PROCESSING AND QUALITY OF RICE-BASED EXTRUDED PRODUCTS

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***in* ENGINEERING**

by

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DECLARATION

The work incorporated in the thesis entitled **"Processing and quality of rice-based extruded products"** was carried out by me at the Department of Grain Science and Technology, Central Food Technological Research Institute, Mysore 570 013, under the guidance of Dr. S. Zakiuddin Ali, Head, Department of Grain Science and Technology, Central Food Technological Research Institute, Mysore and Professor Parimal Chattopadhyay, Department of Food Technology and Biochemical Engineering, Jadavpur University, Calcutta.

I further declare that the work embodied in this thesis has not been submitted for the award of any degree, diploma or other similar title.

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CERTIFICATE

This is to certify that the thesis entitled **"Processing and quality of rice-based extruded products"** submitted by **Smt. Manisha Guha** who got her name registered on 18.01.1994 for the award of Ph.D. (Engineering) degree of Jadavpur University, is absolutely based upon her own -work under the supervision of Dr. S. Zakiuddin Ali and Dr. Parimal Chattopadhyay, and that neither her thesis nor any part of it has been submitted for any degree/diploma or any other academic award anywhere before.

(S. Zakiuddin Ali)	(Parimal Chattopadhyaya)
1. Signature of the Sole Supervisor	2. Signature of Joint Supervisor
Date: 09.01.2000	Date: 10.01.2000

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MANISHA GUHA

CONTENTS

LIST OF TABLES

LIST OF FIGURES

NOMENCLATURE

b*	Colour value in CIELab system indicative of blueness with negative sign
b_o	Constant in the polynomial
b1, b_2... b_k	Constants in the polynomial showing the linear effect of the variables X1, X_2.... X_k, respectively
b_{11}, b_{22}	Constant in the polynomial showing the quadratic effect of the variables X1 and X_2, respectively
b_{313}, b_{323}	Constant in the polynomial showing the cubic effect of the variables X1 and X_2, respectively
b_{12}, b_{221}, b_{212}	Constant in the polynomial showing the interaction effect of the variables X_1 and X_2
BD	Bulk density of the dried extrudates (kg m^{-3})
CPV	Cold paste viscosity (RVU)
d. b	Dry basis
d_g	geometric average of two successive sieve openings (µm)
d_{ga}	geometric average particle diameter of rice flour (µm)
ER	Expansion ratio
F	Statistical F-value
HPV	Hot paste viscosity (RVU)
i	Integers (1,2,3...),
k	Number of variables
k_{av}	Elution constant
K_f	Conductivity of the die (m^3)

MD	Mixing disk
M_w	Molecular weight (average)
n	Screw speed (rpm)
n1	Number of data points for calculating correlation coefficient
N	Number of filled channels
0.D	Optical density
P	Probability level
PV	Peak viscosity (RVU)
Q	Volumetric flow rate (m^3 S^{-1})
R	Correlation coefficient
RPSE	Reverse pitch screw element
RT	Average residence time (s)
RVU	Rapid Viscoanalyser unit (for apparent viscosity)
SME	Specific mechanical energy (kJ kg^{-1})
T	Torque at the screw shaft during extrusion (%)
Ve	Elution volume (ml)
V_o	Void volume (ml)
V_t	Total volume (ml)
Wi	Weight fraction of rice flour retained on ith sieve
WAI	Water absorption index (g/g)
W-B-SS	Warner-Bratzler shear stress (kPa)
WSI	Water solubility index (%)
Xi	Variable in coded level
y	Response function
Z	Power for shearing within the channels (J s^{-1})

Z_c	Power for shearing between the flight tip of one screw and the barrel or bottom of the channels of the other screw ($J\ s^{-1}$)
Z_p	Power needed for pressure build up ($J\ S^{-1}$)
Z_s	Power for shearing between the flight tip of one screw and the barrel or bottom of the channels of the other screw ($J\ S^{-1}$)
Z_w	Power for shearing between the flanks of the flight in the intermeshing region ($J\ s^{-1}$)
Z_t	Total power transmitted from the main motor to the screws ($J\ s^{-1}$)
ε	Random error
μ	Apparent viscosity in the filled channels (Pa s)
μ	Average apparent viscosity in the filled channels (Pa s)
P_B	Bulk density of the individual dried extruded rod ($kg\ m^{-3}$)
λ_{max}	Absorption maximum (nm)

SYNOPSIS

Extrusion cooking is one of the most promising techniques in the field of food technology developed in the recent times. Its advantages are many, which have been well documented in the literature. However, practice of this technology, has remained more an art than the science. Knowledge and database on extrusion cooking is however growing fast. Extrusion cooking is associated with partial or complete gelatinisation of the starch, complex formation, transformation and interactions involving bio-polymers.

Of the two types of extruders (single screw and twin-screw), twin-screw extruder permits a greater flexibility of operation to achieve the desired time, temperature, and shear range for the processed material because of an additional independent variable, viz., screw configuration.

Understanding the extruder behaviour and material flow during extrusion cooking is essential to the design of automation and control systems. Some models have adapted from plastic extrusion with modifications that account for the differences of foods from plastics.

Cereal grains lend themselves as good raw material for preparation of ready to eat snack foods and other products on account of high starch content in them. Extensive work has been reported on extrusion of corn (maize), in comparison to that for wheat and rice. The latter is of significance to India and other Asian countries.

Rice is a popular, nonallergic, glutenfree source of carbohydrate, vitamins, and minerals with little fat. With an annual production of over 120 million tonnes of paddy, rice is the largest crop produced and consumed in India. It is the major supplier of energy, protein and other nutrients in the diet of more than half of the Indian population. Apart from being consumed as whole grain for table purposes in the form of raw milled rice or parboiled milled rice, a considerable quantity of paddy is also converted into many traditional products. Extrusion cooking could give products similar to the traditionally prepared expanded or puffed rice products with advantage of being more hygienic and economic process. However, literature on twin-screw extrusion cooking of rice is rather scanty.

The present study was, therefore, undertaken to screen a larger number of extruder and extrusion variables, and to study their effect on the system parameters during extrusion processing of rice flour, and the quality of resultant extruded products in terms of physical, physicochemical, molecular and functional properties. The results obtained form the basis of this thesis. The thesis has been divided into seven chapters. A brief outline of the same is given below:

CHAPTER I. Extrusion cooking of cereals: a review

A detailed review of the literature on extrusion process, extruders, models in explaining application of extrusion cooking, in general and also with reference to the process of extrusion cooking is presented.

CHAPTER II. Materials and methods - general

This chapter deals with the general methodologies applicable for the entire work with details on the rice cultivars used, their proximate composition, the extruder used, feed preparation for extrusion, extrudate sample preparation for study of different characteristics and the methodology followed for the extrusion and extrudates attributes study. However, the methodologies specific to the work presented in each separate chapter, have been described in the concerned chapter.

CHAPTER III. Screening of variables for extrusion cooking of rice flour employing Plackett-Burman design

Technology of extrusion cooking of foods has been successfully applied to produce a variety of foods during the last two decades. But till now, it is still considered an art. To select a particular variable, or to delete one, is still based on experience, or sometimes, just a guess. This is because of the large number of variables encountered in extrusion cooking. The situation becomes complex if the researcher is interested to know the quantitative effect of these variables on extrusion target parameters.

In this chapter, results of the screening experiments for ten variables, employing Plackett-Burman experimental design, have been reported. Effect of variables on the system parameters and target product parameters without using any die during extrusion processing of rice flour has been studied. The variables included extruder hardware variables (presence or absence of mixing disk and reverse pitch screw element), feed variables (moisture, amylose content, particle size and sugar/salt) and extrusion operating variables (barrel temperature, feed rate and screw speed). The response functions were the extrusion characteristics (torque, net specific mechanical energy and average residence time), product attributes (water solubility index, water absorption index and bulk density), and the viscographic properties of flour slurry (peak viscosity, hot paste viscosity and cold paste viscosity). The results were analysed using coded level (-1 and +1) of variables and fitting to first order regression equations.

The results showed that torque developed during extrusion of rice flour under the experimental conditions ranged between 11.4 and 74.5%, of which low values (~ 25%) were obtained when both reverse pitch screw element (RPSE) and mixing disk (MD) were absent. Among the individual variables, the effect of MD was maximum followed by screw speed, feed rate and RPSE.

The high values (>300 kJ kg^{-1}) for net specific mechanical energy (SME) during extrusion of rice flour was observed when the screw profile included RPSE and / or MD. Among the other variables, amylose content and screw speed imparted marked negative effect. Further, the effect of moisture content was positive followed by the effect of feed rate (negative) and temperature (negative).

The average residence time (RT) of material in the barrel varied between 15.4 and 32.9 s. The presence of RPSE and MD showed the maximum effect on RT. Screw speed, amylose content and feed rate also exerted a marked (negative) effect on RT in that order.

The extrusion of non-waxy (high amylose content, 28.6%) rice flour resulted in low water solubility index (WSI, 3.4 to 20.8%) whereas, that from waxy (very low amylose content, 5.0%) rice variety showed a higher range (12.2 to 65.0%). The co-efficient of regression equations showed the temperature of extrusion had the highest (positive) effect on WSI. RPSE had the next higher (positive) effect on WSI of the extruded product.

Amylose content exerted maximum (positive) effect on water absorption index (WAI. The effect of temperature, MD and RPSE also showed positive effect on WAI, whereas sugar content showed a negative effect.

The bulk density of the extruded products varied widely (170-730 kgm^{-3}). The negative effect of RPSE/MD indicated that their presence markedly decreased bulk density. The feed rate also had a negative effect, whereas sugar exerted a positive effect. The variables that ranked next in exerting effect on BO were salt (-ve effect), amylose (+ve effect) and screw speed (+ve effect).

The variables that showed highest effect on viscosity indices (viz., peak viscosity, PV; hot paste viscosity, HPV; and cold paste viscosity, CPV) were RPSE, MO (both have negative effect) and screw speed (positive effect). Further, PV and HPV were affected negatively by feed rate and moisture content of feed respectively, while CPV was affected (positively) by amylose content.

It could be concluded from the above results that the screw configuration, particularly, the presence of reverse pitch screw element and mixing disk, imparts maximum effect on the extrusion and extrudate characteristics.

Considerable effect was also observed for amylose and moisture content, feed rate, screw speed and barrel temperature. The variables that showed least effect on the response functions were particle size, salt and sugar. The experimental results relating the variables and response function could be fitted well ($0.721 \leq r \leq 0.999$, $P \leq 0.01$) by first order polynomials which indicates the suitability of Plackett-Burman experimental design to evaluate the effect of the individual variables. Thus, the Plackett-Burman experimental design can serve as a useful tool to screen large number of variables and to reduce the number of experiments.

CHAPTER IV. Effect of barrel temperature and screw speed on extrusion parameters and certain physicochemical properties of rice extrudate

Rice flour with 14% moisture content was extruded at different barrel temperatures (80 - 120°C) and screw speed (200-400 rpm) through the twin-screw extruder at a constant feed rate (17 kgh^{-1}) without using a die. The extrusion trial was performed with a screw configuration consisting of forward pitch screw elements, a reverse pitch screw element (near the outlet), and also two kneading blocks.

Effect of these process variables on the extrusion system parameters and the extrudate attributes was determined by using response surface analysis technique. The response functions studied were: a). Torque developed during extrusion, b). Total specific mechanical energy (SME) required during extrusion process, c). Sediment volume, d). *In-vitro* starch digestibility, e). Water absorption index, f). Water solubility index and f). bulk density. These seven response functions were related to the extrusion process variables by a second-degree polynomial, which consisted of linear, quadratic and interaction effect. The method of least squares was used to develop these polynomials and accordingly the response surfaces were developed.

The torque developed during extrusion of rice flour in the present experiments ranged between 38 and 85%. High torque was associated at low screw speed (200rpm). This indicates that rice extrusion is a torque limiting process, particularly if the extruder is operated at low screw speed.

The effect of temperature on torque was rather complex and depended on the level of screw speed. At a high screw speed (400 rpm), increase in the barrel temperature from 80 to 100°C markedly decreased the torque but it remained fairly constant when the temperature was raised beyond 100°C. At a low screw speed (200 rpm), slight decrease in torque was noted with an increase in barrel temperature.

The total specific mechanical energy (SME), defined as the total mechanical energy input to obtain 1kg of extrudate, varied between 317 and 1013 $kJkg^{-1}$. The low values «350 $kJkg^{-1}$) of SME were obtained at high temperature (10O-120°C) and screw speed (300 rpm).

Sediment volume of flour in excess dilute HCI, which serves as an index of the gelatinisation, showed an increase from 7.5 ml for raw to 24.5 to 26.5 ml for all the extruded products, indicating that the degree of gelatinisation in all the extruded samples was markedly high as compared to that reported for parboiled or flaked rice in the literature, which ranged between 8.1 to 19.5 ml.

The in-vitro digestibility of starch in the extruded rice sample was estimated using amyloglucosidase. The results showed that all the extruded rice samples had very high (73.6-87.4%) susceptibility to enzyme degradation as compared to raw rice (12.8%). The extent of susceptibility depended on the conditions of extrusion temperature and screw speed. The digestibility increased with increase in barrel temperature from 80 to 100°C, but showed a decrease for samples extruded at 120°C. The digestibility decreased by 2 to 6 per cent points with increasing screw speed from 200 to 400 rpm.

Water absorption index (WAI) of extruded rice samples ranged between 5.5 to 7.1 gig as compared to unextruded rice (2.6 gig). High WAI (6.1 to 7.1gig) was shown by products extruded at barrel temperature of 80 and 100°C as compared to that at 120°C (5.5 to 5.9 gig). Water solubility index (WSI) was the least (1.7%) for raw rice and increased markedly from 28.0 to 45.5% upon extrusion cooking. It could be inferred that the combined effect of high temperature and high screw speed enhanced the amount of soluble materials in the extrudate.

Bulk density of the extrudates ranged between 172 and 231 $kgm.^{-3}$ Lowest density was obtained at barrel temperature of 100°C and screw speed of 300 rpm.

The analysis of variance (ANOVA) tables were generated for all the seven response functions. The significance of the individual terms in the polynomial was determined statistically by calculating the F - values, and judging them at probability levels (p) of 0.01, 0.05 or 0.10. The correlation coefficients (r), determined to know the relationships between the extrusion characteristics and product attributes, were judged at p = 0.01 when the number of data points (n) was 27.

The detailed statistical analysis using response surface methodology (RSM) generated the coefficients of the second order polynomials for the above seven response functions. The polynomials, developed using the coded level of variables, fitted the experimental results as well as indicated by the high multiple correlation coefficients ($r \geq 0.931$, $P \leq 0.01$).

It is obvious that extrusion characteristics and products attributes are inter-related, and hence, in this chapter linear inter-relationships have been obtained between the extrusion characteristics (torque and SME) and the product attributes (sediment volume, *in-vitro* digestibility, WAI, WSI and bulk density). Significant ($p \leq 0.01$) positive relationships were obtained for torque with WAI and bulk density, whereas, a negative relation was seen with WSI. This means that increasing the torque during processing usually increases the desirable characteristics like WAI but reduces the bulk density and WSI of the extrudates. The torque during extrusion depends on the rheological status of the plasticised mass inside the extruder, and in turn, it is positively related ($r > 0.72$, $P \sim 0.01$) to SME. It was concluded therefore that extrusion at 100°C, 300 rpm needed the least energy (SME 317kJ kg^{-1}) and produced lesser torque (39%) during extrusion of rice flour and yielded product with a desirable quality profile.

CHAPTER V. Effect of barrel temperature and screw speed on pasting behaviour of rice extrudate

The effect of extrusion barrel temperature (80-120°C) and screw speed (200-400 rpm) on the pasting and gelatinisation properties of extruded rice products was studied. The pasting characteristics of the flour slurry were studied using a Rapid Viscoanalyser (RVA), and the viscosity indices observed were the initial viscosity (IV, at 50°C), peak viscosity (PV, highest viscosity during heating phase to 95°C), hot paste viscosity (HPV, viscosity after cooking for 5 min at 95°C), cold paste viscosity (CPV, viscosity after cooling the paste to 50°C) and extent of gelatinisation (GE difference, in percent, of the peak area of processed sample and the raw flour).

Regression equations were generated for the response functions (PV, HPV, CPV and GE) to relate them with the extrusion process variables (barrel temperature and screw speed). The suitability of the regression equations to predict the response functions was judged by determining the multiple correlation coefficient (r). The experimental results were fitted either to a second order or to a third order polynomial such that an r-value of 0.990 was obtained.

The initial viscosity (IV) of extrudates (30-43 RVU) was about ten times higher than that for raw rice (3-4 RVU). The viscosity of the extrudate pastes decreased during the heating phase, in contrast to that of raw rice, which increased.

The peak viscosity (PV) of the extruded rice pastes was between 21 and 33 RVU, very low in comparison to raw rice paste (218 RVU). PV generally decreased with increasing barrel temperature and screw speed. The contour plots showed, however, that at high screw speed (350-400 rpm) for barrel temperature greater than 100°C reverse effect could be observed.

The hot paste viscosity (HPV) of the uncooked rice flour paste was about 80 RVU, whereas, for the extruded rice flour it ranged from 7 to 12 RVU. A fairly linear decrease in HPV was obtained when temperature or screw speed was increased.

The cold paste viscosity (CPV) of extruded products (at the end of cooling to 50OC) was quite low and ranged between 8 and 15 RVU, as against the high value of 177 RVU for raw rice paste. The cold paste viscosity decreased with increasing barrel temperature and screw speed.

The extent of gelatinisation (GE) in the extruded rice samples was high, and ranged between 93.8 and 99.0%. The GE increased with barrel temperature, and reached a maximum (99.0%) at 100°C with a screw speed of 400 rpm. Thereafter, a slight decrease was observed with increasing screw speed and barrel temperature.

It could be concluded from the above results that desirable pasting characteristics (viz., low values of PV, HPV and CPV) for a pre-gelatinized rice flour for use in specialty diet food formulation, could be obtained by extrusion of rice flour at medium to high barrel temperature (100-11O°C) and a high screw speed (400 rpm).

CHAPTER VI. Molecular degradation of starch during extrusion cooking of rice

Molecular changes in starch during extrusion cooking of rice flour (from different varieties of rice) were studied by gel permeation chromatography (GPC), and the results are presented in this chapter.

Rice flour from three different paddy varieties, viz. IR 64, a high amylose (28.6%); Pojobora, an intermediate (22.3%) and Agonibora, a very-low amylose (5.0%, waxy) variety was extruded at different barrel temperature (80-120°C) through a twin-screw extruder at constant screw speed (400 rpm), feed rate (15 kgh^{-1}) and moisture content (20%) using a cylindrical, 5mm diameter die. The extruder screw assembly consisted of forward pitch screw element, a reverse pitch screw element (near the outlet), and also five kneading blocks.

Upon fractionation of rice flour on Sepharose CL-2B gel column, starch in all the rice samples got separated into two main fractions. One was eluted at the void volume (Fraction-I), which contained high molecular weight, branched component of starch i.e. amylopectin, and other that entered the gel and was eluted over a longer elution volume (Fraction II), which contained lower molecular weight, linear component of starch i.e. amylose.

Fraction-I of raw rice formed 61.6%, 66.4% and 85.4% of the total carbohydrate fractionated from IR 64, Pojobora and Agonibora respectively. Upon extrusion, the proportion of Fraction-I decreased considerably with consequent increase in Fraction-I I, indicating that amylopectin was degraded to lower molecular weight components as a result of extrusion cooking due to high thermal and mechanical energy inputs during extrusion. Consequently, the proportion of carbohydrate in Fraction-II increased. Further, a shift of peak of this fraction towards a higher-molecular-weight profile was observed, indicating that relatively larger molecular weight breakdown products from Fraction-I were released into this fraction. The tendency of this shift of Fraction-II peak towards the higher-molecular-weight side increased with increasing barrel temperature of extrusion, and also with lower amylose content.

Among the rice varieties, waxy rice was degraded to a greater extent (decrease by 45% points) as compared to the high amylose variety (decrease by 11% points) under similar extrusion conditions. Over all, it appears that the high molecular weight branched molecules (Fraction-I) was more prone to degradation than the linear ones, perhaps due to the large molecular size of amylopectin which renders it vulnerable to breakdown under severe condition of shear and thermal forces within the extruder. Further, waxy variety was degraded to the maximum extent at lower temperature (80°C) as compared to that for high amylose variety. The degradation increased with increasing barrel temperature for all three varieties. At higher temperature (120OC), however, a reverse trend was observed, which may be due to the formation of resistant starch or starch-lipid I starch-protein complexes at higher temperatures.

The iodine absorption (which is an index of the linearity of the molecule) of the two starch fractions separated on Seahorse CL-2B gel column was also studied. There was a progressive decrease in the iodine absorption of Fraction-II which was accompanied by a continuous decrease in the A_{max} of Fraction-II peak. The A_{max} of Fraction-II decreased from 656 nm to 612 nm, and from 621 nm to 608 nm after extrusion, in case of high and intermediate amylose variety respectively. However, A_{max} of Fraction-II for waxy rice remained more or less constant at 522-520 nm before and after extrusion. The iodine absorption of Fraction-I increased upon extrusion and it was accompanied by an increase in its A_{max}.

CHAPTER VII. Effect of varietal variation of rice and barrel temperature during extrusion cooking on functional properties of the extrudate

The effect of amylose content of rice and barrel temperature of the extruder on functional and textural properties of extrudate upon extrusion cooking of rice flour were studied in detail.

Extruded product obtained after extrusion cooking of the rice flour at different barrel temperature (80-120°C) from three varieties of rice (having a high, 28.6%; an intermediate, 22.3%; and a very low, 5.0%; amylose content) were studied for the functional and textural properties such as, bulk density (P_e), water solubility index (WSI). Water absorption index (WAI), expansion ratio (ER) and Wernere-Bratzler shear stress (W-B-SS).

Regression equations were generated for the response functions (torque, net specific mechanical energy, P_e, WSI, WAI, ER and W-B-SS) to relate them with the extrusion variables (amylose content of the feed and barrel temperature). The experimental results were fitted to a second order polynomial such that a multiple correlation coefficient r-value of 0.903 was obtained.

The torque during extrusion of rice flour ranged between 28 and 51%. High torque was associated with low amylose content feed. The SME varied between 296 and 781 $kJkg^{-1}$ and decreased with increasing barrel temperature in case of all rice varieties. However, extrusion of very-low amylose (waxy) rice required higher SME than other rice at all the barrel temperatures studied.

The bulk density of the extruded rod from three varieties ranged between 371 and 174 kgm^{-3}. In general, bulk density decreased with increasing barrel temperature but waxy variety product showed the least P_e. The WSI of extruded products ranged between 20.7 and 85.3%, increasing with increasing barrel temperature in all rice varieties. Waxy variety, however, yielded maximum WSI (85.3%) value. The expansion ratio of extruded rice products from three varieties ranged between 4.6 and 14.7, increasing with increasing barrel temperature. Here again the highest value (14.7) was obtained in case of waxy variety. The W-B-SS value was also lowest (176 kPa) for the waxy variety confirming the suitability of waxy variety to obtain expanded rice product.

Conclusions

Following conclusions could be drawn from the results presented above:

Plackett-Burman experimental design could be applied to screen the large number of variables in extrusion cooking. Out of ten variables studied, presence of the reverse pitch screw element and the mixing disk in the screw configuration exerted highest influence on the product profile of extruded product. Considerable effect was also observed for amylose and moisture content, barrel temperature, feed rate, and screw speed, whereas particle size and other additives showed lesser effect.

In the case of high amylose variety, barrel temperature showed maximum influenced on system parameters as well as quality attributes of extruded products.

Among the three varieties studied, having different amylose content, waxy rice (amylose content ≤ 5.0) appeared to be highly suitable for producing expanded rice product as it showed the highest expansion ratio, highest water solubility index and other desirable product profile. However, the specific mechanical energy required for extrusion was also high.

Extrusion of rice flour without using a die appears to be an alternative approach to produce processed rice flour. A restriction of die is usually used during extrusion of foods for shaping the final product. However, if the targeted product is to serve as a base material for producing other products (e.g., baby foods or weaning foods), the use of die becomes optional. Further, energy expenditure can be reduced if die is omitted.

CHAPTER I

Extrusion Cooking Of Cereals : A Review

around the outside of the barrel with vents permits heating or cooling of the barrel surface.

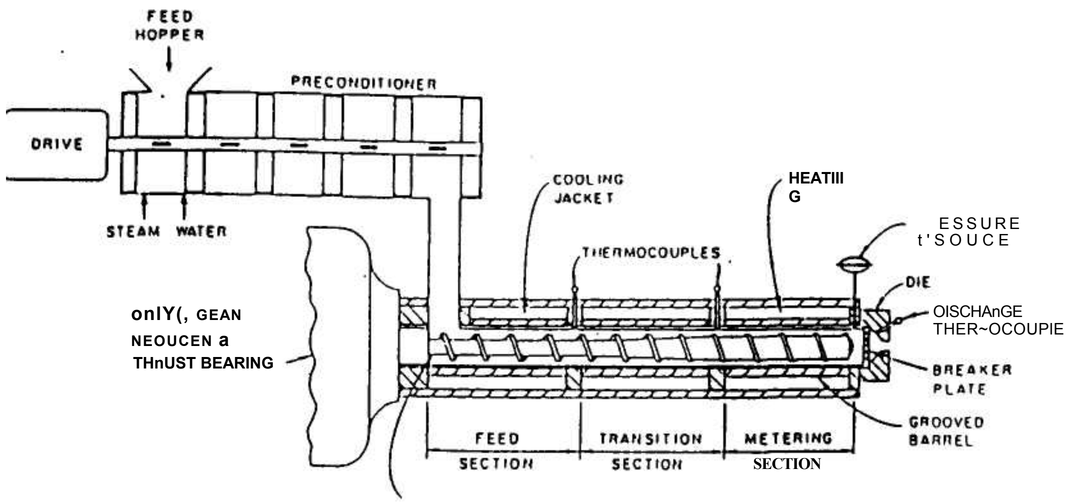


Fig. 1.1. Schematic diagram of a single-screw extruder. (Harper, 1989).

The screw system is the central portion of the extruder that accepts the feed ingredients at the feed port, conveys, works and forces them through the die restriction at the discharge point. The helical metal ribs wrapped around the screw shaft called "flights" convey the material mechanically towards the discharge end. The flights are of different height and shape. At the feed section, the flights are deeper or with greater pitch to ensure easy filling for conveying along the barrel. In the central part of the screw, called the "compression or transition section", the feed material is compacted and converted from a flowing granular or sticky mass to a relatively uniform plasticised dough. This section of the screw is followed by the metering section that has relatively shallow flights of reduced pitch to thoroughly mix, and/or increase the temperature of the material and the shear rate in the channel.

The continuous central shaft, known as the root of the screw, may sometimes be hollow to pass heating or cooling medium. The ratio of the distance between root surface of the screw and internal surface of the barrel, at the beginning of the barrel and at the end, is called "compression ratio", which is an important factor that characterizes the extruder.

The extrusion drive system, which drives the screw, consists of a drive motor and a stand. Continuous variability of the speed is possible through magnetic, electrical or mechanical controls. The motor speed is normally less than 500 rpm. A transmission is used to reduce the speed with a proportional increase in the torque of the motor. The whole extruder assembly is mounted on a frame or a stand that is bolted to the floor. Many times it is equipped with special disassembly devices for ease of functioning and maintenance. The food material leaving the extrusion screw enters the discharge section that normally holds the extruder die, cutters and takes away devices.

The dies have small openings that shape the food material as it flows out of the extruder. The shape of the die varies. The simple one being a hole. Expansion of the extrusion material occurs as the product under high temperature and pressure leaves the die with a rapid release of pressure to ambient conditions. Cutters are used to cut the extruded material coming out from the die. They are employed in combination with the take away devices, and/or the drying or the cooling systems. Drying and cooling section ensures careful decrease in the moisture or temperature of the product while maintaining the textural quality.

1.2.1. Classification of extruders

Rossen and Miller (1973) presented a systematic classification of extruders on the basis of thermodynamic and functional characteristics. Tribelhorn and Harper (1980) later gave a composite overall view of the various main categories as illustrated in Fig.1.2.

A typical pasta extruder has a deep flighted screw and a smooth barrel surface. Due to low shear rate and low screw speed, there is little, if any, cooking of moist semolina. High pressure forming extruder uses products with little larger range of moisture than the pasta extruder and constant temperature is maintained by heating jacket around the barrel and is used for producing pastry dough, cookies and certain candies (Harper and Harmann, 1973). A collet extruder, used for snack production, suitable for relatively dry materials, has a shallow flighted screw in a short grooved barrel, and heat is generated by mechanical energy input. Low-shear cooking extruders are suitable for relatively moist materials such as precooked dough and many pet foods (Linko et al, 1981).

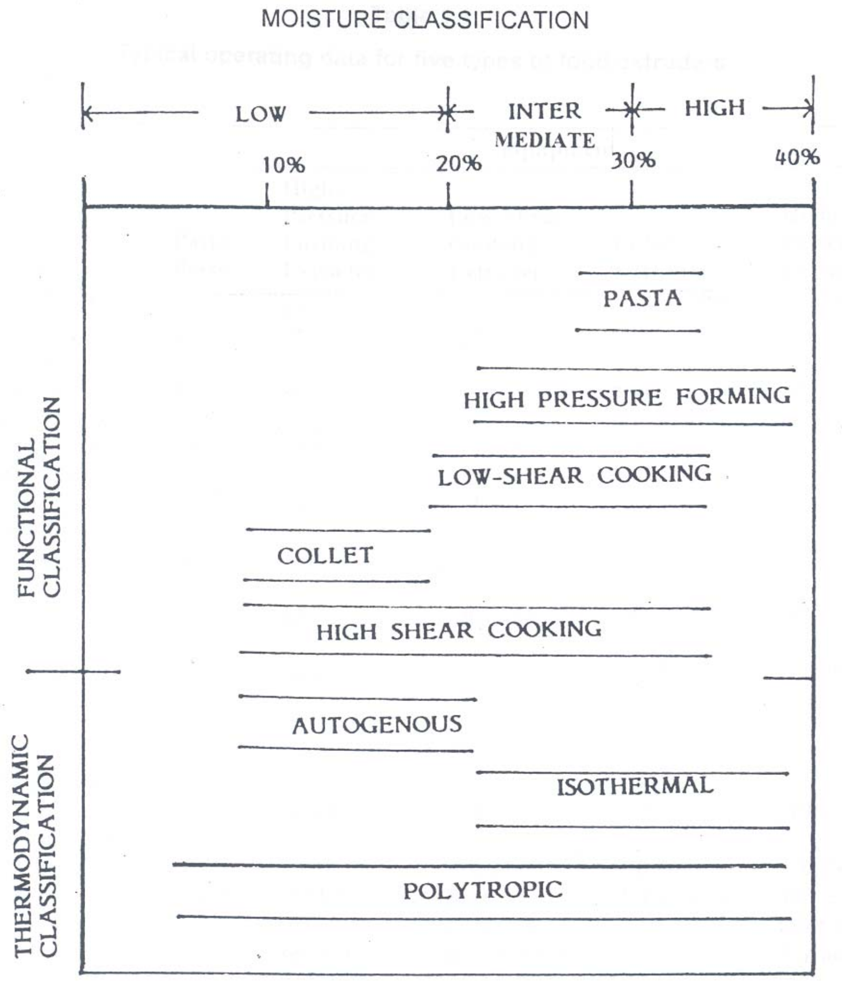


Fig. 1.2. Extruder classification.
(Tribelhorn and Harper, 1980).

