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**THERMO-PHYSICAL AND RHEOLOGICAL PROPERTIES OF MANGO  
PUREE AS INFLUENCED BY SOLUBLE SOLIDS, TEMPERATURE AND  
HIGH PRESSURE TREATMENT**

by

**Anuradha Gundurao**

Department of Food Science & Agricultural Chemistry  
Macdonald Campus  
McGill University  
Montreal

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## ABSTRACT

Two classes of parameters are important in product quality control and process design: thermo-physical and rheological properties. Reliable estimates of thermal properties (thermal conductivity, diffusivity, density, specific heat, and glass transition temperature) are needed to model the rate of heat transfer during food processing. Measurements of rheological parameters like viscosity and elasticity have been recognized as important tools to provide fundamental insights on structural organization of the food. Small amplitude oscillatory testing is useful in evaluation of gel characteristics and texture development in different food products. Proper design of industrial plants, modeling and automation in food process industry require data on these properties for better quality control and improve the shelf life. Thermal processing has been a traditionally accepted method for developing safe and shelf-stable products. However, a disadvantage of thermal treatment is the reduced sensory and nutritional qualities. High pressure (HP) treatment, a novel non-thermal preservation technique, is used for producing high quality products. HP processing can also be used to achieve products with better textural properties and improved nutrition when supplemented with proteins.

This research was carried out in two parts. In first part, experiments with mango pulp were carried out at four temperatures (20, 40, 60 and 80°C) and total soluble solids concentrations (15, 20, 30 and 40°Brix) and their rheological and thermo-physical properties were evaluated and modeled. Thermal properties of mango puree were primarily dependent on the moisture content of the sample, and increased with temperature and decreased with concentration. Density showed a reverse trend. Glass transition temperatures increased with an increase in concentration indicating better stability. Separate models were developed for each thermal property as a function of temperature and concentration ( $R^2 > 0.90$ ). Mango puree exhibited pseudoplasticity during steady shear measurements, and the power law model well described their flow behavior. Consistency coefficient increased with concentration and decreased with temperature. The flow behavior index decreased with concentration and generally increased with

temperature beyond 40°C. Dynamic oscillation shear measurements revealed that mango puree behaved like a weak gel and demonstrated visco-elastic properties.

In the second part, experiments were divided into two parts. In the first part, the original soluble solids in mango puree (28°Brix) was lowered to 20, 23 and 26°Brix and each was supplemented with 2, 5 and 8% of whey protein. Effect of HP treatment at 425, 500, and 575 MPa with 0, 3 and 6 min holding times on rheological properties were evaluated. Pressure treatment resulted in a positive effect on elastic ( $G'$ ) and viscous moduli ( $G''$ ) as well as complex viscosity ( $\eta^*$ ). Values of  $G'$  were higher than  $G''$  demonstrating the product to behave more and more like a gel. The changes in rheological parameters at 500 MPa were modeled based on soluble solids and protein content. In the second part of the experiments, the protein (P) and soluble solids concentrations (S) were simultaneously varied maintaining a total solids content at 28% (same as in the original mango puree) and their combined influence (P/S: 2/26, 3/25, 5/23, 7/21 and 8/20) on the rheology of the product after selected HP treatments (425-575 MPa, 0-6 min) were evaluated. The effects of holding time and pressure level were similar to those observed previously in the first part of this study. However, the positive effect of added protein was over dominated by the negative effect of the simultaneous decrease in the soluble solids concentration, resulting in an overall decreasing effect as protein content increased (with a same magnitude decrease in soluble solids content). Developed models well predicted the combined influence of protein and soluble solids concentration on rheological parameters ( $R^2 > 0.85$ ).

Sensory evaluation of mango puree supplemented with protein (2, 5 and 8%) was performed before and after high pressure treatment (500MPa/3min) using a nine point hedonic scale. The quality parameters chosen were color, sweetness, mouthfeel, flavor and acceptability. Sensory evaluation results indicated that protein enrichment up to 5% level did not adversely affect the sensory qualities.

## RÉSUMÉ

Deux classes des paramètres sont importantes dans le contrôle de qualité de produit et la conception de processus : propriétés thermo-physiques et rhéologiques. Les évaluations fiables des propriétés thermiques (conductivité thermique, diffusivité, densité, chaleur spécifique, et température de transition de verre) sont nécessaires pour modeler le taux de transfert thermique pendant la transformation des produits alimentaires. Des mesures des paramètres rhéologiques comme la viscosité et l'élasticité ont été identifiées en tant qu'outils importants pour fournir des perspicacités fondamentales sur l'organisation structurale de la nourriture. L'essai oscillant de petite amplitude est utile dans l'évaluation des caractéristiques de gel et le développement de texture dans différents produits alimentaires. La conception appropriée des usines industrielles, modeler et l'automation dans l'industrie de processus de nourriture exigent des données sur ces propriétés pour un meilleur contrôle de qualité et améliorent la durée de conservation. Le traitement thermique a été une méthode traditionnellement admise pour développer les produits sûrs et solides sur l'étagère. Cependant, un inconvénient de traitement thermique est les qualités sensorielles et alimentaires réduites. (HP) le traitement à haute pression, une technique non-thermique de conservation de roman, est employé pour produire des produits de haute qualité. Le traitement de HP peut également être employé pour réaliser des produits avec de meilleures propriétés de texture et la nutrition améliorée une fois complété avec des protéines.

Cette recherche a été effectuée dans deux parts. Dans la première partie, des expériences avec de la pulpe de mangue ont été effectuées aux quatre températures (20, 40, 60 et 80°C) et les concentrations en solides solubles totaux (15, 20, 30 et 40°Brix) et leurs propriétés rhéologiques et thermo-physiques ont été évaluées et modelées. Les propriétés thermiques de la purée de mangue dépendaient principalement de la teneur en humidité de l'échantillon, et accru avec la température et diminué avec la concentration. La densité a montré une tendance renversée. Les températures de transition de verre ont augmenté avec une augmentation de la concentration indiquant une meilleure stabilité. Des modèles séparés ont été développés pour chaque propriété thermique en fonction de la température et de la concentration ( $R^2 > 0.90$ ). Le pseudo-plasticity exhibé par purée de

mangue pendant des mesures régulières de cisaillement, et le puits de modèle de loi de puissance ont décrit leur comportement d'écoulement. Le coefficient d'uniformité a augmenté avec la concentration et a diminué avec la température. L'index de comportement d'écoulement a diminué avec la concentration et a généralement augmenté avec la température au delà de 40°C. Les mesures dynamiques de cisaillement d'oscillation ont indiqué que purée de mangue comportée comme un gel faible et des propriétés visco-élastiques démontrées.

Dans la deuxième partie, des expériences ont été divisées dedans à deux parts. Dans la première partie, les solides solubles originaux en purée de mangue (28°Brix) ont été abaissés à 20, 23 et 26°Brix et chacun ont été complétés avec 2, 5 et 8% de protéine de petit lait. L'effet du traitement de HP à 425, 500, et 575 MPa avec 0, 3 et 6 fois tenantes minimum sur les propriétés rhéologiques ont été évaluées. Le traitement de pression a eu comme conséquence un effet positif sur le moduli élastique ( $G'$ ) et visqueux ( $G''$ ) comme des valeurs complexes de la viscosité ( $\eta^*$ ). Les valeurs de  $G'$  étaient plus haut que  $G''$  démontrant le produit pour se comporter de plus en plus comme un gel. Les changements des paramètres rhéologiques à 500 que MPa ont été modélisés ont basé sur les solides solubles et la teneur en protéines. Dans la deuxième partie des expériences, la protéine (P) et les concentrations en solides solubles (S) ont été simultanément changées maintenant un contenu de solides totaux à 28% (mêmes que dans la purée originale de mangue) et leur influence combinée (P/S : 2/26, 3/25, 5/23, 7/21 et 8/20) sur la rhéologie du produit après traitements choisis de HP (425-575 MPa, 0-6 minutes) ont été évalués. Les effets de tenir le niveau de temps et de pression étaient semblables à ceux observés précédemment dans la première partie de cette étude. Cependant, l'effet positif de la protéine supplémentaire était excédent dominé par l'effet négatif de la diminution simultanée de la concentration en solides solubles, ayant pour résultat un effet décroissant global à mesure que la teneur en protéines augmentait (avec une même diminution de grandeur de contenu de solides solubles). Les modèles développés ont bien prévu l'influence combinée de la concentration en protéine et en solides solubles sur les paramètres rhéologiques ( $R^2 > 0.85$ ).

L'évaluation sensorielle de la purée de mangue complétée avec la protéine (2, 5 et 8%) était le traitement avant et après à haute pression exécuté (500MPa/3min) employant



une balance hédonistique de neuf points. Les paramètres de qualité choisis étaient couleur, douceur, effet dans la bouche, saveur et acceptabilité. Les résultats d'évaluation sensorielle ont indiqué que l'enrichissement en protéine jusqu'au niveau de 5% n'a pas compromis les qualités sensorielles.

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## FORMAT OF THESIS

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## Contribution of Authors in Manuscripts

The work stated here was completed by **Anuradha Gundurao**, the candidate, under the supervision of **Dr. Hosahalli. S. Ramaswamy**, Department of Food Science and Agricultural Chemistry, Macdonald Campus of McGill University, Montreal, The authorship for the papers included are:

- 1) Gundurao, A., Ramaswamy, H.S., Ahmed, J. for the paper detailed in Chapter IV "*Effect of concentration and temperature on thermo-physical and rheological properties of mango puree*".
- 2) Gundurao, A. and Ramaswamy, H.S. "*Effect of soluble solids and high pressure treatment on rheological properties of protein enriched mango puree*" which is part of Chapter V.

A. Gundurao is the candidate and the principal author of all papers. Dr. H.S. Ramaswamy is the thesis supervisor under whose guidance the research work was carried out. He was responsible for providing the guidelines and direction for the work and for the final editing of the manuscript. Dr. J. Ahmed, research associate working with Dr. Ramaswamy has contributed to the development of topic in Chapter IV and for assisting in the experimental set-up and helping with drafting of the first paper on rheology.

