mm.

Ground samples were sieved and the fraction falling between 50 and 65 mesh screen was used to prepare a 9% slurry in distilled water (Paton and Spratt 1978). The slurry
was mixed with a spoon for 3 minutes in order to eliminate lumps and then pushed through a sieve with openings equal to 65 mesh before the measurements.

An amount of 90 mL was transferred into the cup with the water bath initially set at 60 °C and shearing was initiated at 128 r.p.m.; a simultaneous recording was made of the slurry temperature and of the torque applied to the paddles. The temperature of the sample was measured with a thermocouple that was located in the bottom of the sample cup.

After a waiting period of 5 minutes to equalize the temperature, the water bath setting was changed to 95 °C. When the water temperature reached 95 °C, a 10 minute period was timed after which the setting was lowered to 25 °C and the temperature of the bath decreased as the cooling water flow was maximized.

The torque and temperature curve was recorded until the sample returned to 25 °C.

The calibration was carried out using Fluid 50 (Brookfield) equal to 50.1 centipoises at 25 °C as standard.

### 3.4.3 Degree of gelatinization

The degree of gelatinization is defined as the ratio of gelatinized starch to total starch in a product and was calculated from spectrophotometric measurements of the amylose - iodine complex formed in an aqueous suspension of sample after solubilisation of starch by alkali.
Wooton et al. (1971) developed this technique to determine the degree of gelatinization in processed foods. Birch et al. (1973) modified it for rice and Owusu-Ansah et al. (1982) for corn starch after extrusion processing.

For determination of the amylose/iodine blue value, 0.2 g of sample was dispersed in 100 mL 0.2 M KOH solution and then gently agitated for 15 min. The slurry was centrifuged and 1 mL aliquots of the supernatant were mixed with 0.4 mL 0.5 M HCl and made up to 10 mL with water. 0.1 mL of iodine reagent (1 g iodine and 4 g potassium iodide per 100 mL) were then added and after mixing the absorbance was read at 600 nm in a Beckman DU-7 spectrophotometer against a reagent blank. The measurement was repeated using 100 mL 0.5 M KOH and 1 mL 0.5 M HCl for neutralization. The ratio of the two absorbances $\alpha_1$ and $\alpha_2$ obtained from each sample enabled the degree of gelatinization to be calculated.

This method was calibrated. Raw corn flour was considered to be ungelatinized. Completely gelatinized corn starch was prepared according to the method described by Chiang and Johnson (1977) and W. Cai (thesis 1992). A 2% suspension of corn meal was autoclaved at 120 °C and 15 psi for 1 hour, dried in a forced air oven at 58 °C and then ground to pass a 65 mesh screen. This material was assumed to have a degree of gelatinization of 100%. Standard samples of 0%, 25%, 50%, 75% and 100% gelatinized corn starch were prepared by mixing the completely gelatinized starch and the raw starch. The calibration equation, obtained by linear regression, with a correlation coefficient $R^2 = 0.99$, was

$$f \times 100\% = 3.455 \frac{\alpha_1}{\alpha_2} - 0.782$$
3.4.4 Water solubility index and water absorption index

Water solubility index (WSI) and water absorption index (WAI) were measured with the method suggested by Anderson et al. (1969). A 2.5 g sample was suspended in 30 mL of water at 30 °C in a 50 mL tared test tube stirred intermittently over a 30 min period, and centrifuged at 2000 g for 15 min. The supernatant liquid was poured carefully into a 50 ml tared beaker and dried in a forced air oven. The remaining gel was weighed. WAI and WSI were calculated as follows:

\[
WSI \, (\%) = \frac{\text{Total solubles}}{\text{Weight of dry sample}} \times 100
\]

\[
WAI = \frac{\text{Weight of swollen gel}}{\text{Weight of dry sample} - \text{Total solubles}}
\]
4. RESULTS AND DISCUSSION

4.1 Flow characteristics

4.1.1 RTD in the preconditioner

To evaluate the effects of several controlling variables on the RTD in the preconditioner, a series of experiments were carried out under selected operating conditions.

The mathematical analysis of the data from the manganese dioxide tracer and the dye colour experiments is discussed in detail in Appendix 1.

The operating conditions along with the mean residence time $t$, the variance $\sigma$ and Pe number are presented in tables 4-1 and 4-2. The Peclet number (Pe) was determined by numerically solving eqn (2.5).

The mean residence time decreases with increasing shaft speed and feed rate. Shaft speed also affects the shape of the distribution as indicated by its variance while the effect of feed rate is negligible.

The values of Pe number $\sim 2.5$ indicate an excessive axial mixing (Todd and Irving, 1969). The slight decrease of Pe number as the feed rate was increased may be due to an increase in degree of fill of the barrel. The relatively constant value obtained for the Peclet number may be explained by the fact that the axial mixing is considered to be more of a screw configuration characteristic than an operating variable (Todd, 1975).

The RTD dependence on shaft speed is shown in figure 4-1. One set of data at 200 kg/h feed rate is reported to illustrate the effect of increasing the speed. It is easily seen that raising the shaft speed shifts the RTD to the left and shortens the mean residence time.
These data also seem to indicate that the shaft speed changes the shape of the distribution as indicated by the variance and by its tendency to narrow the RTD.

The throughput of the preconditioner can be varied over a wide range and is independent of paddle speed. The influence of feed rate on RTD is shown in figure 4-2. The higher the rate, the more narrow the distribution. It seems that at the lower rates when the preconditioner is less filled, there is less tendency for the preconditioner to provide good mixing.

The results also indicate, as shown in the following figures (4-1, 4-2), that a small amount of material is retained in the system for long time.

In figures 4-3 and 4-4 the results for two different paddles configurations are reported. The second configuration is obtained adjusting the paddles in position 5 and 6 from neutral to reverse on all rows.

The data in tables 4-3, 4-4 and the distributions in figures 4-3 and 4-4 indicate that the flow and mixing patterns are similar for both configuration. The one change in paddle configuration investigated had very little effect on RTD.

There was a significant tail with all these series of residence time distribution, showing that some material was held for long time in the so called "dead space".

The material balance on the tracer was checked and for all the runs the recovery was no more than 75%.
Table 4-1. Operating conditions for preparing the samples.

<table>
<thead>
<tr>
<th>Experiment</th>
<th>Feed rate (kg/h)</th>
<th>Shaft speed (Hz)</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>200</td>
<td>20</td>
</tr>
<tr>
<td>2</td>
<td>200</td>
<td>40</td>
</tr>
<tr>
<td>3</td>
<td>200</td>
<td>60</td>
</tr>
<tr>
<td>4</td>
<td>300</td>
<td>20</td>
</tr>
<tr>
<td>5</td>
<td>300</td>
<td>40</td>
</tr>
<tr>
<td>6</td>
<td>300</td>
<td>60</td>
</tr>
<tr>
<td>7</td>
<td>400</td>
<td>40</td>
</tr>
<tr>
<td>8</td>
<td>400</td>
<td>60</td>
</tr>
<tr>
<td>9</td>
<td>500</td>
<td>40</td>
</tr>
<tr>
<td>10</td>
<td>500</td>
<td>60</td>
</tr>
</tbody>
</table>

Table 4-2. Computed data for mean residence time, variance and Pe number.