High-shear extruders, classified as high-temperature short-time (HTST) cooking extruders, are suitable for producing a wide variety of precooked, pre-gelatinized, ready to eat cereal-based foods, textured plant protein, snacks, pet foods and animal feed (Linko et ai, 1981). Typical operating data for different types of food extruders are given in Table 1.1.

Initially, most food extruders were of the single-screw type, often modifications of equipment originally designed for the plastic industry (Harper, 1979). However, because bio-polymer materials have completely different rheological behaviour and mechanical transport properties, special barrel surface structure, and short residence time at high temperatures have been introduced including the twin-screw system (Kim and Rottier, 1980; van Zuilichem et ai, 1980 ; Tribelhorn and Harper, 1980).

Table 1.1 Typical operating data for five types of food extruders

Measurement	Equipment				
	Pasta Press	High-Pressure Forming Extruder	Low-Shear Cooking Extruder	Collet Extruder	High-Shear Cooking Extruder
Feed moisture, %	32	25	28	11	15–20
Product moisture, %	30	25	25	2	4–10
Maximum product temperature, °C	52	80	150	200	180
Screw diameter to flight height, D/H	3–4	4.5	7–15	9	7
No. of parallel screw flights, p	1–2	1	1	2–4	1–3
Screw speed (ω), sec^{-1}	4.5	6.5	10–30	50	70
Shear rate in screw ($\dot{\gamma}$), sec^{-1}	5	10	20–100	140	165
Net mechanical energy input, MJ/kg	0.11	0.14	0.14	0.36	0.40
Steam injection ($\dot{m}_s\lambda$), MJ/kg	0	0	0.11	0	0
Heat transfer (q) through jackets, MJ/kg	(0.04)	(0.04)	0–0.11	0	(0.11)–0
Net energy input to product, MJ/kg	0.07	0.10	0.25–0.36	0.36	0.29–0.40
Product types	Pasta	RTE,[a] pellets, second-generation snacks	Soft moist products, starch, soup bases, RTE	Puffed snacks	Textured vegetable protein, dry pet foods, modified starch

(Harper, 1989).

The main working principle of the twin-screw extruder is same as that of single-screw extruder. However, there are two screws instead of one, which are accommodated in a single barrel unit. The relative direction of the rotation of screw, counter or co-rotating, and the degree of screw intermeshing, are key points of differentiation. Various screw configurations used in twin screw extruders are shown in Fig. 1.3.

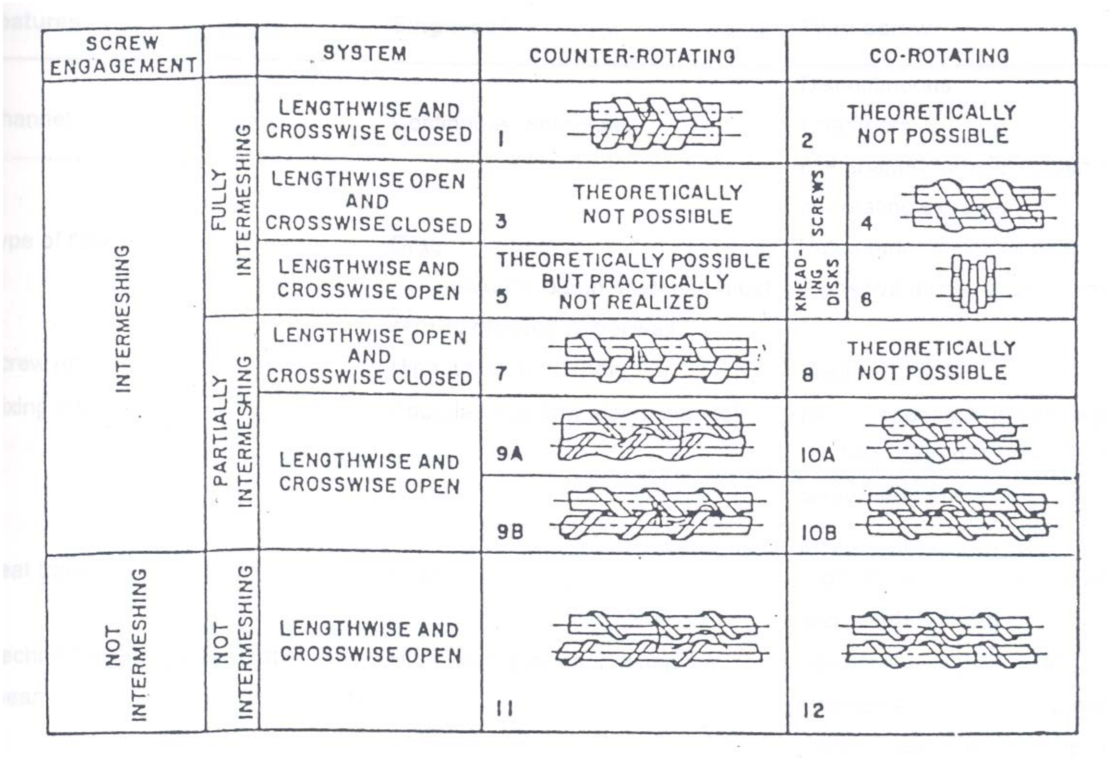


Fig. 1.3. Various screw configurations used in twin-screw extruders.
(Ziminiski and Eise, 1980).

1.2.2. Comparison of single- and twin-screw extruder

A general comparison of single- and twin- screw extruder is shown in Table 1.2.

1.4. ADVANTAGES OF EXTRUSION COOKING

The extrusion cooking process has been used increasingly because of its higher efficiency in comparison with conventional methods involving batch processing and multistage operation (Guy, 1989). The main advantages of extrusion cooking over conventional processes such as baking, autoclaving, etc are briefly listed below:

- Rapid high energy transfer into mass with H.T.S.T advantages
- High capacity with smaller investment and less space taken
- High energy efficiency because less drying required (low moisture cooking)
- Continuous and automated operation, less manpower requirement
- Precise control of residence time and temperature, uniformity of cooking
- No effluents
- Wide range of ingredients (size and consistencies) can be handled
- Diversity of product's shapes

However, the large number of variables which one has to take into consideration with regard to processing parameters, system parameters and raw material parameters, make it a difficult proposition for optimization of extrusion cooking for developing a desired profile for the product. This thus is a limitation of extrusion cooking technology and it is still a matter of skill and art which is in practice by and large in the field.

1.5. APPLICATIONS

Extrusion cooking technology has almost limitless applications in the processing of cereal-based foods and other materials, and is associated with partial or complete gelatinization of the starch, complex formation, transformations and interactions involving biopolymers. The technique may be used to precook, instantize and agglomerate food components. Its various applications in food have been reviewed by Smith (1976), Harper (1979), Hauck (1980) and Linko et al (1981). Various food applications of extrusion cooking as summerised by Cheftel (1990) are tabulated in Table 1.3.

Table 1.3

Food applications of extrusion cooking

Industrial	R&D
CEREALS	
Snacks	
including non expanded "3rd generation" snacks	
Co-extruded snacks and	Instant noodles
Biscuits	e.g. rice or fish noodles
With fruit, cheese, meat filling	
Flat crisp bread	
Breakfast cereals	
• Non expanded nibs:	
Further flattened into flakes	
or puffed products:	
further agglomerated post extrusion	
• Fibre-rich products	
Cereal bars	
Bread crumbs	
Intermediate products	
(=ingredients for the industry)	• flavour encapsulation on maltodextrins
• Precooked flours, starches	
for dry baby foods	• modified glutens
for instant soups	• starch hydrolysis
and puddings	for maltodextrins
• Chemically modified starches	for fermentation
	substrates (beer)
VEGETABLES, LEGUMINOUS AND OTHER SEEDS	
Potato based snacks	Vegetable purees
+ limitation fries	
Texturized vegetable	Moist baby foods
Proteins	(aseptic)
(meat analogues)	Hydrolyzed vegetable
	Proteins
Instant porridges and weaning foods	Roasting of coffee
	And hops (for beer)
full fat soy flour	Nut spreads
	Spices with low microbial load

Industrial	R&D
ANIMAL FEEDS	
Pet foods *Dry rather than moist pet foods*	Continuous gelation/emulsification of high moisture, protein - rich mixtures • *fish mince* • *debonedmeat* • soy *proteins* • *milk proteins*
Fish foods *For fish faming (salmon ...)* *right consistency and density*	Inactivation of antinutrients; destruction of toxins, microorganisms ; increased digestibility of cellulose
Shrimp foods	Processing of leafy fodder Incorporation of
Processing of soy meals *for ruminants, pigs, poultry*	urea into ruminant feeds
SUGAR AND CONFECTIONARIES	
Decrystalization of sucrose	Preparation of boiled sweets, Preparation of marzipan
Preparation of liquorice	Gelled confectioneries *(fruit gums, marshmallow, caramel)*
Conching of chocolate	Non caloric, non cariogenic confectioneries
	Microencapsulation of flavours Instant
	drinks *(cocoa)*
	Flavour generation *By Maillard - type reactions*
FRUITS	
	Fruit cubes *For muesli or snacks*
	Instant food drinks + *tea granules*
	Fruit purees
FATS & OILS	
Stabilization of rice bran *Inactivation of lipase prior to oil extraction*	Pretreatment of oilseeds *prior to oil extraction*
Extraction of fats from meat by-products *In slaughter-houses*	Processing of mustard seeds
	First steps in the preparation offish oils

Industrial	R&D
DAIRY	
Milk proteins in snacks and TVP	Processed cheeses Cheese analogues String cheese Dairy confectioneries *(caramel)*
Caseinates *from acid casein* *=Ingredients with improved functionality*	Spreads, fat analogues Modifications of functional properties of dairy ingredients • *low microbial load* • *partial proteolysis*
MEAT AND FISH	
Extraction of fats from meat by-products	Preparation of fish -cereal snacks
Preparation offish quenelles	First steps in the preparation offish oils Preparation of surimi and surimi-salt mixes *(at low temperature)* Decontamination of blood-cereal mixes Solubilisation of collagen or keratin Development of meat sauce flavours Texturization of mechanically - deboned meat or fish

(Cheftel, 1990).

1.6. EXTRUSION COOKING OF CEREALS

Considering the experience that humankind has had with cereal grains for centuries, it is not surprising that cereals have become popular and nutritious breakfast entree for people of all ages. Although the protein content of breakfast cereals relative to carbohydrate content is small, cereals still make worthwhile contributions to the protein portion of the diet. This is especially true if the protein is protected from damage during processing or if the quantity and quality of the protein are amplified by formulation. The fats provided by breakfast cereals are primarily unsaturated unless the fat moiety is modified by the addition of saturated fats. The carbohydrates of breakfast cereals are mostly starch, plus sugar, if sugar is added to the formulation. A small but physiologically significant portion of the carbohydrates is dietary fibre. Cereal grains are naturally good sources of vitamins and minerals. However, these are frequently added to breakfast cereals to make the cereals even better sources of nutrients. It is also common to fortify breakfast cereals with nutrients that are not found naturally in cereal grains to enhance the nutritional contribution of the cereal and assure the nutritional status of consumers (Robert and Haines, 1990).

All cereals contain a large proportion of starch. The content and the type of starch, apart from process and system variables, therefore affect the properties of extruded products made from them. In its natural form, starch is present in granular state that is insoluble in water, tasteless, and unsuited for human consumption directly. To make it digestible and acceptable it must be cooked. In case of ready-to-eat cereals the cooking is carried out during manufacturing process.

If the cereal is cooked with excess of water and only moderate heat, as in boiling, the starch gelatinizes and becomes susceptible to starch-dissolving enzymes of the digestive system. If cooked with a minimum quantity of water, or without water, but at high temperature, as in toasting and extrusion cooking, non-enzymatic browning reaction between protein and reducing carbohydrate may occur, and there may be some dextinization of starch.

From the time the extrusion cooking process was introduced for food application, preparation of cereal based and starch based products has been the major use. Initially, the extrusion cooking was used for the production of breakfast cereals as cereal flakes to replace the traditional process of making cereal flakes from maize grits. However, later the extrusion cooking process was used for preparation of various cereal based products, such as expanded, fortified and enriched, shaped cereal, precooked instant cereal, infant, weaning and baby foods (Linko et al, 1981). Extrusion cooked corn-soya-milk blends as protein fortified cereal grain products have also been prepared for nutrition intervention programmes (Peplinski and Pfeiffer, 1970). Simultaneous extrusion cooking of cereals and fish meal, and corn / soyabean based infant cereal has also been reported (Harper, 1980). Production of instant and quick cooking noodles and pasta (Tsao et al, 1976), instant dried soup or gravy bases from modified starches with co-extrusion of cereals with meat protein, herbs, spices and vegetables (Hauck, 1980) has also been reported. Various types of snack products,

causing some breakdown of large particles and rapid heat input to the powder by frictional and mechanical effects.

3. A rapid temperature rise within a short distance (2 to 20mm) which raises the mass temperature sufficiently to melt all the crystalline structures within the starch granules.
4. Release of the softened starch granule from the wedge protein and their deformation by the kneading action of the screws under forces of the compression, elongation and shear. This causes changes in the microscopic appearance of the raw materials as air is excluded from the system and the flour is transformed into a plastic or viscoelastic fluid mass.
5. Under further intensive kneading by the screw elements the starch polymers are dispersed from their native aggregates to form a continuous phase. The loss of starch aggregates causes a sharp reduction in fluid viscosity as indicated by lower die pressures. Within the continuous starch phase the proteins are macerated to small globules or rods (<100 micron) and appear as discontinuities, together with the residual granules and the bran platelets in starch phase.
6. In the final pumping section at the die, the viscoelastic fluids tend to lose all their entrapped air and may be extruded as fluid or expanded foams depending on the exit temperatures. At temperature >100°C, bubbles of steam are nucleated in the fluid and are retained by the continuous starch phase. Little further change occurs to the biopolymer structures during this expansion process.

1.6.1. Effect of raw material and its characteristics

Extrusion is a relatively low moisture process operating in the range of 10 - 40 % moisture, w.b. It employs large mechanical energy and heat inputs in highly compressed powders systems which cause the powders to be transformed into fluids (Guy and Horne, 1988; Colonna et ai, 1989). Therefore, characteristics such as surface friction, hardness and cohesiveness of particles become important and in the high solids concentration of melt fluids which are formed within the screw systems, the presence of other ingredients like plasticizers and lubricants cause significant changes to the system variables of the process (Guy, 1994).

The basic structures of the extruded products are formed by transforming and manipulating natural biopolymers, such as starch and certain types of proteins. Cereals

(1989), while studying the effect of wheat quality using 12 different wheat varieties, have reported that the amount of water soluble proteins in the wheat flour influenced the density and hydration behaviour of final extrudates, whereas, the starch content of the wheat flour directly influenced the cold paste viscosity of the Brabender viscogram.

Expansion ratio is one of the crucial parameters in determining the cereal product quality in case of extruded products. Expansion of cereal product has been reported to decrease with increasing amount of protein (Faubion et al, 1982) or lipid (Mercier et al, 1980) and increase with increasing amount of starch and proportion of amylopectin to amylose in starch. Rusnak et al (1980) and Gomez et al (1988) reported that after extrusion and micronizing waxy sorghum flakes not only expanded more but were also extensively gelatinized, lighter in density with uniform distribution of cells and needed less force to break. The presence of amylose in nonwaxy endosperm restricted starch swelling and in turn its expansion (Akingbala and Rooney, 1987; Gomez et al, 1988; Tester and Morrison, 1990).

1.6.2. Effect of process variables

The studies on extrusion cooking have mostly been performed to determine the effect of processing variables on functional properties. Anderson et al (1969a, b; 1970) and Conway (1971a, b) were among the first to describe process conditions in relation to product behaviour. Changing one variable while keeping the others constant gives not much insight into the interactions among the process variables unless many combinations are examined. A solution to this problem is to use a statistically designed multiple factor experiment for economy of experimental points, together with response surface methodology, which has subsequently been used by many workers for optimization of process variables. The main process variables include feed rate, screw configurations, screw designs, screw speed, die design and barrel temperature. These can be directly controlled by the extruder operator.

Feeding

In case of co-rotating extruders the conveying capacity of the extruder generally exceeds the rate at which the material is fed into it. A stable and consistent introduction of feed stocks into the machine is therefore of prime importance. Raw materials can

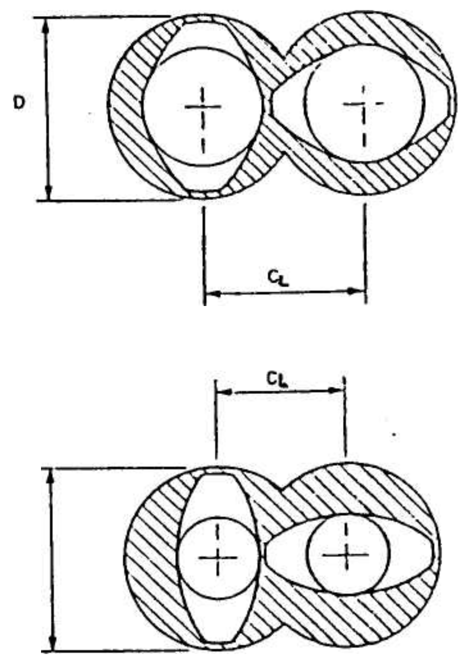


Fig. 1.6. The centre line distance (CL) governs the maximum power transmittable from motor to the shafts and the screw conveying volume. (Frame, 1994).

Screw speed and configuration

Screw speed directly affects the degree of barrel fill, and hence the residence times distribution and the shear stress on the material being extruded. The screw speed is a factor in determining the maximum volumetric output of the extruder and for this reason most extruder manufacturers design machines to run at the maximum speeds mechanically tolerable, usually 400-500 rpm.

The measured torque and die pressure change with screw speed. As most ingredients used in food extrusion are thixotropic or pseudoplastic in nature, there is a linear relationship between speed and torque/pressure. The barrel-fill length decreases with increasing screw speed and die area but increases with feed rate. The normal minimum screw speed range is 70-100 rpm. Below this, the volumetric capacity would be severely limited and would make the majority of food extrusion products costly to manufacture.

The screw configuration in extrusion is a key factor that affects product transformation (Gogoi 1994; Barres et al, 1990; Kirby et al, 1988), residence time distribution (Meuseret al, 1991; Altomare and Ghossi, 1986), product expansion (Sokhey et al, 1994), degree of fill and energy inputs to the materials (Yam et al, 1994; Erdemir et al, 1992; Ollett et al, 1989; and Martelli, 1983). Screw configuration is useful in manipulating resultant attributes of the product.

The location, spacing and number of reverse pitch screw elements (RPSE) and mixing disks (MD) are important during extrusion processing and affect the energy input and residence time. Such process variable influence physico-chemical changes in the material, thereby affecting the product quality. Gogoi et al (1996) studied the effect of RPSE on rice and its blend with fish muscle. Erdemier et al (1992) studied the effect of 18 different screw configuration on specific mechanical energy (SME) subdivided into element mechanical energy (EME) and die mechanical energy (DME) for rice flour. It was observed that changing of element spacing or rotation made little difference in SME values. Kollengode et al (1996) have studied the effect of incorporating 1, 2 or 3 mixing elements during the extrusion of maize, wheat and rice. Shear conversion of corn meal using RPSE (one and two) at low temperature (<60°C) has been shown by Yam et al (1994).

Vergnes et al (1992) studied and computed the residence time and energy distribution in RPSE of twin screw extruder. Their results showed that by modifying the geometrical parameters (length of the RPSE, axial slot width) or the operating conditions (feed rate, screw speed), it is possible to modulate and to control the treatment delivered to the product and to optimize the process in order to achieve a particular degree of transformation.

Gogoi and Yam (1994) studied the relationships between residence time and process variables with three different screw configurations (conveying, mild and severe kneading) on corn meal. They concluded that the most significant process variables affecting mean residence time were screw speed followed by throughput, whereas moisture content, die pressure and die temperature had no significant effect.

The effect of screw configuration on the product properties, die pressure and screw torque in the twin-screw extrusion-cooking of maize grits has been studied by

Kirby et al (1988). Extrudate bulk density, water solubility and water absorption were measured for a set of range of extrusion moisture contents and barrel temperatures for each of four screw configuration of different conveying efficiencies. They concluded that screw configuration is a potent variable in determining product properties. The screw configuration controls the SME input range. Screw configurations of low conveying efficiency give rise to low-viscosity melts, which expand into low-bulk density products. Dead-stopped runs showed that the conveying efficiency controls the degree of fill in the extruder barrel. The mean residence time was increased by decreasing the conveying efficiency of the screws.

Die design

Extrusion die geometry is also important for final product quality. Die design is a complicated art in its own right, and most dies for food products are designed by trial and error (Clark 1978a, b).

van Zuilichem et al (1978) stated that die length-to-diameter does not have a detectable influence on the extrusion of corn grits. The die resists flow, causing back mixing until the extrusion pressure exceeds the die effect. Decreasing the die diameter increased extrusion temperature, degree of expansion, water absorption, and soluble nitrogen of an extruded blend of corn and soybean (Molina et al, 1978). As can be expected, an increase in die diameter decreased starch gelatinization (Chiang, 1975; Chiang and Johnson, 1977). The die shear effect can produce lateral striations when it exceeds the cohesive strength of the product (Rossen and Miller, 1973). Introduction of a discharge die results in additional shear that can be observed by a change in cold paste viscosity (Anderson et al, 1970).

Effect of barrel temperature

Most extruders operate with temperature control and the degree of indirect heating or cooling depends on how the extruder is operated. The pressure differentials and shear stress forces influence reaction rates and generate frictional heat. Barrel heating also generates conductive and convective heat in filled and partially filled zones and the proportion of each heat source depends on the physical and rheological properties of the food, the barrel temperature profile and the available motor power. The motor power usually recorded as torque or amperage is converted into pressure energy, phase transition energy and temperature rise.

When direct expanded products such as ready-to-eat cereals are extruded, the moisture content within the barrel is normally 12-18% depending on the sugar and fat content. The frictional heat generation normally requires the barrel to be cooled with air or water. Extrudate temperatures can reach 180° C. In order to prevent material from burning on the hot barrel surface or inhibit excessive maillard browning or limit the degree of denaturation (of proteins), chilled water can be pumped through a barrel jacket. However, as the material residence time in the extruder is very short, there is little change of significant heat transfer from the bulk of the viscous material.

Reduction in temperature in this case is best achieved by increasing water or oil content (i.e. increasing lubricity) or reducing the degree of shear. This is afforded by reducing the severity of the screw configuration. Various models describing heat transfer have been cited in the literature (Yacu, 1983; 1985).

1.7. APPLICATION OF NUMERICAL MODELS FOR EXTRUSION COOKING

Extrusion cooking technology has been widely used by the food industry since 1960s and is preferred over some conventional methods of processing. However, the physico-chemical changes, molecular rearrangements and interactions among the components of the food system make extrusion cooking a complex food processing operation. Different aspects of low and intermediate extrusion have been addressed by several researchers.

At elevated temperatures biopolymers in foods start loosing their orderly molecular structure. Proteins begin denaturing and starch begins gelatinizing. On initiation of gelatinization and denaturation there is a rapid change in the physical properties of the biopolymer mass due to the formation of new molecular aggregate structures by hydrogen bonding. One of the most significant changes is the rapid rise in the viscosity in the extruder. After the initial rise, viscosity will start to decline as the melt is further heated and mechanically sheared. The initial rise in viscosity is caused by gelatinization of starch resulting in a complete irreversible disappearance of birefringence. Fragmentation and formation of complexes may follow gelatinization in the extruder, depending on the degree of severity of the process, contributing to decrease in the viscosity.

Understanding extruder behaviour and material flow during extrusion cooking is essential to the design of automation and control systems. Some models are adapted from plastic extrusion with modifications that account for the differences of foods from plastics. Plastics can be defined by conventional chemical and physical parameters, as they are highly homogeneous. However, food extrusion utilises a mixture of raw materials in the presence of some water. Modelling is a formidable task in food extrusion for prediction of properties of the melt in the extruder and optimisation of the process. The flow and residence time models may involve highly complicated mass, energy and momentum balances to start with. Response surface methodology is considered a "black-box method" to analyse the influence of variables to PQC (Product quality component) which are dependent variables, within the experimental range.

Several extrusion models for low to intermediate moisture (15 to 40%) starch-based dough exist in the published literature. Harper (1981) developed a model for viscosity as a function of temperature, moisture and shear rate. Cervone and Harper (1978) developed a four-parameter model using temperature, shear rate and moisture content to predict the viscosity of extruded pregelatinized corn flour. Remsen and Clark (1978) included the time-temperature history in their viscosity model along with the shear rate, temperature and moisture content effects. Bhattacharya and Hanna (1986a) established a model with shear rate and moisture effects and tested it with various blends of soya and corn gluten meal.

Wang et al (1990) and Altomare et al (1992) used Harper's model to predict the viscosity changes in wheat flour and rice flour samples at low moisture levels. A model proposed by Morgan et al (1989) incorporates the effect of shear rate, temperature and moisture content as well as time-temperature history and strain history for protein dough. Their model was later tested with various raw materials by Mackey (1989) with some modifications to predict the viscosity of corn starch, potato flour and wheat flour at low to intermediate moisture contents.

Lai and Kokini (1990) modified Harper's viscosity model by including a power dependency to starch degradation in order to successfully predict the changes in viscosity of corn starch. A semi empirical model to predict the viscosity of high-moisture, starch-based system during extrusion was developed by Akdogan et al (1997). Shear rate, temperature, moisture and screw speed was used to describe the viscosity changes. A nonlinear regression analysis was performed to predict the viscosity under experimental conditions. The regression coefficient of the proposed model was 0.93.

1.7.1. General model

Modelling is useful in explaining the influence of extruder geometry or flow rate, scaling up purposes, to predict chemical transformation, the functional properties of the product, and for process control and automation (Linko at el. 1981). Several attempts have been made to apply rheological models for flow rate in the metering section of the screw. Harper (1979) has listed a number of assumptions that are necessary to solve basic flow equations. Tadmore and Klein (1970) developed a simplified model for Q, the

$$Q = G_1 G_3 N F_{dt} \left(1 - \frac{\delta}{H}\right) + \frac{G_2 G_4 F_{pt}}{\mu} \left(\frac{(P_1 - P_2)}{L}\right)(1 + f)$$

Where,

$G_1 \ldots G_4$ = geometric constant for tapered channel

N = Screw RPM

F_{dt}= Total correction factor for drag flow

F_{pt} = Total correction factor for pressure flow

5 = Flight clearance

H = Channel depth of flight height

μ = Viscosity in channel

P_1 = Dough pressure at input end of section under study

P_2 = Dough pressure at output end of section under study

L = Length of the extruder section under study in axial direction

F = correction for pressure flow through flight clearance

This model was proposed for simple single screw plasticating extruder and described the effects of extruder geometry on operational characteristics, assuming laminar, Newtonian, steady, fully developed flow, with no slippage at the barrel wall, incompressibility of the fluid and negligible gravity and internal forces. This model has further been improved to take into account various other processing and thermodynamic aspects for different extruders. Harper (1979), Linko et al (1981), Meuser and van Lengerich (1984), Meuser et al. (1986), Pfaller and Meuser (1988), have reviewed various models and expression for total power input, power dissipation, volume flow rate, velocity profile, viscosity of food material and various other physical and chemical changes in it.

1.7.2. Heuristic and response surface model

Olkku et al (1980a) described the basic changes such as denaturation and plastification in an extrusion cooking process, using a heuristic model in which the product quality indicators are given as functions of certain state profiles. $Y_k = f_k$ (P, T, R, S)

Where, Y_k = Products quality indicator viz., degree of expansion, product colour, water absorption index, etc.

P = Pressure profile

T = Temperature profile

R = Residence time profile

S = Shearing effect profile along the extruder barrel

Roberts and Guy (1986) demonstrated the importance of shear-history dependence of foodstuffs. This is an essential difference between food extrusion and plastic extrusion. An example of Heuristic modeling is the system analytical model used for the extrusion of a starchy material that has been described by Meuser and Van Lengerich (1984).

Another Heuristic sequential model of an extrusion cooker (Linko et al, 1981) deals with the basic transformations that occur during HTST extrusion cooking which are irreversible (Fig.1.7). It depicts the overall effect of extrusion processing on product quality, which is the combined effect of the different functional sections of the extrusion reactor. The response surface methodology (RSM) is **a** statistical method based on regression analysis on quantitative data from appropriate experimental design to construct and simultaneously solve multivariate equation described by the relationship of the dependent variable to product quality characteristics and to process and design parameters (Olkku et al, 1983). The equation can be graphed as response surfaces

the extrusion parameters. The extrusion parameters can be perceived as target parameters. These considerations are described in the Fig. 1.8 (Meuser et al, 1986) which was developed for the flat bread extrusion. This model devised the parameters into three categories; process parameters, system parameters and target parameters. The process parameters are the extrusion variables viz., machine and raw materials.

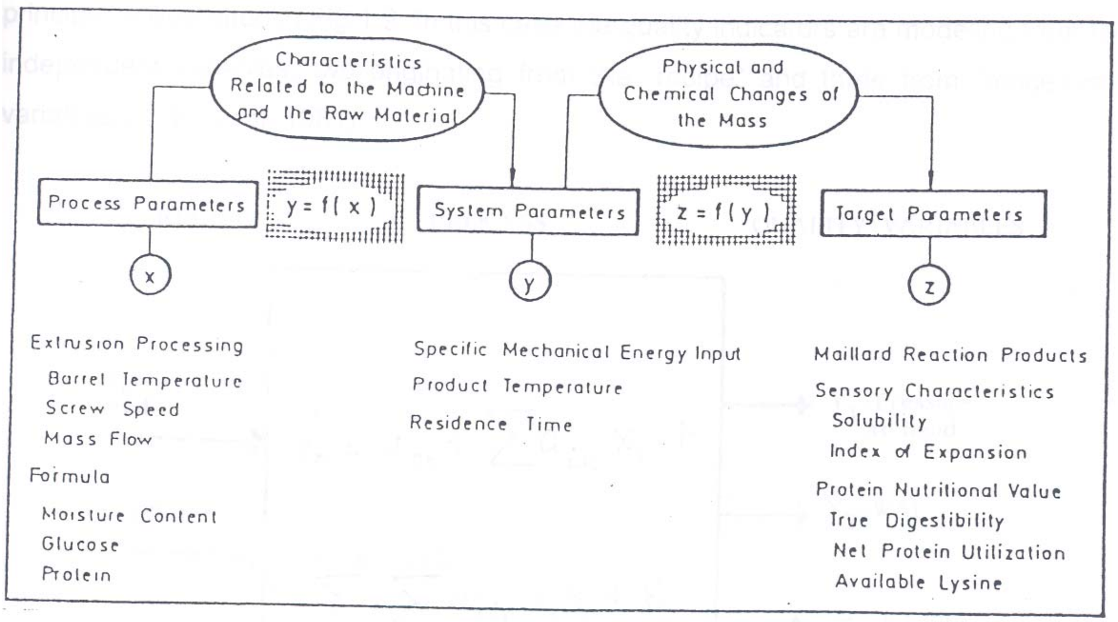


Fig. 1.8. Model used to describe the extrusion cooking process using systems analysis approach. (Meuser et ai, 1986).