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## NOMENCLATURE

$a_n$	Regression constant
A	Constant
$A_K$	Predicted experimental constants
A	Predicted experimental constants
b	Constant
C	Constant
$C^a$	Concentration (emperical constant)
$C_P$	Specific heat capacity ( $\text{kJ kg}^{-1}\text{°C}^{-1}$ )
$C_{PS}$	Specific heat capacity of sample ( $\text{kJ kg}^{-1}\text{°C}^{-1}$ )
$C_{PW}$	Specific heat capacity of water ( $\text{kJ kg}^{-1}\text{°C}^{-1}$ )
$\Delta C_P$	Change in specific heat capacity ( $\text{kJ kg}^{-1}\text{°C}^{-1}$ )
d	Constant
DMTA	Dynamic mechanical thermal analysis
DSC	Differential scanning calorimeter
$D_S$	Displacement of sample
$D_W$	Displacement of water
E	Error
EPR	electronic paramagnetic resonance
$E_a$	Activation energy
exp	Experimental
FAO	Food and agricultural Organization
$G'$	Elastic modulus (Pa)
$G''$	Viscous modulus (Pa)
$G^*$	Complex modulus (Pa.s)
GLT	Glass to liquid transition
GSH	Gastro stimulating hormone
H	Enthalpy ( $\text{kJ kg}^{-1}$ )
H-B	Herschel Bulkley model
HP	High pressure
HPP	High pressure processing
k	Thermal conductivity ( $\text{Wm}^{-1}\text{°C}^{-1}$ )
K	Consistency coefficient ( $\text{Pa.s}^{-n}$ )
$K^*$	Consistency coefficient in oscillation viscosity
$K_C$	Casson plastic viscosity ( $\text{Pa.s}^{1/2}$ )
$K_{OC}$	Casson yield stress ( $\text{Pa}^{1/2}$ )
$K_H$	Herschel-Bulkley Consistency coefficient
m	mass (kg)
M-B	Mizrahi-Berk model
n	Flow behavior index
$n^*$	Flow behavior index in oscillation viscosity
N/n	Number of data points
NMR	Nuclear magnetic resonance
p	Product

$P_{pred}$	Predicted
$q_{exp}$	Experimental value
$q_{pred}$	Predicted value
$Q$	Heat supplied (J)
$R$	Universal gas constant
$s$	sample
$S$	Slope
$S-S$	Sulphur sulphur bond
$\Delta t$	Change in time (min)
$T$	Temperature ( $^{\circ}C$ )
$T_g$	Glass transition temperature ( $^{\circ}C$ )
$TSS$	Total soluble solids
$V$	Volume ( $m^3$ )
$w$	Weight (g)
$WP$	Whey protein
$WPI$	Whey protein isolates
$W_s$	Weight of sample (mg)
$W_w$	Weight of water (mg)
$X$	Corrected actual value
$X^o$	Actual value
$\Delta X$	Increment of X per unit Z
$X_1$	Protein concentration (%)
$X_2$	Pressure applied (MPa)
$X_3$	Holding time (min)
$X_c$	Calculated value
$X_m$	Measured value
$X_w$	Soluble solid content
$Y$	dependent variables
$Z$	coded value
$\alpha$	Thermal diffusivity ( $m^2s^{-1}$ )
$\alpha$ -lac	Lactalbumin
$\beta$ -lg	Lactoglobulin
$\gamma$	Shear rate ( $s^{-1}$ )
$\delta$	Phase shift (deg)
$\eta$	Apparent viscosity (Pa.s)
$\eta^*$	Complex viscosity (Pa.s)
$\theta$	Time (sec)
$\theta_0$	Time correction factor (sec)
$\sigma_0$	Yield stress (Pa)
$\tau$	Shear stress (Pa)
$\tau_{OH}$	Yield stress using Herschel-Bulkley model (Pa)
$\omega$	Angular frequency ( $rads^{-1}$ )

## **CHAPTER I**

### **GENERAL INTRODUCTION**

Mango, also known as “apple of tropics”, is a very popular fruit in the tropics. Commercialization of mango products has enhanced the demand for production and preservation of mango pulp. In addition to the fresh form, mangos are sold in various forms to extend their market potential and meet consumer demand for product diversity and needs. The shelf-life of fresh mango is extended significantly by refrigerated regular and control atmosphere storage as well as appropriate packaging, example modified atmosphere packaging. Mangos are also thermally processed to render them shelf-stable by subjecting them to heat treatment. The more popular mango products are mango pulp, puree, juice and beverages containing mango puree. These are mostly heat processed and packaged in cans for extending their shelf-life. Other products include chutneys, pickles, dried mango puree etc.

The food processing industries need to comply with the consumer demand for improved product quality and enriched nutrition. The primary concern in food processing is to produce safe food by destruction of microorganisms and pathogens, to look for new product development ideas with higher nutritional values and sensory characteristics and increase revenue by employing energy efficient and cost effective technologies. Among the conventional processes, heat treatment has been most widely used technique for food preservation. The microbial destruction process in food is also dependent of product acidity and governs the severity of heat treatment. Fruit beverages are acidic in nature and therefore pasteurization is commonly used for preservation. However, the major limitation of heat treatment on beverages is lowered sensory quality attributing to reduced commercial and consumer values.

Presently, consumers are health conscious and various government legislations have forced the food technologists to produce and serve original like foods with no additives. It leads to exploitation of new technologies that can produce safe and customers oriented foods. High Pressure (HP) processing is one of such technologies that can produce original like foods in terms of color, flavor and safe products at ambient

temperature. Sensory properties like appearance, color and flavor are extremely important for consumer acceptance and enjoyment of food. In addition, the process has benefits on product texture especially using proteins and specific starches. High pressure processing technology is relatively straightforward and has many applications like development of new foodstuffs, manufacture of partially cooked food and prolongation of storage time. By adding proteins to mango puree, a nutritionally enriched product with good consumer appeal and commercial value can be formulated.

Process and product quality control measures need data on thermo-physical and rheological properties of foods. Reliable estimates of thermal properties are essential to understand the rate of heat transfer. Heat and mass transfer are simultaneously involved in processes such as thermal processing, dehydration, concentration or evaporation and extraction. Hence thermal properties play an important role in the quantitative analysis of food processing operation. Models developed for predicting the processing times require data on thermo-physical properties. Rheology is a very powerful tool to describe the structural nature and properties of food products. Most food products are viscoelastic in nature. Even for the weakest structure, flow occurs after the structure exceeds a critical deformation. The rheological tests must be selected according to the material properties to be investigated or the process to be simulated. Rheology plays an important role in process engineering calculations involving wide range of equipments like pump size, pipelines, extruders, heat exchangers etc. It determines ingredient functionality in product development and help in maintaining product intermediate and final quality control. The range of rheological properties provides diverse nature and options in diet. Correlation of thermo-physical and rheological properties of foods to sensory data provides cost effective quality control procedures.

Present literature survey indicates a significant lack of information related to the thermophysical and rheological properties of mango puree as a function of temperature and soluble solids concentration. Further there is no data on the influence of the new processes such as HP processing on the rheological and sensory properties of mango products. Improving the nutritive value of mango based products by protein enrichment without influencing their sensory qualities is a step in the right direction for preparing products that meets the popular demands of the demand.

## **CHAPTER II**

### **GENERAL OBJECTIVES**

The general objectives of the thesis research were to

1. Evaluate the thermo-physical and rheological properties of mango puree as influenced by temperature and soluble solids content.
2. a). Evaluate the rheological properties (dynamic parameters, elastic modulus, viscous modulus and complex viscosity) of protein enriched mango puree as influenced by high pressure, holding time and protein concentration.  
  
b). Evaluate the effect of high pressure treatment on the sensory quality (color, sweetness, mouth feel, flavor, and acceptability) of protein enriched mango puree.



## **CHAPTER III**

### **LITERATURE REVIEW**

#### **3.1 Mango and mango products**

This section introduces mango, mango products, production statistics etc.

##### **3.1.1 Mango cultivars**

Mango also known as “apple of tropics” is one of the highly accepted fruit in the world market for its exotic flavor and taste. The skin color of mango can be green, yellow-orange or even reddish-pink. The mango pulp is capusin yellow in color. Plenty of mango varieties are available with significant variations in color, flavor and texture (pulp content). There are more than 300 varieties of mangoes available in the market. Table 3.1 lists the leading mango producing countries and the cultivars available there. Among the numerous mango varieties, the Indian and Indochinese types are found to be the best. The Indian mangoes have attractive color, exotic flavor and are less fibrous than Indochinese type. Among Indian mangoes ‘Alphonso’ mangoes are considered as the best cultivars, which is also called ‘King of mangoes’. Badami, Happus, Khader, Appas and Kagdi Happus are the other names given to Alphonso mango. This fruit is medium in size, ovate oblique in shape and orange yellow in color. Alphonso mango has been widely used for canning purpose.

##### **3.1.2 Post harvest handling and storage of mangoes**

Mangoes are generally handpicked or plucked at mature stage and ripened for optimum quality. A picker pole with knife and a bag is usually used as a harvest aid. Mango pulp turns light yellow to orange on maturity and the seed starts hardening. Injuries to the peel or to the stalk end during harvesting serve as path for invasion of microorganism and lead to rotting of the fruits. About 25-40% of post harvest loss of mangoes is estimated from harvesting to storage due to improper harvesting, handling, transportation, and storage.

The fruits are graded according to their size, weight, color and maturity. Wooden boxes are commonly used for packaging and transportation of mango fruits. Polythene lining is used to reduce ventilation and to maintain humidity, which results in lesser loss in weight, color and shrinkage during storage. Wrapping of fruits individually with paper and packing in honeycomb structure helps in getting optimum ripening with reduced spoilage. The mature green fruits are stored at room temperature for about 4-10 days depending upon the variety. Precooling, chemical treatments and low storage temperature extends the shelf life of fruits (<http://www.horticultureworld.net/mango-india.htm>).