<table>
<thead>
<tr>
<th>Experiment</th>
<th>$\bar{t}$ (s)</th>
<th>$\sigma^2$ (s$^2$)</th>
<th>Pe</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>365</td>
<td>31765</td>
<td>7.22</td>
</tr>
<tr>
<td>2</td>
<td>228</td>
<td>19858</td>
<td>3.92</td>
</tr>
<tr>
<td>3</td>
<td>120</td>
<td>5470</td>
<td>3.96</td>
</tr>
<tr>
<td>4</td>
<td>298</td>
<td>36759</td>
<td>3.48</td>
</tr>
<tr>
<td>5</td>
<td>201</td>
<td>20632</td>
<td>2.46</td>
</tr>
<tr>
<td>6</td>
<td>109</td>
<td>5481</td>
<td>2.93</td>
</tr>
<tr>
<td>7</td>
<td>189</td>
<td>20556</td>
<td>1.94</td>
</tr>
<tr>
<td>8</td>
<td>100</td>
<td>5019</td>
<td>2.53</td>
</tr>
<tr>
<td>9</td>
<td>165</td>
<td>19610</td>
<td>1.07</td>
</tr>
<tr>
<td>10</td>
<td>94</td>
<td>4982</td>
<td>2.02</td>
</tr>
</tbody>
</table>
Figure 4-1. The effect of shaft speed on RTD at 200 kg/h
Figure 4-2. The effect of feed rate on RTD at 40 Hz
Table 4-3. Operating conditions for preparing the samples (changed configuration).

<table>
<thead>
<tr>
<th>Experiment</th>
<th>Feed rate (kg/h)</th>
<th>Shaft speed (Hz)</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>200</td>
<td>20</td>
</tr>
<tr>
<td>2</td>
<td>200</td>
<td>40</td>
</tr>
<tr>
<td>3</td>
<td>200</td>
<td>60</td>
</tr>
<tr>
<td>4</td>
<td>300</td>
<td>20</td>
</tr>
<tr>
<td>5</td>
<td>300</td>
<td>40</td>
</tr>
<tr>
<td>6</td>
<td>300</td>
<td>60</td>
</tr>
<tr>
<td>7</td>
<td>400</td>
<td>40</td>
</tr>
<tr>
<td>8</td>
<td>400</td>
<td>60</td>
</tr>
<tr>
<td>9</td>
<td>500</td>
<td>60</td>
</tr>
</tbody>
</table>

Table 4-4. Computed data for mean residence time, variance and Pe number (changed configuration).

<table>
<thead>
<tr>
<th>Experiment</th>
<th>$i$ (s)</th>
<th>$\sigma^2$ (s$^2$)</th>
<th>Pe</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>383</td>
<td>43797</td>
<td>5.48</td>
</tr>
<tr>
<td>2</td>
<td>237</td>
<td>21179</td>
<td>4.00</td>
</tr>
<tr>
<td>3</td>
<td>130</td>
<td>5805</td>
<td>4.65</td>
</tr>
<tr>
<td>4</td>
<td>324</td>
<td>37570</td>
<td>4.30</td>
</tr>
<tr>
<td>5</td>
<td>206</td>
<td>20560</td>
<td>2.70</td>
</tr>
<tr>
<td>6</td>
<td>120</td>
<td>4994</td>
<td>2.49</td>
</tr>
<tr>
<td>7</td>
<td>192</td>
<td>19393</td>
<td>2.32</td>
</tr>
<tr>
<td>8</td>
<td>98</td>
<td>4920</td>
<td>2.44</td>
</tr>
<tr>
<td>9</td>
<td>96</td>
<td>4798</td>
<td>2.37</td>
</tr>
</tbody>
</table>
Figure 4-3. The effect of paddle configuration on RTD at 400 kg/h and 40 Hz
Figure 4-4. The effect of paddle configuration on RTD at 500 kg/h and 60 Hz
These results suggested that the particles tend to segregate inside the preconditioner. Although differences in the size, density, shape and resilience of the constituent particles of a mixture can give rise to segregation, difference in particle size is by far the most important of these (Williams, 1990).

The effect of difference in particle size was tested by varying the feed material. All of the experimental runs were carried out using the same operating conditions (450 kg/h feed rate and 60 Hz paddle speed) and the same amount of tracer (40 g of MnO₂). Sampling along the length of the preconditioner chamber at the end of each run enabled us to calculate a total mass balance on the tracer.

Table 4-5 and Fig. 4-5 show the changes of the distribution for the three different feed materials. It seems that mean residence time is slightly affected by the particle size. Increasing the particle size of the feed material resulted in a higher variance or greater spread of the distribution and poorer mixing.

The low recovery indicates that a large quantity of tracer is still in the preconditioner after the sampling period, as shown in table 4-6 and may leave later under different regimens. Especially in the case of corn meal and corn grits, which are different in particle size from the manganese dioxide, a large amount of tracer is caught in the "dead" space, creating a long tail. The tail portion of the RTD is an important factor for product quality. A protracted residence time allows a portion of the product to be overprocessed.
Table 4-5. Computed data for mean residence time, variance, Pe number, and manganese dioxide recovery after the sampling period, for different feed materials.

<table>
<thead>
<tr>
<th>Feed material</th>
<th>$\bar{t}$ (s)</th>
<th>$\sigma^2$ (s$^2$)</th>
<th>Pe</th>
<th>MnO$_2$ Recovery</th>
</tr>
</thead>
<tbody>
<tr>
<td>Corn Flour</td>
<td>121</td>
<td>5941</td>
<td>3.59</td>
<td>94.7%</td>
</tr>
<tr>
<td>Corn Meal</td>
<td>128</td>
<td>8339</td>
<td>2.47</td>
<td>55.5%</td>
</tr>
<tr>
<td>Corn Grits</td>
<td>136</td>
<td>11224</td>
<td>1.27</td>
<td>35.8%</td>
</tr>
</tbody>
</table>
Figure 4-5. C(t) distribution for feed materials of different particle sizes
Table 4-6. Tracer concentration (p.p.m.) along the preconditioner channel after the sampling period (8 minutes) for two different feed materials.

<table>
<thead>
<tr>
<th>Position</th>
<th>Corn Meal (p.p.m.)</th>
<th>Corn Flour (p.p.m.)</th>
</tr>
</thead>
<tbody>
<tr>
<td>FL</td>
<td>1214</td>
<td>17.3</td>
</tr>
<tr>
<td>FR</td>
<td>1.94</td>
<td>21.4</td>
</tr>
<tr>
<td>ML</td>
<td>14.6</td>
<td>10.9</td>
</tr>
<tr>
<td>MR</td>
<td>13.5</td>
<td>12.9</td>
</tr>
<tr>
<td>EL</td>
<td>22.8</td>
<td>10.5</td>
</tr>
<tr>
<td>ER</td>
<td>27.3</td>
<td>18.7</td>
</tr>
<tr>
<td>DIS</td>
<td>28.5</td>
<td>17.9</td>
</tr>
</tbody>
</table>

FL = front left side  
FR = front right side  
ML = middle left side  
MR = middle right side  
EL = end left side  
ER = end right side  
Front = entrance to preconditioner  
End = discharge
Dead Stop

In order to have a better understanding of the dead space, the volume inside the preconditioner where the material is held for longer time, some "dead stop" runs were carried out with all the three different feed materials.

During these runs the preconditioner was stopped every minute and after opening, samples were collected in different positions along the barrel. In table 4-7 ((a),(b),(c)) the measured concentrations (p.p.m.) are shown.