The system parameters are the mechanical and the thermal energy inputs into the mass as well as their residence time in the extruder, while the target parameters are the various extrudate characteristics, viz., solubility, expansion and bulk density of the extrudates. Since the energy input also partly determines the reaction behaviour of the mass, a functional relationship exist between process parameters and target parameters as described in a more detailed model (Mauser et al, 1986; Pfaller and Meuser, 1988; Yeh and Jaw, 1998).

1.7.4. Steady-state modelling

This modelling is used to predict chemical reaction, functional properties after processing and state indicators during processing. This model has two apparently different philosophies. One, the empirical black box approach and other, a basic physical approaches. Both have their own merits and demerits.

The black box approach is based on response surface methodology. The principle is illustrated in Fig.1.9. In this case the quality indicators are modelled from five independent variables, two originating from the "recipe" and three from "processing" variables (Linko et al, 1981).

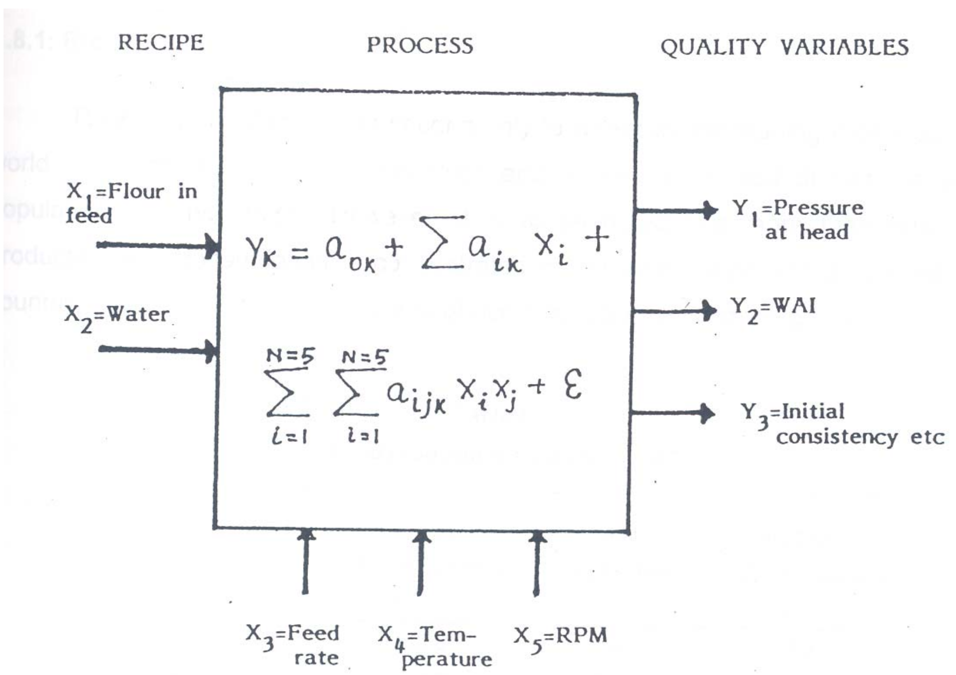


Fig.1.9. Principle of empirical black box response surface modelling (RSM) of quality or state indicators from recipe and processing variables. The equation in the box is a second-order polynomial in sum notation (Olkku, 1981).

This approach is beneficial when little, if anything is known of the real form of the function in the box. A problem, which is also encountered when using polynomial models, is the difficulty that arises when physical and chemical interpretations are needed. However, they can be applied to optimization by linear programming or simulation.

1.7.5. Dynamic modelling

This type of modelling is required for process control and stabilization. The basis here is a steady-state operation point and a given set of process state indicators at that point. For example, if mass pressure and temperature are taken as state indicators in an operation point, the process state point is indicated by screw speed, barrel temperature and feed rate for a given recipe. If any deviation is observed from the desired values of state indicators, the control takes over and changes a process variable so that the desired values of state indicators are reached.

1.8. RICE PRODUCTS AND SCOPE FOR EXTRUSION COOKING OF RICE

1.8.1. Rice

Rice *(Oryza sativa,* L.) is second only to wheat as the leading food crop of the world. It is grown in over 100 countries and is the staple food of half the world's population (Juliano, 1985). However, it is to be noted that more than 90% of its production and consumption is concentrated in the south, east and southeast Asian countries. Table 1.5 shows the spread of rice production around the globe.

Table 1.5 World production of paddy in 1997

Area/Continent	Area (Million hectares)	Yield (kg/hectare)	Production (Million tonnes)
World	149.8	3827	573.3
Asia	134.2	3904	523.9
China	31.3	6331	198.5
India	42.2	2915	123.0
N. America	1.8	5593	10.3
S. America	5.5	3278	18.0
Europe	0.44	6076	2.6
Africa	7.5	2212	16.5
Oceania	0.17	8010	1.3
Australia	0.16	8244	1.3

(FAO, 1997).

The countries contributing most to the world's rice production are China (33%) and India (22%). In India it is the largest produced and consumed staple with an annual production of more than 120 million tonnes of paddy. It is the major supplier of energy, protein and other nutrients in the diet of more than half of the Indian population.

1.8.2. Rice products

Rice is utilised throughout the world mainly for food purposes. It is generally consumed as whole grain in the form of raw milled rice or parboiled milled rice as table rice after boiling/cooking in water. However, a considerable quantity of rice is also converted into many products and marketed in various rice consuming countries. These include flaked rice, expanded rice, popped rice, extruded rice products, breakfast cereals, quick cooking rice, instant rice, ready-to-eat cereal, infants foods, fermented foods and various types of snack foods like rice cakes, granola snacks, rice fries, puddings, crispies, crackers and noodles (Kelly, 1985; Bean and Nishita, 1985; Burns and Gerdes, 1985; Juliano and Sakuria, 1985; Hsieh and Luh, 1991a, b; Wang 1991). But for a few of these products, majority of them do not exist in the Indian market.

Use of rice in processed foods in the Western countries, especially in USA, has shown substantial increase in the recent past. Its use in ready-to-eat cereal has consistently accounted for over half of the processed food use of rice in USA during 1980s. No other product has recorded this large an absolute increase in its consumption (Meyers, 1994). On account of technological advancement, many of the traditional products from rice in the industrialized countries are also being produced at commercial scale particularly in the Fareast and southeast Asian countries.

Extrusion cooking could give products similar to the traditionally prepared expanded or puffed products. Products from wheat and corn have been prepared and many corn-based extruded products are marketed in the western countries. However, due to the compositional differences between the ingredients of the grain, specially with reference to starch and protein, the properties of the rice extrudates are expected to be different from those based on other cereals, like wheat and corn. With more and more food industries opting for twin-screw extruders for better control of products and other technical advantages, it is necessary to have enough data on extrusion of rice, to provide a better knowledge-base for preparation of products with the desired quality parameters.

1.9. SCOPE AND OBJECTIVES OF THE PRESENT WORK

As mentioned earlier, a considerable quantity, (more than 10 million tonnes) of paddy is converted annually into rice products like expanded rice, popped rice and flaked rice in India. These rice products are quite popular as ready-to-eat whole grain products. In addition, they are also used for preparation of various snack foods and breakfast dishes in the Indian subcontinent. These products owe their popularity to the fact that they are reasonably priced, pre-cooked convenience products.

These products are produced manually in batches normally, of 1-2 kg each, in rather unhygienic conditions in the traditional, tiny or cottage-scale processing units, that account for about 90% of the rice products presently produced in India. The quality of products is also not uniform and the processes are not efficient. The main draw back of these products is the presence of a significant quantity of residual sand (in which paddy/rice is roasted/puffed during processing) as contamination.

To overcome these problems, it is desirable that newer and improved technologies be explored to replace them. Extrusion cooking lends it self as an alternate processing technology for this purpose. The present work was therefore undertaken to address this problem. It is envisaged that extrusion cooking could not only provide an alternative to the traditionally practiced inefficient processing for puffed and expanded products, it would also enable use of varieties other than those specifically identified as suitable for production of these products. In other words, it would eliminate the "variety specificity" barrier. Chinnaswammy and Bhattacharya (1983); Murugesan and Bhattacharya (1991) have identified the basis of such varietal specificity for the production of expanded rice and popped rice respectively in the traditional processing technologies for these products.

Extensive research work on the extrusion cooking of cereal, particularly of corn and wheat, has been conducted all over the world, a review of which has already been presented in the preceding pages. However, literature on extrusion cooking of rice is rather scanty. Similarly, there are very few extruded rice products in the market than extruded corn or wheat products. Since the consumption of rice products has increased substantially in the recent years, study of the rice processing is also becoming a priority area for grain researchers. In recent years, a few studies have been reported on the effect of extrusion variables on the product characteristics of extruded rice using a single-screw extruder (Kim and Maga, 1990; Bhattarcharya and Prakash, 1994; Yeh and Jaw, 1999).

Studies using twin-screw extrusion of rice flour are also limited (Bhattacharya and Choudhury, 1994; Pan et al, 1992; Kumagai et al, 1987). Since twin-screw extruders provide better control of product quality and offer other technological advantages over single screw extruder (see review), adoption of twin-screw extruder for production of extruded products is always advisable. It is also necessary to generate sufficient data, for a better comprehension and understanding of rice based systems. However, a large number of variables (extruder hardware variables, feed variables, operating variables etc.) need to be controlled and optimized. This becomes a limitation for extrusion cooking technology for its wider adoption in the food industry.

Extrusion cooking could be considered as a specialization area of food technology because of the complexity of the interactive effects, which are inherent in the system. General predictive modelling is very difficult because ingredients are diverse and can vary considerably. Modelling tends to be product specific. New product development demands carefully planned experimental design, good expertise and orientation.

The present study was therefore undertaken with the following objectives:

a). To screen a large number of extruder and extrusion variables, quantitative determination of the effect of the variables on the system parameters as well as on the target product parameters during extrusion processing of rice flour.

b). To study the effect of the extrusion variables on extrusion system parameters and product attributes, and also to determine the inter-relationships between system parameters and product attributes during extrusion cooking of rice flour in a twin-screw extruder.

c). To study the changes in the macromolecular properties of starch during extrusion cooking of rice flour.

CHAPTER 11

Materials And Methods - General

2.1. Rice flour

A high amylose paddy cultivar, IR 64, was procured from local market. An intermediate amylose paddy cultivar, Pojo bora and a very low amylose (i.e. waxy) variety cultivar, Agoni bora, were procured from Assam Agricultural Research Institute, Titabar, Assam, India. The paddy was cleaned, fumigated and stored in cold room in metallic containers, and used as when required. All these three paddy cultivars were milled to about 8% degree of polish at pilot plant rice mill at CFTRI using rubber roll sheller for dehusking and huller for polishing (debraning). The milled rice was ground to flour in a hammer mill and used for extrusion trials. Analysis for the proximate composition parameters of rice powder from these three varieties were done as per AOAC methods (AOAC, 1984) and the results are shown in Table 2.1.

Table 2.1
Proximate composition of rice flour

Parameter (%)	Cultivar		
	IR64	Pojo bora	Agoni bora
Moisture	11.7 ± 0.2	12.6 ± 0.2	12.6±0.2
Protein [a]	6.7 ± 0.1	7.1±0.1	7.8 ± 0.1
Fat	0.7 ± 0.1	0.7 ± 0.1	0.9 ± 0.1
Ash	0.4 ± 0.1	0.4 ± 0.1	0.4 ± 0.1
Carbohydrate[b] Amylose	80.5	79.2	78.8
Equivalent[C]	28.6	22.3	5.0

[a] N X 5.95

" Calculated by difference

[c]Also generally called "total amylose" or "amylose" content (Reddy et al, 1993).

2.2. Extruder

A co-rotating, fully intermeshing, twin-screw extruder (Werner and Pfleiderer, Model ZSK 30, New Jersey, USA) with screw diameter of 30.8mm (shown in Fig 2.1), was employed with or without using a die. The length-to-diameter (L/D) ratio of the extruder was 30.8: 1. The extruder screw consisted of forward pitch screw elements, reverse pitch screw elements (generally near the outlet) and also kneading blocks. The actual screw configurations used in extrusion trial are described in successive chapters. The extruder was provided with a digital display for torque (T%) developed during extrusion. The feeding was maintained at desired feed rate using a volumetric twin-screw feeder. The feeder had co-rotating twin-screws and was developed at the Institute to convey granular or powdery materials.

Fig. 2.1. Twin-screw extruder (Werner & Pfleiderer).

2.3. Preparation of feed for extrusion

Rice flour was ground to pass through **a** British Standard (BS) Sieve of desired mesh size. Water was added by sprinkling to adjust the required moisture content and mixed thoroughly in a Sigma mixer. Care was taken to avoid lumping. The flour was packed in double-walled polyethylene bags and allowed to equilibrate overnight at 4-S°C in a refrigerator. Before extrusion, the feed was allowed to come to ambient temperature (2S-30°C) and was remixed for 3 min after checking its moisture content.

Standard potato amylose (100 mg, d.b.) was taken in a 100ml conical flask and wetted with distilled alcohol (1 ml). Alkali (10 ml, *1N* NaOH) was added gently and the contents mixed. The solution was boiled for 3 min in a water bath and cooled. About 50 ml of distilled water and acid (7.5 ml, 1N HCl) were added to the solution to partially neutralise it and the volume was made upto 100 ml in a volumetric flask. This solution could be stored in a refrigerator upto 10 days.

To 1 ml of the above standard solution, 50 ml of distilled water, 1 ml acetic acid (1N) and 2 ml iodine solution were added and the volume made upto 100 ml. The colour developed was read against a blank at 630 nm in a spectrophotometer. The amylose content of sample (%, d.b.) was calculated as follows :

$$\text{Amylose content (\%, d.b.)} = \frac{\text{O.D. of rice flour dispersion}}{\text{O.D. of standard amylose solution}} \quad \frac{\text{Weight of standard amylose (mg)}}{\text{Weight of rice flour (mg)}} \quad \times \; 100$$

Materials and methodologies specific to the objectives of the different parts of the studies are described in the respective chapters oriented for the purpose in the following presentation.

CHAPTER III

Screening Of Variables For Extrusion Cooking Of Rice Flour Employing Plackett-Burman Design

3.1. INTRODUCTION

The technology of extrusion cooking of foods has been successfully applied to produce a variety of foods and specialty ingredients during the last two decades. However, a practical problem which a researcher faces in the beginning of the study is the existence of a large number of variables. This makes the situation complex if the researcher is interested to know the quantitative effect of these variables on target parameters. Hence, the researcher tries to reduce the number of extrusion trials, mostly based on previous experience, by reducing the number of variables, may be by conducting some preliminary trials - basically the screening experiments. Till now, it is still an art to select a particular variable (or delete one). The basis of such act is still experience, or sometimes, just a guess.

However, most of the development of extrusion process applications is based on empirical studies, at least in the beginning. The easiest and the most commonly used approach practiced for long is employing response surface methodology (RSM) (Stanley et al, 1972; Bhattacharya and Prakash, 1994) though handling of a large number of variables (e.g., more than 8 or 10) is extremely difficult. These results are product and machine specific, and the conclusion is limited within the scope of the investigation (Lue et al, 1994). On the other hand, a system analytical model has also been applied (Meuser, 1987). This model distinguishes between process parameters (screw speed, barrel temperature, feed rate, moisture content of the feed, etc) and system parameters (specific mechanical energy, die pressure, residence time, etc) as well as influenced target parameters (expansion ratio, water solubility index, water absorption index, bulk density etc). The other approaches include application of artificial neural networking (ANN) (Linko et al, 1992) and dimensional analysis (Bhattacharya and Hanna, 1986b). But the problem of a large number of variables comprising of extruder hardware variables, ingredient variables and extrusion operation variables is yet to be solved. It is logical to have a screening experiment (at the beginning of the trials) that will allow having a feel of the quantitative effects of the variables, usually large in number.

Therefore, scope exists to have a systematic processing of the large number of variables to reduce them to a sizable number say, four or five. The experimental design model of Plackett-Burman can serve as an efficient tool though in food system, its application is rather rare. However, Arnoldsson and Kaufman (1994); Chan and Kavanagh (1992) have reported some applications though not in extrusion technology.

A restriction of die is usually used during extrusion of foods for shaping the final product. However, if the targeted product is a modified ingredient (such as, modified starch/cereal) for developing other food products (e.g., baby food or weaning food), the use of die becomes optional. Further, energy expenditure can be reduced if die is omitted. A separate study on this aspect has been carried out and presented in chapter IV and V.

The objectives of present work was thus the application of the Plackett-Burman experimental design technique to screen large number of extruder and extrusion variables (10 in the present case), and quantitative determination of the effect of these variables on the system parameters and target product parameters without using any die during extrusion processing of rice flour. The variables included extruder hardware variables, feed variables and extrusion operation variables. The system parameters and the target product parameters were also studied.

3.2. EXPERIMENTAL

3.2.1. Rice

A low-amylose (Agoni bora) and a high amylose (IR 64) varieties were used. They were milled, and ground to flour in a hammer mill as described under Section 2.1. The proximate composition of rice powders and total amylose content (Table 2.1) have been reported under Section 2.1.

The feed powder was sieved by using a 355 /lm aperture (45BS) sieve to categorise into two fractions viz., a coarse (retained on the sieve), and a fine (that passed through the same sieve). The geometric average diameter of the particle in rice flour, as determined by sieve analysis (Farrall, 1976) was 438 and 257/lm respectively for coarse and fine fractions. The geometric average particle diameter (dg_a) of rice flour was calculated employing Eq (1):

$$\log(d_{ga}) = \frac{\sum[W_i * \log(d_g)]}{\sum W_i} \qquad (1)$$

3.2.2. Extruder and extrusion cooking

Details of the extruder have been described under Section 2.2. The extruder screw used for the present study consisted of forward pitch screw elements (of varying pitches), and a reverse pitch screw element (near the outlet), and also, three kneading blocks (Table 3.1) for the screw configuration with mixing disks (MD) and reverse pitch screw elements (RPSE) (yes-yes, according to experimental design, discussed later). For other configurations, these MD and RPSE were replaced with 20/20 forward pitch screw elements.

Table 3.1

Screw profile used for extrusion trials with mixing disks and reverse pitch screw element

Screw element details (Pitch/Length of screw)	Total length (mm)
42/42	268
28/28	336
28/14	14
KB 45/5/14*	14
20/20	80
KB 45/5/14*	14
20/20	80
KB 45/5/14*	14
20/20	100
-20/10**	10
20/20	20

* Mixing disks
** Reverse pitch screw element (negative pitch)

According to the experimental design (discussed later), the temperature of the extruder barrel was maintained at 100° or 140°C throughout the barrel; the screw speed of the extruder was maintained at 200 or 400 rpm, while the feed rate was maintained at 10 or 20 kgh^{-1} using a volumetric gravity feeder. The moisture content of the feed was either12.0:t0.1 or 20:t0.1% (d.b.). Extrusion trials were repeated twice.

After extrusion, the extrudate was dried in **a** tray drier at 40°C for 2 h to 6.1-6.4% (d.b.) moisture content, ground in a laboratory grinder to pass through 180μm aperture sieve and used for the determination of various quality characteristics, such as, Water solubility index (WSI), Water absorption index (WAI), Bulk density (BO) and pasting profile indices viz. Peak viscosity (PV), Hot paste viscosity (HPV) and Cold paste viscosity (CPV).

3.2.3. Plackett-Burman experimental design for extrusion variables

An experimental design, based on Plackett-Burman theory (Akhnazarova and Kafarov, 1982), was employed (Table 3.2) to study the effect of a total of 10 variables (at two levels each) on 9 response functions. The total number of experiments was 12. The variables included extruder hardware variables (MO and RPSE), feed variables (moisture, sugar and salt contents, and particle size of the feed), and extrusion operation variables (barrel temperature, feed rate and screw speed). The discrete variables, MO and RPSE, were assigned a code of +1 when they were used in the screw profile, or -1 when they were absent. The experimental design, in actual level of variables, is shown in Table 3.3.

The response functions were the extrusion characteristics, such as, torque (T), net specific mechanical energy (SME), average residence time (RT»), the product attributes, such as, water solubility index (WSI), water absorption index (WAI), bulk density (BO), and the viscogram indices such as, peak viscosity (PV), hot paste viscosity (HPV) and cold paste viscosity (CPV). All the variables were used at two levels each denoted by coded levels of -1 and +1.

Table 3.2

Plackett-Burman experimental design in coded level of variables

Expt No.	X_1	X_2	X_3	X_4	X_5	X_6	X_7	X_8	X_9	X_{10}
	Temp	SS	FR	MC	Amy	Par Size	Salt	Sugar	RPSE	MD
1	1	-1	1	-1	-1	-1	1	1	1	1
2	1	1	-1	1	-1	-1	-1	1	1	1
3	-1	1	1	-1	1	-1	-1	-1	1	1
4	1	-1	1	1	-1	1	-1	-1	-1	1
5	1	1	-1	1	1	-1	1	1	1	1
6	1	1	1	-1	1	1	-1	1	-1	-1
7	-1	1	1	1	-1	1	1	1	1	1
8	-1	-1	1	1	1	-1	1	1	1	1
9	-1	-1	-1	1	1	1	-1	1	1	-1
10	1	-1	-1	-1	1	1	1	1	1	1
11	-1	1	-1	-1	-1	1	1	1	1	1

Symbols:

Temp:	Barrel temperature
SS:	Screw speed
FR:	Feed rate
MC:	Moisture content
Amy:	Amylose content
Par size:	Particle size
Salt:	Salt content in feed
Sugar:	Sugar content in feed
RPSE:	Reverse pitch screw element
MD:	Mixing disk

Table 3.3

Experimental design in actual level of variables

Expt No	X_1	X_2	X_3	X_4	X_5	X_6	X_7	X_8	X_9	X_{10}
	Temp	SS	FR	MC	Amy	Par Size	Salt	Sugar	RPSE	MD
1	140	200	20	12	5	257	2	10	YES	NO
2	140	400	10	20	5	257	0	10	YES	YES
3	100	400	20	12	28.6	257	0	0	YES	YES
4	140	200	20	20	5	438	0	0	NO	YES
5	140	400	10	20	28.6	257	2	0	NO	NO
6	140	400	20	12	28.6	438	0	10	NO	NO
7	100	400	20	20	5	438	2	0	YES	NO
8	100	200	20	20	28.6	257	2	10	NO	YES
9	100	200	10	20	28.6	438	0	10	YES	NO
10	140	200	10	12	28.6	438	2	0	YES	YES
11	100	400	10	12	5	438	2	10	NO	YES
12	100	200	10	12	5	257	0	0	NO	NO

3.2.4. Torque (T) and net specific mechanical energy (SME)

The torque (T%) developed during extrusion has been mentioned under Section 2.4.2. The total specific mechanical energy (SME) input during extrusion has also been mentioned under Section 2.4.3.The energy for rotating the screws without feed was subtracted from the total SME with feed to obtain net SME values for extrusion.

Average residence time (RT)

The average residence time of the feed in the extruder was determined using phycocyanin, a natural blue dye. The extrudates were collected at regular intervals of 3 seconds, and the samples, after immediate cooling, were determined for their colour, measured using a UV-visible spectrophotometer, (Shimadzu Corporation, Japan, model

2100) with a reflectance attachment. As the dye comes out over a considerable period of time, a plot of time *vs* colour content was constructed from which RT was determined corresponding to the highest colour value. The b"-values, indicating the blueness of the sample with negative values, were used to know the content of the blue dye in the extrudates. The reported values are the mean of five determinations.

Water solubility index (WSI); Water absorption index (WAI) and Bulk density (BO)

The above parameters were analyzed by procedures as reported under Section 2.4.4 and 2.4.5. Mean of three replicates is reported.

3.2.5. Sample preparation for pasting study and viscography

The dried and ground (to pass through a 80 mesh BS sieve) extrudate (about 6% moisture content d.b.,) and unextruded samples were exposed in Petri dishes inside a desiccator, maintained at 25°C and 65% relative humidity (maintained by a saturated solution of $NaNO_2$), for two weeks to equilibrate moisture such that the moisture contents of the product was in the range of 12 and 13% (d.b.). The viscographic profile was obtained using a Rapid Visco-Analyser (RVA, Newport Scientific, Narabeen, Australia) as per the procedure of the manufacturer's instructions using set standard 2 programme. Three gram sample (14% moisture basis) was mixed with 25 ml distilled water and transferred quantitatively to the aluminium sample holder can (Walker et ai, 1988; Deffenbaugh and Walker, 1990). The slurry was cooked through a definite heating/cooling regime. Initially, it was held at 50°C for 3 minutes, and then heated at the rate of 6°C/min from 50°C to 95°C, held at 95°C for 5 min, and finally cooled at the rate of 6°C/min to 50°C, and held for 1 min.

The parameters that were obtained from the viscogram included:

a) The peak viscosity (PV), i.e., the highest viscosity attained by the paste during the heating phase

b) Hot paste viscosity (HPV), i.e., the viscosity of the paste at the end of the heating phase at 95°C

c) Cold paste viscosity (CPV), i.e., the viscosity of the paste at the end of the cooling phase at 50°C. All viscosity values reported are the average of triplicate measurements and are reported in rapid viscograph units (RVU).

3.2.6. Statistical analysis

The experimental results (response function, y) were fitted to first order multiple regression equations (Eq 2) using coded level (-1 or +1) of the variable (x_I).

$$y = b_0 + \sum_{i=1}^{k} b_i x_i + \varepsilon \quad (2)$$

The coefficients of the polynomial are represented by b_0 (constant term), b_1 b_2...." b_k (linear effect) and s (random error). The coefficients of the polynomial were obtained by using Gauss-Jordan method (Jain et al, 1995). The multiple correlation coefficient (r), determined to know the extent of fitting of the regression model, was judged at a probability (p) level of 0.01. These developed regression equations were used to produce the plots for the response functions.

3.3. RESULTS AND DISCUSSION

The experimental results for the response functions (torque, net specific mechanical energy, average residence time, water solubility index, water absorption index, bulk density, peak viscosity, hot paste viscosity and cold paste viscosity) are presented in Table 3.4. The coefficients of the first order multiple regression equations (in coded level of variables) are shown in Table 3.5. Sample response surfaces (Fig. 3.1 - 3.9), based on the effects of important variables (in actual level of variables) are cited to aid in visualisation of their effects.

The magnitude of the multiple correlation coefficients (usually above 0.93, $p \sim 0.01$) of the first order equations (Eq 2) show that these equations can adequately predict the response functions in relation to the variables. In addition, based on the sign (+ or -) of the coefficients in coded level of variables (Table 3.5), the effect of the individual variables can be categorised as a positive or negative effect. Furthermore, the magnitude of these coefficients indicates the relative effects of the individual variables on the response functions. Hence, variables that impart strong effects (judged by comparing the magnitudes) can be identified to perform the second stage of experiments; thus, the number of variables can be reduced. These indicate that Plackett-Burman design can be employed for screening of a large number of variables in the field of extrusion technology to reduce the number of experiments. The effect of variables studied on the response function is discussed in the subsequent sections.

3.3.1. Torque (T)

The torque during extrusion ranged between 11.4 and 74.5%, of which low values (≤ 25%) were obtained when both reverse pitch screw element (RPSE) and mixing disk (MD) were absent (Table 3.4). Among the individual variables, the effect of MD was maximum followed by screw speed, feed rate and RPSE (Table 3.5). The presence of MD and RPSE markedly increased the torque. On the other hand, an increase in the screw speed reduced the torque, but feed rate had an opposite effect. The sample response surface (Fig. 3.1) for torque was drawn as a function of feed rate and screw speed with or without mixing disk (remaining variables were constant at a coded level of zero except for RPSE which was equal to -1, i.e., without using reverse pitch screw element). It shows that torque increases with increase in feed rate having mixing disk in the screw profile but decreases with an increase in screw speed.

It could therefore be seen that the use of RPSE/MD increases the residence time and also the shearing forces. Hence, the food material inside the extruder offers more resistance leading to an increase in torque. An increase in screw speed or a decrease in feed rate reduces the degree of fill in the extruder causing torque to be at low level. The torque during extrusion usually decides the capacity of the extruder. The torque (T) applied to the screw' s shaft is given by Martelli (1983):

$$T = Z_t/n \qquad (3)$$

Where the total power (Z_t) is given by:

$$Z_t = N(Z+Z_c+Z_s+Z_w)+ Z_P \qquad (4)$$

Table 3.4

Experimental values of the response functions

Expt No	Torque (%)	SME (kJ kg-[1])	WSI (%)	WAI (g/g)	BO (kg m^{-3})	PV (RVU)	HPV (RVU)	CPV (RVU)	RT (s)
1	53.4	328.2	55.6	1.8	302.3	9	10	13	28.5
2	48.2	1083.5	65.0	2.0	395.7	5	4	5	29.3
3	51.9	599.7	15.2	6.7	230.3	38	22	28	25.2
4	74.5	495.0	55.6	2.5	288.3	10	9	12	21.0
5	16.5	79.2	3.4	2.5	730.0	182	90	216	17.8
6	16.6	41.2	8.2	2.5	709.5	174	96	192	15.4
7	39.0	396.0	32.4	5.1	618.7	18	12	18	24.6
8	65.9	426.8	20.2	5.0	489.3	42	12	29	19.6
9	29.3	274.0	13.7	4.9	710.7	74	19	44	24.8
10	59.0	744.5	20.8	5.7	170.0	30	16	22	32.9
11	18.8	153.0	14.4	2.4	594.0	150	90	146	21.7
12	11.4	9.1	12.2	2.6	720.3	158	95	158	16.6
IR-64	-	-	2.3	2.6	-	211	102	227	-
Agoni bora	-	-	7.6	2.7	-	196	86	124	-

As no die was used during extrusion, Z_p equals zero due to absence of pressure build-up at die. Now, an increase in screw speed (n) reduces torque (Eq 3) provided Z_t remains constant. Increasing screw speed reduces the number of filled length (N) of the screw but increases the power expenditure for shearing (Z, Zc, Z_s and Z_w). Therefore, an increase in screw speed results in a drastic decline in N/n values (due to an increase in "n" compounded by a fall in the length of the filled channels) which is possibly much higher than the effect of power (Z) terms, and hence, the torque decreases. Incorporation of the RPSE in the screw profile reduces the conveying efficiency of the feed with simultaneous increase in N and torque (Kirby et al, 1988). Besides, MD is likely to increase other power terms, particularly Z_s. The net result is an increase in torque. An increase in feed rate increases the number of filled channel (N) and consequently, the torque increases. A low amylose feed is generally sticky in nature and usually it tends to bind with the screw and barrel surfaces. Therefore, it is not likely to affect N but increases Z, Zc, Z_s and Z_w - leading to an increase in torque during extrusion. As stickiness of a dough depends on the moisture content (Bhattacharya and Narasimha, 1997; Noguchi et al, 1976), it is expected that a minimum moisture level is necessary for the dough to show stickiness. At a low moisture content (12%), the feed does not behave as a sticky one, and hence, the torque is low, whereas, with high moisture content (20%) torque values are high.