**Table 3.1 Mango cultivars available in major mango producing countries.**

<b>Country</b>	<b>Cultivars</b>
Bangladesh	Aswina, Fazli, Gopal Bhog, Himsagar, Khirsapati, Langra, Kishan Bhog, Kohinoor, Kua Pahari, Mohan Bhog
Brazil	Bourbon, Carlota, Coracao, Espada, Itamaraca, Maco, Magoada, Rosa, Tommy Atkins
China	Baiyu, Guixiang, Huangpi, Huangyu, Macheco, Sannian, Yuexi No. 1
Egypt	Alphonso, Bullock's Heart, Hindi Be Sennara, Langra, Mabrouka, Pairie, Taimour, Zebda
India	Alphonso, Baganapalli, Bombay, Bombay Green, Chausa, Dashehari, Fazli, Fernandian, Himsagar, Kesar, Kishen Bhog, Langra, Mallika, Mankurad, Mulgoa, Neelum, Pairi, Samar Behisht Chausa, Suvarnarekha, Totapuri, Vanraj, Zardalu, Amrapali, Bangalora, Gulabkhas
Indonesia	Arumanis, Dodol, Gedong, Golek, Madu, Manalagi, Cengkir, Wangi
Mexico	Haden, Irwin, Kent, Manila, Palmer, Sensation, Tommy Atkins, Van Dyke
Myanmar	Aug Din, Ma Chit Su, Sein Ta Lone, Shwe Hin Tha
Pakistan	Anwar Ratol, Baganapalli, Chausa, Dashehari, Gulab Khas, Langra, Siroli, Sindhri, Suvarnarekha, Zafran
Philippines	Carabao, Manila Super, Pico, Binoboy, Carabao, Dudul, Pahutan, Senora
Thailand	Nam Doc Mai, Ngar Charn, Okrong, Rad, Choke Anand, Kao Keaw, Keow Savoey, Pimsenmum

### **3.1.3 Benefits of mango to human health**

Mangoes are perfect to replenish salts, vitamins and energy after physical exercise. The vitamin C in the mango enhances the absorption of iron and helps in preventing anemia. Taking Mango regularly makes the complexion fair and the skin soft and shining. Vitamin C and Calcium present in mangoes helps in tightening the capillary vessels and prevents bleedings of inner parts of body. It is also used as medicine for dry cough, fever, and gastric problems. Mango is an excellent natural source of  $\beta$ -carotene; the content rises even after being picked before ripening. The content of carotenes is very high also in the dried fruit, and retains high levels for at least six months after harvest.

### **3.1.4 Statistics of mango production**

India is the largest producer of mango with 41% of total world production followed by China (13%) and Thailand (6%). India also produces 55% of mangoes in Asia. Mexico leads the export market of mango with 23% of world export followed by India (20%) and Brazil (15%). Thailand dominates the production of mango pulp with 95% of world production. Export of mango juice is lead by Egypt with 95% of world export followed by China (4%) (FAO, 2003). Top 10 mango producing countries in 2004 are shown in Table 3.2.

### **3.1.5 By-products of mango**

Both ripe and unripe mangoes have been utilized to make various products. Ripe mango pulp converts to juice, nectar and leather while pickles, salads, chutney and mango powder are manufactured from unripe mango. The development of mango beverage industry has enhanced the demand for canned mango pulp. In addition, pulps and purees are also frozen, dried, canned or cooked for further processing or value added products like jams, preserves and jellies. Mango puree is also gaining popularity as an ingredient in foods such as ice creams, yogurt and bakery items for their striking flavor and color.

**Table 3.2 Top ten mango producing countries in the world (FAO, 2004).**

<b>Country</b>	<b>Mango production (10<sup>3</sup> Mt)</b>
India	10,800
China	3,622
Thailand	1,750
Mexico	1,503
Pakistan	1,072
Philippines	890
Brazil	845
Indonesia	800
Nigeria	730
Egypt	327

### **3.1.6 Production of mango puree**

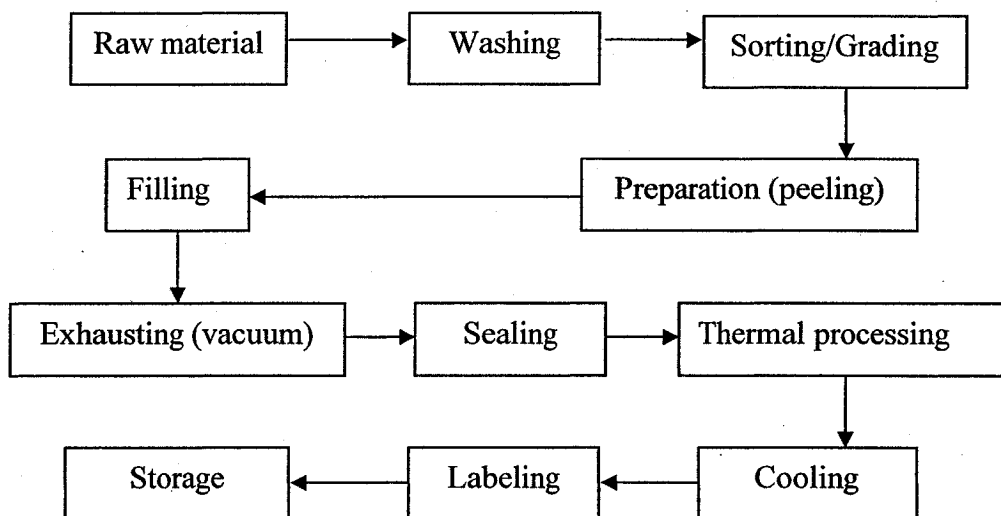
Harvesting at proper maturity is a very important step in thermal processing. Mango selected for processing should be at the soft-ripe stage to have good taste, color and flavor. The fruit should be thoroughly washed to remove dirt, dust, mold spores and other foreign matters. Water temperature should be low during washing to reduce leaching and to keep firmness of fruit. The fruits are then sorted to remove inferior or damaged produce. This could be done manually or by using new technologies like magnetic resonance imaging. The fruits are then size-sorted and undersized fruits are used for baby foods or for concentrates.

Hot water peeling and steam peeling are the most practical method used for mango peeling. The flesh is disintegrated from mango using a paddle pulper. The seeds, residual peels and large part of fibers are separated from pulp by centrifugation or by screening. The composition of mango flesh used for making puree depends on cultivars, climate condition, postharvest storage and treatment of the fruits. Usually the soluble solids content ranges between 15-20°Brix, acidity between 0.1- 1.1% and pH from 2.6 to

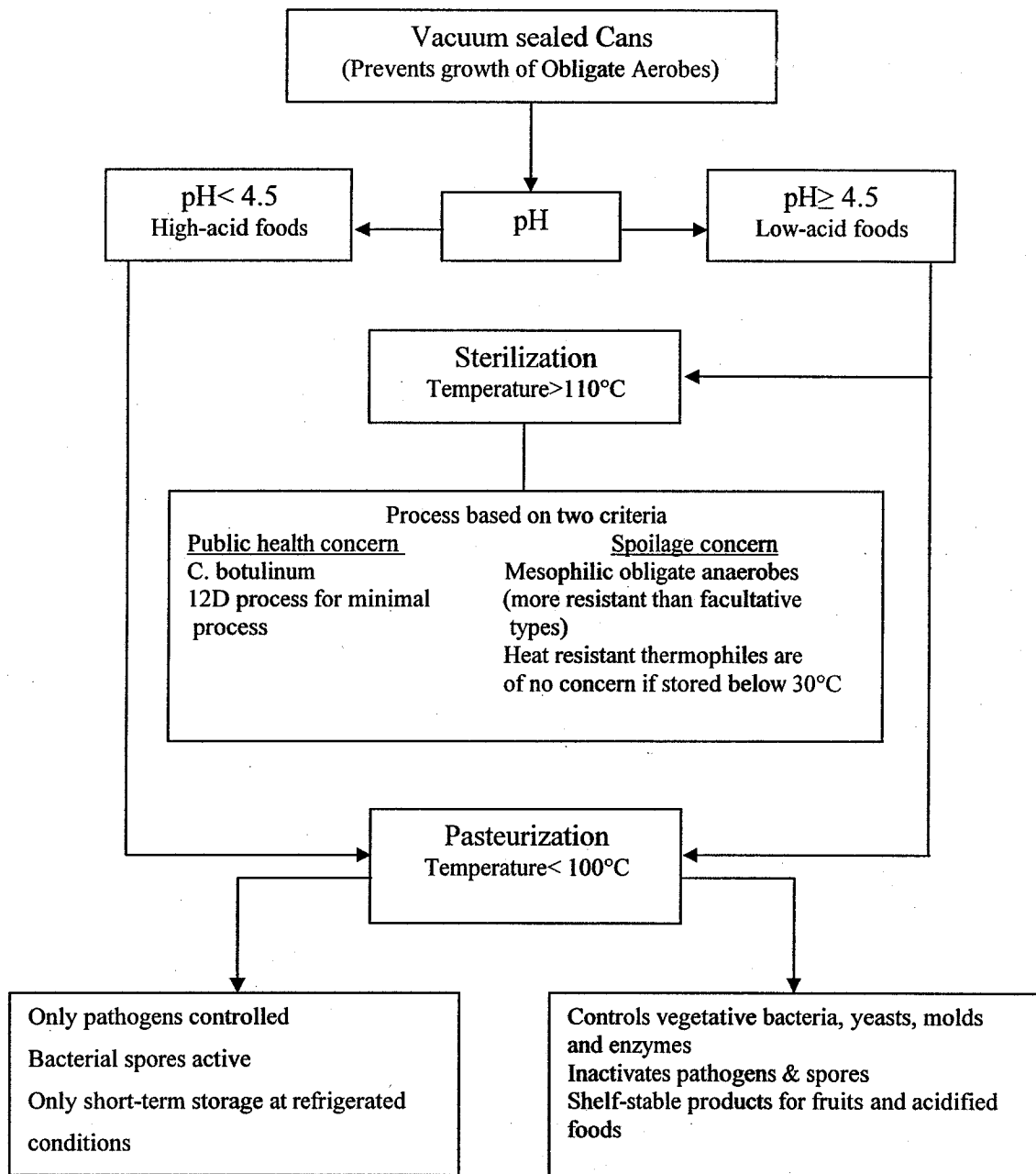
5.8. For safety and quality purposes, pH in mango puree is lowered with low acidity (<4.0) by adding citric acid and color is added before heat treatment (Wu & Sheu, 1996).