The results show that in the right side the tracer concentration is lower than in the left side, indicating an uneven mixing.

In the case of corn grits the tracer seems trapped in the front zone, showing the poor forwarding capability of the preconditioner probably due to the large particle size.

From these results it is clear that the preconditioner does not provide good mixing, since at the end of the five minutes sampling period a considerable amount of tracer is still inside and its distribution is not uniform along the channel.

The preconditioner is not suitable for product formulations that require feed materials with different granulation unless an appropriate paddle configuration able to improve residence time and mixing is found.
Table 4-7. Dead Stop: Tracer Concentration (p.p.m.) along the preconditioner channel

(a): Corn Flour

<table>
<thead>
<tr>
<th>Time (m)</th>
<th>FL</th>
<th>FR</th>
<th>ML</th>
<th>MR</th>
<th>EL</th>
<th>ER</th>
<th>DISC</th>
</tr>
</thead>
<tbody>
<tr>
<td>0</td>
<td>1.5</td>
<td>1.44</td>
<td>2.22</td>
<td>1.78</td>
<td>1.84</td>
<td>4.48</td>
<td>4.08</td>
</tr>
<tr>
<td>1</td>
<td>756</td>
<td>242</td>
<td>931</td>
<td>661</td>
<td>519</td>
<td>989</td>
<td>923</td>
</tr>
<tr>
<td>2</td>
<td>199</td>
<td>149</td>
<td>418</td>
<td>586</td>
<td>800</td>
<td>546</td>
<td>624</td>
</tr>
<tr>
<td>3</td>
<td>165</td>
<td>112</td>
<td>203</td>
<td>240</td>
<td>537</td>
<td>366</td>
<td>288</td>
</tr>
<tr>
<td>4</td>
<td>55.7</td>
<td>35.4</td>
<td>126</td>
<td>239</td>
<td>223</td>
<td>288</td>
<td>101</td>
</tr>
<tr>
<td>5</td>
<td>25</td>
<td>17</td>
<td>76.3</td>
<td>211</td>
<td>128</td>
<td>193</td>
<td>74.5</td>
</tr>
</tbody>
</table>

(b): Corn Meal

<table>
<thead>
<tr>
<th>Time (m)</th>
<th>FL</th>
<th>FR</th>
<th>ML</th>
<th>MR</th>
<th>EL</th>
<th>ER</th>
<th>DISC</th>
</tr>
</thead>
<tbody>
<tr>
<td>0</td>
<td>2.13</td>
<td>4.56</td>
<td>4.4</td>
<td>5.09</td>
<td>4.88</td>
<td>5.38</td>
<td>6.16</td>
</tr>
<tr>
<td>1</td>
<td>731</td>
<td>165.4</td>
<td>556</td>
<td>656</td>
<td>619</td>
<td>706</td>
<td>626</td>
</tr>
<tr>
<td>2</td>
<td>190.3</td>
<td>88.3</td>
<td>142</td>
<td>188</td>
<td>206</td>
<td>211</td>
<td>197</td>
</tr>
<tr>
<td>3</td>
<td>78.9</td>
<td>9.29</td>
<td>44.5</td>
<td>78.9</td>
<td>107</td>
<td>86</td>
<td>70.5</td>
</tr>
<tr>
<td>4</td>
<td>69.1</td>
<td>5.81</td>
<td>22.5</td>
<td>48.6</td>
<td>68.6</td>
<td>55.1</td>
<td>32.5</td>
</tr>
<tr>
<td>5</td>
<td>50.7</td>
<td>4.13</td>
<td>12.9</td>
<td>17.1</td>
<td>44</td>
<td>23.8</td>
<td>26.6</td>
</tr>
</tbody>
</table>

(b): Corn grits

<table>
<thead>
<tr>
<th>Time (m)</th>
<th>FL</th>
<th>FR</th>
<th>ML</th>
<th>MR</th>
<th>EL</th>
<th>ER</th>
<th>DISC</th>
</tr>
</thead>
<tbody>
<tr>
<td>0</td>
<td>1.9</td>
<td>2.5</td>
<td>2</td>
<td>3.08</td>
<td>4.46</td>
<td>4.11</td>
<td>2.53</td>
</tr>
<tr>
<td>1</td>
<td>855</td>
<td>742</td>
<td>193</td>
<td>300</td>
<td>596</td>
<td>457</td>
<td>165</td>
</tr>
<tr>
<td>2</td>
<td>548</td>
<td>662</td>
<td>88.3</td>
<td>324</td>
<td>143</td>
<td>379</td>
<td>258</td>
</tr>
<tr>
<td>3</td>
<td>129</td>
<td>485</td>
<td>62.5</td>
<td>47.1</td>
<td>150</td>
<td>129</td>
<td>102</td>
</tr>
<tr>
<td>4</td>
<td>108</td>
<td>103</td>
<td>23</td>
<td>33.3</td>
<td>62.9</td>
<td>90.1</td>
<td>85</td>
</tr>
<tr>
<td>5</td>
<td>75</td>
<td>58</td>
<td>11</td>
<td>16.5</td>
<td>53.6</td>
<td>35.1</td>
<td>64</td>
</tr>
</tbody>
</table>

FL= front left side
FR= front right side
ML = middle left side
MR = middle right side
EL = end left side
ER = end right side
DISC = discharge
Front = entrance to preconditioner
The difficulty of mixing can be reduced by making the sizes of the components as close as possible or by adding a small amount of liquid to the mixture in order to prevent segregation.

Since in preconditioning steam and water are used to preheat and prehumidify the feed materials their effect on the residence time distribution and on the quality of mixing was investigated.

The steam and water injection seems to slightly decrease the mean residence time and affect the shape of the distribution, as shown in figure 4-6. The peak occurs at the same time but in the case with steam injection the peak is sharper.

The material balance on the tracer showed that 80% of the amount initially injected is recovered. Steam and water have a positive effect on the quality of mixing, reducing the quantity of tracer caught inside the preconditioner but 20% of the tracer is still inside, producing a long tail.

A new analytical method was developed in order to analyze the RTD and to obviate the problem of particle size. This problem was overcome by using a tracer with the same granulation as the feed material. The feed material was coated with the food color FD&C No.40. An amount of 200g of feed material coated with 10 g of red dye gave the best peak and tail resolution.

Table 4-8 and Fig. 4-7 show the changes of the distribution for the three feed materials.
Figure 4-6. The effect of water (90 kg/h) and steam (22.5 kg/h) injection on RTD at 450 kg/h and 60Hz.

\[ \bar{i} = 107; \sigma = 7257.01; \text{Pe} = 1.5 \]
Table 4-8. Computed data for mean residence time, variance, Pe number, red dye recovery after the sampling period, for different feed materials.

<table>
<thead>
<tr>
<th>Feed material</th>
<th>$\bar{t}$ (s)</th>
<th>$\sigma^2$ ($s^2$)</th>
<th>Pe</th>
<th>Tracer Recovery</th>
</tr>
</thead>
<tbody>
<tr>
<td>Corn Flour</td>
<td>89</td>
<td>2219</td>
<td>5.94</td>
<td>93.9%</td>
</tr>
<tr>
<td>Corn Meal</td>
<td>71</td>
<td>1466</td>
<td>5.66</td>
<td>91%</td>
</tr>
<tr>
<td>Corn Grits</td>
<td>36</td>
<td>611</td>
<td>7.20</td>
<td>92%</td>
</tr>
</tbody>
</table>
Figure 4-7. Residence time distribution for feed material of different particle sizes
In Fig. 4-7, it seems that the mean residence time and variance decreased with increasing particle size. The Pe number also indicates that the axial mixing becomes less important, leading to a better product homogeneity.