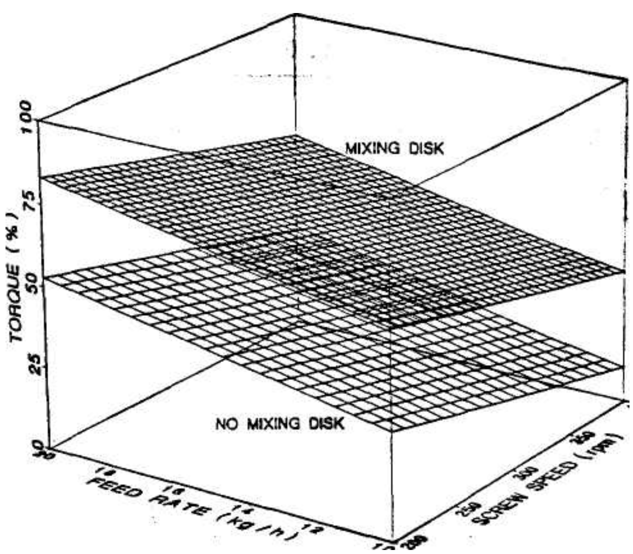


Fig. 3.1. Sample response surfaces for torque during extrusion of rice flour as a function of feed rate and screw speed with or without mixing disk.

Table 3.5

Coefficients of the regression equations for the response functions

Coefficient	Torque	SME	RT	BD	WSI	WAI	PV	HPV	CPV
b_0	38.07	236.20	21.29	475.67	26.28	3.43	77.17	40.00	98.74
b_1	2.10	-67.56	-0.68	-41.67	8.48	-0.63	-8.83	-2.50	27.41
b_2	-10.88	-147.91	-2.58	29.17	-3.38	-0.30	23.33	13.17	52.94
b_3	10.14	-76.50	-0.74	-77.50	4.73	0.10	-22.67	-12.33	-20.99
b_4	4.71	127.36	-0.46	19.83	5.23	-0.13	-16.00	-14.83	-21.16
b_5	-2.79	-171.39	-2.30	32.33	-12.72	1.10	12.83	2.50	39.58
b_6	-0.33	-85.38	0.51	-2.00	-2.32	0.02	4.83	1.17	-0.22
b_7	1.52	51.30	1.09	-34.67	-2.02	-0.08	0.68	-0.83	-4.85
b_8	-1.29	-65.90	0.15	59.17	3.20	-0.38	-1.50	-1.50	0.85
b_9	8.84	347.15	6.36	-114.33	7.30	0.55	-42.17	-25.33	-78.62
b_{10}	15.18	371.88	3.79	-114.67	5.57	0.60	-31.33	-14.50	-61.59
r**	0.982	0.721	0.918	0.938	0.999	0.932	0.989	0.999	0.953

** Significant at p ~ 0.01

Variables associated with:

b_1: Barrel temperature

b_2 Screw speed

b_3 Feed rate

b_4: Moisture content

b_5: Amylose content

b_6: Average particle size

b_7: Salt content in feed

b_8: Sugar content in feed

b_9: Reverse pitch screw element

b_{10}: Mixing disk

3.3.2. Net specific mechanical energy (SME)

The high values (> 300 kJ kg^{-1}) for net specific mechanical energy (SME) during extrusion of rice flour were observed when the screw profile included RPSE and/or MD. Table 3.5 shows that both MD and RPSE exerted the maximum effect on SME. Among the other variables, amylose content and screw speed imparted marked negative effect. The response surface (Fig. 3.2) shows high SME values in the presence of MD. Increase in screw speed or amylose content reduced the SME. Further, the effect of moisture content was positive (Table

3.5) followed by the effect of feed rate (negative) and temperature (negative).

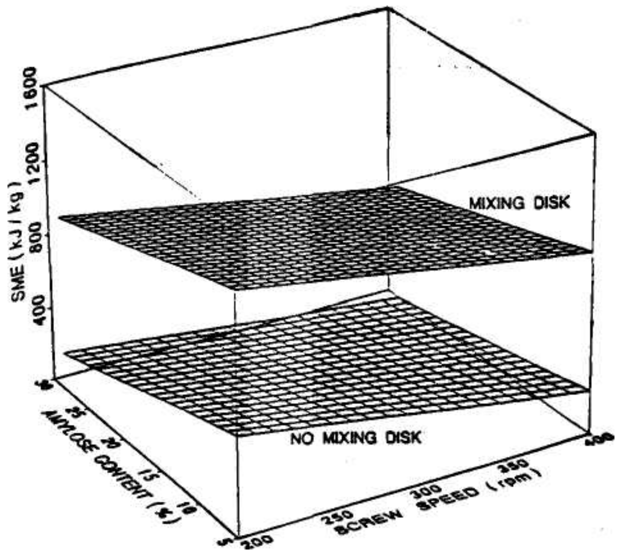


Fig. 3.2. Response surfaces for net specific mechanical energy (SME) during extrusion of rice flour at different amylose content of the feed and screw speed in presence or absence of mixing disk.

The variation of SME during extrusion can be explained by considering the effect of the variables (Eq 5) for power expenditure (Martelli, 1983).

$$Z_t = f\left(\frac{Q^2 \mu}{K_f}, \overline{\mu}, N, n^2\right) \qquad (5)$$

In the present experiment, the effect of Kf is omitted as die was absent and hence the equation reduces to:

$$Z_t = f\left(Q^2 \mu, \overline{\mu}, N, n^2\right) \qquad (6)$$

It appears from Eq 6 that when screw speed (n) is increased (by keeping the feed rate constant), $Z_t \propto n^2$, and $Z_t \propto N$. This follows that an increase in screw speed quadratically increases Z_t but at the same time the number of filled channel (N) decreases; a decrease in N proportionately reduces Z_t. Possibly they compensate each other. On the other hand, u. and p are also proportional to Z_t (Eq 6). As the extrusion feed are pseudoplastic dough (Harper et al, 1971; Levine, 1983; Bhattacharya et al, 1992), an increase in screw speed increases shear rate and subsequently decreases u, and jl. The net result is a decrease in Z_t and SME values with increase in screw speed.

3.3.3. Average residence time (RT)

The average residence time of material in the barrel varied between 15.4 and 32.9s (Table 3.4). The presence of RPSE and MD showed maximum influence on the RT. Screw speed, amylose content and feed rate also exerted a marked effect on RT in that order. The use of RPSE and/ or MD markedly increased the RT which confirms the findings of Yam et al (1994); Gogoi et al (1996); Altomare and Ghossi (1986). The effects of screw speed, amylose content (Fig. 3.3) and feed rate were negative (Table 3.5).

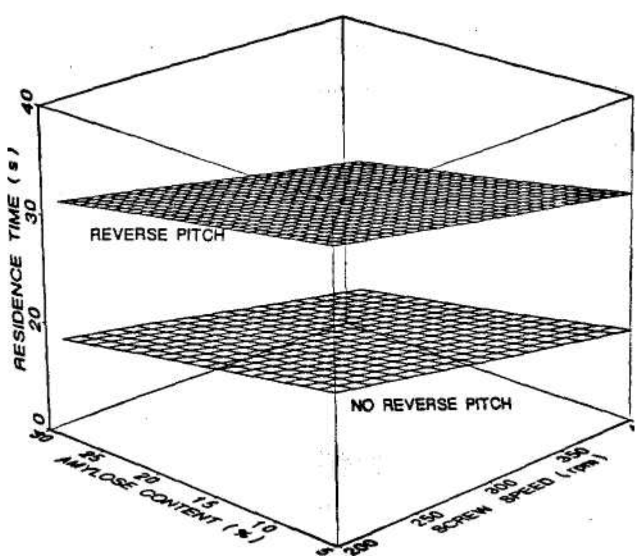


Fig. 3.3. Average residence time of the feed inside the extruder as a function of amylose Content and screw speed when reverse pitch screw element was employed.

The presence of RPSE and/or MD markedly affect RT values as these screw elements restrict or even oppose (specially in case of RPSE) the forward flow of the material. These elements enhance efficient transfer of thermal energy causing product transformation (Colonna et al, 1983) leading to expansion (Lee and McCarthy 1996) at discharge. Increasing the screw speed or feed rate reduces the RT values of which the former has more effect. Yeh et al (1992) found similar results but reported more effect for feed rate than screw speed for extrusion of wheat flour, and indicated that it was due to approaching the effect of a plug flow.

An increase in the amylose content of the feed decreases stickiness (Bhattacharya et ai, 1982), and hence, improves its flowability inside the extruder which in turn reduces the RT values.

3.3.4. Water solubility index (WSI) and water absorption index (WAI)

The raw rice (non-extruded) from high amylose variety (IR 64) had a low WSI (about 2%), whereas, that from the low amylose variety (Agoni bora) was high (7.5%). The WSI of rice increased upon extrusion cooking. Further increase was less (Table 3.4) from high amylose variety (WSI between 3.4 to 20.8%) whereas, that from low amylose variety showed a higher range (between 12.2 to 65.0 %). The coefficients of the regression equations (Table 3.5) shows the temperature of extrusion had the highest (positive) effect on WSI indicating that an increase in barrel temperature increases WSI markedly. Similar effects of amylose content and temperature on WSI of rice have been reported by earlier workers too (Mercier and Feillet, 1975). Amylose content of the feed exerted a negative effect, which indicated that the use of high amylose variety yields low WSI. RPSE had the next higher (positive) effect, i.e., incorporation of RPSE in the screw profile enhanced WSI of the extruded products.

The representative response surface (Fig. 3.4) shows the effect of barrel temperature and amylose content of the feed on WSI of extrudates with two screw profiles, i.e., with or without the use of RPSE. This figure indicates that WSI values for products are distinctly higher when RPSE was used in the screw profile. Similar results were also observed for rice-fish mince blend (Gogoi et al, 1996). The other important variables were MD and moisture content, which gave positive effects.

The extrusion of non-waxy high amylose rice flour results in low WSI (Pan et al, 1992; Gomez and Aguilera, 1983) as high-amylose rice takes long time for gelatinization. A high value of WSI is desirable in cooked extruded products, such as, ready-to-eat snacks, breakfast cereal and porridge. A combination of high level of barrel temperature (140OC), moisture content of the feed (20%) in the presence of RPSE and MD using a feed with low amylose (5%) is found suitable to achieve high WSI values. Use of RPSE and MD increases the severity of the mechanical degradation (Yam et al, 1994). Hence, in a shear field, aided by enhanced mixing, large starch molecules (amylopectin, a highly branched high molecular weight structure) degrade to a large number of small size molecules. This process is further aided by the thermal energy input caused by increased barrel temperature leading to an increase in solubility.

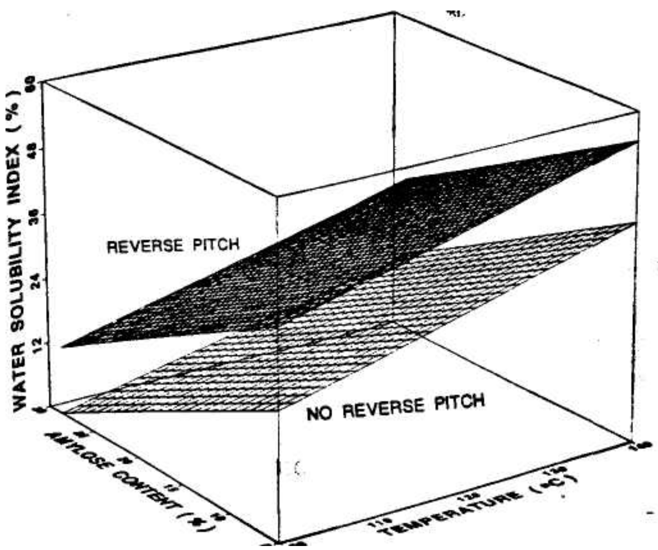


Fig. 3.4. Water solubility index (WSI) of the product obtained by extrusion at different barrel temperature and amylose content with or without reverse pitch screw element.

Amylose content exerted maximum (positive) effect on WAI (Table 3.5). The effect of amylose content on WAI was reported to be positive for rice flour extrudates (de Mosqueda et al, 1986). The effect of temperature, MD and RPSE also showed positive effects. The response surface (Fig. 3.5) showed that the extrudates showed higher WAI when MD was present in the screw profile. An increase in sugar content also reduced the WAI values.

The negative effect of sugar on WAI of extruded rice samples can be explained by starch-sugar complex formation and by a decrease of available water for starch to gelatinise as sugar readily absorbs water. The formation of such complex is a temperature dependent phenomenon, and therefore, an elevation in the same reduces WAI.

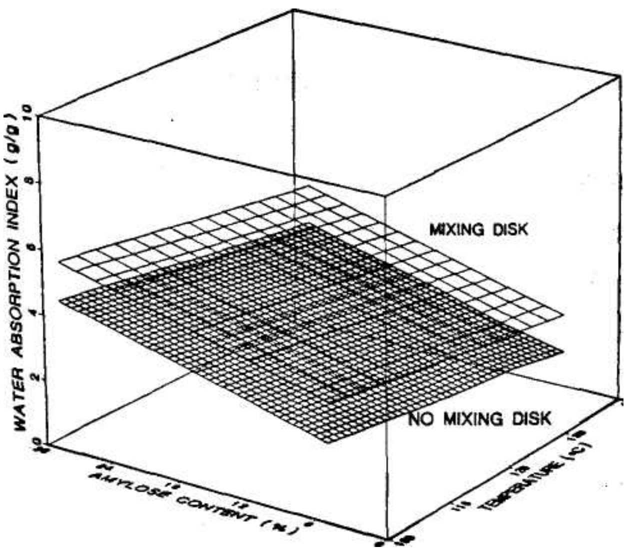


Fig. 3.5. Water absorption index (WAI) of the extruded products obtained by extrusion trials at different barrel temperature and amylose content with or without mixing disk.

3.3.5. Bulk density (BO)

The bulk density of the extruded products varied widely (170-730 kg m^{-3}), (Table 3.4). Lowest BD values were obtained when the screw profiles comprised of RPSE and/or MD (Table 3.5). The negative effect of RPSE/MD indicated that their presence markedly decreased bulk density. The feed rate also had a negative effect, whereas sugar exerted a positive effect (Fig. 3.6). The variables that ranked next in exerting effect on SO were salt (-ve effect), amylose (+ve effect) and screw speed (+ve) effect.

The use of RPSE and MD in the screw profile increases the average residence time (Table 3.4), and consequently, the shearing forces inside the extruder increase. Increase in mechanical and thermal energy (when screw speed and barrel temperature are increased) inputs thus leads to lowering of the product density due to enhancement of gelatinization. Gogoi et al. (1996) have mentioned that extrudates for a screw configuration without RPSE always had higher bulk densities compared to those produced with it; as severity of the screw configuration was enhanced, the product density decreased. During extrusion of rice meal and wheat starch, Lee and McKarthy (1996); Hoseney et al (1992) respectively found that an increase in screw speed increased product expansion (and hence bulk density decreased). Increasing feed rate increases the degree of fill in the extruder (Altomare and Ghossi, 1986) and the tendency towards plug flow increases (Yeh et al, 1992). Hence, heat transfer from the barrel to the material becomes efficient leading to enhanced gelatinization, and consequently the bulk density decreases. The use of **salt** possibly also improves gelatinization as it increases the thermal conductivity of the feed and aids in efficient heat transfer. Sugar in the feed enhances caramelization and form complexes with starch leading to a decrease in gelatinization, and therefore, BD increases.

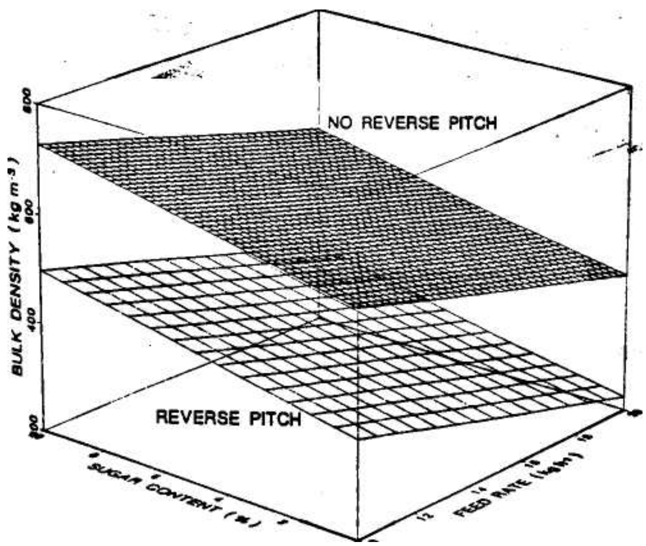


Fig. 3.6. Bulk density of the extrudates obtained at different feed rate and sugar content when the reverse pitch screw element was provided in the screw profile.

3.3.6. Viscographic profile

The different viscosity indices, viz., peak viscosity (PV), hot paste viscosity (HPV) and cold paste viscosity (CPV) of the extruded products, as read from the RVA viscogram, are shown in Table 3.4 along with those of non-extruded rice samples. The variables that affected these viscosity parameters most were RPSE, MO (both have negative effect), and screw speed (positive effect) (Table 3.5). Further, the PV and HPV were affected negatively by feed rate and moisture content of the feed respectively, while CPV was affected (positively) by amylose content. Figures 3.7, 3.8 and 3.9 show the response surfaces for PV, HPV and CPV, respectively with or without the use of RPSE. Incorporation of RPSE in the screw profile markedly reduced these viscosity parameters.

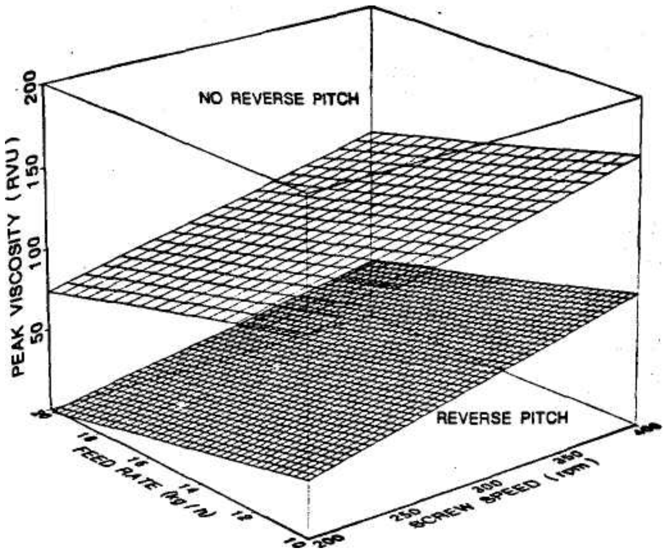
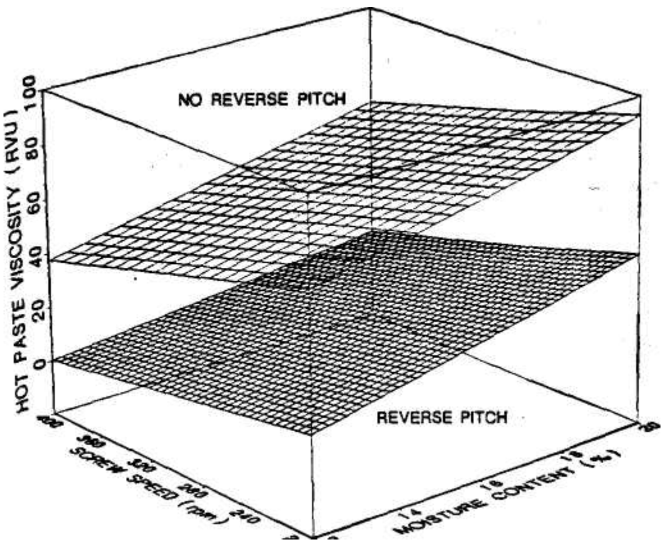


Fig. 3.7. Peak viscosity (PV) of the extruded product obtained by extrusion at different screw speed and feed rate with or without reverse pitch screw element.

The high values for peak viscosity (PV) in the viscogram of extrudates indicates the presence of high proportion of ungelatinized (uncooked) starch. On the contrary, a low PV is associated with a high extent of cooking obtained with high moisture content (20%) in the feed with a screw profile consisting of RPSE and MD. The effect of screw speed and feed rate are opposing to each other, of which the effect of screw speed is pronounced. This means that a proper combination of screw speed in relation to feed rate is necessary to obtain the desirable low PV values.

Structural breakdown with loss of granular integrity and disintegration of the starch granules occur when thermal gelatinization and mechanical damage occur (El-Dash, 1981). Hence, pregelatinized starch granules lose their ability to swell upon heating in water during viscographic (RVA) studies, which results in low HPV values. Similarly as the intensity and degree of cooking (gelatinization) increases, the HPV also decreases (Tipples, 1980).

Extrusion cooking of cereal flour is a typical example of low moisture gelatinization process because the moisture content in feed hardly crosses 20%. As moisture content was increased from 12 to 20% in the present study, gelatinization was expected to increase with subsequent decrease in HPV values. The presence of RPSE and/or MD increased residence time, and extent of shearing (discussed earlier) aids to the process of gelatinization, decreasing HPV consequently.



Fig, 3.8. Hot paste viscosity (HPV) of the extruded product obtained by extrusion at different screw speed and feed moisture content with or without reverse pitch screw element.

Cold paste viscosity (CPV) reflects the extent of starch retrogradation that occurs during the cooling phase. CPV values, apart from depending on the presence of RPSE and MD, and on screw speed and moisture content, increase with an increase in amylose content and barrel temperature. Starch with low moisture content, extruded at a high temperature, results in extrudates characterised by a low degree of retrogadation (El-Dash et al, 1984). As high amylose can produce more retrograded starch (Cruzy Celis et al, 1996), CPV increases.

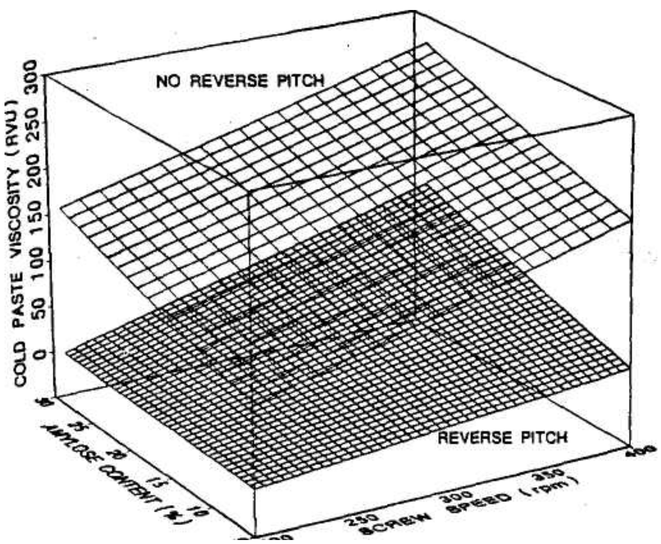


Fig. 3.9. Cold paste viscosity (CPV) of the extruded product obtained by extrusion at different screw speed and amylose content of feed with or without reverse pitch screw element.

3.4. SUMMARY

Large number of variables in the food processing operations including extrusion technology is a limitation for the appropriate application and standardization of the technology. A screening experiment with ten extrusion variables using Plackett-Burman experimental design has been applied for the extrusion of rice flour. The variables included, extruder hardware variables (mixing disk and reverse pitch screw elements), feed variables (amylose content, moisture, sugar, salt and particle size) and extrusion operating variables (barrel temperature, feed rate and screw speed). The extrusion trials were conducted on rice flour employing a twin-screw extruder without using a die.

The response functions were the extrusion characteristics (torque, net specific mechanical energy and average residence time), product attributes (water solubility index, water absorption index, and bulk density), and the viscographic indices (peak viscosity, hot paste viscosity and cold paste viscosity). The results were analysed using coded level of variables (-1 and +1) by fitting to first order regression equations.

The present work showed that the screw configuration, particularly the presence of reverse pitch screw element and mixing disks, exerts maximum effect on the extrusion and extrudate characteristics.

Considerable effects were also observed for amylose and moisture content, feed rate, screw speed and barrel temperature. The variables that possessed least effect on the response functions were particle size, salt and sugar contents.

The extrusion of non-waxy (high amylose content) rice flour resulted in low WSI of the product. As high values of WSI are desirable in cooked extruded products, such as, ready-to-eat snacks, breakfast cereal and porridges, a combination of high levels of barrel temperature (140°C) in the presence of RPSE and MD using a low amylose (5%) variety is considered desirable. The experimental results relating the variables and response function could be fitted well (0.721 $ r $ 0.999, p $ 0.01) by first order polynomials which indicates the suitability of the Plackett-Burman model to evaluate the effect of the individual variables.

CHAPTER -IV

Effect Of Barrel Temperature & Screw Speed On Extrusion Parameters And Certain Physicochemical Properties Of Rice Extrudate

4.1. INTRODUCTION

As mentioned in earlier chapter, extensive work on the extrusion cooking of cereals, particularly corn and wheat has been carried out. On the contrary, literature on extrusion processing of rice is sparse. Further, fewer extruded rice products are available compared to corn and wheat. However, rice being the staple in Asian countries, rice based extruded products are expected to find a good market in these countries. The advantages of rice are that it is non-allergenic, gluten-free, and low in sodium and fat content (Dziezak, 1991). In addition, rice products impart a fatty mouthfeel and texture (Anon, 1991), and therefore, can be used for developing foods with low fat content. Rice products are also gaining popularity in European and American markets in recent years (Tuley, 1992).

A few studies have been conducted in the recent past using a single screw extruder on the effect of extrusion variables on product characteristics (Kim and Maga 1990; Bhattacharya and Prakash, 1994). Studies on twin-screw extrusion of rice flour are however, limited (Kumagai et al, 1987; Pan et al, 1992; Bhattacharya and Choudhury 1994). With more and more food industries opting for twin-screw extruders for bettel control of product profile and other technical advantages, it is necessary to have enough data and information on extrusion of rice to develop rice based extruded products. It is also important to correlate the extrusion conditions with the product characteristics.

The role of die during extrusion is basically in shaping of the product. It would be quite appropriate not to use the die if the target is to have a cooked product that will be used subsequently for other purposes, such as developing food for babies or for using it as base material for producing another product.

The objectives of the present work were: a) to study the effect of the extrusion variables (temperature of barrel and screw speed) on extrusion system parameters (torque and specific mechanical energy) and product attributes (sediment volume, *in-vitro* starch digestibility, water absorption index, water solubility index, bulk density), and

b) to determination of relationships between system parameters and product attribute during extrusion cooking of rice flour without using a die.

4.2. EXPERIMENTAL

4.2.1. Rice

The milled rice (cultivar IR 64) was ground to flour in a hammer mill as describe under Section 2.1. The particle size distribution of the rice flour, as determined using British Standard (BS) sieves, is shown in Table 4.1. The '+' sign indicates the quantity of material that is retained by the particular sieve whereas '_' denotes the quantity passing through. The proximate composition of rice has been presented in table 2.1 under Section 2.1.

Table 4.1 Particle size distribution of rice flour

BS sieve Particle size	Quantity (%)
+20	0.0
-20/+28	0.2
-28/+32	4.9
-32/+45	32.9
-45/+60	17.1
-60/+80	18.0
-80/+100	7.8
-100/+200	16.1
-200	3.2

4.2.2. Extruder and extrusion cooking

The extruder used has been described under Section 2.2. The extruder screw consisted of forward pitch screw elements, and a reverse pitch screw element (near the outlet), and also, two kneading blocks. No die was used. Screw profile used for present extrusion work is described in Table 4.2.

The temperature of the extruder barrel was maintained at 80, 100 or 120°C throughout the barrel. The screw speed used was 200, 300 or 400 rpm while the feed rate was maintained constant at 17 kgh^{-1} using a volumetric gravity feeder. The moisture content of the feed was 14.2 ± 0.1% (d.b). All extrusion trials were repeated once. The extrudate samples were prepared as mentioned under Section 2.4.

Table 4.2

Screw profile used for extrusion trials

Type of screw Element	Screw element details Pitch/lenqth	Total length (mm)
Forward pitch	42/42	220
	42/21	42
	28/14	14
	28/28	140
Kneading block*	45/5/14	14
Forward pitch	28/28	196
	20/20	60
Kneading block *	45/5/14	14
Forward pitch	20/20	180
Reverse pitch	-20/10	10
Forward pitch	20/20	60

* The kneading blocks consist of 5 mixing disks placed at an angle of 45° and have a total length of 14 mm

4.2.3. Extrusion characteristics

The torque (T%) developed during extrusion was obtained as mentioned under Section 2.4.2 and the total specific mechanical energy (SME) input during extrusion was estimated using equation (1) as mentioned under Section 2.4.3 as follows:

$$SME = \frac{\text{rpm of screw (run)}}{\text{rpm of screw (rated)}} \times \frac{\text{\% torque (run)}}{100} \times \frac{\text{motor power (rated)}}{\text{production capacity}}$$

4.2.3.1. Sediment volume

Sediment volume of the extrudate was determined according to the method 01 Bhattacharya and Ali (1976). Two-gram (d.b.) extruded rice flour was taken in a 50 m glass-stoppered measuring cylinder and 40 ml of 0.05N HCl was added with gentle shaking. The cylinder was stoppered and the slurry mixed by repeated inversions. A drop or two of amyl alcohol was added on top to disperse the froth and the cylinder was left undisturbed. The sediment volume was read after 4 h. Mean of three replicates was reported.

4.2.3.2. *In-vitro* starch digestibility

In-vitro digestibility of starch in the extruded sample was estimated according to the method of Holm et ai, (1985) using amyloglucosidase (from *Aspergillus oryzae,* Grade V, Catalogue No. A-9268, Sigma, USA). Preliminary studies were conducted to ascertain optimum enzyme-substrate ratio and the time of treatment. To 100 mg of sample dispersed in 10 ml of distilled water, 10 ml acetate buffer (pH 4.5, containing 7 units of enzyme) was added. The mixture was incubated at 55°C for 6 hours in a shaker water bath. The reducing sugar produced at the end of the treatment was analysed by using 3,5-dinitro salicylic acid (Bernfeld, 1955). A standard curve using glucose was prepared and the extent of hydrolysis was calculated as the proportion of starch (% glucose equivalent) converted to glucose. The number of replicates were two.

Water absorption index (WAI), Water solubility index (WSI) and Bulk density (BO)

The above parameters were determined by the procedures as described under Sections 2.4.4 and 2.4.5. The reported values are the means of three replicates.