### 3.1.7 Preservation of mango puree

Canning is the most commonly used technique to heat-sterilize mango puree. Figure 3.1 shows the typical mango puree canning operation. Thermal processing is proven to be one of the most effective techniques for preservation of mango and its products. The adequacy of the process depends on the reduction in the amount of spoilage causing bacteria and the inactivation of most heat resistant enzymes present. It is very important to estimate the level and rate of destruction/inactivation accomplished by the process. This is attained by obtaining the thermal destruction kinetics and the product heating profile. Figure 3.2 is the schematic of thermal processing principles (Ramaswamy & Abbatemarco, 1996).



**Figure 3.1 Typical mango puree canning operation**



**Figure 3.2 Schematic of thermal processing principles**

### 3.1.8 Enrichment of mango puree

Mango puree is an excellent source of  $\beta$ -carotene and vitamin C (Table 3.3). It has substantial amount of calcium, phosphorus, potassium and sodium. However, mango contains minimum amount of fat and the energy value is significantly low. It is also a low source of protein. Mixed with a protein of high biological values, mango puree could achieve a nutritionally balanced food with good consumer appeal and commercial values.

**Table 3.3 Nutritional value of mango per 100g of edible portion**

<b>Composition</b>	<b>Percentage</b>
Moisture (g)	81.0
Protein (g)	0.6
Fat (g)	0.4
Minerals (g)	0.4
Fibre (g)	0.7
Carbohydrates (g)	16.9
Energy (Kcal)	74
Calcium (mg)	14
Phosphorus (mg)	16
Carotene ( $\mu$ g)	2743
Vitamin C (mg)	16
Potassium (mg)	270
Sodium (mg)	26

### **3.2 Physical properties**

One of the main objectives of food processing is to provide a shelf stable and nutritious food. It is also important to maintain quality of product both during production and storage. Consistency, color, flavor and texture are some of quality factors, which enhance market and consumer value. For food industries to be technically and economically feasible, it is important to develop a product with a good balance between safety and quality. This balance can be obtained with the knowledge about the physical and chemical properties of the product. Among these, the thermo-physical and rheological properties are one of the most important. Models developed by thermo-physical properties are important to evaluate the rate of heat flow and predict processing time. Rheological properties help in analysis of flow properties and fluid handling during processing. Both thermo-physical and rheological properties are useful in determining ingredients functionality, shelf life testing and in maintaining final product quality control.

#### **3.2.1 Thermal properties**

Knowledge of thermal properties like thermal conductivity ( $k$ ), thermal diffusivity ( $\alpha$ ), specific heat ( $c$ ) and density ( $\rho$ ) are vital for quantitative analysis of foods undergoing thermal processing. The formulation of the product and the thermal properties of its components determine the thermal response characteristics under the applied heating conditions. Thermal processing specialist should know the thermal properties of each component while applying that knowledge in the design of processes to ensure food safety and optimum quality (McGinnis, 1987). Singh (1982) discussed the importance of using the component thermal property values in numerical or analytical solutions to heat transfer in composite foods. Thermal property values are reported in the literature for a number of selected food materials (Ramaswamy & Tung, 1981; Nije et al., 1998; Azoubel et al., 2004). But for an ever-increasing inventory of processed and/or manufactured food products, thermal property data are always in demand from both industrial and research point of view (McGinnis, 1987).



### ***3.2.1.1 Thermal conductivity***

The importance of thermal conductivity lies in the prediction and/or control of heat flux during processing, evaluation of thermophysical properties like thermal diffusivity, heat penetration coefficients, and to determine processing time. They are necessary for maintaining quality of food and efficiency of the equipment (Fontana et al., 2001).

#### ***3.2.1.1.1 Methods of determination***

The two methods used to measure thermal conductivity of food materials are the steady state (guarded hot plate, concentric cylinder, concentric sphere, etc) and the unsteady state or transient methods (line heat source method). The steady state techniques have been used to obtain accurate thermal conductivity values (Mathur & Lele, 2001). Some well-established standard techniques for example the guarded hot plate method need large sample size and longer temperature equilibration time which lead to moisture migration in the sample (Reddy & Datta, 1994). So they are not always well suited for food samples. Rao and Rizvi (1986) in their review on the different techniques of thermal conductivity measurement concluded that the line heat source probe is most suitable for food applications. It is preferred because of its simplicity, speed and requirement of relatively small sample size (Ramaswamy and Tung, 1981; Reddy & Datta, 1994). Reidy and Rippen (1969) and Sweat (1974) described these methods. The probe technique was originally developed for measuring the thermal conductivity of insulated material (Reidy and Rippen, 1971) and its application was then extended to food system. The most notable thermal conductivity compilations have been by Quashou (1970) and Choi and Okas (1985) (cited by Reddy & Dutta, 1994).

Unsteady state measurements involve dynamic response of a food system measured by a step change or an impulse. During a step change, the system is subjected to a new parameter such as temperature, pH or color. Whereas, if an impulse is used, the parameter is perturbed only for a short time and then it goes to the old values for example like injecting a dye or acid shot. Measurement of the thermal conductivity using heated probe is based on the principle that the temperature rise at a point close to a line heat

source in a semi infinite solid subjected to a step change heat source is a function of time, the thermal properties of solid and the source strength (Mathur & Lele, 2001).

### 3.2.1.1.2 Mathematical basis for thermal conductivity measurement

The history and development of a mathematical basis for the line-heat source method of conductivity determination is presented extensively in the literature (Mohsenin, 1980; Rao & Rizvi, 1986; Rahman, 1996). Considering only radial conduction heat flux in an infinite cylinder, the change in temperature 'T' at a point close to line heat source following a suddenly initiated constant heating rate, between times  $\theta_1$  and  $\theta_2$  is given by the following equation

$$T(\theta_1) - T(\theta_2) = \left( \frac{Q}{4\pi k} \right) \ln \left( \frac{\theta_1}{\theta_2} \right) \quad (3.1)$$

Thermal conductivity 'k' can then be determined from equation (3.1) knowing the heat source strength 'Q' and the temperature versus time relationship (McGinnis, 1987). Use of a time correction factor ( $\theta_0$ ) has been widely accepted as a means of compensating for the finite diameter and unique thermal properties of the line heat source. The time correction factor ( $\theta_0$ ) is subtracted from each time value, and the slope (S) of the time corrected temperature rise versus  $\ln(\theta - \theta_0)$  plot can be used in the calculation of k, giving

$$k = \frac{Q}{4\pi S} \quad (3.2)$$

where

$$S = \frac{T(\theta_1) - T(\theta_2)}{\ln \left( \frac{\theta_1 - \theta_0}{\theta_2 - \theta_0} \right)} \quad (3.3)$$

### *3.2.1.1.3 Errors and corrections*

Some authors (Sweat, 1986; Rahman, 1996) have suggested that proper probe design for example; making probe size very small (~0.66 mm diameter) would purge the need for a time correction factor for most measurements. Also, it should be considered that the time correction would be greater for larger heaters since the deviation from the line source theory is greater for larger heaters. It has been suggested that  $\theta_0$  is not required for samples at temperatures above freezing as the heat capacity of sample would be about the same as food sample. Probe constants, which are obtained by calibration of probe, can also be used instead of  $\theta_0$  (Rahman, 1996).

The finite sample size can also cause error if the sample boundaries experience temperature gradient during the measurement. The required sample diameter depends on thermal diffusivity of sample and test duration. Sweat and Haugh (1972) suggested a sample diameter of at least 1.9 cm for food samples having thermal diffusivity of the same order of magnitude as that of water (cited by Mohsenin, 1980). Increase in sample size and short measurement time would minimize the finite size error. The expression  $4\alpha\theta/d^2$  (four times the Fourier number) has also been used to estimate the minimum sample size where 'd' is the shortest distance between the probe and the sample boundary, and 'θ' is the time since the heater was energized. The error is noticeable when this expression is greater than 0.6 (Mohsenin, 1980).

Excessive test duration can also cause error due to convection and moisture transfer. Fibreglass wool by 1% to weight of sample can be used to reduce convection at elevated temperatures (Romero, 1998). Sweat (1995) used 0.5% agar gel to minimize convection effects. High power supply can also cause temperature gradient, convection and moisture migration. Sweat (1986) suggested that the power level should be selected so as to have high correlation between time-ln (temperature) curve.

### *3.2.1.1.4 Thermal conductivity and fruit juice*

Many works are available on thermal conductivity (k) of fruit juices. Azoubel (2005) used probe method to measure thermal conductivity (k) of cashew juice at

different concentrations and found that 'k' decreases with increase in juice concentration. Romero et al. (1998) found similar results for orange juice and Shamsudin et al. (2005) for guava juice. They also show that temperature has small effect on thermal conductivity compared to water content. Very little data is available on thermal conductivity of mango puree.

### 3.2.1.2 Specific heat capacity

Specific heat is the mass heat capacity defined as the heat capacity of body per unit mass of the body.

$$C = \frac{Q}{(w)\Delta t} \quad (3.4)$$

where C is specific heat ( $\text{Jg}^{-1}\text{C}^{-1}$ ), w is weight (g) Q is the heat supplied (J) and  $\Delta t$  is the temperature difference ( $^{\circ}\text{C}$ ) (Mohsenin, 1980). Most of the food processing unit operation is heat or energy intensive. So, specific heat is required to calculate the heat load in food processing operation (Rahman, 1996).