The material balance on the tracer gives a good recovery value, indicating that after a sampling period approximately of 3 or 4 minutes less than 10% of the tracer is still inside the preconditioner.

These results show that mixture of particles of different sizes causes segregation inside the preconditioner and consequently poor quality of mixing. This was confirmed by Williams (1990) who suggested that in cases where segregation is known to be a problem, the mixer chosen should be one which relies mainly on a convective action, the most common type being the ribbon blender.

Since this general conclusion is not supported by any studies reported in the literature, the mechanical design of the preconditioner's paddles and its effect on the quality of mixing should be investigated further.

**Theoretical models**

Depending on whether flow is close to plug or mixed flow, different kinds of models are available.

Bouvier (1996) used the tanks-in-series model to represent the experimental RTD curves. He found that the preconditioner presents an intermediate/low degree of mixing and moves from a plug flow behavior at low shaft speed to a mixed flow behavior at high shaft speed.

In the present study the experimental RTD curves were initially compared for their fit to the dispersion and tanks in series model (equations 2-12 and 2-13).
No satisfactory fit was obtained because the residence time distributions for preconditioners have a long tail, due to material held in dead space that can not be described by these simple models.

The deviations observed in the data from the theoretical dispersion and tanks in series models suggested the use of a mixed model which divides the real system into different flow behavior sections. There are two approaches to the representation of dead space. The first is to define the region as completely stagnant so the material trapped there does not exit from the system. In this model the dead volume contributes to the total volume but not to the exit age distribution.

The alternative definition of dead space allows a slow interchange or crossflow between this region and the active fluid. Thus elements from stagnant space do eventually appear in the exit stream. Both types of models were considered in this research.

The multistage Wolf-Resnick model which did not allow material caught in the dead space to pass out of the preconditioner was fitted to three of the experimental residence time distributions obtained using the dye tracer. The mean residence time, calculated as the first moment of the experimental distribution was used as the estimate of the model parameter, \( \bar{t} \), in each case.

The coefficients \( b \) and \( \varepsilon \) (equation 2-14) were estimated by the direct search optimization technique (Luus and Jaakola, 1973) employing random numbers and search region contraction. Description of the technique and mathematical analysis of the data are presented in Appendix 1.

In table 4-9 the values estimated for \( b \) and \( \varepsilon \) are presented along with the number of stages, \( n \), and the performance index given by the square of the deviation between \( F \)
(cumulative residence time distribution) expected from the model and measured F values.

The deviation of $b$ from unity indicates that a state of perfect mixing has not been achieved in the preconditioner due to plug flow and/or dead space. The term $\varepsilon$ is a measure of the phase shift in the system and the fact that $\varepsilon/\ddot{\theta}$ is $>0$ indicates that the system lags behind that expected for perfect mixing due to plug flow.

For the corn grits $\varepsilon/\ddot{\theta}$ is $<0$, so the system allowed partial short circuiting. It is assumed that part of the entering feed passes to the outlet with an infinite velocity without mixing.

The multistage model gave a good representation of the tail of the distribution.

In figure 4-8 the cumulative exit age curve, the F curve, fitted from the model is compared with the experimental data obtained for corn meal.

The crossflow model was fitted to the same three experimental distributions analysed for the multistage model and the estimates of the parameters are summarized in table 4-10.

Levich et al.1967 derived the exit age distribution for these flow patterns which consist of $n$ stages of active and dead space regions. He showed that, for large $n$, the distribution can be approximated as a Gaussian curve and an exponential decay curve.

The general form of the exit age curve was as follows:

\begin{equation}
E(t) = (1 - f_D) \frac{1}{\sqrt{2\pi \sigma^2}} \exp\left[ -\frac{(t-t_a)^2}{2\sigma^2} \right] + \frac{f_D}{4\tau_a} \left[ 1 - \text{erf}\left( \frac{t_a - t}{\sqrt{2\tau_a}} \right) \right]
\end{equation}  (4-1)
Table 4-9 Parameter Estimates for Wolf-Resnick Multistage Model

<table>
<thead>
<tr>
<th>Feed material</th>
<th>n</th>
<th>b</th>
<th>$\frac{e}{f}$</th>
<th>Performance Index</th>
</tr>
</thead>
<tbody>
<tr>
<td>Corn Flour</td>
<td>3</td>
<td>3.5</td>
<td>0.016</td>
<td>$0.202 \times 10^{-2}$</td>
</tr>
<tr>
<td>Corn Meal</td>
<td>3</td>
<td>2.88</td>
<td>0.018</td>
<td>$0.125 \times 10^{-2}$</td>
</tr>
<tr>
<td>Corn Grits</td>
<td>3</td>
<td>2.87</td>
<td>-0.054</td>
<td>$0.184 \times 10^{-1}$</td>
</tr>
</tbody>
</table>

Table 4-10 Parameter Estimates for Levich Model

<table>
<thead>
<tr>
<th>Feed material</th>
<th>$t_a$ (s)</th>
<th>$t_d$ (s)</th>
<th>$f_d$</th>
<th>$\sigma^2$ (s$^2$)</th>
<th>Performance Index</th>
</tr>
</thead>
<tbody>
<tr>
<td>Corn Flour</td>
<td>60</td>
<td>258</td>
<td>0.36</td>
<td>1.29</td>
<td>$0.780 \times 10^{-4}$</td>
</tr>
<tr>
<td>Corn Meal</td>
<td>60</td>
<td>226</td>
<td>0.3</td>
<td>1.14</td>
<td>$0.108 \times 10^{-3}$</td>
</tr>
<tr>
<td>Corn Grits</td>
<td>20</td>
<td>198</td>
<td>0.4</td>
<td>3.18</td>
<td>$0.5297 \times 10^{-4}$</td>
</tr>
</tbody>
</table>
Figure 4-8. Comparison of Cumulative Exit Age Distribution, $F(t)$, predicted by the Wolf and Resnick model and experimental observation for corn meal
Figure 4-9. Comparison of Exit Age Distribution, E(t), predicted by the Levich model and experimental observations for corn grits.
for $t>t_a$

$$E(t) = (1 - f_D) \frac{1}{\sqrt{2\pi \sigma^2}} \exp\left[\frac{(t-t_p)^2}{2\sigma^2}\right] + \frac{f_D}{t_d} \exp\left(-\frac{t}{t_d}\right)$$  \hspace{1cm} (4-2)

These equations describe the flow pattern as characterized by two mechanisms. The first term is common to both equation and it describes the behavior of the major fraction, $(1-f_D)$, which pass through as a symmetric plug. The second term in equation 4-1 is small and according to Levich et al. (1967) it can be neglected. A small fraction of the material, $f_D$, is delayed in the dead space and goes through the preconditioner according to the second exponential relationship in equation 4-2.

The mean residence time in the active volume, $t_a$, corresponded to the time at which the peak occurred in the residence time distribution. The value for $t_d$, the average delay time, was obtained by measuring the rate of exponential decay in the tail.

The fraction of material delayed in dead space, $f_D$, and the variance $\sigma^2$, were estimated using again direct search optimization technique.