4.2.4. Experimental design and statistical analysis

The extrusion process variables (X_1, i.e., temperature of barrel; viz. 80, 100 and 120°C and X_2, i.e., screw speed; viz. 200, 300 and 400 rpm) were coded to the levels of -1, 0 and +1 such that a total of 9 experiments were conducted. All experiments were repeated once. The measurements of the dependent variables reported were the mean of three observations each. The response functions (y) were: a) torque during extrusion, b) total SME, c) sediment volume, d) *in-vitro* starch digestibility, e) water absorption index, f) water solubility index, and g) bulk density. These seven response functions were related to the extrusion process variables by a second degree polynomial (Eq 2) which consisted of linear, quadratic and interaction effects; the method of least squares was used to develop these polynomials (Little and Hills, 1978), and accordingly, the response surfaces were generated. The optimization of the response functions was done as per the procedure described by Bhattacharya and Prakash (1994).

$$y = b_o + b_1 X_1 + b_2 X_2 + bn\, fi + b1_2 X1\, X_2 + b_{22} X_2^2 + \epsilon \qquad ..\text{-}(2)$$

The coefficients of the polynomial are represented by b_o (constant term), b1 and b_2 (linear effect), b_{11} and b_{22} (quadratic effect), b_{12} (interaction effect) and $_E$ (random error). The barrel temperature is denoted by X1 and the screw speed by X_2 in coded level of variables.

The analysis of variance (ANOVA) tables were generated for all the seven response functions. The significance of the individual terms in the polynomial was determined statistically by calculating the F-values, and judging them at probability levels (p) of 0.01, 0.05 or 0.10. The correlation coefficients (r), determined to know the relationships between the extrusion characteristics and product attributes, were judged at p=0.01 when the number of data points (n1) was 27.

4.3. RESULTS AND DISCUSSUION

4.3.1. Torque

The torque during extrusion of rice flour ranged between 38 and 85% (Fig. 4.1) and high torque values were associated at low screw speeds (200 rpm). This indicates that is a torque limiting process, particularly if the extruder is operated at low screw speed. Increase in screw speed decreased the magnitude of torque due to a) decrease in the filled length and b) increase in the shear rate that reduces the apparent viscosity of the extrudate as the mass inside the extruder behaves as a pseudoplastic material that shows shear thinning behaviour (Harper, 1981; Bhattacharya et al, 1992) The effect of temperature on torque is rather complex and depends on the level of screw speed. At high screw speed (400 rpm), increase in the barrel temperature from 80 to 100°C markedly decreased the torque. However, beyond 100°C it remained fairly constant. At low screw speed (200 rpm), a slight decrease in torque was noted upon increase in barrel temperature. The present results are in agreement with those reported by Bhattacharya and Prakash (1994) for a blend of rice and chickpea when barrel temperature showed a linear (negative) effect with torque.

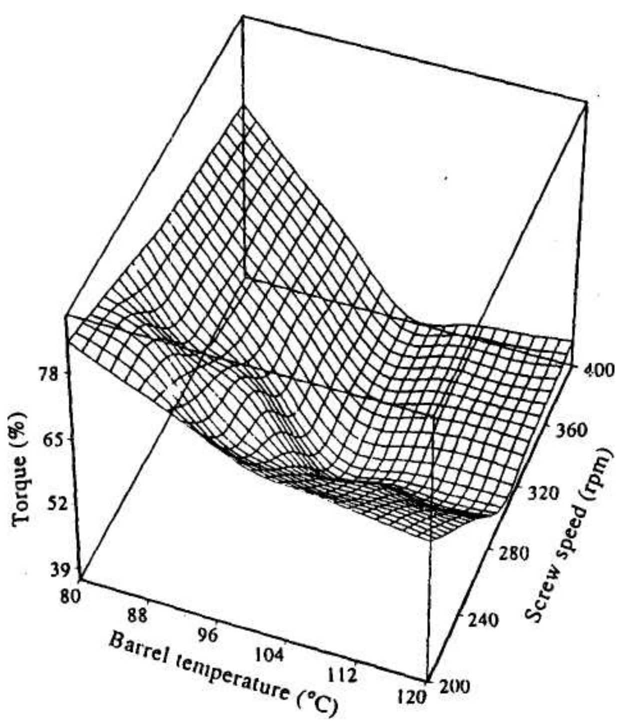


Fig. 4.1. Response surface for torque during extrusion of rice flour.

4.3.2. Specific mechanical energy (SME)

The total specific mechanical energy (SME), defined as the total mechanical energy input to obtain 1 kg of extrudate, varied between 317 and 1013 $kJkg^{-1}$ (Fig. 4.2). Low values « 350 $kJkg^{-1}$) of SME were obtained at high temperature (100-120cC) and screw speed (300 rpm). It seems logical to mention that increase in screw speed increases the SME values as the latter is directly proportional to screw speed (Eq 1) provided the torque is constant. As torque also decreases markedly with elevation in screw speed, the effect of screw speed becomes complex and shows a curvilinear relation with SME. An increase in temperature generally decreases the energy values which may be explained on the basis of gelatinization of starch (the main ingredient of rice flour) and apparent viscosity of the mass inside the extruder. The extent of *in-vitro* digestibility (discussed later), an indirect index of gelatinization extent, was high even at 80°C, and any further increase in temperature was therefore expected to reduce the apparent viscosity of the plasticised mass.

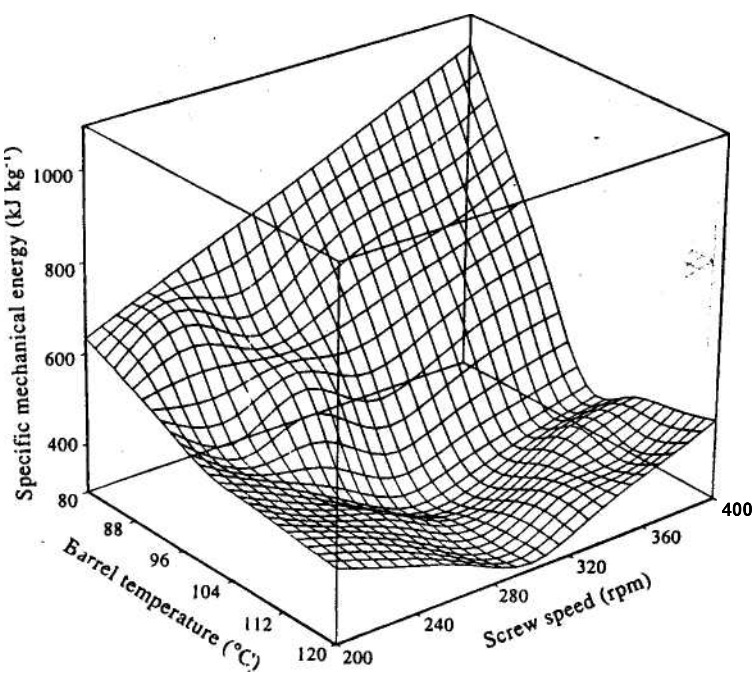


Fig. 4.2. Response surface for total specific mechanical energy (SME) during extrusion of rice flour.

4.3.3. Sediment volume

Volume of the sediment of a processed rice product, such as, parboiled rice c flaked rice in excess of dilute HCI acid has been proposed to serve as an index of th gelatinization by Bhattacharya and Ali (1976). The sediment volume showed **a** increase from raw to mild parboiled, to severely parboiled rice to flaked rice and to **pre** gelatinized starch. The latter occupying the full liquid volume as gel.

In the present studies, all the extruded products showed very high sediment volume that ranged between 24.5 to 26.5 ml (Fig. 4.3) compared to non-extruded rice (7. ml). However, low values among them were shown by extruded samples produced at

high temperature. It is therefore, apparent that the degree of gelatinization in all the sample was markedly high as compared to that which could be seen in either parboiled or flake rice that ranged between 8.1 to 19.5 ml as reported by Bhattacharya and Ali (1976).

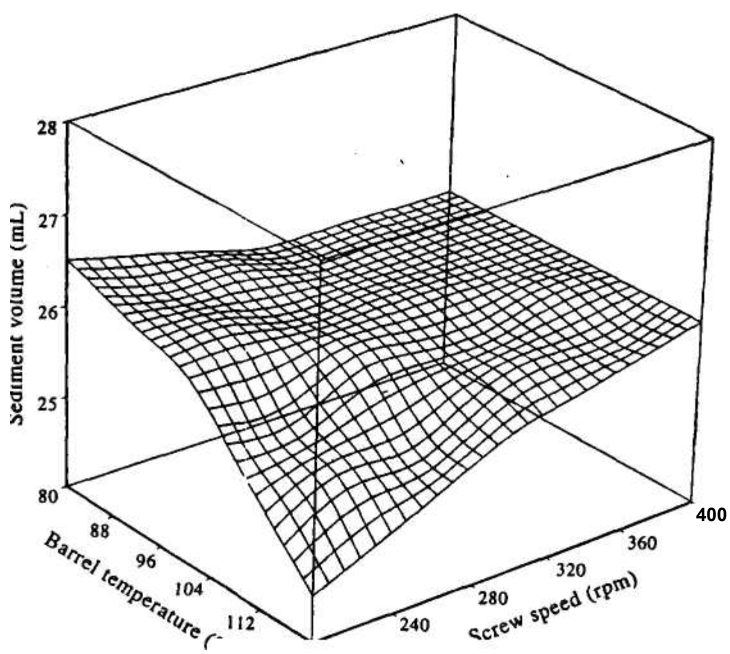
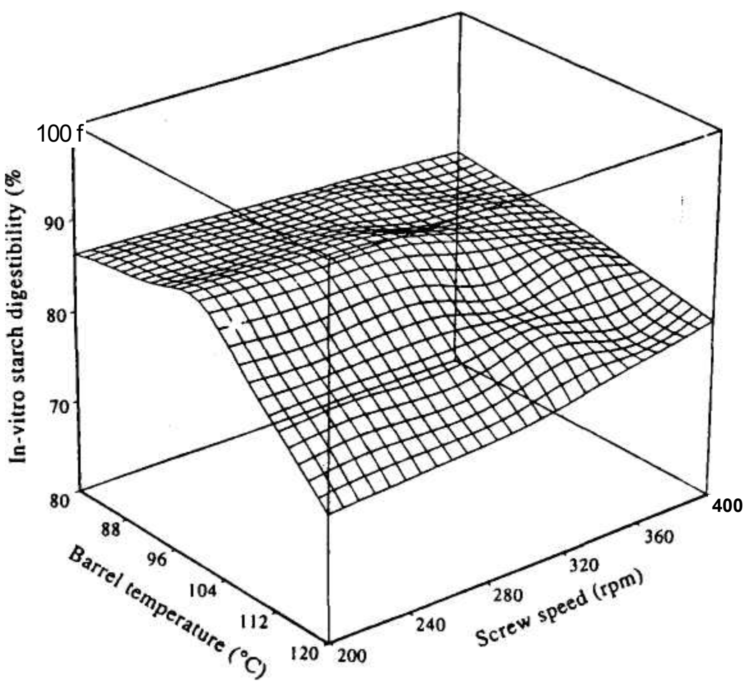


Fig.Fig.4.3. Sediment volume of the extruded product obtained by extrusion at different barrel temperatures and screw speeds.

4.3.4. *In-vitro* starch digestibility

The results on the *in-vitro* starch digestibility studies (Fig. 4.4) showed that the extruded rice samples had very high susceptibility (73,6-87.4%) to enzyme degradation than that of raw rice (12.8%). However, the extent of susceptibility differed among samples extruded under different conditions of extrusion temperature and screw speed. Digestibility increased with barrel temperature from 80 to 100°C, but showed a decrease for samples extruded at 120°C with increasing screw speed from 200 to 400 rpm. The digestibility decreased by 2 to 6 per cent points when the screw speed was increased from 200 to 400 rpm for samples extruded at 80 and 100°C. However, at 120°C, not only was the overall digestibility less, as compared to that at lower temperatures, but showed an increase with increasing screw speed by about 3 per cent points. Mercier and Feillet (1975) while investigating the effect of different processes on starch digestibility in different cereals, observed that the severe the process, the greater was the digestibility. Iikimani et al, (1990); Chiang and Johnson (1977) have reported an increase in the digestibility of starch with increasing extrusion temperature and a decrease with increasing screw speed, This decrease was explained on the basis of low residence time in the extruder at high screw speed.



Fig, 4.4. *In-vitro* digestibility of starch of the extruded product obtained by extrusion at different barrel temperatures and screw speeds.

The reversal of the trend at 120°C observed in the present work may be due to retrogradation or reassociation of gelatinised starch or formation of amylose-lipid complex, starch-protein complex or resistant starch. Formation of these complexes are known to reduce the susceptibility of starch to enzyme hydrolysis (Mercier, 1980; Eerlingen et al, 1994).

4.3.5. Water absorption index (WAI) and water solubility index (W51)

Water absorption index was least (2.6 g/g), as expected, for the raw rice flour and increased upon extrusion to a maximum of 7.1 g/g (Fig. 4.5). High WAI was shown by products extruded at barrel temperature of 80 and 100°C (6.1 to 7.1 g/g) as compared to that to (5.5 to 5.9 g/g). Decrease in WAI has been reported by Anderson et al, (1969a), and by Mercier and Feillet (1975). This may be related to the degradation of starch that causes a reduction in the water holding capacity of the molecules as a result of decrease in the molecular size. At a low shear rate (low screw speed) and/or low temperature, one can expect more undamaged polymer chains and a greater availability of hydrophilic groups which can bind more water resulting in higher values of WAI (Gomez and Aguilera, 1983).

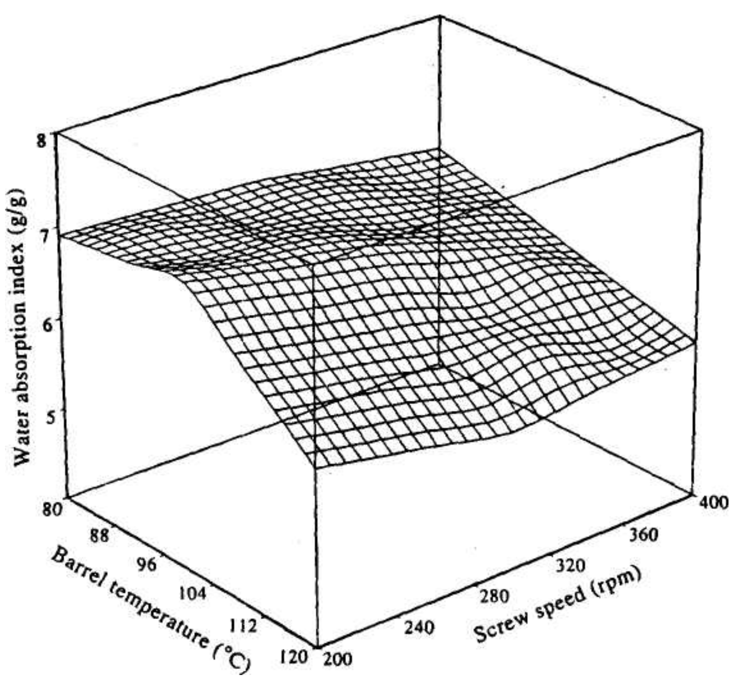


Fig. 4.5. Water absorption index of the product obtained by extrusion at different barrel temperatures and screw speeds.

At any temperature, samples extruded at lower screw speed (200 rpm), showed relatively high WAI values than at higher screw speeds (300 and 400 rpm). This is perhaps due to high residence time at low screw speed permitting enhanced extent of cooking.

Water solubility index (WSI) was the least (1.7%) for the raw rice and increased markedly from 28.0 to 40.5% upon extrusion cooking (Fig.4.6). However, the trends observed in this case was reverse of that observed in the case of WAI with respect to the effect of barrel temperature and screw speed, viz., samples extruded at higher temperatures and screw speeds showed high WSI values. Similar results have been reported by other workers also (Conway, 1971b; Artz et al, 1990; Badrie and Mellowes, 1991 and Ralet et al, 1991). The WSI depends on quantity of soluble matter which increases due to the degradation of starch. Wen et al, (1990) indicated that screw speed had a direct effect on polysaccharide size distribution. A higher screw speed (increasing the shear) resulted in more fragmentation than a lower screw speed.

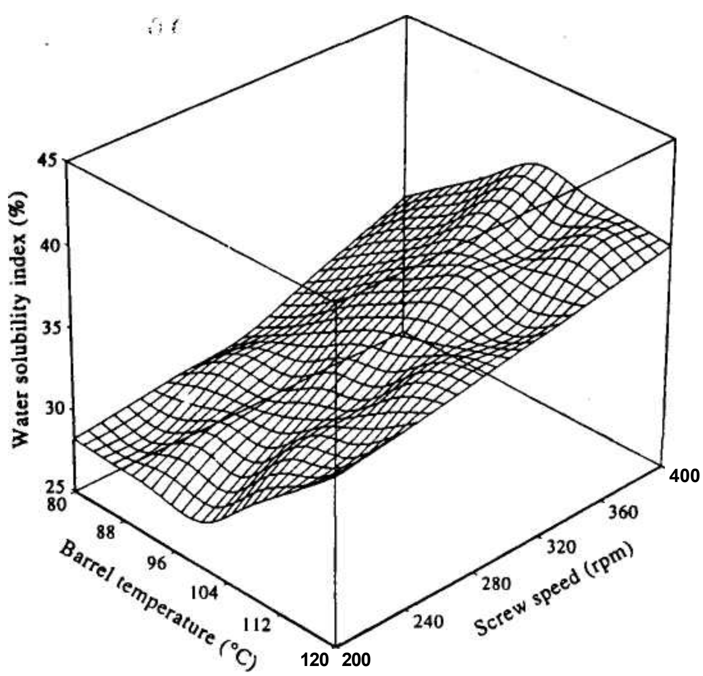


Fig. 4.6. Water solubility index of the product obtained by extrusion at different barrel temperatures and screw speeds.

A degradation of amylose and amylopectin molecules of manioc starch through chain splitting has been reported by Colonna et al, (1983). Similar results were also observed in case of wheat starch (Davidson et al, 1984). It could therefore, be inferred that that combined effect of high temperature and high screw speed enhanced the amount of soluble material in the extrudate.

4.3.6. Bulk density

The extrudates showed bulk densities in the range of 172 and 231kg m^{-3} (Fig 4.7) Lowest density was obtained at a temperature of 100°C and screw speed of 200-300 rpm. The response surface showed a decrease in bulk density with elevation if temperature upto 100°C. Further increase in temperature increased the bulk density marginally. An increase in screw speed decreased the product density. It is logical to assume that high temperature enhanced the level of thermal input leading to complete gelatinization even when the screw speed was high (and hence, low residence time inside the extruder). A combination of high temperature and high screw speed yields a production with least density. The other reason may, be the structural breakdown that possibly occurs in the high-shear environment leading to low density product.

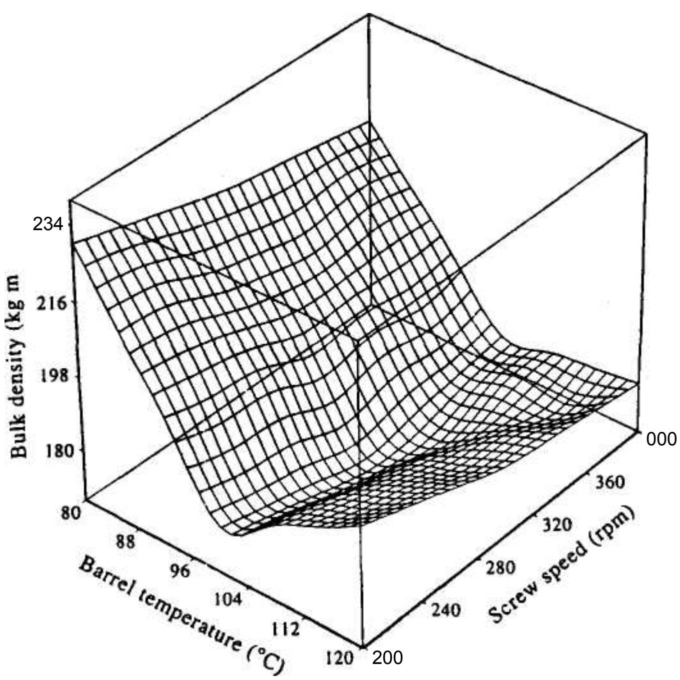


Fig. 4.7. Bulk density of the extruded obtained at different barrel temperatures and screw speeds.

4.3.7. Statistical analysis

The detailed statistical analysis using response surface methodology (RSM) generated the coefficients (Table 4.3) of the second order polynomials for the response functions (torque, SME, bulk density, WAI, WSI, sediment volume and *in-vitro* starch digestibility). The polynomials, developed using the coded level of variables, fitted the experimental results well as indicated by the high multiple correlation coefficients ($r \geq 0.931$, $P \leq 0.01$).

Values presented in the Table 4.3 show that the torque during extrusion was negatively related ($p < 0.05$) to the linear effects of temperature (X_1) and screw speed (x_v indicating that an increase in either of the variables decreased the torque values (Fig.4.1). The SME decreased with an increase in temperature (Fig. 4.2) at low temperature levels, as it's linear effect was negative. On the other hand, at high temperatures the SME values increased with positive quadratic effect ($p < 0.10$). Sediment volume decreased linearly with an increase in temperature (Fig. 4.3). The interaction term (temperature X screw speed) was also significant ($p < 0.05$) showing that the effect of temperature on sediment volume depends on screw speed. The effect of temperature on *in-vitro* digestibility was curvilinear as both of its linear and quadratic effects are significant ($p < 0.05$ and $p < 0.10$, respectively). An increase in temperature reduced the digestibility values (Fig. 4.4). The WAI depended mostly on the temperature ($p < 0.05$). An increase in temperature decreased WAI values (Fig. 4.4). The effect of screw speed was similar to that of temperature but showed a lesser effect ($p < 0.10$). Solubility index varied linearly (positive effect) with screw speed (Fig. 4.6). The bulk density of the extrudates was mainly dependent on the temperature, as its linear as well as quadratic effects are highly significant ($p < 0.01$). Screw speed had a linear negative effect. An increase in screw speed therefore decreased the bulk density (Fig. 4.7).

Table 4.3 also presents the optimum conditions of temperature and screw speed for the response functions. Saddle points were obtained for WAI, WSI and for sediment volume, whereas, for *in-vitro* starch digestibility, the optimum condition was outside the experimental region. The generalised optimum extrusion conditions in respect to minimum torque and SME during extrusion, and bulk density of the product, were the use of high barrel temperature (107-110°C) in combination with medium to high level (301- 407 rpm) of screw speed.

Table 4.3

Coefficients of the polynomials relating the response functions and the extrusion variables (barrel temperature: X1 and screw speed: X2) in coded level of variables

Coefficients	Torque	SME	Bulk density	WSI	WAI	Sediment volume	In-vitro digestibility
b_0	41.689	333.700	172.444	33.311	6.611	26.000	86.289
b_1	-13.333**	-196.683	-17.000	3.133	-0.616**	-0.416	-4.617
b_2	-11.950	-40.366^{NS}	-7.833	-3.583**	-0.266*	0.166^{NS}	-0.400^{NS}
b_{11}	14.967-	226.250*	28.334**	0.567^{NS}	-0.416^{NS}	-0.250^{NS}	-5.383
b_{12}	-3.325^{NS}	-98.624^{NS}	-1.250^{NS}	-0.575^{NS}	0.049^{NS}	0.500**	1.900^{NS}
b_{22}	10.816**	100.400^{NS}	3.833^{NS}	0.983^{NS}	0.033^{NS}	0.000^{NS}	-0.333^{NS}
r	0.981**	0.961**	0.991**	0.931**	0.954**	0.966**	0.943**
Targeted Optimum Condition	Minimum	Minimum	Minimum	Maximum	Maximum	Maximum	Maximum
Optimum Condition	Achieved	Achieved	Achieved	Saddle point	Saddle point	Saddle point	Outside experimental Range
Optimum Temperature (°C)	110.3	108.8	106.5	-	-	-	-
Optimu scew speed (rpm)	363.1	301.4	407.4	-	-	-	-

*Significant at p≤ 0.10

**Significant at p ≤ 0.05

***Significant at p ≤ 0.01

NS Non-significant at p = 0.10

4.3.8. Inter-relationship between system parameters and product attributes

It is obvious that extrusion characteristics and product attributes are inter-related, and hence, a system analytical model has been proposed by some workers (Meuser and Wiedmann, 1989). In an extension of the model, linear inter-relationships have been obtained between the extrusion characteristics (torque and SME) and the product attributes (sediment volume, *in-vitro* digestibility, WAI, WSI and bulk density). Significant ($p < 0.01$) positive linear relationships were obtained for torque with WAI (Fig. 4.8A) and bulk density (Fig. 4.8B), whereas a negative relation exists with WSI (Fig. 4.8C). This means that increasing the torque during processing usually increases the desirable characteristics like WAI but reduces the bulk density and water solubility index of the extrudates. The torque during extrusion depends on the rheological status of the plasticised mass inside the

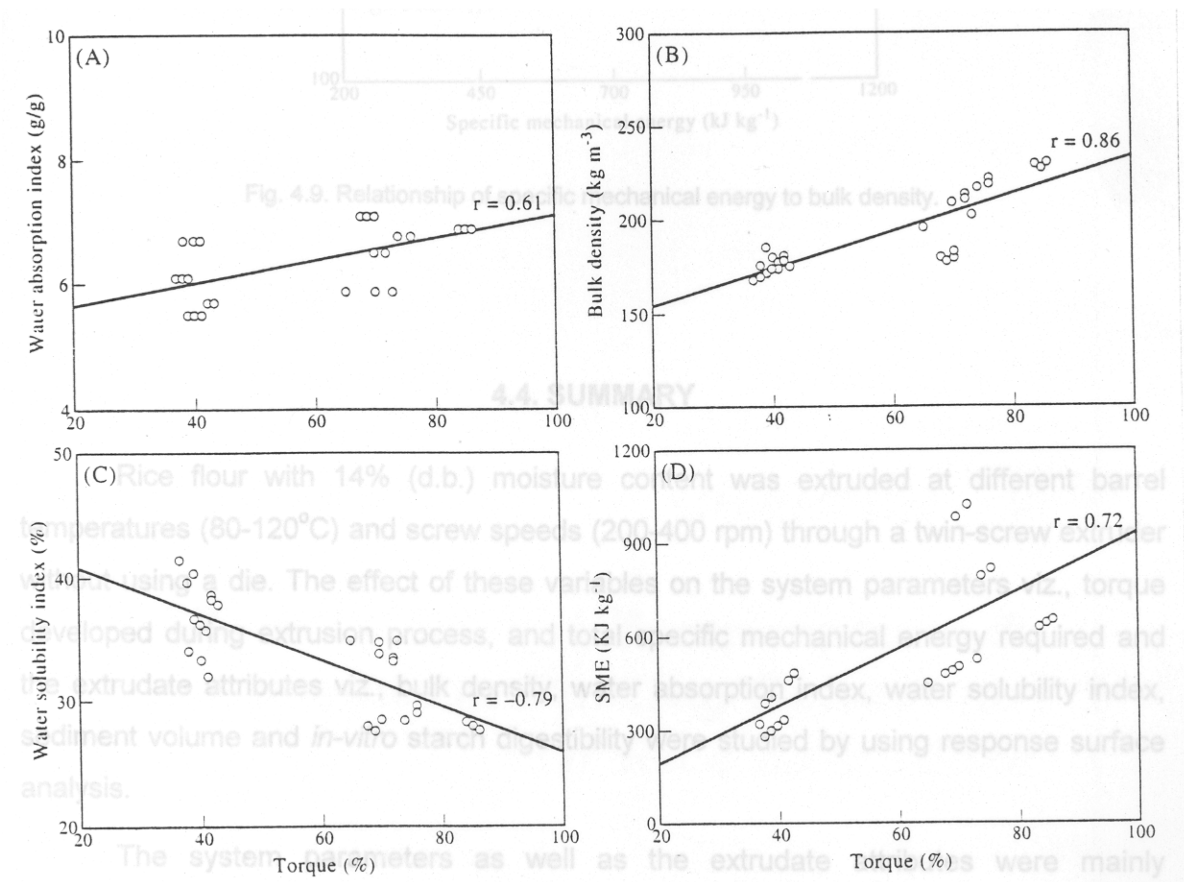


Fig. 4.8. Relationship of torque to (A) water absorption index, (B) bulk density, (C) water solubility index and (D) specific mechanical energy.

Operation of the extruder at a high torque becomes highly energy intensive and product loses its solubility in water. In such a situation, intermediate values seem to be practical On the other hand, SME showed a moderate but positive significant (r = 0.78, P ≤ 0.01; relationship with bulk density (Fig. 4.9), showing that to obtain a low density (≤175 kg m^{-3}: product, a low SME (300-350 kJ kg^{-1}) is appropriate. Further increase of input energy) leads to a decrease in product density and reduces solubility.

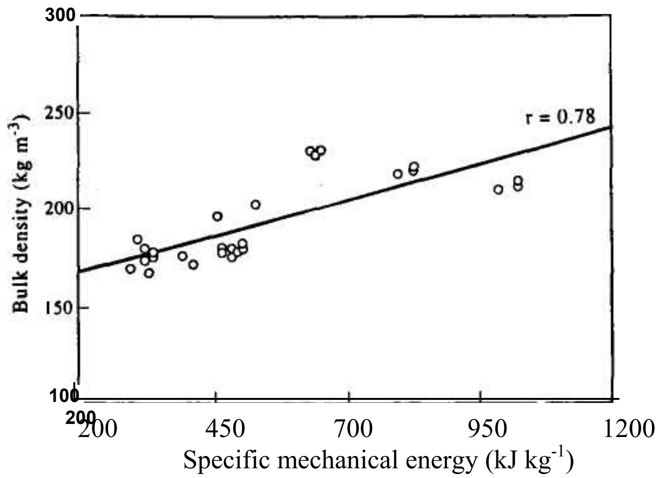


Fig. 4.9. Relationship of specific mechanical energy to bulk density.

4.4. SUMMARY

Rice flour with 14% (d.b.) moisture content was extruded at different barrel temperatures (80-120°C) and screw speeds (200-400 rpm) through a twin-screw extruder without using a die. The effect of these variables on the system parameters viz., torque developed during extrusion process, and total specific mechanical energy required and the extrudate attributes viz., bulk density, water absorption index, water solubility index, sediment volume and *in-vitro* starch digestibility were studied by using response surface analysis.

The system parameters as well as the extrudate attributes were mainly dependent on temperature, whereas the screw speed imparted a lesser effect. At low barrel temperature (80°C) and low screw speed (i.e., low shear rate, 200 rpm) in a twin-screw extruder although yielded extrudate with acceptable cooking and *in-vitro* starch digestibility indices, it had high bulk density, and required high specific mechanical energy (643 to 1012 kJ kg^{-1}) for extrusion, and generated high torque (71 to 85%). High temperature (120°C) on the other hand, required less SME (322 to 497 kJ kg^{-1}) and produced less torque (approximately 40%) during extrusion, particularly at high shear (300-400 rpm) but had comparatively decreased *in-vitro* starch digestibility. Optimum extrusion conditions for obtaining minimum torque, specific mechanical energy (SME) and bulk density were determined. A positive linear relationship (correlation coefficient $r=0.78$, $p \leq 0.01$) existed between SME and bulk density indicating that it is possible to obtain low density extrudate with low SME. Extrusion at 100°C, 300 rpm needed the least energy (SME 317 kJ kg^{-1}) and produced lesser torque (39%) during extrusion and yielded a product with desirable product profile. Extrusion of rice flour without a die appears to be an alternative approach to produce processed rice flours with high water absorption index and *In-vitro* starch digestibility.