Differential scanning calorimeter (DSC) is the most commonly used instrument to measure specific heat. The DSC method works by comparing the heat flow rate into a sample material to that of a standard material when both are subjected to the same linear rate of temperature increase (Dagga & Kolbe, 1997). The specific heat for the sample material can then be obtained as a function of temperature. DSC is well known for its simplicity, rapid and accurate measurement with small amount of sample. Total enthalpy change will also result from DSC as shown by Iso et al. (1991) for fish and chicken paste. Dagga & Kolbe (1997) calculated the specific heat of surimi paste at cooking temperature by ratio method (equation 3.5) using DSC heat capacity software

$$C_p = \frac{E_q}{Hm} \quad (3.5)$$

where H is the enthalpy, m is the mass of sample.

Zainal et al. (2001) used DSC to determine specific heat of pink guava juice using the equation

$$C_{PS} = \frac{W_W D_S C_{PW}}{W_S D_W} \quad (3.6)$$

where  $C_p$ ,  $W$  and  $D$  are specific heat capacity, weight and recorded displacement (or heat flow) for juice sample (s) and water (w), respectively. Constenla et al. (1989) used the same method for calculating the specific heat of clarified apple juice. It has been shown for fruit juices that specific heat is influenced by both temperature and concentration.

### 3.2.1.3 Thermal diffusivity

Thermal diffusivity is the ability of the product to respond to temperature change. Its relationship with temperature change is similar to that of thermal conductivity to heat change in the product. Thermal diffusivity of material is measured by the equation:

$$\alpha = \frac{k}{C\rho} \quad (3.7)$$

where  $\alpha$  is thermal diffusivity ( $\text{m}^2 \text{s}^{-1}$ ),  $k$  is thermal conductivity ( $\text{W m}^{-1} \text{K}^{-1}$ ),  $\rho$  is density ( $\text{kg m}^{-3}$ ) and  $C$  ( $\text{J kg}^{-1} \text{K}^{-1}$ ) is specific heat capacity.

Thermal diffusivity can be estimated by direct or indirect method. Indirect evaluation of  $\alpha$  is done using experimental values of thermal conductivity, specific heat and density. Direct measurement of thermal diffusivity is usually determined from the solution of one dimensional unsteady state heat transport equation with the appropriate boundary conditions. Analytical method is used for finite or infinite bodies and numerical techniques for irregular bodies (Rahman, 1996). The analytical solutions depend on relative importance of internal and external heat transfer resistance, which intern depends on Biot number. Biot number greater than 40 indicates negligible external resistance whereas less than 0.2 shows negligible internal heat resistance. For Biot number varying between 0.2-40, both internal and external resistance to heat transfer is important (Singh, 1992).

Many methods (Dickerson method; Hayakawa method; half cooling method; Moore and Bilanski method) have been described for direct measurement of thermal diffusivity (Rahman, 1996). Drouzas et al. (1991) used both direct and indirect method for determination of thermal diffusivity in granular starch and found that the indirect

method yielded more accurate values than direct measurement. Many researchers (Mohsenin, 1980; Ramaswamy & Tung, 1981; McGinnis, 1987; Constenla et al., 1989; Njie et al., 1998) have used indirect method to calculate  $\alpha$  from experimental values of  $\rho$ ,  $k$ ,  $C_p$  and equation 3.7.

Thermal diffusivity of liquid foods is mainly affected by change in moisture content and temperature. Thus, prediction of thermal diffusivity as a function of temperature and water content can be used in thermal analysis of food processing operations. Martenz (1980) studied the affect of water, protein, temperature, fat and carbohydrates on thermal diffusivity and found that water and temperature influence major than other factors.

#### ***3.2.1.4 Density***

Density of a food material is a ratio of its mass divided by the volume at a particular temperature. Knowledge of the density of foods is important in separation processes and differences in density can have important effects on the operation of size reduction and mixing equipment. This is particularly important in fluids where differences in density cause convection currents to be established (Fellows, 2000).

#### ***3.2.1.5 Glass transition temperature***

Glass transition temperature ( $T_g$ ) or glass to liquid transition (GLT) is the name given to phenomena observed when a glass is changed into a super-cooled melt during heating, or to the reverse transformations during cooling. Both are non-crystalline states; but while the glass is a rigid solid, the supercooled melt that is observed between glass transition and melting point can be a viscoelastic “rubber” in the case of a polymeric material, or a mainly viscous liquid, for low molecular weight materials (Meste et al., 2002). The glass transition corresponds to the temperature zone where the viscosity reaches the value of  $10^{10}$ - $10^{12}$  (Champion et al., 2000). Research on  $T_g$  for food systems can be seen since 1960s. However, a huge variety of possible applications in food science and technology was highlighted by Levine and Slade in the 1980s (Slade & Lavine, 1988; Lavine and Slade, 1986).

The glass transition temperature  $T_g$  is usually referred as single temperature even though it occurs over a temperature range due to distribution of relaxation times or transitions due to different components. It has thermodynamic characteristics of a second-order phase transition (Roos et al., 1996). Glass transition involves no latent heat (Sperling, 1986) but the transition can be detected by observing changes in various dielectric, mechanical and thermodynamic properties (Wunderlich, 1981; Sperling, 1986). These changes allow observation of the transition with various methods, which may detect any of the changes as a function of frequency, temperature, or time (Roos, 1995).

#### *3.2.1.5.1 Glass transition determination*

Most popular methods to determine the temperature range of  $T_g$  currently are DSC method that detects change in heat capacity ( $\Delta C_p$ ) occurring over the transition temperature range (Kalichevsky et al., 1992; Roos, 1995) and mechanical spectroscopy (DMTA) method which measure the effect of a sinusoidally varying stress on dynamic moduli (Maste et al., 1992; Goff, 1995). The two techniques could provide significantly different values (Biliaderis et al., 1999). The discrepancies may be understood by 1) differences induced by the analysis of experimental data, for example, definition of  $T_g$  in the DSC curve and choice of feature in DMTA; 2) differences attributed to the coupling of different structural units with the imposed perturbations, which may differ with respect to nature of the stress or the experimental time. It is therefore not possible to have unique  $T_g$  (Meste et al., 2002). The important discrepancies between published values of  $T_g$  may, in addition, originate from uncertainties in the water content and/or chemical degradation (Vanhal & Blond, 1999). Calorimetric or spectroscopic techniques have some limitations in terms of sample size and shape, and water content control.

Impedance spectroscopy studies the variations of the dielectric constant as a function of temperature or/and frequency and is observed as particularly effective for the study of secondary relaxations in products with low water contents. It provides a good alternative to mechanical spectroscopy for very brittle products (Jouppila & Roos, 1994). Some other methods that allow the determination of molecular mobility in heterogeneous systems are NMR (nuclear magnetic resonance) and EPR (electronic paramagnetic

resonance), which measure the mobility of nuclei and spin probes respectively and have been recognized as potential tools for the study of glass transition (Ruan & Chen, 1998; Champion et al., 2000). For practical purposes, the method of measurement should be chosen according to the application.

#### *3.2.1.5.2 $T_g$ and food products*

The major discrepancy between amorphous food materials and synthetic polymers is found in their chemical composition. Foods are complex mixtures of solids and water, while polymers are composed of repeating units of well-characterized molecules (Roos et al., 1996). For long time the knowledge about glass transition was mainly concerned with non-food materials (mineral glasses, natural and synthetic polymers). From what is known so far about food products and ingredients, it appears, however, that essential features are similar. What makes the food material different is on one hand the frequent heterogeneity in structure and on the other the predominant role of the water as a plasticizer. The study of the different ingredients separately in model systems allows the determination of the temperature/water content area where the physical properties of the system may evolve sharply with temperature (Champion et al., 2000).

Recent works have focused on evaluating glass transition temperature of fruits and fruit based products at low, medium and high moisture range. Sa & Sereno (1994) measured  $T_g$  of grape and strawberry equilibrated at different water activity levels and showed increase in water activity decreases  $T_g$ . Xu et al. (2001) measured influence of  $T_g$  and viscosity on cryostability of frozen concentrated orange juices produced by enzymatic process and squeezing process. It has been shown that some physico-chemical changes in juices during storage were controlled by diffusion. The lower molecular weight components were responsible for the  $T_g$  while the high molecular weight carbohydrates significantly affected the viscosity of orange juice. Rizzalo et al. (2003) evaluated the  $T_g$  of blueberry juices through carbohydrate addition and said  $T_g$  shifted to higher temperatures by addition of maltose and no significant changes in the transition temperatures were noted with storage time.



#### *3.2.1.5.3 Shelf life and quality control*

Temperature, time and water content are the main factors controlling the quality and stability of food during processing and storage. Water content in fresh or high moisture foods forms an admirable media for reaction and diffusion. As  $T_g$  is highly sensitive to water content in foods, the glass transition concept will be a powerful tool for understanding the mechanisms of processes in food products and for controlling their shelf life (Champion et al., 2000). The glass transition temperature is considered as a reference temperature. Below  $T_g$ , the food is stable with very little or no mobility of molecules but changes may occur above this temperature due to the transitional mobility of molecules. The difference between  $T_g$  and the storage temperature  $T$  controls the rate of physical, chemical and biological changes (Eskin & Robinson, 2001). The variations of mechanical and transport properties in the glass transition range could contribute to a better control of some food processing operations such as drying and freeze-drying, extrusion and flaking (Champion et al., 2000).

Recent publications have aimed at a deeper understanding of the basic physical aspects of glass transition in order to determine its real impact in food technology (Simatos et al., 1996; Jang et al., 2001; Rizzola et. al., 2003). Proper determination of  $T_g$  is very important for multi domain food products like cereals with raisins, ice cream with cones. The glass transition concept could be applied to maintain texture difference by designing a confectionary with soft core and rigid coating to limit the transfer of water from one domain to another. Storage at  $T_g$  helps in better retention of flavor. Collapse phenomena, resulting from a change in structure or viscous flow causing stickiness, caking, and loss of porosity can be prevented below  $T_g$ . Preservation of structure and activity of proteins during freeze-drying or storage needs them to be stored below its  $T_g$ . Glass transition concept also helps in maintaining the chemical and microbial stability of concentrated and intermediate moisture foods (Metse et al., 2002).