In figure 4-9 the exit age distribution $E(t)$, fitted from the model is compared with the experimental distribution obtained for corn grits.

The Levich model did not represent the tail region well and tends to predict longer residence time than experimentally measured.

The tail region represents a relatively important portion of the total mass, in fact according to the results in the table 4-10, the fraction of material trapped in the dead space, $f_D$, varies from 0.3 to 0.4.
Based on this analysis, it appeared clear that we can describe the flow pattern as characterized by an active volume, where the material moves by dispersed plug flow and by dead space regions that are responsible of the long tail in the distribution.

4.1.2 RTD in the extruder

Fig. 4-10 shows the residence time distribution for the extruder measured using the operating conditions reported in table 3-2. The distribution obtained with the red dye tracer presented a peak that is sharper and earlier in time than that obtained with manganese dioxide, as shown in table 4-11. It seems that the Manganese dioxide stays longer inside the extruder as indicated by the wider distribution and by the lower Pe value. This behaviour can be explained by the phenomenon of segregation due to the difference in particle size between the tracer and corn meals, even though water and steam injection should help prevent segregation inside the extruder.

The velocity profile in a twin-screw extruder having the screw configuration illustrated in table 3-1 is very complex. Hence, mixed models which divide the barrel into different flow behaviour sections are more appropriate in describing RTD. According to Balke (1985) and Agur (1986) the plug-flow/stirred tank model given by eq.(4-3) describes adequately the flow characteristics inside a twin screw extruder.
The extruder is considered as a pipe with no axial dispersion (plug flow) representing the conveying section followed by a stirred tank with perfect distributive mixing representing the mixing section.

\[ E(t) = A e^{\frac{(t-\delta)}{\tau}} \]  \hspace{1cm} (4-3)

\[ E(t) = 0 \quad t < \delta \]

Where \( A \) is the preexponential factor, \( \delta \) is the time delay (in the pipe), and \( \tau \) is the time constant (in the stirred tank). The value of \( \delta \) is obtained as the time at which the peak occurs and can be viewed as a measure of the conveying performance of the screw sections of the extruder. The value of \( \tau \) represents the time constant of the mixing sections of the extruder and is obtained by fitting equation (4-3) to the experimental distribution using direct search optimization. In figure 4-11 it is shown the comparison between the experimental residence time distribution and the model, while in table 4-12 the parameters fitted from the experimental distribution are presented.

It is clear that there is a significant difference in the extruder conveying performance using the two tracers while the difference in the mixing section, indicated by the parameter \( \tau \), seems unimportant.
Figure 4-10. Comparison of Exit Age Distribution, $E(t)$, measured with FD&C Red 40 and manganese dioxide
**Table 4-1**  | Computed data for mean residence time, variance, Pe number, tracer recovery after the sampling period for the extruder

<table>
<thead>
<tr>
<th>Tracer</th>
<th>$\bar{t}$ (s)</th>
<th>$\sigma^2$ (s²)</th>
<th>Pe</th>
<th>Tracer Recovery</th>
</tr>
</thead>
<tbody>
<tr>
<td>MnO₂</td>
<td>91</td>
<td>3198</td>
<td>3.86</td>
<td>89%</td>
</tr>
<tr>
<td>FD&amp;C No40</td>
<td>71</td>
<td>1012</td>
<td>8.83</td>
<td>90%</td>
</tr>
</tbody>
</table>

**Table 4-12**  | Parameter estimates for plug flow-stirred tank model

<table>
<thead>
<tr>
<th>Tracer</th>
<th>$\delta$</th>
<th>A</th>
<th>$\tau$</th>
<th>Performance Index</th>
</tr>
</thead>
<tbody>
<tr>
<td>MnO₂</td>
<td>80</td>
<td>0.032</td>
<td>32.12</td>
<td>$0.7413 \times 10^{-3}$</td>
</tr>
<tr>
<td>FD&amp;C No40</td>
<td>60</td>
<td>0.023</td>
<td>29.43</td>
<td>$0.6134 \times 10^{-4}$</td>
</tr>
</tbody>
</table>


Figure 4-11. Comparison of Exit Age Distribution, $E(t)$, predicted by plug-flow-stirred tank model and experimental observation with $\text{MnO}_2$. 
To understand the effect of the preconditioner on the flow distribution inside the extruder, the residence time distribution was calculated by measuring the concentration of the tracer after it passed through the preconditioner-extrusion system.

From the results in Fig.4-12 it is evident that the presence of the preconditioner strongly affects the flow characteristics. The shape of the distribution is similar to the one found for the preconditioner alone but the distribution has been shifted to the right.

In order to investigate the quality of mixing the residence time distribution measured with two different tracers were compared as shown in table 4-13.

The Pe number and the variance indicated that with the red dye the distribution is narrower. So it is clear that in the case of the FD&C no 40 we obtained better mixing with a shorter residence time.

These results were also confirmed by fitting the experimental observation with the Wolf and Resnick model.

In table 4-14 and figure 4-13 are shown the parameter estimates and the comparison of cumulative exit age distribution function, $F(t)$.

In the case of Manganese dioxide the axial mixing, which allows a larger variance in the distribution, becomes more important along with a time lag as indicated by the parameter $b$ and $\varepsilon$. 
Figure 4-12. Comparison of Exit Age Distribution, $E(t)$, measured with FD&C Red 40 for preconditioner (PC), for extruder (EX) and for preconditioner-extruder (PC-EXT)

1.03
0.025
0.02
0.015
0.01
0.006
0.005
0.004
0.003
0.002
0.001
0.000
0
0
20
40
60
80
100
120
140
160
180
200
Time (s)
Table 4-13  Computed data for mean residence time, variance, Pe number and tracer recovery after the sampling period for the preconditioner-extruder system

<table>
<thead>
<tr>
<th>Tracer</th>
<th>$\bar{t}$ (s)</th>
<th>$\sigma^2$ (s²)</th>
<th>Pe</th>
<th>Tracer Recovery</th>
</tr>
</thead>
<tbody>
<tr>
<td>MnO₂</td>
<td>177</td>
<td>6346</td>
<td>8.74</td>
<td>90%</td>
</tr>
<tr>
<td>FD&amp;C No40</td>
<td>149</td>
<td>2014</td>
<td>20.8</td>
<td>91%</td>
</tr>
</tbody>
</table>

Table 4-14  Parameter estimates for the Wolf and Resnick model

<table>
<thead>
<tr>
<th>Feed material</th>
<th>n</th>
<th>b</th>
<th>$e_i/\bar{t}$</th>
<th>Performance Index</th>
</tr>
</thead>
<tbody>
<tr>
<td>MnO₂</td>
<td>3</td>
<td>3</td>
<td>0.056</td>
<td>0.067</td>
</tr>
<tr>
<td>FD&amp;C No40</td>
<td>3</td>
<td>5.08</td>
<td>0.042</td>
<td>0.1084 \cdot 10^{-2}</td>
</tr>
</tbody>
</table>
Figure 4-13. Comparison of Cumulative Exit Age Distribution, $F(t)$, predicted by the Wolf and Resnick model and experimental observation with FD&C No 40.
The practical implication of these results is that if the distribution is broad, a portion of the product may receive excessive or insufficient cooking, even though the average heat treatment is correct for the product. This factor has to be taken into account in the design of preconditioning in cooking extrusion process. The flow pattern inside the preconditioner should be characterized by good mixing, with a short residence time.