CHAPTER - V

Effect Of Barrel Temperature & Screw Speed On Pasting Behaviour Of Rice Extrudate

5.1. INTRODUCTION

The use of extrusion in the food industries has increased over the past two decades mainly because of greater demand for convenience foods. The introduction of twin-screw extruders has widened the scope of food extrusion technology for the manufacture of many cereal-based products including ready-to-eat breakfast cereals, infant food formulations, snack foods and modified starches. Despite the increased use of extrusion processing on starch-based products, information on the effect of extrusion variables on the viscosity characteristics of the products is meagre. Extrusion of starch appears to be a simple technological process, but the control of the finished product characteristics is rather complicated. This is because of the complex nature of food and the large number of variables involved in the process (El-Dash, 1981). Earlier studies were carried out with the aim of producing specific food products using single screw extruders (Anderson et al, 1969a; Lawton et al, 1972; Conway, 1971a; Conway and Anderson, 1973; Owusu-Ansah et al, 1983; Bressani et al, 1978). Research on the twin-screw extrusion of starches and cereal flours has shown that extrusion variables and ingredients markedly alter the physico-chemical properties of the finished product (Charbonniere et al, 1973; Mercier, 1977; Mercier and Feillet, 1975; Ryu et al, 1993).

Pasting properties of extrudates are important when pregelatinized extrudate flours are used further as base material for different industrial food products. Changes in viscosity of extrudate powders produced under various operating conditions have been studied by Lawton et al, (1972) to find the factors affecting pasting properties. Mason and Hoseney (1986) investigated the effect of operating variables on cold paste and hot paste viscosities and swelling peak area (calculated from the amylograph pasting curve) of extrusion cooked wheat starch, by using a Brabender viscoamylograph. They concluded that the hot paste viscosity depends on die temperature, screw speed and barrel temperature, whereas, cold paste viscosity was affected by moisture content and feed rate. They also reported that swelling peak area positively correlated with the cold paste viscosity.

In comparison to the conventional method of determining the pasting behaviour of starch and/or starch-rich cereal flour by Brabender viscoamylograph, the Rapid Viscoanalyser (RVA) requires a small amount of sample and short time to produce pasting curves (Walker et al, 1988) and yields essentially similar information. However, the use of the RVA or even a viscoamylograph has not yet been widely reported for determining the pasting properties of rice extrudates. The pasting parameters generated from the RVA provide a relative measure of starch gelatinization, disintegration, swelling and gelling ability (Ryu et al, 1993). There exists a need therefore, to study the pasting properties of rice extrudate by employing the RVA for characterising the extrudate viscosity profile, which could allow process operators to monitor extruder systems and decide on the extrusion conditions required to prepare rice extrudates for special industrial food uses.

The present work was undertaken with an objective of studying the effect of the extrusion variables, viz. barrel temperature and screw speed, on the viscosity characteristics of extrudate rice flour during pasting from 50°c to 95°C and cooling back to 50°C, using a Rapid viscoanalyser (RVA). The viscographic parameters studied during the pasting programme were the peak, hot paste and cold paste viscosities, as well as the extent of gelatinization.

5.2 EXPERIMENTAL

Rice flour

The rice flour (cultivar IR 64) used in the present study was the same as described under Section 4.2.1.

Extruder and extrusion cooking

Details on the extruder used and the extrusion cooking conditions employed were the same as reported in the previous Chapter (Section 4.2.2).

5.2.1. Sample preparation for pasting studies and viscography

The sample preparation for pasting studies and viscography measurement have been carried out as described under Section 3.2.S.

The parameters that were noted from the viscogram included: a) Initial Viscosity (IV), i.e. the viscosity obtained at 50°c at the beginning of the RVA run,

b) Peak Viscosity (PV), c) Hot Paste Viscosity (HPV) and d) Cold Paste Viscosity (CPV) as mentioned under Section 3.2.4.

The area under the peak occurring on the down-slope of viscograph curve during the heating phase of the pasting curve (swell peak area) exhibited by the extruded samples in comparison to that of raw rice (as 100%) indicates the ungelatinized portion. The extent of gelatinization (GE) was therefore the value after subtracting this value from 100. The swell peaks are evident for extrudates suspensions on viscogram appearing in several publications (Anderson et al,1969a; Gomez and Aguilera, 1983; Kim, 1984). To measure area under the peak, a line was drawn tangentially from the inflections at the beginning and end of the peak (Mason and Hoseney, 1986). The peak area was then determined using a planimeter. All viscosity values reported are the averages of triplicate measurements and are reported in rapid viscoanalyser units (RVU).

5.2.2. Statistical analysis

Regression equations were generated for the response functions (PV, HPV, CPV and GE) to relate them with the extrusion variables (barrel temperature, X1, and screw speed, X_2), using the method of least squares (Little and Hills, 1978). The suitability of the regression equations to predict the response functions was judged by determining the multiple correlation coefficient (r). The experimental results were fitted either to a second order (Eq 1) or a third order (Eq 2) polynomial such that an r-value of 0.990 was obtained. These equations were used to develop the plots for the response functions.

$$y = b_0 + b_1 X_1 + b_2 X_2 + b_{11} X^2 + b_{12} X_1 X_2 + b_{22} X_2^2 + \varepsilon \qquad ...(1)$$

$$y = b_0 + b_1 X_1 + b_2 X_2 + b_{11} X^2 + b_{12} X_1 X_2 + b_{22} X_2^2$$
$$b_{313} X_1^3 + b_{221} X_1^2 X_2 + b_{212} X_1 X_2^2 + b_{323} X_2^3 + \varepsilon \qquad ... (2)$$

The coefficients of the polynomial are represented by b_o (constant term), b1 and b_2 (linear effect), b11 and b_{22} (quadratic effect), b_{313} and b_{323} (cubic effect), $b_{12} b_{221}$ and b_{212} (interaction effect) and E (random error). The barrel temperature and screw speed are denoted by X1 and X_2, respectively.

The analysis of variance (ANOVA) tables were generated and the multiple correlation coefficients (r), determined to know the relationships between the extrusion characteristics and the product attributes, judged at p=0.01 (n_1=27).

5.3. RESULTS AND DISCUSSION

Pasting profiles of raw rice and extrudates under different conditions of barrel temperature and screw speed are presented in Figs. 5.1 and 5.2 respectively. The initial viscosity (IV) of extrudates (30-43 RVU) was about ten times higher than that for raw rice (3-4 RVU). This is expected, as extruded samples are already gelatinized, which enables them to hydrate faster and to a greater extent than the raw ungelatinized sample. The viscosity of the extrudate pastes decreased during the heating phase (Fig. 5.2), in contrast to that of raw rice. This behaviour is also not unexpected. Heating slurries of completely gelatinized materials is known to cause a decrease in the viscosity leading to the 'thinning' of the slurry (Schweizer et al., 1986). However, during the heating phase, there was a decline in the rate of thinning, which could be ascribed to the counter-effect of swelling of the residual, partially gelatinized material. In samples extruded at low screw speed (low shear rate) at 80 and 100°C, the paste showed a rise in the viscosity curve during this phase, indicating that the proportion of uncooked or partially cooked material was slightly higher in these cases, which swells as it gets gelatinized and absorbs water producing a rise in viscosity of the slurry. However, when the proportion of this ungelatinized or partially cooked material was less, breakdown due to shear occurred sooner, and the thinning continued during pasting regime.

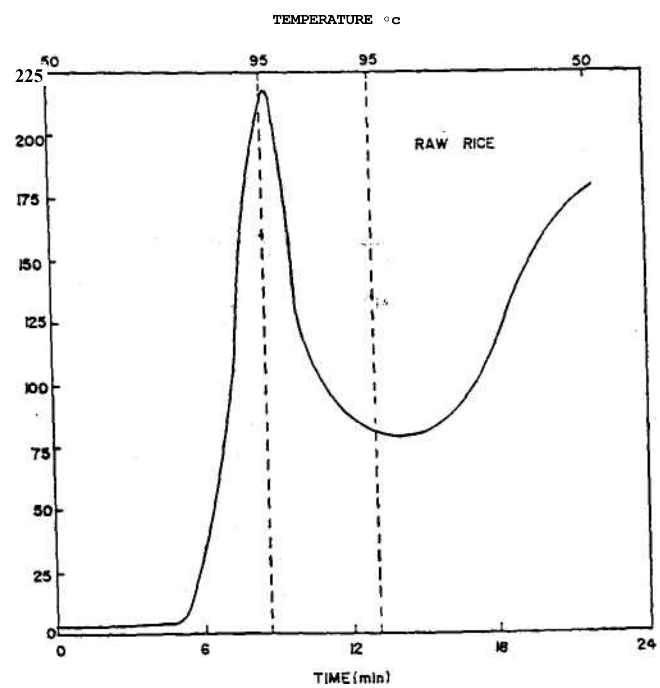


Fig. 5.1. Representative RVA pasting curve for raw rice. Viscosity values are expressed in Rapid visco-amylograph (RVU) units.

The rise in viscosity at the end of the cooling phase, i.e. the difference between CPV and HPV, gives an indication of the gelling properties due to retrogradation of the starch. This was notably minimal for all the extrudate samples. The viscosity rose only by 1 to 4 RVU for extrudates in comparison to a 97 RVU rise for raw rice. This suggests that starch in extruded rice had undergone thermal and mechanical degradation to an extent which was not conducive to retrogradation or gelling and the slurry remained thin. Cai and Diosady (1993) and Cai et al. (1995) have also observed similar trends for wheat extrudates.

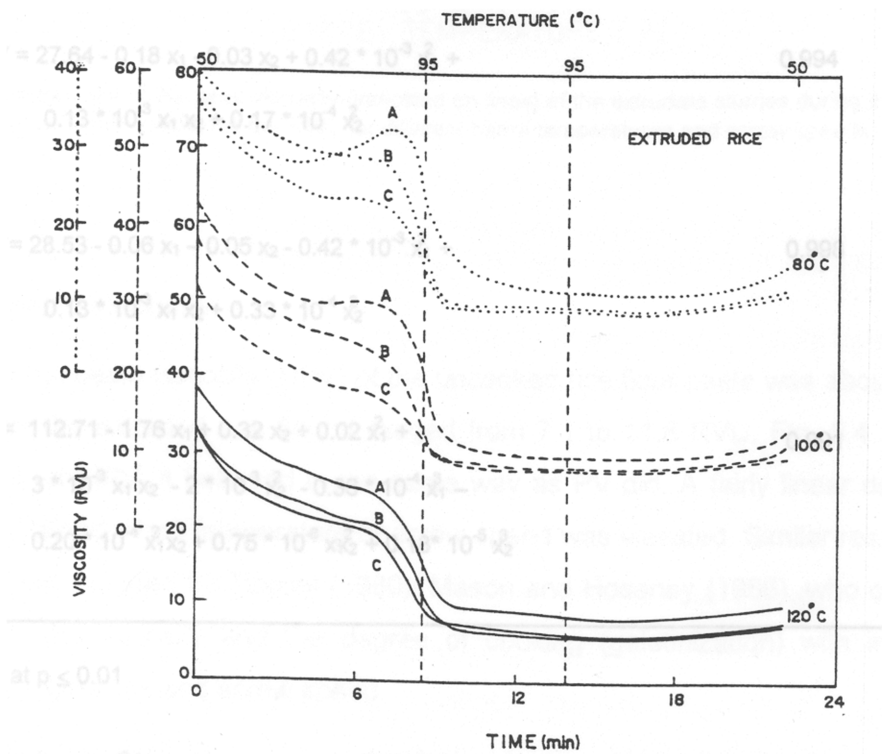


Fig. 5.2. Representative RVA pasting curves for extruded products obtained with different barrel temperature (80.100 and 120°C) and screw speed (A: 200, B: 300 and C: 400 rpm). The respective scales for extruded products are: 0 to 40 RVU for 80°C, 0 to 60 RVU for 100°C and 0 to 80 RVU for 120°C.

Table 5.1 shows the regression equations relating the pasting curve indices (PV, HPV and CPV) and extent of gelatinization (GE) as a function of temperature and screw speed. A high correlation coefficient ($r \geq 0.991$, $P \leq 0.01$) indicates the validity of the equations to predict the response functions.

Table 5.1
Regression equations relating pasting curve indices (PV, HPV, CPV) and extent of gelatinization (GE) of rice extrudate with barrel temperature and screw speed of extruder

Regression equations	r^{**}
$PV = -21.73 + 2.64\,x_1 - 0.15\,x_2 - 0.02\,x_1^2 - 0.01\,x_1 x_2 + 2 * 10^{-3}\,x_2^2 + 0.72 * 10^{-5}\,x_1^3 + 0.05 * 10^{-3}\,x_1^2 x_2 + 0.25 * 10^{-5}\,x_1 x_2^2 - 0.29 * 10^{-5}\,x_2^3$	0.996
$HPV = 27.64 - 0.18\,x_1 - 0.03\,x_2 + 0.42 * 10^{-3}\,x_1^2 + 0.13 * 10^{-3}\,x_1 x_2 + 0.17 * 10^{-4}\,x_2^2$	0.994
$CPV = 28.53 - 0.06\,x_1 - 0.05\,x_2 - 0.42 * 10^{-3}\,x_1^2 + 0.13 * 10^{-3}\,x_1 x_2 + 0.33 * 10^{-4}\,x_2^2$	0.998
$GE = 112.71 - 1.76\,x_1 + 0.32\,x_2 + 0.02\,x_1^2 + 3 * 10^{-3}\,x_1 x_2 - 2 * 10^{-3}\,x_2^2 - 0.36 * 10^{-4}\,x_1^3 - 0.20 * 10^{-4}\,x_1^2 x_2 + 0.75 * 10^{-6}\,x_1 x_2^2 + 0.18 * 10^{-5}\,x_2^3$	0.991

** Significant at $p \le 0.01$

Symbols:

xi : Barrel temperature (°C) X2 :
Screw speed (rpm)

5.3.1. Peak viscosity

The peak viscosities (PV) of the extruded rice pastes were between 21 and 33 RVU, very low in comparison to raw rice paste (218 RVU). PV generally decreased with increasing barrel temperature and screw speed (Fig. 5.3). The tendency was reversed only at high screw speeds (350-400 rpm) for barrel temperatures greater than 100°C.

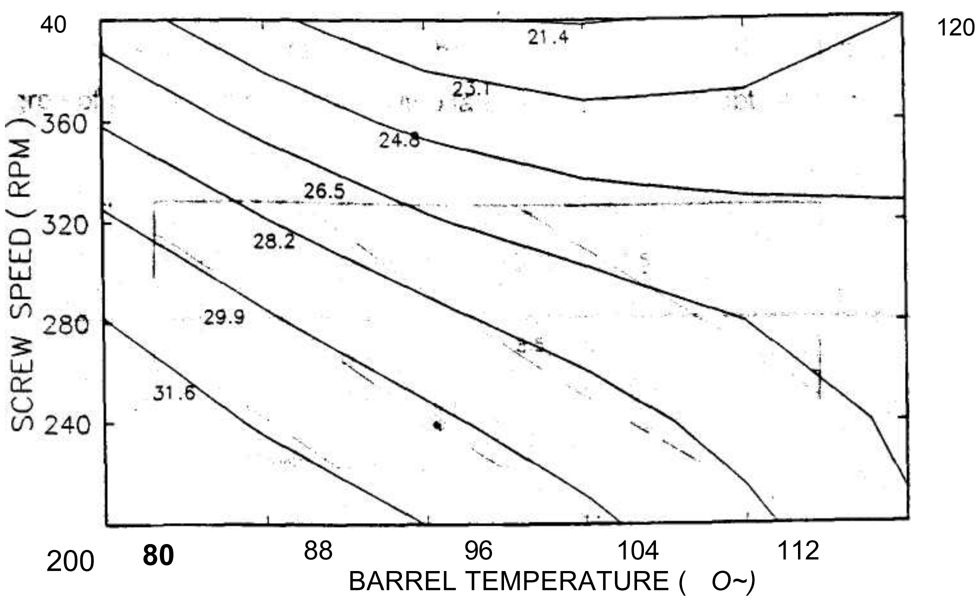


Fig. 5.3. Contour plot of the peak viscosity (indicated on lines) of the extrudate slurries during the heating phase while pasting in the RVA, for different barrel temperatures and screw speeds.

5.3.2. Hot paste viscosity

The hot paste viscosity (HPV) of the uncooked rice flour paste was about 80 RVU, whereas, for the extruded rice flour it ranged from 7.1 to 11.8 RVU. Fig. 5.4 shows the contour plot for HPV; it behaved in the same way as PV did. A fairly linear decrease in HPV was obtained when temperature or screw speed was elevated. Similar results for PV and HPV were reported by Tipples (1980); Mason and Hoseney (1986), who observed a decrease in the intensity and the degree of cooking (gelatinization) with increase in die/barrel temperature and screw speed.

Application of thermal and mechanical energy could result not only in a structural breakdown of starch granules but also in molecular degradation with subsequent loss of integrity and disintegration (Cai and Diosady, 1993; Gomez and Aguilera, 1983; Holm et al, 1988 a, b). Pregelatinized and degraded extruded starch granules therefore lose their ability to swell upon heating in water, resulting in low PV and HPV. Generally, the intensity and the extent of breakdown of starch will depend on the type of starch, mechanical shear, temperature and chemical agents present during the gelatinization (El-Dash et al, 1984).

Since PV is inversely related to the degree of gelatinization, it is evident that lowest degree of gelatinization (data is cited later in Fig. 5.6) was obtained at 80°C while

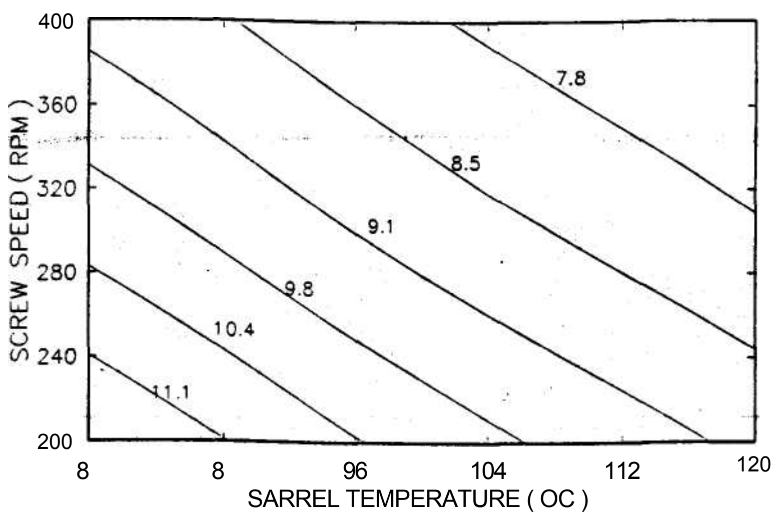


Fig. 5.4. Contour plot of the hot paste viscosity (indicated on lines) of the extrudate slurries during the heating phase while pasting in RVA, for different barrel temperatures and screw speeds.

the maximum gelatinization was obtained at 100°C. It may thus, be concluded that the degree of gelatinization and peak viscosity of extruded rice product could be effectively controlled by controlling the extruder barrel temperature and screw speed.

5.3.3. Cold paste viscosity

The cold paste viscosity (CPV) of extruded products (at the end of cooling to 50°C) ranged between 8.0 and 15.0 RVU (Fig. 5.5), whereas, for raw rice paste it was 177 RVU. The difference amongst the various extruded samples was thus extremely low, i.e. only 7 RVU. This trend was also seen in the case of HPV, in which the difference was less than 4. These insignificant differences perhaps could be ignored, with an overall conclusion that there was hardly any difference amongst the samples studied with respect to these properties. However, one can still see that a trend existed in the extent of this difference.

A better perception of the extent of retrogradation and gelling ability could be indexed by the rise in viscosity from HPV to CPV, which has been termed as "Total set back" by Bhattacharya and Sowbhagya 1978. The total set back values thus lay between

1 to 3 RVU in comparison to 97 RVU for raw rice, highlighting that the molecular degradation was to such an extent that it did not permit a strong "gelling" network upon cooling to 50°C.

The cold paste viscosity decreased with increasing barrel temperature and screw speed; similar results on the effect of temperature were reported for corn grits (Anderson et al, 1970) and on the effect of screw speed on corn starch (Owusu-Ansah et al, 1983).

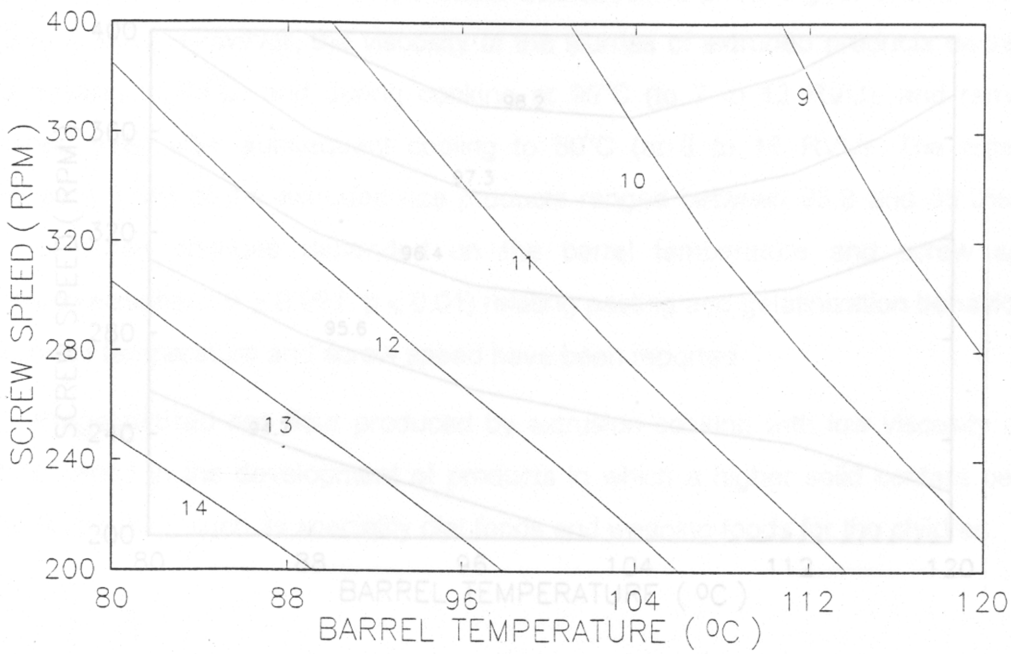
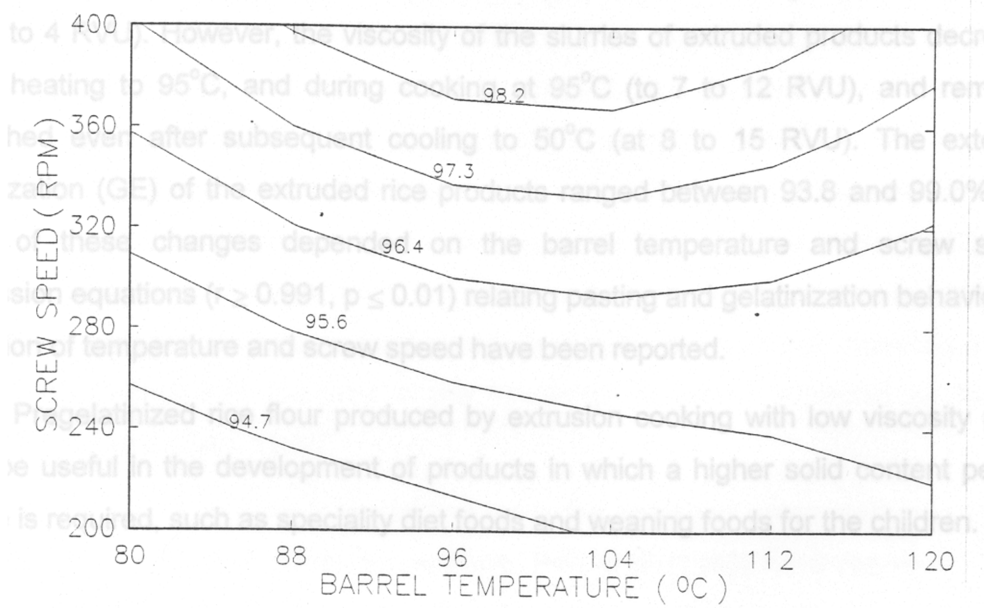


Fig. 5.5. Contour plot of the cold paste viscosity (indicated on lines) of the extrudate slurries during the cooling phase while pasting in RVA, for different barrel temperatures and screw speeds.

5.3.4. Extent of gelatinization (GE)

The extent of gelatinization (GE) of the extruded rice samples was high, and ranged between 93.8 and 99.0% (Fig. 5.6). However, within this range, an increase in screw speed appeared to result in higher GE values, which could be attributed to the low residence time at elevated screw speeds. Initially, GE increased with barrel temperature, and reached a maximum (99.0%) at 100°C with a screw speed of 400 rpm. Thereafter, a slight decrease was observed with increasing screw speed and barrel temperature. Similar results have been reported for corn starch by Owusu-Ansah et al, (1983), who observed that maximum gelatinization occurred at 100°C when the feed had a moisture content of 23%, which however, decreased slightly with further increase in barrel temperature. This could perhaps also be due to a higher extent of degradation produced by application of a higher mechanical (shear) force on account of the screw profile selected, or due to reduced swelling on account of the formation of resistant starch (Russell et ai, 1989), starch-lipid complexes (Mercier et ai, 1980), starch-protein complexes or even retrograded amylose (Cruzy Celis et ai, 1996).



5. 6. Contour plot of the extent of gelatinization (indicated on lines) of the extrudate slurries determined from the pasting behaviour In RVA, for different barrel temperatures and screw speeds.

The desirable pasting characteristic of a pregelatinized rice flour for use in speciality diet food formulations, for increasing the calorie density in these diets the low viscosity upon making slurries for preparation. Indexing this in the viscographic terms it would mean that the extruded rice flour paste should have low values of PV, HPV and CPV. These are obtainable when the extent of gelatinization is high, by extrusion at a high screw speed (400 rpm) coupled with a medium to high barrel temperature (100-110°C).

5. 4. SUMMARY

The effect of extrusion barrel temperature (80-120°C) and screw speed (200-400 rpm) on the pasting and gelatinization properties of extruded rice products was studied. A twin-screw extruder was employed without using a die. The pasting study was conducted using a Rapid Viscoanalyser (RVA), and the parameters determined were the initial viscosity (IV), peak viscosity (PV), hot paste viscosity (HPV) and cold paste viscosity (CPV).

The extrusion process parameters (barrel temperature and screw speed) markedly affected the pasting properties and the extent of gelatinization of rice flour. The initial viscosity of the extrudate suspensions was about 10 times higher than that of raw rice (3 to 4 RVU). However, the viscosity of the slurries of extruded products decreased during heating to 95°C, and during cooking at 95°C (to 7 to 12 RVU), and remained diminished even after subsequent cooling to 50°C (at 8 to 15 RVU). The extent of gelatinization (GE) of the extruded rice products ranged between 93.8 and 99.0%. The extent of these changes depended on the barrel temperature and screw speed. Regression equations ($r \geq 0.991$, $P \leq 0.01$) relating pasting and gelatinization behaviour as a function of temperature and screw speed have been reported.

Pregelatinized rice flour produced by extrusion cooking with low viscosity profile could be useful in the development of products in which a higher solid content per unit volume is required, such as speciality diet foods and weaning foods for the children.

CHAPTER - VI

Molecular Degradation Of Starch During Extrusion Cooking Of Rice

6.1. INTRODUCTION

Extrusion cooking involves conversion of shear and frictional energy to heat, which is used to cook the feed material (Hauck, 1981). A large variation occurs in product viscosity due to heat and pressure in the extruder. The changes in molecular and crystalline properties of starchy foods during extrusion cooking are highly dependent upon the condition of the extrusion and post extrusion drying. Processes involving high pressure / temperature and high shear have been known to cause partial or complete destruction of crystalline structure of starch, and considerable macro-molecular degradation, as shown in the case of puffing and popping of rice (Chinnaswamy and Bhattacharya, 1986; Murugesan and Bhattacharya, 1989), drum drying and extrusion cooking of wheat (Colonna et al, 1984; Schweizer and Reimann, 1986; Abdel- Aal et al, 1992). The type and severity of thermal processing employed therefore, imparts specific properties to the respective starch-based products. Changes in the two components of starch viz., amylose and amylopectin (which are present in different proportion in the native starch granules specific to the source materials and the cultivar) are reflected in changes in the functional properties exhibited by the end product (Jane and Chen, 1992).

An important variable in controlling extrusion operation parameters and product properties is the extruder barrel temperature. Pan et al (1992) have reported that the power consumption of the extruder and the shear force were more affected by the barrel temperature than by the screw speed. They also showed that the variety (rice having amylose content between 0.1% and 26%) plays an important role in determining the type and properties of extruded products. Cai et al (1995) studied the degradation of wheat starch at different barrel temperature. Wen et al (1990) have conducted detailed studies on starch fragmentation of corn meal under 15 different extrusion variables including barrel temperature.

The effect of barrel temperature on the macromolecular and functional properties of corn starch has also been studied by Chinnaswamy et al (1989); Chinnaswamy and Hanna (1990) and Wen et al (1990). In comparison to corn and wheat, very little work has been reported on extrusion of rice. Granular and molecular structure of rice starch differs from those of corn and wheat, so the properties of rice extrudate are expected to be different from corn and wheat extrudate. The process of extrusion generally involves both thermal treatment and mechanical shearing in which tissue damage is likely to occur altering the products qualities further. Gap in the information on the effects of barrel temperature on thermal and molecular properties of extrudate from rice, varying in the amylose / amylopectin ratio, thus warrants studies to focus on these aspects.

The present chapter reports the results of the work carried out to address the above aspects, specifically directed towards elucidation of macromolecular changes in starch taking place during extrusion of rice as a function of barrel temperature. The main objectives of the work were:

a) To study the product profile in relation to the amylose / amylopectin content of feed, and

b) To understand the macromolecular properties of extrudates.

6.2. EXPERIMENTAL

6.2.1. Rice

Rice flour prepared from three paddy cultivars (IR 64, Pojo bora and Agoni-bora) was used. The flour was passed through a 24mesh (8S) sieve and used for extrusion cooking. The proximate composition of rice flour has already been presented (Table 2.1, Chapter II).

6.2.2. Extruder and Extrusion cooking

The Werner and Pfleiderer extruder (see Section 2.2) was used with a die having a cylindrical aperture of 5 mm diameter. The extruder screw consisted of forward pitch screw element, a reverse pitch screw element (near the outlet), and also five kneading blocks (Table 6.1). The temperature of the extruder barrel was maintained at 80, 100 or 120°C throughout the last three zones of the barrel, whereas the first two zones (feed end) were maintained at 50°C. The screw speed, feed rate and moisture content of the feed were maintained constant at 400 rpm, 15 kgh^{-1} and 20 ± 0.2% respectively. All extrusion trials were repeated at least once.