#### **3.2.2 Rheology**

Parameters from rheological tests are interwoven, complement each other and describe properties of formulations during processing, application, storage and they can

even be translated to economic value. Various factors affecting the rheological behavior of fruit purees and concentrates include temperature (Oomah et al., 1999), soluble solids concentration, particle size (Pelegrine et al., 2002), addition of enzymes (Bhattacharya and Rastogi, 1998) and pH (Dik and Ozilgen, 1994).

#### ***3.2.2.1 Steady state rheology***

Rheological parameters like flow behavior index, consistency coefficient and viscosity are important for calculation in any process involving fluid flow for e.g. pump sizing, extraction, filtration, extrusion and purification. They play an important role in the analysis of flow conditions in food processes such as pasteurization, evaporation, drying and aseptic processing. The viscosity of fruit purees can be significantly affected by variables such as shear rate, temperature, pressure and time of shearing. For Newtonian fluids, at constant temperature and pressure, the viscosity does not vary with the shear rate. For most non-Newtonian fluids, the viscosity decreases with an increase in shear rate, giving rise to pseudoplasticity or shear-thinning behavior (Barnes et al., 1989; Marcotte et al., 2001). It has been recognized that pseudoplasticity presents an irreversible structural breakdown and the decrease in viscosity occurs as a result of change in molecular alignment that takes place within a substance.

#### ***3.2.2.2 Yield stress***

Yield stress ( $\sigma_0$ ) is defined as the minimum shear stress required for initiating flow. It is one of the important behaviors while studying rheological properties. Barnes & Walters (1985) have argued regarding the presence of yield stress that sufficient time and sensitive equipment would make everything flow. However there is no doubt that  $\sigma_0$  is an engineering reality (Harnett and Hu, 1989) that strongly influences the process engineering calculations.

There are many ways to evaluate the yield stress for fluid like substances and no single best technique. Extrapolation of shear stress-shear rate curve is the most common method to obtain the yield stress. This value will strongly influence the rheological models like Bingham, Herschel-Bulkley, etc. selected to represent the data (Ofoli et al.,

1987). An alternative numerical procedure is to plot apparent viscosity versus shear stress and determine  $\sigma_0$  from the point (related to zero shear rate) where  $\eta$  becomes infinite (Steffe, 1992). Cheng (1986) in his review about yield stress problem has written that the magnitudes of measured values are closely associated with creep, stress growth, thixotropy and the time of these transient responses. He has also described a concept of static and dynamic yield stresses that has great value in rheological testing of fluid foods on the idea that there can be more than one type of structure in a thixotropic fluid. One strong structure serves to define the equilibrium flow curve and the other structure, the weaker one, forms over a certain period of time when the sample is at rest. Combined, the two structures cause a resistance to flow, which determines the static yield stress (Steffe, 1992). This type of behavior has been observed for many food products such as apple sauce, banana and peach baby food, mustard, tomato ketchup (Canovas and Peleg, 1983). A true value of yield stress is very important to properly design food processing systems like those required in thermal processing.

### ***3.2.2.3 Dynamic rheology***

The dynamic rheology also called as small amplitude oscillatory testing has gained great research interest in recent years. Here, the viscoelastic behavior is measured by subjecting the samples to oscillatory motion controlling the level of stress to be applied. The magnitude of stress applied depends on the viscoelastic nature of the substance. Investigation on dynamic rheology leads to various properties of materials like dynamic viscosity ( $\eta'$ ), complex viscosity ( $\eta^*$ ), complex modulus ( $G^*$ ), elastic modulus ( $G'$ ) and viscous modulus ( $G''$ ).  $G'$  is the ratio of stress in phase with strain to the strain. Here the stress components are in phase with the strain. When the stress component is  $90^\circ$  out of phase with shear strain we get viscous (loss) modulus ( $G''$ ), which can be defined as ratio of stress out of phase with the strain to the strain (Bourne, 2002).

Dynamic testing has become a common method for studying the viscoelastic behavior of food. It is a non-destructive way of measuring rheological parameters when the oscillations are of small amplitude. It is very useful in gel strength evaluation, protein coagulation observation, and texture development in different food products. It is also important for evaluation of storage stability and in obtaining better sensory perception.

Scientists and engineers have found it as powerful tool for product development (Steffe, 1992).

### ***3.2.2.4 Rheological models***

#### ***3.2.2.4.1 Steady shear rheological models***

Various models (Power Law, Herschel Bulkley, Bingham, Casson model) have been employed to indicate steady flow characteristics of food and food products. Some of the models that have been used extensively are the simple Power Law model with two parameters, the Casson Model with two parameters and yield stress and the Mizrahi-Berk model (M-B) with three parameters and yield stress (Pelegrine et al., 2002). Several researches used Power Law model to describe rheological behavior of fruit pulps, juices and puree (Rao et al., 1985, Pelegrine et al., 2002). The Ostwald-de-Waele (Power Law) model is described as

$$\tau = K \dot{\gamma}^n \quad (3.8)$$

where  $\tau$  is the shear stress (Pa),  $\dot{\gamma}$  the shear rate ( $s^{-1}$ ), K the consistency coefficient ( $Pa \cdot s^{-n}$ ) and n the flow behavior index of the fluid. Casson model demonstrates the effect of suspended material and is extensively used for measuring the viscosity of chocolate and some filled fluids (Bourne, 2002). M-B model is an attempt to match these two effects. Some of foods show yield stress, an important rheological parameter, which cannot be described using Power law.

Casson proposed following model, which includes yield stress:

$$\tau^{1/2} - K_{OC} = K_C \dot{\gamma}^{1/2} \quad (3.9)$$

where  $\tau$  is the shear stress (Pa),  $\dot{\gamma}$  is the shear rate ( $s^{-1}$ ),  $K_{OC}$  is Casson yield stress ( $Pa$ )<sup>1/2</sup> and  $K_C$  the plastic viscosity of Casson ( $Pa \cdot s$ )<sup>1/2</sup>.

Mizrahi and Firstenberg (1975) tested the applicability of the M-B model and the Herschel-Bulkley model as shown below to a series of 60<sup>0</sup>Brix concentrates, varying the relative viscosity of the 11<sup>0</sup>Brix serum and the pulp content

$$\tau - \tau_{OH} = K_H \dot{\gamma}^{nH} \quad (3.10)$$

Vitali and Rao (1984) reported that the M-B model was slightly superior to the H-B model.

Understanding the need for studies on various models in engineering operations like pasteurization and sterilization is as important as to comprehend the role of various constituents in the foods. In the former, the effect of shear rate and temperature must be available in the single expression. In the latter, the effect of temperature and concentration must be combined with the effect of shear rate (Vitali & Rao, 1984).

The effect of temperature on apparent viscosity can be described by the Arrhenius relationship

$$\eta_a = \eta_{\infty} \exp\left(\frac{E_a}{RT}\right) \quad (3.11)$$

where  $\eta$  is the apparent viscosity (Pa.s). Combination of Arrhenius model for the consistency coefficient and Power law model would incorporate the effect of temperature for fluids obeying Power law as

$$\tau = K_{TH} \exp\left(\frac{E_{aH}}{RT}\right) \dot{\gamma}^n \quad (3.12)$$

where  $E_a$  is the activation energy,  $R$  is universal gas constant and  $T$  is the temperature. This equation was used to describe data on tomato concentrates (Rao et al., 1981), and Guava puree (Vitali and Rao, 1984)

For the combined effect of temperature and concentration at constant shear rate a power dependence on concentration was proposed by Harper and El-Sahrigi (1965) as

$$\eta_a = \eta_{ac} C^a \quad (3.13)$$

This was suitable for tomato concentrates (Rao et al., 1981). It may also be written with the consistency coefficient substituted in place of the apparent viscosity (Rao et al., 1985; Steffe, 1992).

#### 3.2.2.4.2 Dynamic rheological models

The ratio of viscous modulus to elastic modulus describes the loss factor or the tangent of phase shift given as equation below

$$\tan(\delta) = \frac{G''}{G'} \quad (3.14)$$

The complex modulus ( $G^*$ ) depends on  $G'$  and  $G''$  as

$$G^* = \sqrt{(G')^2 + (G'')^2} \quad (3.15)$$

and complex viscosity is the ratio of complex modulus and angular frequency given as equation (3.16), where  $\omega$  is the angular frequency (Steffe, 1992)

$$\eta^* = \frac{G^*}{\omega} \quad (3.16)$$

#### 3.2.2.5 Super positioning of steady shear and complex viscosity

It has long been known that polymeric liquids show similarities between steady shear and dynamic rheological properties (Bistany & Kokini, 1983). Cox and Merz (1958) predicted the relationship between dynamic viscosity and steady shear viscosity. According to them at higher shear rates the dynamic viscosity comes closer or equals the steady shear viscosity, which can be explained as follows (Bistany & Kokini, 1983)

$$\eta(\dot{\gamma}) = \eta^*(\omega)_{\omega=\dot{\gamma}} \quad (3.17)$$

where  $\eta(\dot{\gamma})$  is the steady shear viscosity,  $\eta^*(\omega)$  is the complex viscosity and  $\omega$  is the angular frequency and  $\dot{\gamma}$  is the shear rate.

Many efforts have been made on applying Cox-Merz rule to food. But this rule may not be applicable all times due to the complexity of food. Rao & Cooley (1992) applied Cox-Merz rule on tomato pastes and found that this rule was not applicable. But multiplying angular frequency  $\omega$  by a shift factor brought the two parameters close to each other.