4.2 Paste viscosity

In order to fully understand the preconditioning-extrusion process in terms of industrial practice, it was important to investigate the relationships between the structural modifications and functional properties. The paste viscosity, the degree of gelatinization, and the water absorption and water solubility indices were selected as representative functional properties. The characteristics of the paste viscosity curve were significantly affected by extrusion processing as indicated by Figure 4-14. Unprocessed corn flour has a negligible paste viscosity until the gelatinization temperature range is reached, while the extruded samples had significant initial viscosities but the maximum temperature viscosities and the final value after cooling were much lower.

The paste viscosity curve for each sample was defined in terms of torque at three points (initial cold, maximum temperature and final torque at the end of cooling cycle). These data were tabulated in Appendix 3.

We first investigated the difference in paste viscosity in the corn flakes produced with the traditional method and with the cooking extrusion process.

From the paste viscosity curve in Figure 4-14 it seems clear that the difference is small,
with the exception of the final viscosity. The extruded product had a lower final viscosity indicating a higher extent of degradation. This is due to the effect of the two degradation mechanisms present in the extruder, thermal and mechanical. While in the case of the pressure cooker only the effect of thermal degradation is available.

Varying the mechanical and thermal degradation in an extruder can be performed by changing the ratio of steam injection.

Some runs were conducted at different steam ratios and some extrudate samples and some corn flakes were analysed for paste viscosity.

The viscosity at the maximum temperature and the final value after cooling to 25 °C seemed to be the most sensitive to the extent of degradation, as shown in figure 4-15.

At higher steam feed rate, when the extent of mechanical degradation was higher, the final viscosity was lower for both the ropes and the corn flakes. This suggested that starch in the extruded sample had undergone thermal and mechanical degradation (Davidson, 1983, Cai et al. 1995, Whalen et al. 1997) to an extent which was not conducive to retrogradation or gelling.

The viscosity at maximum temperature is related to the degree of gelatinization and it was reported to decrease as the degree of cooking (gelatinization) increased (Mason and Hoseney, 1986). This is due to the fact that pregelatinized extruded starch granules lose their ability to swell upon heating in water resulting in low peak viscosity.
Figure 4-14. Paste viscosity curve for unprocessed corn flour, extruded corn flakes and traditional corn flakes.
Figure 4-15. Paste viscosity for corn flakes obtained with different steam ratio.
The changes in the viscosity at maximum temperature were small until a value of steam ratio equal to 8% of the feed rate was reached. This result showed that maximum gelatinization occurred at 8% steam and that with lower steam injection the degree of gelatinization decreased slightly with respect to the case with no steam. With no steam the low moisture content resulted in higher viscosity, increasing high shear stress and thus increasing the degree of gelatinization (Gomez and Aguilera, 1983).

4.3 Degree of gelatinization

The results found for the paste viscosity were confirmed by measuring the degree of gelatinization on the same samples. The results, presented in appendix 4, show the effect of steam injection on the gelatinization of the extrudate and of the final product. Convection of energy by steam injection differs from the other inputs because it contributes not only energy but moisture to the system. Water is a reactant in gelatinization and it also affects the process time - gelatinization proceeds more easily in the presence of excess moisture (Fast, 1990). Since some hygroscopic ingredients, such as salt and sugar, compete with starch for moisture, reducing the effective concentration available for gelatinization, the runs were conducted by feeding the extruder only with corn meal and water.

The degree of gelatinization initially increased with an increase in steam injection and then decreased slightly with further steam injection to 5% for both extrudates and final
products. This may be due to the formation of resistant starch and of starch-protein complexes (Russell et al. 1989).

The degree of gelatinization at low moisture content, in the case without steam injection, was very high, because of the development of high stresses within the product.

The difference in gelatinization between traditional and extruded corn flakes was also investigated. The extruded corn flakes have a higher degree of gelatinization because the high shear developed inside the extruder alter the starch structure through the mechanical breakdown of starch molecules, resulting in the production of dextrins and other short-chain species (Cai et al. 1995).

In addition to modifying the molecular structure of the starch, mechanical and thermal stresses in the extruder produced changes in physical properties which affect functionality, such as water absorption and water solubility.

4.4 Changes in functional properties

The water solubility and water absorption index are used by industry because they can be measured quickly and easily by the same procedure (Anderson et al. 1969,a,b and Mercier and Feillet, 1975).

WSI and WAI analysis are very useful in determining whether starch granules are in a swollen state or have been degraded or depolymerized. Water solubility gives specific information about degradation while water absorption is more related to the swelling capability of granules.

The changes in WAI for the range of the extrusion conditions used were not significant, except in the case of 8% steam, ranging only between 5.6 and 5.8 for corn flakes and between 4.3 and 5.4 for extrudates, as shown in appendix 5. While the change in WSI
confirmed once again the results obtained for paste viscosity and degree of gelatinization. Water solubility increased with increased steam injection because of structural modifications in the starch granules resulting from the mechanical and thermal stresses applied during the extrusion process (Fig.4-16).

The fact that at 8% steam injection the value for WAI was lower may be due to the fact that at high degree of gelatinization the starch granules lose the capability of swelling and the molecular starch structure, weakened by mechanical and thermal shearing, breaks up increasing the starch solubility. In the case without steam injection the solubility was higher because the low moisture content resulted in a higher degree of gelatinization (Gomez and Aguilera, 1983 and Bhattacharyya and Hanna, 1987).

The results found for the extruded and the traditional corn flakes seemed to be anomalous because, while the difference in water absorption is slightly significant, the WSI for the extruded product is lower. This may due to the fact that the extruded product formed a strong gel and it was difficult to remove the water trapped in the gel by centrifugation. Since only a small amount of supernatant was recovered the WSI was low for these samples.
Fig. 4-16. The effect of steam injection on WSI for the extrudate and the corn flakes.
5. CONCLUSIONS

Despite their widespread use in food extrusion, the design as well as the operating conditions of preconditioners have remained highly empirical.

The aim of this research program was to develop a detailed understanding of the effects of preconditioning on the physical, chemical, functional properties of extruded products and to compare these with the traditional process.

First, we measured experimentally the residence time distribution in order to investigate the effect of the operating conditions on the flow patterns and the effect of the preconditioner on the extruder flow characteristics.

The changes in some functional properties were related to the modification of structural properties that takes place during the cooking-extrusion process. The important results that have emerged are:

1. The residence time distribution in the preconditioner depends on feed rate and paddle speed. Different distributions were found using two different tracers. The preconditioner does not provide a uniform mixing due to segregation, especially in presence of large differences in particle sizes in the feed stream.

The Pe number and the variance indicated that with the red dye a better mixing with a shorter residence time was obtained.

The effect of segregation is also significant for the preconditioner-extrusion system, since the distribution inside the extruder strongly depends on the conditions experienced by the material in the preconditioner.
The uniformity of mixing is important for the characteristics of the final product, since part of the product may receive excessive or insufficient cooking.

2. The material flow inside the preconditioner can be described by two mechanisms. Part of the material moves directly through an active volume with a residence time distribution similar to a dispersed plug flow. The remaining part is delayed in a dead space region, that creates a long tail in the distribution. The Wolf and Resnick multistage model was shown to adequately describe this flow behavior, since it represents the tail of the distribution very well.