The rice extrudates after drying were ground to flour (as mentioned under Section 2.4) to pass through a 100mesh (BS) sieve

Table 6.1

Screw profile used for extrusion trials

Types of screw element	Screw element Details	Total Length (mm)
Forward pitch	42/42	187
	42/21	21
	28/14	14
	28/28	168
	28/14	14
Kneading block[s]	45/5/20	20
Forward pitch	20/20	120
Kneading block[b]	45/5/14	14
Forward pitch	20/20	80
Kneading block[b]	45/5/14	14
Forward pitch	20/20	120
Kneading block[b]	45/5/14	14
Forward pitch	20/20	80
Kneading block[b]	45/5/14	14
Forward pitch	20/20	40
Reverse pitch	-20/20	10
Forward pitch	20/20	20

The Kneading blocks are composed of five mixing disks placed at an angle of 45° and have a total length of 20 mm for kneading block[s] and 14 mm for kneading block[b].

Defatting

Rice flour used for gel permeation chromatography (GPC) was first defatted by refluxing with 85% methanol in a Soxhlet apparatus for 18-20 h, as described by Sowbhagya and Bhattacharya (1971).

6.2.3. Gel permeation chromatography (GPC) of total starch

Gel permeation chromatography of starch was carried out essentially as per the method of Chinnaswamy and Bhattacharya (1986) using flour itself, rather than isolated starch, as isolation of starch from the processed sample was extremely difficult. About 100 mg defatted flour was taken in a 100ml conical flask, One millilitre distilled alcohol and 10 ml of 1N NaOH were added to it and left overnight. Next morning the mixture was heated under nitrogen atmosphere for 10 min on a boiling water bath with occasional mixing. After cooling, the dispersion was neutralized with the 1*N* hydrochloric acid using phenolphthalein as an indicator. The solution was filtered through a G-4 sintered glass filter. When perfectly dispersed, the solution became clear, though rather opalescent. The carbohydrate content of the dispersion was measured by phenol-sulphuric acid reagent method (Dubois et al, 1956). The flour dispersion containing exactly 10 mg (d,b.) carbohydrate was fractionated by ascending chromatography on a Sepharose Cl-2B gel column (Pharmacia, 1.6 x 70 cm) operating with the peristaltic pump at a flow rate of 15 ml/h, using distilled water containing 0.02% sodium azide as eluent. Three millilitre fractions were collected.

The void volume (V_o) and the total volume (V_J) of the gel column were determined using isolated waxy starch and glucose respectively by noting their elution volumes, The molecular weight of the peaks of GPC fractions were determined from the elution volume, read on a curve prepared by plotting the elution volumes of dextran standards (T_{20} to T_{2000}, Pharmacia Fine Chemicals, Sweden) of different molecular weights.

6.2.4. Chemical analysis

Carbohydrate content of the each 3ml sub-fraction was estimated by phenol-sulphuric acid reagent method (Dubois et al, 1956). To an aliquot (0,5 ml) of each subfraction, 0.5 ml distilled water and 1 ml of 5% phenol (w/v) were added. Five millilitre concentrated sulphuric acid was added rapidly on to the liquid. The solution was mixed thoroughly using a cyclo-mixer and cooled. The absorbance of the characteristic yellow-orange colour developed was measured at 490 nm against a reagent blank in a Shimadzu UV-1601 spectrophotometer substituting distilled water for the sample. The carbohydrate content was determined using standard glucose (dried in vacuum oven at

50°C for 20 h) solution (10 mg/100ml). The glucose equivalent obtained was multiplied with the factor 0.9 to express as starch or anhydroglucose (Lyne, 1976). An overall mass balance was calculated to ensure that the recovery of carbohydrate from **the** column was reasonable (see results).

To the aliquot (2.5 ml) of each subfraction, remaining after estimation of carbohydrate, 0.2 ml of 0.2% iodine (2 g of 1_2 and 20 g of KI per litre) was added for determination of amylose equivalent. The colour developed was read in a Shimadzu UV-1601 spectrophotometer at 630 nm against iodine blank. The amylose equivalent (total amylose) was calculated against the absorbance of a standard potato amylose solution (prepared as described under the Section 2.5) and treated similarly with iodine.

The absorption maximum ($_{Amax}$) of the iodine-polysaccharide complex was also determined by scanning the solution between 400 to 800 nm using a Shimadzu UV-1601 spectrophotometer. The gel chromatography was carried out at least in duplicate and the mean values are reported.

6.3. RESULTS AND DISCUSSION

6.3.1. Macromolecular degradation

Gel permeation chromatography (GPC) profile of starch

The macromolecular changes in starch as a result of extrusion were studied by gel permeation chromatography (GPC) of the extruded products.

Upon fractionation on Sepharose CL-2B gel column, starch in all rice samples was separated into two main fractions, a high molecular weight fraction eluting at the void volume (Fraction-I) and the other, a relatively low molecular weight one, that entered the gel and was eluted over a wide range (Fraction-II). Fraction-I, the high molecular weight void volume fraction, is generally considered as the branched component of starch i.e. amylopectin, and Fraction-II, mainly the linear component i.e. amylose (Biliaderis et al, 1979, 1981; Chinnaswamy and Bhattacharya, 1986). The recovery of carbohydrates in the chromatographed fractions ranged between 73.5% and

96.8% with a mean of 85.1%. Figs. 6.1 to 6.3 show chromatograms of raw an extrudate products from three different varieties.

Table 6.2 shows data on the proportion of carbohydrate in Fraction-I, and Fraction-II and the molecular weight of peak of Fraction-II.

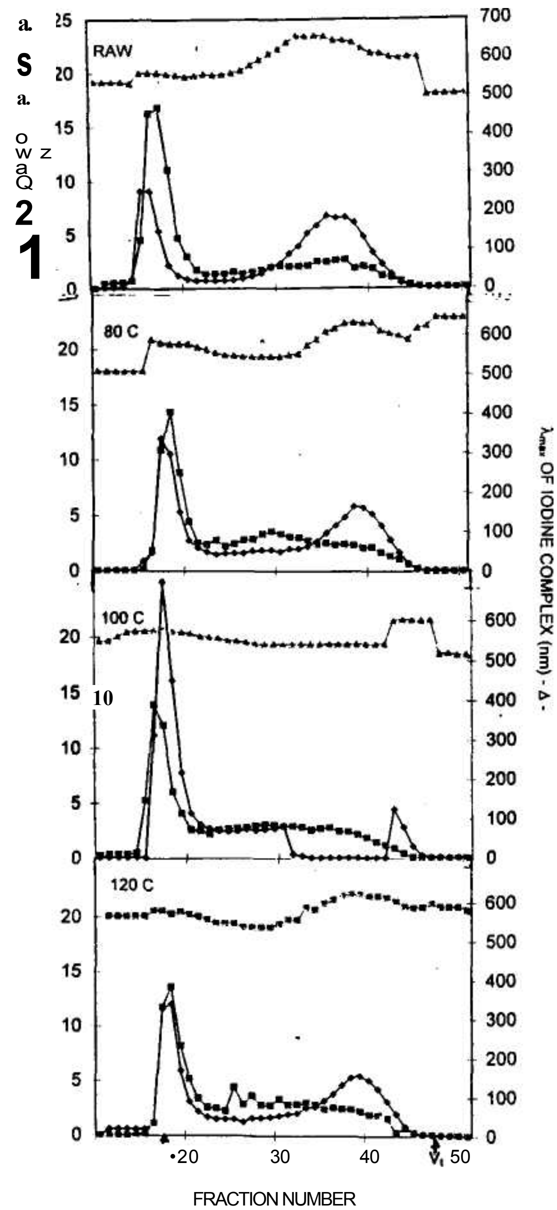


Fig. 6.1 GPC profiles for raw and extruded rice flour at different barrel temperatures from IR 64 variety.

Fraction-I represented 61.6%, 66.4% and 85.4% of the material eluted from the column for the unprocessed raw rice flour from IR 64, Pojo bora and Agoni bora cultivars respectively. The most obvious result of these chromatograms was the reduction in the amount of void volume carbohydrate in all the extruded products relative to unprocessed rice flour. Depending on the extrusion barrel temperature and the rice varieties, only 40.4% to 55.3% of the sample was excluded by the gel in comparison to 61.6 to 85.4% for the unprocessed rice flour (Table 6.2). The present values are in corroboration to the observations published by Colonna and Mercier (1983) for extruded manioc starch, and Davidson et al (1984) for wheat starch. It is apparent therefore, that significant degradation of starch occurred during extrusion processing.

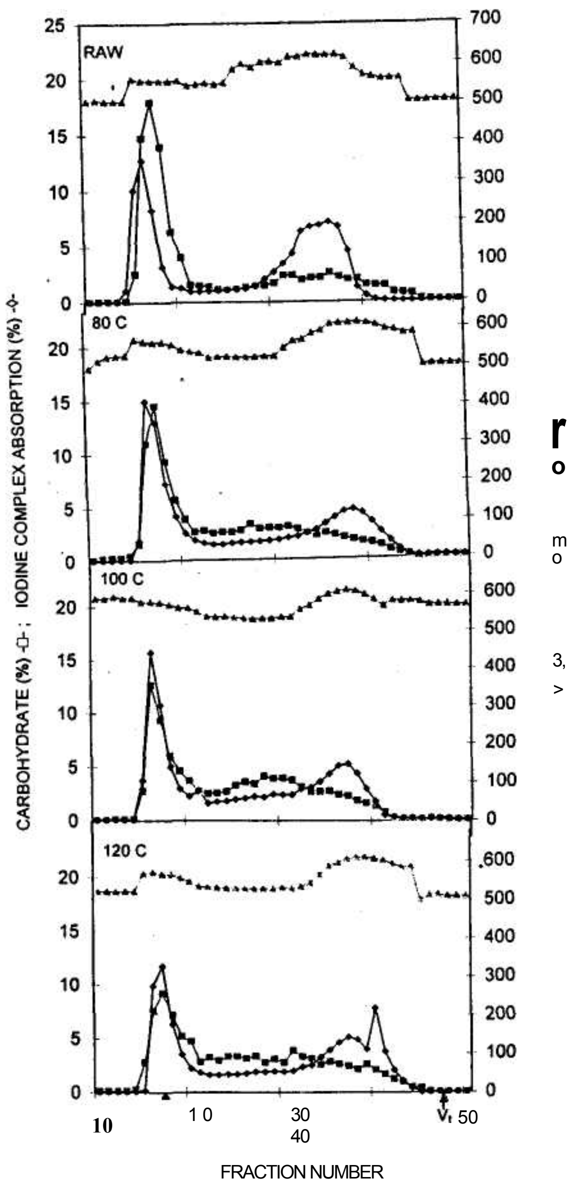


Fig. 6.2 GPC profiles for raw and extruded rice flour at different barrel temperatures from Pojo bora variety.

It could also been seen from Table 6.2, that the carbohydrate content in Fraction-I of the total starch decreased for all the varieties with consequent increase in Fraction-II to 49.8%, 55% and 59.6% respectively. Further, among the three varieties, the reduction of Fraction-I was maximum (45% points) in case of waxy rice variety. In case of high amylose variety (IR 64) on the other hands, the decrease was low (11% points). Since the degraded products of high molecular weight component were eluted along with the low molecular weight products as one broad peak, it could not be ascertained whether the amylose (linear fraction, Fraction-II) also underwent degradation.

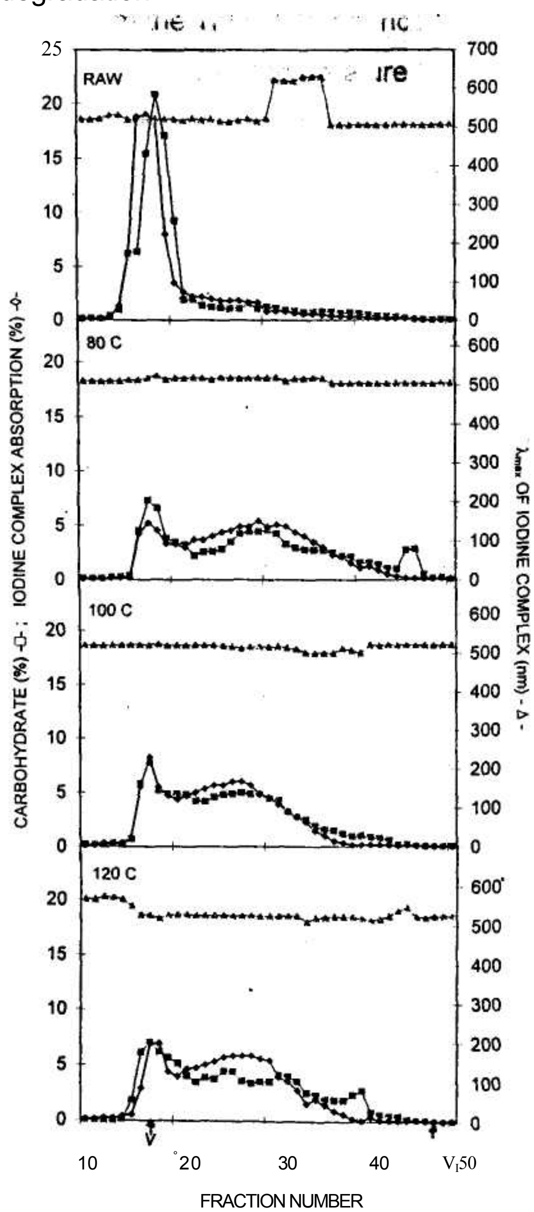


Fig. 6.3 GPC profiles for raw and extruded rice flour at different barrel temperatures from Agoni bora variety.

The extent of molecular degradation of starch in rice flour depends not only on the severity of extrusion but also on the variety of rice. As noted above, waxy rice variety was degraded to the maximum extent as compared to the high amylose variety under similar extrusion cooking conditions. Overall, it appears that the high molecular weight branched molecules (Fraction-I) were more prone to degradation than the linear ones (Fraction-II). This may be due to the large molecular size of Fraction-I, which renders it vulnerable to degradation under severe conditions of shear and thermal forces within the extruder.

It could be also seen from the Table 6.2 that rice flour of waxy variety Agoni bora was degraded to the maximum at lower temperature (80 °C) as compared to that for high amylose variety. The degradation increased with increasing barrel temperature for all three varieties whereas at higher temperature at 120°C, reverse trends could be observed. This may be due to formation of resistant starch or starch-lipid I starch-protein complexes at higher temperatures.

Further, it could also be noted from Figs. 6.1 to 6.3 and Table 6.2 that the peak of the Fraction-II of all extruded rice samples were eluted at a slightly lesser elution volume (i.e. lower K_{av}) than that of unprocessed rice flour, indicating a shift of this fraction towards a higher molecular weight profile. The tendency of this shift of Fraction-II peak towards the higher molecular weight side increased with increasing barrel temperature of extrusion and also with lower amylose content variety. The average molecular weight of peak of Fraction-II also increased with the increasing barrel temperature. This indicated that the degraded intermediate products had larger molecular size.

Table 6.2

Proportion of carbohydrate content (starch) in different fractions (Fr-I, Fr-II) and average molecular weight of peak of Fr-" of raw and extruded products from three rice varieties

Variety	Barrel temperature (°C)	Carbohydrate (%), in Fr I	Fr II	Kay Fr-II	Mw(10^5) Fr-II
IR	Nil (Raw)	61.6	38.4	0.56	9.5
	80	54.4	45.6	0.53	12.0
	100	50.2	49.8	0.48	18.0
	120	52.6	47.4	0.42	29.0
Pojo bora	Nil (Raw)	66.4	33.6	0.56	9.5
	80	55.3	44.7	0.45	27.0
	100	45.0	55.0	0.41	31.0
	120	47.3	52.7	0.37	40.0
Agoni bora	Nil (Raw)	85.4	14.6	0.44	24.0
	80	40.4	59.6	0.41	31.0
	100	43.0	57.0	0.35	47.0
	120	43.2	56.8	0.31	65.0

6.3.2. Iodine binding nature of GPC fractions

Table 6.3 presents data on the distribution of iodine absorbance in GPC fractions of total starch, and absorption maxima of Fraction-I and II of raw and extruded rice products from the three varieties studied. The distribution of iodine absorption of the two starch fractions separated on Sepharose CL-28 gel column, showed a progressive increase in Fraction-I in case of IR 64 and Pojo bora varieties. Thus about 33% and 42% of total iodine absorption by Fraction-I in raw rice in these varieties increased to 65% and 52% respectively in the extruded samples. The proportion of iodine absorption in Fraction-II consequently decreased and was accompanied by a continuous decrease in the $A_{max}$of Fraction-II peak. The $A_{max}$of raw rice of these varieties thus decreased from 656 nm and 621 nm to 612 nm and 608 nm for extruded product respectively. However, the $A_{max}$for Fraction-II of Agoni bora remained approximately constant at 522 nm before and after extrusion.

Table 6.3

Distribution of iodine absorbance in GPC fractions of total starch and absorption maxima of Fr-II of raw and extruded rice products.

Variety	Barrel temperature (°C)	I2-absorbance Fr I	(%) Fr II	Amax of Fr-I peak	X max of Fr-II Peak
IR64	Nil (Raw)	32.7	66.6	561	656
	80	42.1	57.9	574	631
	100	65.0	35.0	577	612
	120	44.4	55.6	574	621
Pojo bora	Nil (Raw)	42.1	57.9	557	621
	80	51.7	48.3	574	617
	100	50.1	49.9	575	608
	120	42.5	57.5	574	612
Agoni bora	Nil (Raw)	87.0	13.0	520	522
	80	39.9	60.1	525	520
	100	56.7	43.3	526	518
	120	52.7	47.3	530	523

The absorption maxima ($_{Ama}$x) of iodine complex of starches of the Fraction-I of all the three varieties showed an increase after extrusion cooking (Table 6.3). This increase was more in nonwaxy variety than waxy. Raw (unprocessed) rice of IR 64 and Pojo bora varieties had A_{max} of 561 nm and 557 nm respectively which increased to 576 nm and 574 nm after extrusion respectively. The A_{max} of Fraction-I of Agoni bora (waxy), on the other hand, showed a lesser increase from 520 nm for raw rice to 530 nm for extruded samples.

It could be concluded from the results presented in Table 6.3 that the A_{max} of Fraction-I increased after extrusion and that of Fraction-II decreased. Similar to the observations made by Chinnaswamy et al (1989) with regard to the extrusion of corn

of Fraction-II for waxy rice remained more or less constant at 522-520 nm before and after extrusion. The iodine absorption of Fraction-I increased upon extrusion and it was accompanied by an increase in its A_{max}.,

CHAPTER - VII

Effect Of Varietal Variation Of Rice & Barrel Temperature During Extrusion Cooking On Functional Properties Of The Extrudate

7.1. INTRODUCTION

A large proportion of all human food energy is derived from of cereal or starch-based products (Harper, 1981). Apart from providing energy, starch also contributes to the texture and structure of the food we eat. Gelatinization, molecular degradation and / or reassociation during the processing to product govern the final textural and functional profile of the product. Extrusion cooking is no exception. Under high pressure and shear conditions that exist in the cooking extruder, starches can be liquefied without enzymatic hydrolysis (Suzuki et al, 1976; Olkku and Linko, 1977). Mercier and Feillet (1975) have reported that cereal starches were rendered soluble in cold water using a twin-screw extruder at 170°C to 200°C. Wheat starch was also broken down to low-molecular-weight sugars at high temperature and pressure (Lorenz and Johnson, 1972; Chiang and Johnson, 1977).

The subject of starch extrusion has been reviewed by many workers (Linko et al, 1981; Fontanet et al, 1997 and Grenus et al, 1993). It has been shown that pure starches give maximum expansion upon extrusion cooking. Other transformations that occur during extrusion processing of starch result in changes in the functional properties, such as water absorption capacity, water solubility, breaking strength and rheological behaviour of flour slurry of the extruded product.

The changes in functional properties of starchy foods during extrusion cooking are highly dependent upon the conditions of extrusion and variety of starch source. The severity of extrusion cooking and the composition of starch in the variety of rice cultivar employed, therefore, impart specific properties to the respective starch-based extruded product.

The objective of this study was to evaluate the effect of varietal variation in feed (i.e. amylose content of rice) and extrusion temperature on functional and textural properties of extrudates.

7.2. EXPERIMENTAL

7.2.1. Rice

Rice flour used for the extrusion cooking was the same as described in the in previous chapter under Section 6.2.1. ~

7.2.2. Extruder and extrusion cooking

The extruder and the extrusion cooking conditions were also same as described in the previous chapter under Section 6.2.2.

7.2.3. Bulk density (P_s) of extruded product

The bulk density (P_B) of individual, dry cylindrical extrudate rods was estimated by determining the mass and volume of the rod pieces. The volume of the cylindrical rod was calculated by determining the average diameter of the individual rod piece and its length by using a dial thickness gauge having a resolution of 0.01mm. At least 10 replicates were run by collecting random samples and average value was taken for computation.

7.2.4. Expansion ratio (ER)

The expansion ratio (ER) of the dried extruded rod was measured as the ratio of the cross-sectional area of the extruded rods to that of the die hole through which it was extruded. The ER values were obtained from ten random samples of five observations on each sample and averages were reported.

7.2.5. Warner- Bratzler shear stress (W-B SS)

The Werner-Bratzler shear force for shearing the dry extrudates across their cross-section was determined by dividing the maximum force during shearing by the corresponding cross-sectional area of the extrudate with a Werner - Bratzler shear attachment to the Instron Universal Testing Machine (Model No. 4301, Instron Corporation, Canton, Massachusetts, USA). The crosshead speed of 100 mm/min and

Table 7.1

Effect of amylose content in rice and extrusion barrel temperature
on some functional properties of extrudates

Variety	Temp .(°C)	P_B (kg m^{-3})	WSI (%)	WAI (g/g)	ER	W-B SS (kPa)	SME (kJ kg^{-1})	T (%)
IR64	80	371±3.6	26.0±0.6	6.1±0.3	4.6 ± 0.4	539±4.6	443±1.7	35±1.7
	100	280±2.0	35.8 + 0.3	7.0±0.5	8.8 ± 0.3	353±3.0	338±2.6	30 + 1.0
	120	196±1.7	34.0±1.1	6.4±0.3	10.8±0.9	402±2.6	296±3.5	28±0.6
Pojo bora	80	327±2.6	26.7±1.4	6.7±0.4	6.0 ± 0.4	519±2.6	655±4.3	45 ± 0.9
	100	239±2.0	31.8±2.1	5.8±0.8	12.3±0.6	245±3.4	591±2.6	42±0.6
	120	237±2.6	32.0±1.3	5.8± 0.9	11.0±0.7	314±3.5	570±2.6	40±1.0
Agonibora	80	362±2.6	78.8±2.4	1.9±0.6	5.6±0.2	343±3.6	781±3.6	51±0.8
	100	271±1.7	83.5±3.4	1.4±0.6	8.5±0.5	333±2.8	696±1.6	47 ± 0.6
	120	174±2.6	85.3±1.4	1.2±o.3	14.7±0.7	176±3.6	718±1.7	48±0.8

7.3.3. Bulk density (P_B)

The bulk density of the rice extrudate from three different varieties ranged between 371 and 174 kgm^{-3}. The bulk density decreased with increasing barrel temperature. Similar observations have been reported by many researchers (Gogoi et ai, 1996; Bhattarcharya and Choudhury, 1994). In general, bulk density decreased with decreasing amylose content of the feed (Table 7.1). High amylose variety (IR 64) yielded a product with highest bulk density of 371 kgm^{-3} at 80°C, and Agoni bora (waxy) variety resulted in the least bulk density of 174 kgm^{-3} when extruded at 120°C barrel temperature.

7.4. SUMMARY

The effect of barrel temperature (80-120°C) during extrusion and the amylose content (5-28.6%) of feed (rice cultivar) on extrusion system parameters in a twin-screw extruder and extrudate characteristics was studied. The feed rate (15 kgh^{-1}), moisture content (20.0 % ± 0.2) of feed, and the screw speed (400 rpm) were kept constant. The extrusion system parameters studied were net specific mechanical energy (SME) and the torque (T).

The bulk density (P_B), water solubility index (WSI), water absorption index (WAI) expansion ratio (ER) and Warner-Bratzler shear stress (W-B-SS), as affected by the barrel temperature and varietal variation of feed, were investigated.

Experimental data on system parameters and extrudate characteristics were fitted to a second-degree polynomial. Regression equations ($r \geq 0.904$, $p \leq 0.01$) that relate extrusion system parameters and extrudate characteristics to barrel temperature and varietal variation were reported.

The bulk density and W-B-SS generally decreased with increasing barrel temperature and decreasing amylose content of the feed. Solubility index (85.3%) and net specific mechanical energy (781$kJkg^{-1}$) were highest in case of low amylose variety of rice. The torque during extrusion decreased with increasing barrel temperature. Low amylose rice variety (Agoni bora) yields an extruded product with least bulk density (174 kgm^{-3}) and with a least W-B-SS value (176kPa). Expansion ratio (14.7) of the extrudate suggested that a barrel temperature of 120°C is desirable to generate an expanded rice product from waxy rice cultivar.

CONCLUSIONS

Following conclusions could be drawn from the results presented in the preceding chapters on the extrusion cooking of rice flour using a twin screw extruder covering a wide range of variables and analysis of the extruded product characteristics:

The Placket-Burman experimental design could serve as an effective tool to screen a large number of variables and to reduce the number of experiments. Ten variables were used in the initial experiments (barrel temperature, feed rate, screw speed, presence or absence of mixing disk and reverse pitch screw elements, feed moisture, particle size, total amylose content, sugar and salt) with application of the above design to identify the main determinants of the extrusion characteristics, (torque, T; specific mechanical energy, SME; and average residence time, RT), and product attributes (water solubility index, WSI; water absorption index, WAI; bulk density, BO and viscosity indices) as response functions.

The response functions were mostly affected by the reverse pitch screw element and mixing disk indicating the importance of the screw profile on extrusion and extruded characteristics. Considerable effect was also observed for amylose and moisture content, barrel temperature, feed rate and screw speed, whereas particle size and other additives showed lesser effect

From the detailed studies on the extrusion cooking of rice flour of a high amylose variety at constant feed rate and moisture content with variable barrel temperature and screw speed, it emerged that the system parameters (torque and SME) and extrudate attributes (WSI, WAI, BO, Sediment volume and *in-vitro* starch digestibility) were mainly dependent on temperature. The screw speed imparted lesser effect. A positive linear relationship existed between SME and BO.

A study of pasting behaviour of flour slurry from extruded product using Rapid Viscoanalyser showed that on account of gelatinization and its extent, the pasting profile was entirely different. The viscosity was high at relatively low temperatures but decreased during heating and showed very little rise during subsequent cooling in comparison to flour slurry from raw rice.

A study of the molecular profile of starch from raw rice and extruded product from different varieties of rice varying in amylose content by using gel permeation chromatography showed that the high molecular weight component (amylopectin) was degraded into lower-molecular-weight components. The degradation was high when the proportion of amylopectin was high (waxy rice). Maximum degradation occured in waxy rice and at lower temperatures in comparison to non-waxy rice.

Among the three varieties studied, having different amylose content, waxy rice therefore appeared to be highly suitable for producing expanded rice products as it showed the highest ER, highest WSI and other desirable product profile. However, the specific mechanical energy required for extrusion was also high.

The above studies have also revealed that if the extruded products are to be used for production of pregelatinized, but high calorie density, finished products like baby food and weaning foods, the use of a die in the extruder is not essential and also would be preferable as the energy expenditure could be reduced.

BIBLIOGRAPHY

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Abdel-Aal, E.-S.M., Sosulski, F.W., Adel, A., Shehata, Y., Youssef, M.M. and Ibave, J.L. 1992. Effect of extrusion cooking on the physical and functional properties of wheat, rice and fababean blends. Lebensm. Wiss. u. Technol. 25: 21.

Akdogan, H., Rui, L. and Oliveira, J.C. 1997. Rheological properties of rice starch at high moisture contents during twin-screw extrusion. Lebensm. Wiss. u. Technol. 30: 488.

Akhnazarova, S. and Kafarov, V. 1982. Experiment Optimization in Chemistry and Chemical Engineering. MIR publishers. Moscow. pp: 151.

Akingbala, J.O. and Rooney, L.W. 1987. Paste properties of sorghum starch. J.Food Proc. Preserv. 1 1 : 13.

Altomare, R.E. and Ghossi, P. 1986. An analysis of residence time distribution patterns in a twin-screw extruder. Biotechnol. Prog. 2:157.

Altomare, R.E., Anelich, M. and Rakos, R. 1992. An experimental investigation of the rheology of the rice flour dough with an extruder-coupled slit die rheometer. In: Food Extrusion Science and Technology. J.L. Kokini, Ho, C. and M.V. Karwe (eds). Marcel Dekker, Inc. NY. pp: 233.

Anderson, R.A., Conway, H.F., Pfeifer, V.F. and Griffin, L.E.J. 1969a. Gelatinization of corn grits by roll and extrusion cooking. Cereal Sci. Today. 14: 4.

Anderson, R.A., Conway, H.F., Pfeifer, V.F. and Griffin, L.E.J. 1969b. Roll and extrusion cooking of grain sorghum grits. Cereal Sci. Today. 14: 372.

Anderson, R.A., Conway, H.F. and Peplinski, A.J. 1970. Gelatinization of corn grits by roll-cooking, extrusion cooking and steaming. Staerke. 22:130.

Anon. 1991. Rice-derived ingredient produces fatty texture and mouthfeel for use in low-fat applications. Food Technol.45: 264.

AOAC. 1984. Official Methods of Analysis. 14th edition. Association of Official Analytical Chemists, Arlington, VA.

Arnoldsson, K.C. and Kaufman, P. 1994. Lipid class analysis by normal phase high performance liquid chromatography, development and optimisation using multivariate method. Chromatographia. 38: 317.

Artz, W.E., Warren. C. and Villota, R. 1990. Twin-screw extrusion modification of a corn fibre and corn starch extruded blend. J. Food Sci. 55: 746.

Badrie, N. and Mellowes, W.A. 1991. Effect of extrusion variables on cassava extrudates. J. Food Sci. 56: 1334.

Bain, B.K. 1979. Extrusion Today. Cereal Foods World. 25:136.

Barres, C., Vergnes, B., Tayeb, J. and Della Valle, G. 1990. Transformation of wheat flour by extrusion cooking: Influence of screw configuration and operating conditions. Cereal Chern. 67: 427.

Bean, M.M. and Nishita, K.D. 1985. Rice flours for baking. In: Rice: Chemistry and Technology. B.O. Juliano (ed). Second edition, Am. Assoc. Cereal Chern. Inc., St. Paul, Minn. USA. pp: 539.

Bernfeld, P. 1955. Amylases, alpha and beta. In: Methods in Enzymology. S.P. Colowick and N.O. Kaplan (eds). Vol. I. Acad. Press. NY. pp: 149.

Bhattacharya, K.R. and Ali, S.Z. 1976. A sedimentation test for pregilatinized rice products. Lebensm. Wiss. u. Technol. 9: 36.

Bhattacharya, K.R. and Sowbhagya, C.M. 1978. On viscograms and viscography, with special reference to rice flour. J. Texture Stud. 9: 341.

Bhattacharya, K.R., Sowbhagya, C.M. and Indudhara Swamy, Y.M. 1982. Quality profile of rice: A tentative scheme for clarification. J. Food Sci. 47: 564.