#### **3.2.2.6 Rheology and fruit puree**

Numerous studies have been carried out on the rheological properties of fruit puree (Vitali and Rao, 1982; Manohar et al., Ahmed et al., 2005). Several studies are available on the steady shear rheological properties of mango puree/paste (Gunjal and Waghmare, 1986; Ahmed et al., 2005). Generally, mango puree has been described as shear thinning or pseudoplastic material. Consistency coefficient (K) has been found to decrease with increase in shear rate and temperature. Flow behavior index (n) increase with shear rate but has not shown particular trend with temperature. Recent papers have shown the presence of yield stress in mango puree (Bhattacharya & Rastogi, 1998; Ahmed et al., 2005). Very less data is available on the dynamic properties and application of Cox-Merz rule to mango puree.

### **3.3 Thermal processing**

#### **3.3.1 Thermal processing of fruit products**

There has always been a great interest in applying heat treatment for developing new products. Heat processing provides safe, quality foods, provided enzymes are inactivated. The key advantages of heat processing are (Fellows, 2000):

1. Relatively simple control of processing conditions
2. Foods that are shelf stable at room temperature can be produced
3. Improvement in the availability and digestibility of proteins and gelation of starches and proteins.

But the main disadvantage of thermal processing is reduced sensory and nutritional properties especially in products like delicate fruits and fruit products. Fruits

are commodities with special organoleptic properties that must be carefully preserved. Food additives like color and flavor are usually added to improve appearance, flavor and quality of thermally processed products.

### **3.3.2 Thermal processing of whey protein**

Understanding the mechanism of interaction of whey protein concentrates/isolates with different food products plays an important role in food industry. Whey proteins have very good ability to gel on heating and therefore been utilized as ingredients in formulated foods. During gelation, proteins unfold and the exposed hydrophilic groups bind large quantities of water and increase viscosity. This behavior is useful in the formation of high viscous products like jams, fruit jellies and protein shakes. Pre-heated whey protein dispersion is used in cold setting whey protein products like yogurts, by gelling at low temperatures. Heat caused denaturation is also highly used in cheese making.

However, heat treatment of whey proteins also has some disadvantages like insolubilization, which is a major obstacle for the inclusion of whey proteins in heated beverages. A quality defect associated with whey protein added fruit beverages and other liquid food during their production and storage is the formation of sediment. This can be the result of interactions between heat-labile whey proteins and some of the fruit components like pectin, even when pH below 3.5-3.7 where whey proteins are heat-stable (Patocka et al., 1986; Iordache & Jelen, 2003).

Structure and solubility of whey proteins is usually affected by heat treatments. Heat treatment of proteins and other nutrients can induce various chemical reactions like Maillard browning which leads to nutritional, sensory and safety deterioration in certain foods (DeMan, 1996; Hayashi et al., 1989; Knorr et al., 2002; Sekai et al., 2005). Fuente et al. (2002), reviewed on the aggregation of whey proteins affected by temperature, protein concentration and pH. Various binding mechanisms like covalent disulfide and non-covalent bonds are important in the formation of whey protein aggregates (Schokker et al., 2000). When heated above 65-75°C, whey proteins in aqueous solution may either precipitate due to production of heat-induced insoluble aggregates or may form viscoelastic gels at higher protein concentrations (Iordache & Jelen, 2003).



### **3.4 Non-thermal processing**

Today's consumer demands foods that are fresh, safe and natural. There is always a challenge for designing a process, which preserves the natural qualities of product with minimal heat treatment. High pressure processing (HPP), pulse electric field, irradiation, and membrane filtration are some of the non-thermal techniques, which are of great interest. But there is an increasing trend towards the application of HPP, a minimal or non-thermal technology, which prolongs the shelf life of foods without the detrimental effects found in thermal treatment (Sekai et al., 2005). HPP opens a new dimension in food processing not only by inactivating microorganisms but also by preserving the fresh quality of products. HPP offers opportunities for creating new texture with better functional properties in different foods. Application of high pressure is instantaneous, uniform and independent of sample volume. As the operation can be maintained at low or ambient temperature, there is narrow chance of changing the chemical properties of product.

#### **3.4.1 High pressure processing (HPP)**

Studies on the applications of HP in food technology are seen from end of 19<sup>th</sup> century (Hite 1899). But the real interest in HP processing developed only after 1970, may be due to lack of suitable equipment before that time. This interest has increased considerably over last decade or so (Balci & Wilbey, 1999). HPP is being widely used in food industry for food preservation, gelatinisation, enzyme inactivation and pressure-shift freezing and thawing.

##### ***3.4.1.1 Principles of HPP***

The general behavior of biological system under HP is governed by two basic principles:

##### ***Isostatic principle (Pascal's law)***

It states that application of HP is independent of sample size or geometry. The product undergoes homogeneous treatment instantaneously and in short time. Thus, over processing, a major concern in thermal processing is overcome.

### *Le Chateliers principle*

According to this principle, whenever stress is applied to a system in equilibrium, the system reacts as to counteract the applied stress. This promotes reduction in volume of sample under HP. Such reactions result in inactivation of microorganisms or enzymes and textural changes in foods (Balci & Wilbey, 1999; Huppertz et al., 2002). Small molecules like vitamins and flavor components contributing to nutritional and sensory quality are not affected as only non covalent bonds (hydrogen, ionic and hydrophobic bonds) are sensitive to HP treatment.

#### **3.4.1.2 Advantages of HPP**

HPP has many advantages (Ramaswamy et al., 2004)

1. Application of HP is uniform regardless of size and shape of sample, overcoming under and/or over processing.
2. As the covalent bonds are not affected by pressure, development of flavor alien to product is prevented, maintaining the natural quality of the product.
3. It is applied at very low or room temperature thereby minimizing the amount of thermal energy needed for food products during conventional processing.
4. It is time or mass independent; it reduces processing time.
5. It is environmentally friendly; energy efficient and there are no waste products.

However, like other process, HPP also has certain limitations,

1. Very high pressure is necessary to inactivate food enzymes and bacterial spores, which are highly resistant to pressure.
2. Most pressure processed foods need to be stored at low temperatures to retain their sensory qualities.

#### **3.4.1.3 HP and whey protein**

Dietary proteins with high disulfide content are highly resistant to digestion (Astwood, 1996).  $\beta$ -lg, which is one of the major constituents of whey protein, resists gastric digestion and remains intact in the stomach (Mahe et al., 1991; Hagemeister &

Antila, 1994). It is also shown that  $\beta$ -lg is less susceptible to peptic, tryptic or chymotryptic digestion than other milk proteins (Otani, 1992).

#### *3.4.1.3.1 Denaturation*

Pressurization of whey protein for nutritional applications has been focused upon enzymatic hydrolysis at elevated hydrostatic pressures for substrates such as  $\beta$ -lg that cannot be readily hydrolysed at ambient conditions (Hosseini et al., 2002). Stapelfeldt et al. (1996) reported that in vitro hydrolysis of  $\beta$ -lg by pepsin was facilitated by exposure of  $\beta$ -lg to pepsin at pressure as 300MPa. Hosseini et al. (2002) has shown that most of the secondary structure in whey proteins were unaffected by the application of pressure up to 400MPa. The structural changes of proteins are due to the disruption of noncovalent bonds while covalent bonds remain unaffected (Mozhaev et al., 1994). The non disruption of covalent bonds ensures the retention of essential vitamins and nutrients (Tedford et al., 1999). Hayakawa et al. (1992) using spectrofluorometry demonstrated the conformations of BSA and ovalbumin remained fairly stable up to 400MPa. Repeated pulse cycling of pressures up to 400 MPa is required for the protein denaturation and irreversible changes in the secondary structure of whey proteins including a major drop in the S-S content (Hosseini et al., 2002).

#### *3.4.1.3.2 Applications*

Since the allergenic reactions are dependent on antibody recognition of specific epitopes on the protein molecule, irreversible changes in the protein conformation induced by high pressure might result in a reduction of allergies (Hosseini et al., 2002). Physiological properties like stimulation of immune system function, antioxidative and glutathione stimulating activities (GSH) related to whey protein may be due to the release of bioactive peptides from digestion (Gattegno et al., 1988; Nicodemo et al., 1999; Meisel et al., 1989). It is possible that the combined and repeated cycling of pressurization modes could increase the digestibility of whey proteins via disruption of the S-S bonds. This enhances the release of more bioactive peptides such as  $\gamma$ -

glutamylcysteine that allow for a more rapid intracellular uptake of cysteine towards GSH synthesis (Hosseininia et al., 2002).

#### ***3.4.1.4 HP and food products***

First high pressure processed food, a fruit jam, was introduced in Japanese market (Mertens & Deplace, 1993). Combination of very high pressure (700-1000MPa) and temperature (70-90°C) is been recently used for sterilization of low-acid foods. Strawberry jam prepared by high pressurization not only maintains original color and flavor but also retains 95% of the vitamin C originally present in fruit. The texture of several fruits and vegetables has been shown to undergo slight firming as a result of HP treatment. This effect can be used to induce hardening without using chemical additives to protect texture and original form of fruits

HP has also been used to induce gelation of different kinds of surimi. Better quality gels have been produced from pollack, sardine, skipjack and tuna at 400 MPa. Pressure induced gels are smoother, more elastic and organoleptically superior to heat induced gels (Ramaswamy et al., 2004).

#### ***3.4.1.5 Commercial HP processed food products***

A number of HP processed fruit products are now available including orange juice, apple juice, apple cider, applesauce, strawberry juice, avocado halves/puree, jams/jellies, lemonade, fruit purees and fruit smoothies. Beef, chicken, cod (both dried and salted), guacamole, mussels, oysters (shucked, in-shell, and shooters), ham, ready-to-eat meal kits (beef and chicken), salsa, seafood salads are some of HP processed commercial meat products. But HP processed mango puree or its by-products are yet not available.