3. The changes in functional properties were interpreted in terms of modifications at the molecular level. When the extent of degradation increased, the viscosities at maximum temperature and at the end of the cooling cycle were lower. The increase in amount of steam injected increased the extent of degradation. In the case without steam injection the low moisture content resulted in higher viscosity and higher degree of gelatinization.

4. The measurement of the degree of gelatinization confirmed the effect of the steam injection on the mechanical and thermal degradation.

5. The changes in WAI for the range of the extrusion conditions used were not significant. Water solubility increases with increased steam injection because of structural modifications in starch granules resulting from the mechanical and thermal stresses applied during the extrusion process.
6. Some of the functional properties were measured in order to compare the characteristics of the extruded and traditional corn flakes.

The extruded product had a lower final viscosity indicating a higher extent of degradation and higher degree of gelatinization. This is due to the effect of the two degradation mechanisms present in the extruder, thermal and mechanical. While in the case of pressure cooker, used in the traditional process, only the thermal degradation effect is present.

These results help to have a better understanding of the effect of preconditioning on the final quality of the extruded products and provide the information needed to optimize the preconditioning-extrusion process.
6. RECOMMENDATIONS

As result of this research program the following recommendations can be made:

1. The residence time distribution results showed that mixture of particles of different sizes causes segregation inside the preconditioner and consequently poor quality of mixing. Further studies could lead to the design of a paddle configuration able to improve residence time and quality of mixing.

2. The changes in functional properties were related to the structural modification of starch during cooking extrusion. Future experimental work should include a detailed analysis of the degradation of starch polymer structure. High performance liquid chromatography (HPLC) has been shown to be a very effective technique for the determination of changes in the molecular weight distribution of simple systems such as starch. It would be interesting to expand the study of molecular degradation to a more complex food system, such as corn flour and corn flakes formulations.

3. The study of preconditioning could include the effect on oat, wheat, rice, barley and rye, all grains used in formulating breakfast cereals. The presence of protein, fat, fiber and the different amylose/amylopectin ratios will affect the rheology, the gelatinization, the functional properties thus the finished product quality.

4. The current state of food extrusion control in the industry is described as manual feedback control. Direct measurement of product quality such as moisture, colour,
expansion, and cell size is very limited and general. Future studies should be extended to quality control via on-line measurement of quality-determining extrusion parameters. A dynamic comprehensive model could be developed to predict the uncertainty in process measurements and to describe the relationships among operating variables.

5. Many researchers noted that paste viscosity could be used as a control variable for the extrusion process. The use of rapid and easy viscosity measurement, such as the Rapid ViscoAnalyser (RVA), should be investigated further.
7. REFERENCES


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FAST, R.B. and CALDWELL, E.F. 1990 Breakfast cereals and how they are made. American Association of Cereal Chemistry, St. Paul, MN.


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WANG, S.S., CHIANG, W.C., YEN, A.I., ZHOA, B., and KIM, I.H. Kinetics of phase transition of waxy corn starch at extrusion temperature and moisture contents. J. of Food Sci. 54: 1298-1301, 1326


8. APPENDIXES
Appendix 1. Residence time distribution calculations

A pulse of tracer was injected into the preconditioner and into the extruder and the downstream concentration of tracer was monitored as a function of time. The exit age distribution, $E(t)$ was given by:

$$E(t) = \frac{C(t)}{\int_{0}^{t} C(t) dt}$$

Where $C(t) =$ concentration of tracer in the exit stream.

And the integral $A = \int_{0}^{\infty} C(t) dt$ was calculated using Simpson's Rule.

The tracer concentration was measured for discrete time intervals, $\Delta t$. The cumulative exit age distribution, $F(t)$, the mean residence time and the variance were calculated as follows:

$$F(t) = \frac{\sum_{0}^{i} C(t) \Delta t}{A}$$

$$\bar{t} = \frac{\sum_{0}^{i} t C(t) \Delta t}{A}$$

$$\sigma^2 = \frac{\sum_{0}^{i} t^2 C(t)}{A} - \bar{t}^2$$
The Pe number was calculated by numerically solving the following equation:

\[(\sigma / \tilde{\tau})^2 = 2 \left( \frac{Pe - 1 + e^{\nu}}{Pe^2} \right) \]

**Fitting Model Parameters**

The parameters were estimated by the LJ direct search optimization technique. The method is based on using a number of randomly chosen test points over some region and contracting the region after every iteration, always starting the iteration with the best point found from the previous iteration as the centre of the region.

Computations were performed on a Pentium 120 and the total computation time was a few seconds. To carry out the parameter estimation, NPTS (number of random points per iteration) = 100, NIT (number of iterations) = 301, RED (region contraction factor) = 0.95 were chosen.

The performance index is given by the square of deviation between \( F \) (cumulative residence time distribution) expected from the model and \( F \) experimentally measured values.
The area under the curve is given by:

\[ A = \Delta t/3 \left( c_0 + 4c_1 + 2c_2 + \cdots + 2c_{n-2} + 4c_{n-1} + c_n \right) \]
Appendix 2. Operating conditions

Traditional method

Process Flowsheet

Dry ingredient delivery and handling

Steam

Liquid ingredient delivery and handling

Blending

Cooking

Surge Buffer

Drying

Tempering

Flaking

Toasting

Cooling

Packing Line
### Moisture content

<table>
<thead>
<tr>
<th>Process</th>
<th>Moisture Content</th>
</tr>
</thead>
<tbody>
<tr>
<td>At the end of cooking process</td>
<td>30%</td>
</tr>
<tr>
<td>Before drying</td>
<td>28%</td>
</tr>
<tr>
<td>At the end of tempering process</td>
<td>18.5%</td>
</tr>
<tr>
<td>At the end of toasting process</td>
<td>3.2%</td>
</tr>
</tbody>
</table>

### Flaking Conditions:

- Pivot roll inlet temperature: 29 °C
- Pivot roll outlet temperature: 31 °C
- Fixed roll inlet temperature: 23 °C
- Fixed roll outlet temperature: 20 °C

### Toaster Conditions

- Zone 1 Conveyor Speed: 285 rpm
- Zone 2 Conveyor Speed: 271 rpm
- Cooling Conveyor Speed: 20 rpm
- Zone 1 Temperature: 185 °C
- Zone 2 Temperature: 175 °C
## Appendix 3. Paste viscosity results