Bhattacharya, M. and Hanna, M.A. 1986a. Viscosity modelling of dough in extrusion. J. Food Technol. 21: 167.

Bhattacharya, M. and Hanna, M.A. 1986b. Mathematical modelling of food extruder. Lebensm. Wiss. u. Technol. 19: 34.

Bhattacharya, M. and Hanna, M.A. 1987. Textural properties of extrusion-cooked corn starch. Lebensm. Wiss. u. Technol. 20: 195.

Bhattacharya, S. 1989. Extrusion cooking of food and its possible application in Indian food industry. Indian Food Ind. 8:1.

Bhattacharya, S. and Prakash, M. 1994. Extrusion cooking of blends of rice and chickpea flour: A response surface analysis. J. Food Engg. 21: 315.

Bhattacharya, S. and Choudhury, G.S. 1994. Twin-screw extrusion of rice flour: Effect of extruder length-to-diameter ratio and barre! temperature on extrusion parameters and product characteristics. J. Food Proc. Preserv. 18: 389.

Bhattacharya, S., Das, H. and Bose, A. N. 1992. Rheological behaviour during extrusion of blends of minced fish and wheat flour. J. Food Engg. 15:123.

Bhattacharya, S. and Narasimha,H.V. 1997. Puncture and tress relaxation behaviour of blackgram (Phaseolus mungo) flour-based papad dough. J. Food Proc. Engg. 20: 301.

Biliaderis, C.G., Grant,D.R. and Vose,J.R. 1979. Molecular weight distribution of legume starches by gel chromatography. Cereal Chem. 56: 475.

Biliaderis, C.G., Grant,D.R. and Vose,J.R. 1981. Structural characterization of legume starches. I. Studies on amylose, amylopectin and beta-limit dextrins. Cereal Chem. 58: 496.

Bressani, R., Braham, J.E., Elias, L.G., Ceuvas, R. and Molina, M.R. 1978. Protein quality of whole corn, whole soybean mixture processed by a simple extrusion cooker. J. FoodSci. 43: 1563.

Burns, E.E. and Gerdes, D.L. 1985. Canned rice foods. In: Rice: Chemistry and Technology. B.O. Juliano (ed).Second edition, Am. Assoc. Cereal Chem. Inc., St. Paul, Minn. USA. pp: 557.

Cai, W. and Diosady, L.L. 1993. A model for gelatinisation of wheat starch in a twin-screw extruder. J. Food Sci. 58: 872.

Cai, W., Diosady, L.L. and Rubin, L.J. 1995. Degradation of wheat starch in a Twin-screw extruder. J. Food Engg. 26: 289.

Cervone, N.W. and Harper, J.M. 1978. Viscosity of an intermediate dough. J. Food Proc. Engg. 2:105.

Chan, K.Y. and Kavanagh, P.E. 1992. Application of Plackett-Burman design and linear programming to light-duty liquid detergent formulation. J. Am.Oil Chem.Soc. 69: 690.

Chandrasekhar, M.R. 1988. Role of extrusion cooking in food processing. IFCON-1988 Proceedings. Assn. Food Scientists and Technologists (India), Mysore, India. Publication. pp: 49.

Charbonniere, R., Duprat, F. and Gulibot, A. 1973. Changes in various starches by cooking extrusion processing. II. Physical structure of extruded products. Cereal Sci. Today. 18; 9.

Cheftel, J.C. 1990. Extrusion cooking: Operating principles, research trends, and food applications. In: Processing and Quality of Food. P. Zeuthen, J.C. Cheftel, C. Eriksson, T.R. Gormley, P. Linko and K. Paulus (eds). Elsevier Sci. Publishers, London. pp: 1.60.

Chiang, C.Y. 1975. Gelatinization of starch in extruded products. Diss. Abstr. 36: 636.

Chiang, C.Y. and Johnson, J.A. 1977. Gelatinization of starch in extruded products. Cereal Chem. 54: 436.

Chinnaswamy, R. and Bhattacharya, K.R. 1983. Studies of expanded rice. Physicochemical basis of varietal differences. J. Food Sci. 48: 1600.

Chinnaswamy, R. **and Bhattacharya,** K.R. 1986. Characteristics of gel chromatographic fractions of starch in relation to rice and expanded rice-products qualities.Staerke. 38: 51.

Chinnaswamy, R., Hanna, M.A. and Zobel, H.F. 1989. Microstructural, physiochemical, and macromolecular changes in extrusion-cooked and retrograded corn starch. Cereal Foods World. 34: 415.

Chinnaswamy, R. and Hanna, M.A. 1990. Macromolecular and functional properties of native and extrusion cooked corn starch. Cereal Chem. 67: 490.

Clark, J.P. 1978a. Dough rheology in extrusion cooking. Food Technol. 32: 73.

Clark, J.P. 1978b. Texturization by extrusion. J. Texture Stud. 9:109.

Colonna, P. and Mercier, C. 1983. Macromolecular modifications of manioc starch components by extrusion cooking with or without lipids. Carbohydr. Polym. 3: 87.

Colonna, P., Melcion, J.P., Vergnes, B. and Mercier, C. 1983. Flow, mixing and residence time distribution of maize starch within **a** twin-screw extruder with a longitudinally-split barrel. J. Cereal Sci. 1:115.

Colonna, P., Doublier, J.L., Melcion, J.P., Monredon, F.De. and Mercier, C.1984. Extrusion cooking and drum drying of wheat starch. I. Physical and macromolecular modifications. Cereal Chem. 61: 538.

Colonna, P., Tayeb, J. and Mercier, C. 1989. Extrusion cooking of starch and starchy products. In: Extrusion Cooking. C. Mercier, P. Linko and J. Harper (eds). Am. Assoc. Cereal Chern. Inc., St. Paul, Minn. USA. pp: 247.

Conway, H.F. 1971a. Extrusion cooking of cereals and soybeans. Part I. Food Prod. Dev. 5: 27.

Conway, H.F. 1971b. Extrusion cooking of cereals and soybeans. Part II. Food Prod. Dev. 5: 14.

Conway, H.F. and Anderson, R.A. 1973. Protein-fortified extruded food product. Cereal Sci.Today. 18: 94.

Cruzy Celis, L.P., Rooney, L.W. and McDonough, C.M. 1996. A ready-to-eat breakfast cereal from food-grade sorghum. Cereal Chem. 73: 108.

Davidson, V.J., Paton, D., Diosady, L. and Rubin, L.J. 1984. A model for mechanical degradation of wheat starch in a single-screw extruder. J. Food Sci. 49:1154.

Deffenbaugh, L.B. and Walker, C.E. 1990. Use of the rapid visco-analyzer to measure starch pasting properties. Part II: Effect of emulsifiers and sugar emulsifier interactions. Starke. 42: 89.

de la Gueriviere, J.F. 1976. Principles of the extrusion-cooking process. Application to starchy foods. Bull. Anc. El. Ec.Fr. Meun. 276: 305.

de Mosqueda, M.B., Perez, C.M., Juliano, B.O., del Rosario, R.R. and Bechtel, D.B. 1986. Varietal differences in properties of extrusion-cooked rice flour. Food Chem. 19: 173.

Dubois,M., Gilles, K.A., Hamilton, J.K., Rebers, P.A. and Smith,F. 1956. Colorimetric method for dete~ination of sugars andrelated substances. Anal. Chem. 28: 350.

Dziezak, J.D. 1989. Single- and twin-screw extruder in food processing. Food Technol. 43: 163.

Dziezak, J.D. 1991. Romancing the kernel: A salute to rice varieties. Food Technol. 45: 74.

Eerlingen, R.C., Jacobs, H. and Delcour, J.A. 1994. Enzyme-resistant starch. V. Effect of retrogradation of waxy maize starch on enzyme-susceptibility. Cereal Chem. 71: 351.

El-Dash, A.A. 1981. Application and control of thermoplastic extrusion of cereals for food and industrial uses. In: Cereals: A Renewable Resource: Theory and Practice. Y. Pomeranz, and L. Munch (eds).). Am. Assoc. Cereal Chem. Inc., St. Paul, Minn. USA. pp: 165.

El-Dash, A.A., Gonzales, R. and Ciol, M. 1984. Response surface methodology in the control of thermoplastic extrusion of starch. In: Extrusion Cooking Technology. R. Jowitt (ed). Elsevier Applied Sci. publishers, NY. pp: 51.

Erdemir, M.M., Edwards, R.H. and Mc Carthy, K.L. 1992. Effect of screw configuration on mechanical energy transfer during twin-screw extrusion of rice flour. Lebensm. Wiss. u. Technol. 25: 502.

FAO. 1997. FAO Production Year Book, FAO. Rome. 51: 64.

Farrall, A.W. 1976. Food Engineering Systems. Vol.Operations. The AVI publishers. Westport.m CC. pp: 233.

Faubion, J.M. and Hoseney, R.C. 1982. High-temperature short-time extrusion cooking of wheat starch and flour. II: Effect of protein and lipid on extrudate properties. Cereal Chem. 59: 533.

Faubion, J.M., Hoseney, R.C. and Seib, P.A. 1982. Functionality of grain components in extrusion. Cereal Foods World. 27: 212.

Fontanet, I., Davidou,S., Dacremont, C. and Meste, M.L. 1997. Effect of water on the mechanical behaviour of extruded flat bread. J. Cereal Sci. 25: 303.

Frame N. D. 1994. Operational characteristics of the co-rotating twin-screw extruder. In: The Technology of Extrusion Cooking. Blackie Acad. And Professional. pp: 1.

Giovanni, M. 1983. Response surface methodology and product optimization. Food Technol. (Chicago). 37: 41.

Gogoi, B.K. 1994. Effects of reverse screw elements on energy inputs, residence times and starch conversion during twin-screw extrusion of corn meal. Ph.D Thesis, Rutgers Univ, New Jersey.

Gogoi, B.K. and Yam, K.L. 1994. Relationships between residence time and process variables in a corotating twin-screw extruder. J. Food Engg. 21:177.

Gogoi, B.K., Oswalt, A.J. and Choudhury, G.g. 1996. Reverse screw element (s) and feed composition effects during twin-screw extrusion of rice flour and fish muscle blends. J. Food Sci. 61: 590.

Gomez, M.H. and Aguilera, J.M. 1983. Changes in the starch fraction during extrusion-cooking of corn. J. Food Sci. 48: 378.

Gomez, M.H., Waniska, R.D., Rooney, L.W. and Lusas, E.W. 1988. Extrusion- cooking of sorghum containing different amounts of amylose. J. Food Sci. 53:1818.

Grenus, K.M., Hsieh, F. and Huff. H.E. 1993. Extrusion and extrudate properties of rice flour. J. Food. Engg. 18: 229.

Guilbot, A. and Mercier, C.1974. Nouveaux produits provenant de l'amidon de pomme de terre: Leur obtention et leur application. French patent. 74-14-373.

Guy, R.C.E. and Horne, A.W. 1988. Extrusion and co-extrusion of cereals. In: Food Structure: Its Creation and Evaluation. J.M.V. Blanshard and J.R. Mitchell (eds). Butterworth Press, London. pp: 331.

Guy, R.C.E. 1989. The use of wheat flours in extrusion cooking. In: Wheat is Unique. Y. Pomeranz, (ed). Am. Assoc. Cereal Chem. Inc., St. Paul, Minn. USA. pp: 369.

Guy, R.C.E. 1994. Raw materials for extrusion cooking processes. In: The Technology of Extrusion cooking. N.D. Frame (ed). Blackie Acad. and Professional. London. pp: 52.

Harper, J.M., Rhodes, T.P. and Wanniger, L.A (Jr). 1971. Viscosity model for cooked cereal dough. Chern. Engg. Prog. Symp. Ser. 67: 40.

Harper, J.M. and Harmann, D.V. 1973. Research needs in extrusion cooking and forming. Trans. ASAE. 16:941.

Harper, J.M. 1979. Food extrusion. Crit. Rev. Food Sci. Nutr. 1 1 : 155. FV

Harper, J.M. 1980. Cereal-fish protein blends. LEC Newsletter 4:1.

Harper, J.M. 1981. Extrusion of Food. Vol. II. CRC Press. Boca Raton, Florida. pp:1.

Harper, J.M. 1989. Food extruders and their applications. In: Extrusion cooking. C. Mercier, P. Linko and J.M. Harper (eds). Am. Assoc. Cereal Chern. Inc., St. Paul, Minn. USA. pp: 1.

Hauck, B.W. 1980. Marketing opportunities for extrusion cooked products. Cereal Foods

World. 25: 594.

Hauck, B.W. 1981. Control of process variables in extrusion cooking. Cereal Foods World. 26: 170.

Holm, J., Bjorck, I., Asp, N.G., Sjoberg, L.B. and Lundquist, I. 1985. Starch availability *in vitro* and *in vivo* after flaking, steam-cooking and popping of wheat. J. Cereal Sci. 3: 193.

Holm, J., Bjorck, I. and Eliasson, A.C. 1988a. Effects of thermal processing of wheat and starch: I. Physico-chemical and functional properties. J. Cereal Sci. 7. 249.

Holm, J., Bjorck, I. and Eliasson, A.C. 1988b. Effects of thermal processing of wheat and starch: II. Physico-chemical and functional properties. J. Cereal Sci. 8. 145.

Hoseney, R.C., Mason, W.R., Lai, C.S. and Guetzlaff, J. 1992. Factors affecting the viscosity and structure of extrusion-cooked wheat starch. In: Food Extrusion Science and Technology. J.L. Kokini, C.T. Ho and M.V. Karwe (eds). Marcel Dekker, NY. pp: 277.

Hsieh, F. and Luh, B.S. 1991a. Breakfast rice cereals and baby foods. In: Rice Utilisation. B.S. Luh (ed). Vol.II, AVI-VAN Nostrand Reinhold, NY.pp: 177.

Hsieh, F. and Luh, B.S. 1991b. Rice snack foods. In: Rice Utilisation. B.S. Luh (ed). Vol.II, AVI-VAN Nostrand Reinhold, NY.pp: 225.

Indudhara Swamy, Y.M., Ali, S.I. and Bhattacharya, K.R. 1971. Hydration of raw and parboiled rice and paddy at room temperature. J. Food Sci. Technol. 8: 20.

Jain, M.K., Iyengar, S.R.K, Jain, R.K. 1995. Numerical Methods for Scientific and Engineering Computation. 3rd Edition, New Age Int. Ltd. New Delhi, India. pp: 78.

Jane, J.-L. and Chen, J.-F. 1992. Effect of amylose molecular size and amylopectin branch chain length on paste properties of starch. Cereal Chern. 69: 60.

Juliano, B.O.1985. Production and utilisation of rice. In: Rice: Chemistry and Technology. B.a. Juliano (ed). Second edition, Am. Assoc. Cereal Chern. Inc., St. Paul, Minn. USA. pp: 1.

Juliano, B.O. and Sakuria, J. 1985. Miscellaneous rice products. In: Rice: Chemistry and Technology. B.a. Juliano (ed) .Second edition, Am. Assoc. Cereal Chem. Inc., St. Paul, Minn. USA. pp: 569.

Kelly, V.J. 1985. Rice in infant foods. In: Rice: Chemistry and Technology. B.a. Juliano (ed). Second edition, Am. Assoc. Cereal Chem. Inc., St. Paul, Minn. USA. pp: 525.

Kim, J.C. and Rottier, W. 1979. Veranderungen der Eigenschaften von Aestivum-weizen-Griess durch Extrusion. Getreide. Mehl Brot. 33:188.

Kim, J.C. and Rottier, W. 1980. Modification of aestivum wheat semolina by extrusion.

Cereal Foods World. 24: 62.

Kim, J.C. 1984. Effect of some extrusion parameters on the solubility and viscograms of extruded wheat flour. In: Thermal Processing and Quailtiy of Foods. P. Zeuthen, J.C. Cheftel, C. Eriksson, M. Jul, H. Leniger, P. Linko, G. Varela and G. Vos (eds). Elsevier Applied Sci. Publishers, NY. pp: 251.

Kim, C.H. and Maga, J.A. 1990. Wild rice in extruded products. Lebensm. Wiss.u. Technol. 23; 349.

Kirby, A.R., Cliett, A.L., Parker, R. and Smith, A.C. 1988. An experimental study of screw configuration effects in the twin-screw extrusion-cooking of maize grits. J. Food Engg. 8:247.

Kollengode, A.N., Sokhey, A.S. and Hanna, M.A. 1996. Physical and molecular properties of reextruded starches as affected by extruder screw configuration. J. Food Sci. 61:596.

Kumagai, H., Lee, B.H. and Yano, T. 1987. Flour treatment to improve the quantity of extrusion-cooked rice-flour products. Agric. Bioi. Chem. 51: 2067.

Lai, L.S. and Kokini, J.L. 1990. The effect of extrusion operating conditions on the on-line apparent viscosity of 98% amylopectin (amilo) and 70% amylose (Hylon 7) corn starches during extrusion. J. Rheology. 34:1245.

Launay, B. and Lisch, J.M. 1983. Twin screw extrusion cooking of starches: Behaviour of starch pastes, expansion and mechanical properties of extrudates. J. Food Engg. 2: 259.

Lawton, B.T., Henderson, G.A. and Derlatka, E.J. 1972. The effect of extruder variables on gelatinisation of corn starch. Can. J. Chem. Engg. 50:168.

Lee, S.Y. and McCarthy, K.L. 1996. Effect of screw configuration and speed on RTD and expansion of rice extrudate. J. Food Proc. Engg. 19:153.

Lelievre, J. 1976. Theory of gelatinization in a starch-water-solute system. Polymer. **17:** 854.

Levine, L. 1983. Estimating output and power of food extruders. J. Food Proc. Engg. 6:1.

Likimani, T.A., Maga, J.A. and Sofas, J.N. 1990. The rate of starch hydrolysis in extruded corn I soybean products. Lebensm. Wiss. u. Technol. 23: 226.

Lin, J.K., and Armstrong, P.J. 1990. Process variables effecting residence time distribution of cereals in an intermeshing, counter-rotating twin-screw extruder. Trans. ASAE. 33: 1971.

Linko, Y.Y., Vuorinen, H., Olkku, J. and Linko, P. 1980. The effect of HTST-extrusion on retention of cereal a-amylase and on enzymatic hydrolysis of barley starch. In: Food Process Engineering: Enzyme Engineering in Food Processing. P. Linko and J.

Larinkari (eds). Vol.2. Applied Sci. Publishers, London. pp: 210.

Linko, P., Colonna, P. and Mercier, C. 1981. High-temperature, short-time extrusion cooking. In: Advance Cereal Science Technology. Y. Pomeranz (ed). Vol.4. Am. Assoc. Cereal Chem. Inc., St. Paul, Minn. USA. pp: 145.

Linko, P., Uemura, K. and Eerikainen, T. 1992. Neural networks in fuzzy extrusion control. ICHEME Symp. Ser. 126: 401.

Little, T.M. and Hills, F.J. 1978. . Design and Analysis. In: Agricultural Experimentation. John Wiley and Sons, **NY.** pp: 247.

Lorenz, K. and Johnson,J.A. 1972. Starch hydrolysis under high temperatures pressures. Cereal Chem. 49: 616.

Lue, S., Hsieh, F. and Huff, H. E. 1994. Modeling of twin-screw extrusion cooking of corn meal and sugar beet fiber mixtures. J. Food Engg. 21: 263.

Lyne, F.A. 1976. Determination of starch in various products. In: Examination and anlysis of starch products. J.A. Radley (ed). Appplied sci. publishers. London. pp: 167.

Mackey, K.L. 1989. A generalised viscosity model for the cooking extrusion of starch based products. Doctoral Dissertation, Department of Food Science and Human Nutrition, Michigan State University, East Lansing, Michigan.

Martelli, F.G. 1983. Twin-screw Extruders: A basic understanding. AVI-Van Nostrand Reinhold Company. NY. pp: 43.

Mason, W.R and Hoseney, R.C. 1986. Factors affecting the viscosity of extrusion-cooked wheat starch. Cereal Chem. 63. 436.

Matz, S.A. 1976. Snack Food Technology. The AVI Publishers. West port, CT. pp: **1.**
Mercier, C. and Feillet, P. 1975. Modification of carbohydrate components by extrusion-cooking of cereal products. Cereal Chem. 52: 283.

Mercier, C. 1977. Effect of extrusion cooking on potato starch using a twin-screw French extruder. Starke, 29: 48.

.Mercier, C. 1980. Structure and digestibility of cereal starches by twin-screw extrusion-cooking. In: Food Process Engineering. P. Linko, Y. Malkki, J. Olkku and J. Larinkari (eds). Vol. I. Applied Sci. Publishers. London. pp: 795.

Mercier, C., Charbonniere, R., Grebaut, J. and de la Gueriviere, J.F. 1980. Formation of amylose-liquid complexes by twin screw extrusion cooking of manoic starch. Cereal Chem. 57: 4.

Meuser, F. and van Lengerich, B. 1984. System analytical model for the extrusion of starch. In: Thermal Processing and Quality of Foods. P. Zeuthen, J.C. Cheftel, C. Eriksson, M. Jul, H. Leniger, P. Linko, G. Varela and G. Vos (eds). Elsevier Applied Sci. Publishers. NY. pp: 175.

P. Linko, Y. Malkki, J. Olkku and J. Larinkari (eds). Applied Sci. Publishers, London. pp:791.

Olkku,J., Hassinen, H., Antila, J., Pohjanpalo, H. and Linko, P. 1980b. Automation of HTST-extrusion cooker. In: Food Process Engineering.Vol 1. Food Processing Systems. P. Linko, Y. Malkki, J. Olkku and J. Larinkari (eds). Applied Sci. Publishers, London. pp: 777.

Olkku, J. 1981. Extrusion processing - A study in basic phenomena and application of system analysis.m In: Developments in Food Preservation. S. Thorne (ed). Applied Sci. Publishers. London. pp: 177.

Olkku, J., Hagqvist, A. and Linko, P. 1983. Steady state modelling of extrusion cooking employing response surface metheodology. J. Food Engg. 2:105.

Cliett, A.L., Li, Y., Parker, R. and Smith, A.C. 1989. A comparative study of the conveying performance of screws in a twin-screw corotating extrusion cooker. J. Food Engg. 10: 165.

Owu3u-Ansah, J., Van de Voort, F.R. and Stanley, S.W. 1983. Physico-chemical changes in cornstarch as a function of extrusion variables. Cereal Chern. 60: 319.

Pan, 8.S., Kong, M.S. and Chew, H.H. 1992. Twin-screw extrusion for expanded rice products: Processing parameters and formulation of extrudate properties. In: Food Extrusion Science and Technology. J.L. Kokini, C.T. Ho and M.V. Karwe (eds). Marce! Dekker. NY. pp: 693.

Peplinski, A.J. and Pfeiffer, V.F. 1970. Gelatinization of corn and sorghum grits by steam-cooking. Cereal Sci. Today. 15:144.

Pfaller, W. and Meuser, F. 1988. Effects of specific raw material quality characteristics on the extrusion cooking behaviour of maize and rye flour products. Getreide Mehl Brot. 42: 245.

Ralet, M.C., Thibault, J.F. and Della Valle, G. 1991. Solubilization of suger-beet pulp cell wall polysaccharides by extrusion-cooking. Lebensm. Wiss. u. Technol. 24: 107.

Reddy, K.R., Ali, S.Z. and 8hattacharya,K.R. 1993. The fine structure of amylopectin and its relation to the texture of cooked rice. Carbohydr. Polym. 22: 267.

Remsen, C.H. and Clark, J.P. 1978. A viscosity model for a cooked dough. J. Food Technol. 21:39.

Robert, O.N. and Haines, 8.L. 1990. Cereal Nutrition. In: Breakfast Cereals and How They are Made. B.F. Robert and F.C. Elwood (eds). Am. Assoc. Cereal Chern. Inc., St. Paul, Minn. USA. pp:301.

Roberts, S.A. and Guy, R.C.E. 1986. Instabilities in an extrusion-cooker: A simple model. J. Food Engg. 5: 7.

Rossen, J.L. and Miller, R.C. 1973. Food extrusion. Food Technol. 27: 46.

Rusnak, B.A., Chou, C.-L. and Rooney, L.W. 1980. Effect of micronizing on kernel charecteristics of sorghum varieties with different endosperm type. J. Food Sci. 45: 1529.

Russell, P.L., Berry, C.S. and Green well, P. 1989. Characterisation of resistant starch from wheat and maize. J. Cereal Sci. 9:1.

Ryu, G.H. Neumann, P.E. and Walker, C.E. 1993. Pasting of wheat flour extrudates containing conventional baking ingredients. **J.** Food Sci. 58: 567.

Schweizer, T.F. and Reimann, S. 1986. Influence of drum-drying and twin-screw extrusion cooking on wheat carbohydrates. I. A comparison between wheat starch and flours of different extraction. J. cereal Sci. 4:193.

Schweizer, T.F., Reimann, S., Solms, J., Eliasson, A.C. and Asp, N.G. 1986. Influence of drum-drying and twin-screw extrusion cooking on wheat carbohydrates, II, Effect of lipids on physical properties, degradation and complex formation of starch in wheat flour. J. Cereal Sci. 4: 249.

Seiler, K. and Seibel, W. 1978. Herstellung ballas toffangereicherter Extrudate auf Getreidebasis. Gordian. 78: 284.

Smith, O.B. 1976. Extrusion cooking. In: New Protein Foods. M. Altschul (ed). VO"2. Acad. Press Inc. **NY.** pp: 86.

Sokhey, A.S., Kollengode, A.N. and Hanna, M.A. 1994. Screw configuration effects on corn starch expansion during extrusion J. Food Sci. 59: 895.

Sowbhagya, C.M. and Bhattacharya, K.R. 1979. Simplified determination of amylose in milled rice. Staerke. 31: 159.

Stanley, D.W., Cumming, D.B. and deMan, J.M. 1972. Texture-structure relationships on texturized soy protein. I. Textural properties and ultrastructure of rehydrated spun fibres. Can. Inst. Food Sci. Technol. J. 5:118.

Stevens, D.F. and Elton, G.A.H. 1971. Thermal properties of the starch/water system. I. Measurement of heat of gelatinization by differential scanning calorimetry. Staerke. 23:8.

Suzuki, K., Kubota, K., Omichi, M. and Hosaka, H. 1976. Kinetic studies on cooking of rice. J. Food Sci. 41: 1180

Tadmore, A. and Klein, I.1970. Engineering principles of plasticating extrusion. AVI-van Nostrand Reinhold, NY.pp: 1

Tester, R.F. and Morrison, W.R. 1990. Swelling and gelatinization of cereal starches. I. Effects of amylopectin, amylose and lipids. Cereal Chem. 67: 551.

Tipples, K.H. 1980. Uses and applications. In: The Amylograph Handbook. W. Shuey and K. Tipples (eds). Am. Assoc. Cereal Chern. Inc., St. Paul, Minn. USA. pp: 15.

Tribelhorn, R.E. and Harper, J.M. 1980. Extrusion-cooker equipment. Cereal Foods World. 25: 134.

Tsao, T.F., Beetner, C., Loranz, K. and Frey, A. 1976. Extrusion processing of instant rice spaghetti. Lebensm. Wiss. u. Technol. 9: 96.

Tuley, L. 1992. The rice revolution. Food Rev. 19: 13.

van Lengerich, B., Meuser, F. and Pfaller, W. 1989. Extrusion cooking of wheat products. In: Wheat is Unique. Y. Pomeranz, (ed). Am. Assoc. Cereal Chern. Inc., St. Paul, Minn. USA. pp: 395.

van Zuilichem, D.J., Stolp, W. and Conzales, R.J. 1978. Future trends and limitations in food extrusion. In: Cooking Extrusion. Proceeding of Symp. Zentralfachschule der Deutschen Suesswarenwirtschaft, Solingen, Germany.

van Zuilichem, D.J., Bruin, S., Janssen, L.P.B.M. and Stolp, W. 1980. Single-screw extrusion of starch- and protein-rich materials. In: Food Process Engineering. Food Processing Systems. P. Linko, Y. Malkki, J. Olkku and J. Larinkari (eds). Vol.1. Applied Sci. Publishers, London, pp: 745.

Vergnes, B., Barres, C. and Tayeb, J. 1992. Computation of residence time and energy distributions in the reverse screw element of a twin-screw extrusion-cooker. J. Food Engg. 16: 215.

Vincent, M.W. 1980. Cooker extruder saves space, water and energy. Food Flavorings Ingredients Packing and Processing. 1: 23.

Walker, C.E., Ross, A.S., Wrigley, C.W. and McMaster, G.J. 1988. Accelerated starch-paste characterization with the Rapid viscoanalyzer. Cereal Foods World. 33: 491.

Wang, S.M., Bouvier, J.M. and Gelus, M. 1990. Rheological behaviour of wheat flour dough in twin-screw extrusion cooking. Int. J. Food Sci. Technol. 25: 129.

Wang, H.H. 1991. Fermented rice products. In: Rice Utilisation. B.S. Luh (ed). Vol.II, AVI-van Nostrand Reinhold, NY.pp: 195.

Wen, L.F., Rodis, P. and Wasserman, B.P. 1990. Starch fragmentation and protein insolubilization during twin-screw extrusion of corn meal. Cereal Chem. 67: 268.

Wiedmann, W. 1990. Control of extrusion cooking. In: Processing and Quality of Foods. Vol.1. High temperature I Short time (HTST) Processing. P. Zeuthen, J.C. Cheftel, C. Eriksson, T.R. Gormley, P. Linko and K. Paulus (eds). Elsevier Sci. Publishers, London. pp: 1.237

Willard, M.J. 1989. Snack food ingredient and method for making same. U.S. Pat. 4,876,101. In: The Chemistry and Technology of Cereals as Food and Feed. S.A. Matz(ed). Second edition. AVI-van Nostrand Reinhold, NY.pp: 713.

Yacu, W. A. 1983. Modelling of a twin-screw corotating extruder. In: Thermal Processing and Quality of Foods. P. Zeuthen, J.C. Cheftel, C. Eriksson, M. Jul, H. Leniger, P. Linko, G. Varela and G. Vos (eds). Elsevier Applied Sci. Publishers. NY. pp: 62.

Yacu, W. A. 1985. Modelling a twin screw co-rotating extruder. J. Food Engg. 8: 1.

Yam, K.L., Gogoi, B.K., Karwe, M.V. and Wang, 5.5. 1994. Shear conversion of corn meal by reverse screw elements during twin-screw extrusion at low temperature. J. Food Sci. 59: 113.

Yeh, A,I., Hwang, 5.J. and Guo, J.J. 1992. Effects of screw speed and feed rate on residence time distribution and axial mixing of wheat flour in a twin screw extruder. J. Food Engg. 17: 1.

Yeh, A.I., and Jaw, Y.M. 1998. Modelling residence time distributions for single screw extrusion process. J. Food Engg. 35: 211.

Yeh, A.I. and Jaw, Y.M. 1999. Effects of feed rate and screw speed on operating characteristics and extrudate properties during single-screw extrusion cooking of rice flour. Cereal Chem. 76: 236.

Ziminiski, R.D. and Eise, K. 1980. Twin-screw extruder mechanisms in food processing. Werner and Pfleiderer Corp. Ramsey, NY. pp: 1.

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