<table>
<thead>
<tr>
<th>Sample</th>
<th>Sample concentration (dry matter basis) (%)</th>
<th>Initial cold viscosity (centipoises)</th>
<th>Maximum Temperature viscosity (centipoises)</th>
<th>Final viscosity (centipoises)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Raw Corn Flour</td>
<td>8.6</td>
<td>0</td>
<td>330</td>
<td>486</td>
</tr>
<tr>
<td>Trad. Corn flakes</td>
<td>8.9</td>
<td>24.4</td>
<td>49.1</td>
<td>94.3</td>
</tr>
<tr>
<td>Extr. Corn Flakes</td>
<td>8.9</td>
<td>33</td>
<td>40.9</td>
<td>85.2</td>
</tr>
<tr>
<td>Ropes 0%</td>
<td>8.7</td>
<td>44</td>
<td>165</td>
<td>315</td>
</tr>
<tr>
<td>Ropes 1%</td>
<td>8.8</td>
<td>34.9</td>
<td>151.2</td>
<td>301</td>
</tr>
<tr>
<td>Ropes 3%</td>
<td>8.6</td>
<td>157</td>
<td>88</td>
<td>209</td>
</tr>
<tr>
<td>Ropes 5%</td>
<td>8.7</td>
<td>145</td>
<td>93.5</td>
<td>218</td>
</tr>
<tr>
<td>Ropes 8%</td>
<td>8.8</td>
<td>110</td>
<td>67</td>
<td>198</td>
</tr>
<tr>
<td>Corn Flakes 0%</td>
<td>8.6</td>
<td>130</td>
<td>66.5</td>
<td>165</td>
</tr>
<tr>
<td>Corn Flakes 1%</td>
<td>8.7</td>
<td>157</td>
<td>73</td>
<td>185</td>
</tr>
<tr>
<td>Corn Flakes 3%</td>
<td>8.7</td>
<td>157</td>
<td>77</td>
<td>171</td>
</tr>
<tr>
<td>Corn Flakes 5%</td>
<td>8.6</td>
<td>148</td>
<td>78</td>
<td>169</td>
</tr>
<tr>
<td>Corn Flakes 8%</td>
<td>8.6</td>
<td>101</td>
<td>44</td>
<td>113</td>
</tr>
</tbody>
</table>
### Appendix 4. Degree of gelatinization results

<table>
<thead>
<tr>
<th>Product</th>
<th>Degree of gelatinization (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Traditional corn flakes</td>
<td>89</td>
</tr>
<tr>
<td>Extruded corn flakes</td>
<td>99.6</td>
</tr>
<tr>
<td>Corn Flakes 0% steam</td>
<td>89.8</td>
</tr>
<tr>
<td>Corn Flakes 1% steam</td>
<td>80.1</td>
</tr>
<tr>
<td>Corn Flakes 3% steam</td>
<td>82.3</td>
</tr>
<tr>
<td>Corn Flakes 5% steam</td>
<td>88.6</td>
</tr>
<tr>
<td>Corn Flakes 8% steam</td>
<td>91</td>
</tr>
<tr>
<td>Ropes 0% steam</td>
<td>77.1</td>
</tr>
<tr>
<td>Ropes 1% steam</td>
<td>73.2</td>
</tr>
<tr>
<td>Ropes 3 % steam</td>
<td>72.9</td>
</tr>
<tr>
<td>Ropes 5 % steam</td>
<td>76.6</td>
</tr>
<tr>
<td>Ropes 8 % steam</td>
<td>80</td>
</tr>
</tbody>
</table>
## Appendix 5. WAI and WSI results

### Raw Corn Flour

<table>
<thead>
<tr>
<th></th>
<th>WSI</th>
<th>WAI</th>
<th>Extruded Corn Flakes</th>
<th>Traditional Corn Flakes</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>2.1%</td>
<td>2.3</td>
<td>17.1%</td>
<td>20.5%</td>
</tr>
</tbody>
</table>

### Corn Flakes

<table>
<thead>
<tr>
<th>Corn Flakes</th>
<th>0% steam</th>
<th>1% steam</th>
<th>3% steam</th>
<th>5% steam</th>
<th>8% steam</th>
</tr>
</thead>
<tbody>
<tr>
<td>WSI</td>
<td>15.7%</td>
<td>13.1%</td>
<td>13.6%</td>
<td>14.4%</td>
<td>16.2%</td>
</tr>
<tr>
<td>WAI</td>
<td>5.6</td>
<td>5.8</td>
<td>5.6</td>
<td>5.6</td>
<td>7.1</td>
</tr>
</tbody>
</table>

### Ropes

<table>
<thead>
<tr>
<th>Ropes</th>
<th>0% steam</th>
<th>1% steam</th>
<th>3% steam</th>
<th>5% steam</th>
<th>8% steam</th>
</tr>
</thead>
<tbody>
<tr>
<td>WSI</td>
<td>5.1%</td>
<td>4.2%</td>
<td>8.3%</td>
<td>9%</td>
<td>10.4%</td>
</tr>
<tr>
<td>WAI</td>
<td>4.3</td>
<td>4.7</td>
<td>5.6</td>
<td>5.4</td>
<td>3.8</td>
</tr>
</tbody>
</table>
### Appendix 6. List of symbols often used in this thesis

<table>
<thead>
<tr>
<th>Symbol</th>
<th>Quantity</th>
<th>Units</th>
</tr>
</thead>
<tbody>
<tr>
<td>A</td>
<td>preexponential factor (plug-flow stirred tank model)</td>
<td></td>
</tr>
<tr>
<td>b</td>
<td>parameter in Wolf and Resnick model</td>
<td></td>
</tr>
<tr>
<td>C(t)</td>
<td>tracer concentration at time t</td>
<td>p.p.m.</td>
</tr>
<tr>
<td>$D_a$</td>
<td>axial dispersion coefficient</td>
<td>cm²s⁻¹</td>
</tr>
<tr>
<td>$E(t)$</td>
<td>residence time distribution function</td>
<td></td>
</tr>
<tr>
<td>$E(\theta)$</td>
<td>normalized residence time distribution function</td>
<td></td>
</tr>
<tr>
<td>$F(t)$</td>
<td>cumulative residence time distribution function</td>
<td></td>
</tr>
<tr>
<td>$f_d$</td>
<td>fraction of material delayed in the dead space</td>
<td></td>
</tr>
<tr>
<td>$F(\theta)$</td>
<td>normalized cumulative residence time distribution function</td>
<td></td>
</tr>
<tr>
<td>L</td>
<td>barrel length</td>
<td>m</td>
</tr>
<tr>
<td>n</td>
<td>power law index, eq.2-10</td>
<td></td>
</tr>
<tr>
<td>N</td>
<td>number of CSTRs in series</td>
<td></td>
</tr>
<tr>
<td>P</td>
<td>pressure</td>
<td>Pa</td>
</tr>
<tr>
<td>Pe</td>
<td>Peclet number</td>
<td></td>
</tr>
<tr>
<td>t</td>
<td>time</td>
<td>s</td>
</tr>
<tr>
<td>$t_a$</td>
<td>mean residence time in active region (Levich model)</td>
<td></td>
</tr>
<tr>
<td>$t_d$</td>
<td>mean residence time in the dead space (Levich model)</td>
<td></td>
</tr>
<tr>
<td>$\bar{t}$</td>
<td>mean residence time (Levich model)</td>
<td>s</td>
</tr>
<tr>
<td>u</td>
<td>axial velocity</td>
<td>m/s</td>
</tr>
<tr>
<td>x</td>
<td>rectangular coordinate (cross-channel direction)</td>
<td></td>
</tr>
</tbody>
</table>
\( y \)  
rectangular coordinate (channel depth direction)

**Greek letters**

\( \delta \)  
time delay in the pipe (plug-flow stirred tank model)  
\( \text{s} \)

\( \varepsilon \)  
phase shift

\( \theta \)  
normalized time

\( \mu \)  
viscosity  
\( \text{centipoise} \)

\( \sigma^2 \)  
variance of residence time distribution  
\( \text{s}^2 \)

\( \tau \)  
time constant in stirred tank  
\( \text{s} \)

or shear stress (eq. 2-8 and 2-9)

**Abbreviations**

RTD  
residence time distribution

RTE  
ready to eat

WAI  
water absorption index

WSI  
water solubility index

EX  
extruder

PC  
preconditioner

PC-EX  
preconditioner extruder