#### ***3.4.1.6 HP and mango/mango by-products***

Otero et al. (2000) evaluated the modifications in microstructure of peach and mango due to conventional freezing and high-pressure-shift freezing. With the high-pressure-shift method, samples were cooled under pressure (200 MPa) to -20°C without

ice formation and then pressure was released to atmospheric pressure (0.1 MPa). Uniform and rapid ice nucleation throughout the volume of the specimen was observed due to high level of super cooling (approximately 20°C). This method maintained the original tissue structure to a great extent. As there was no thermal gradient during pressurization, high-pressure-shift freezing prevented quality losses due to freeze-cracking or large ice crystal presence.

Boynton et al. (2002) vacuum-sealed the sliced mangoes and processed at 300 MPa and 600 MPa for 1 min. Fresh mango flavor declined and off-flavor increased during storage at 3°C, however, color, texture, and other sensory attributes changed slightly. Pressure treatments also slightly reduced fresh mango flavor and increased off flavor and sweetness.

Ahmed et al. (2005) applied pressure (100-400MPa) on both fresh and canned mango puree to study the change in rheological properties. Canned mango was very little affected by pressure compared to fresh puree. Hiremath (Masters thesis, 2005, Dept. Food Science, McGill University) showed that the pressure treated (550MPa) mango juice maintained their superior quality throughout the 60-day test period and no microbial growth was observed at 4°C. Both above-mentioned works show that color is minimally affected by pressure.

#### ***3.4.1.7 Effect of HP on rheological properties of protein***

It is already well established that protein structure, product texture and thus rheological parameters are susceptible to modification by static HPP. Microstructure and rheology of whey protein added fruit products are influenced by factors such as protein concentration, pH and soluble solids in product. Zasytkin et al. (1996) reported that  $\beta$ -lg gels can be stabilized against syneresis by the addition of xanthan and confirmed that a more elastic protein gel network was formed on addition of the polysaccharide. The gel strength of HP induced whey protein concentrate gels as a function of pH has been studied by various researchers (Kanno et al., 1997; Walkenstrom & Hermansson, 1997). The optimum pH for the formation of HP induced gels with the highest hardness was pH 9, while the lowest hardness occurred in the acidic pH range. The combined use of elevated temperature and pressure was found to give variable results, which was highly

dependent on the kind of protein under study (Van Camp et al., 1996). Whey protein concentrate formed gels whose strength increased with an increase in pressurization temperature. The improvement was more noticeable at high protein concentration where stronger gels were obtained by pressurization (400 MPa at 50°C for 30 min). HP treatment is regarded as a milder processing operation than thermal processing. Strong emulsion gels have been produced from concentrated emulsion samples following treatment at > 70°C for 5 min (Dickenson & James, 1998). Furthermore, the increase in viscoelasticity caused by moderate heating (65°C for 5 min) resembled the change in rheological behavior induced by severe pressure treatment of 800 MPa for 60 min.

Ngarize et al. (2005) compared the deformation in rheological properties of egg albumen and whey protein isolate gels induced by heating (90°C for 30 min) to those induced by HP (400–800 MPa for 20 min). Significant differences in appearance and textural properties between heat and pressure induced gels were observed. Pressure induced whey and egg albumen gels were glossy and smooth in appearance with a rubbery texture compared with heat-treated gels, which were less glossy but were harder and more brittle. In addition, pressure-treated gels had significantly lower values of force at maximum compression and Young's modulus than gels heated at 90°C for 30 min. Pressure-treated gels also tended to lose water easily upon compression due to the presence of large aggregates with irregular pores. Okamoto et al. (1990) on egg albumen and yolk, paste of rabbit meat, fish and soy protein reported similar findings with pressure-treated gels being characterized by soft glossy gels while heat-treated gels were harder. The gel strength increased with increasing pressure for both whey and egg albumen proteins.

## **CONNECTING STATEMENT**

Chapter IV is prepared as a manuscript “Effect of concentration and temperature on thermo-physical and rheological properties of mango puree” for submission to a refereed journal and is authored by Gundurao, A., Ahmed, J. and Ramaswamy, H.S.

Gundurao is the candidate and the principal author of the paper. Ramaswamy is the thesis supervisor under whose guidance the research work was carried out. Ahmed contributed to the development of topic and for helping with drafting of the paper.

This chapter focuses on the evaluation of thermal and rheological properties of mango puree as affected by soluble solids concentration and temperature.

## **CHAPTER IV**

# **EFFECT OF CONCENTRATION AND TEMPERATURE ON THERMO-PHYSICAL AND RHEOLOGICAL PROPERTIES OF MANGO PUREE**

### **4.1 Abstract**

Thermo-physical and rheological and properties of mango pulp were evaluated at four temperatures (20, 40, 60 and 80°C) and total soluble solids concentrations (15, 20, 30 and 40°Brix). Thermal properties were primarily dependent on the moisture content of the sample, and increased with temperature and decreased with concentration. Density showed a reverse trend. Glass transition temperatures increased with an increase in concentration indicating better stability. Separate models were developed for each thermal property as a function of temperature and concentration ( $R^2 > 0.90$ ). Mango puree exhibited pseudoplasticity during steady shear measurements, and the power law model well described their flow behavior. Consistency coefficient increased with concentration and decreased with temperature. The flow behavior index decreased with concentration and generally increased with temperature beyond 40°C. Dynamic oscillation shear measurements revealed that mango puree behaved like a weak gel and demonstrated visco-elastic properties.

### **4.2 Introduction**

Mango is one of the important fruits of tropical countries and is consumed as whole as well as processed forms like pulp, juice, soft drinks, nectar, leather etc. Mango has been the most studied fruit in the tropics. India holds the top position in mango production with 40% of world production and export (FAO, 2004). Alphonso is one of the best mango variety produced in India, which is relished for its attractive color, exotic flavor and superb delicacy. Mango puree - a semi-finished form has been utilized by food



processing industries to make various food formulations. Sometimes sugar is added to increase the total soluble solids of pulp/puree and acidified to reduce the pH to enhance the shelf life of the product.

Reliable estimates of thermal properties are essential to understand the rate of heat transfer during food processing including cooking, drying, freezing, pasteurization, sterilization. Proper design of industrial plants, modeling and automation of food process industries require these properties to obtain better quality control and improve the shelf life of the products. Some of the important thermo-physical properties are thermal conductivity, diffusivity, density, specific heat, and glass transition temperature. These parameters vary with temperature and product concentration.

Thermal conductivity and diffusivity are affected by moisture content and temperature. Both moisture and temperature change significantly during food processing and, therefore, estimation and availability of thermal property data could help process industries. Various techniques (steady state: guarded hot plate, concentric cylinder and heat flux method; transient techniques: line heat source, thermal comparator technique) are available to measure thermal conductivity ( $k$ ) of food materials. However, line heat source is the simplest among those, which needs small sample volume and provides accurate results with pre-calibration (Sweat, 1986). The author recommends probe length to diameter ratio of greater than 25 and very small diameter (0.66mm) could eliminate finite size correction factor and produce accurate result. Specific heat is another important thermal property that helps to better understand the heat transfer phenomenon. Empirical equations estimating specific heat verify the dependency of specific heat on water content of food components (Romero & others, 1998; Saravacos & Kostaropoulos, 1995). DSC has been frequently used to measure specific heat of various food products (AbuDagga et al., 1997; Zainal et al., 2001).

Glass transition is a powerful tool for understanding quantification of water mobility in foods and for controlling the shelf life of products. The temperature below  $T_g$  of any food product has been considered to be stable, and there will be no considerable change in physio-chemical or biological qualities. The changes from the glassy state to the rubbery state occur as a second order phase transition at a temperature known as the glass transition temperature ( $T_g$ ).  $T_g$  is product specific (Roos and Karel, 1991; Slade and

Levine, 1991) and is a function of moisture content. Determination of glass transition temperature is more complicated with food products because of their chemical and microstructural complexity. Glass transitions often extend over a large temperature range, due to a broad distribution of relaxation times and/or because of unresolved transitions corresponding to different phases in multi-component mixture. If normal  $T_g$  of product is very low or not practical to achieve, it may be increased by addition of sugar, starch or gums that have very high glass transition temperature (Torreggiani et al., 1999; Slade & Lavine, 1991).

Rheology has important roles in food product development, quality control, sensory evaluation, design and evaluation of the process equipment. Measurements of rheological parameters have been recognized as an analytical tool to provide fundamental insights on the structural organization of food and play a significant role in fluid heat transfer. Numerous literatures have cited on effect of temperature and concentration on flow behavior of mango puree (Manohar et al., 1990, Bhattacharya et al., 1998, Guerrero et al., 1998, Ahmed et al., 2005) and these authors reported the flow behavior was characterized by the Herschel-Bulkley model. The process parameters like temperature, concentration, shear rate and pressure significantly affect the rheological properties.

Most concentrated structured fluids exhibit strong viscoelastic behaviours at small deformations. With the advent of rheometer, oscillation dynamic measurement replaces steady flow measurement of fluid foods with less texture damage, work at considerably low angular frequency, and repeatability. The dynamic rheological properties in terms of elastic ( $G'$ ) and viscous modulus ( $G''$ ) are used to characterize the viscoelastic properties of fluid foods. In qualitative terms the oscillatory curves give a fingerprint of the state of the microstructure, in the same way as does an NMR or an infrared spectrum (Barnes, 2000). Limited information is available on dynamic measurement of mango puree as function of temperature and concentration.

Considering the importance of all these parameters for mango puree for process industries the objective of this work is to evaluate the thermo-physical and rheological properties as function of temperature and concentrations.

## **4.3 Materials and methods**

### **4.3.1 Material**

Canned mango pulp (Cv. Alphonso produce of India and marketed by Phoenicia Products Inc. Montreal, Canada) was chosen as the product that has exotic flavor and commercial demand for manufacturing of processed products. All the experiments were conducted at four selected temperatures (20, 40, 60 and 80°C) and total soluble solids (TSS) content (15, 20, 30 and 40°Brix). The original TSS of the pulp was 28°Brix. Distilled water was used to dilute the pulp to 15 and 20°Brix while sucrose was added to concentrate the sample to 30 and 40°Brix.

The total soluble solids and pH were measured by a hand refractrometer (Atago, Japan) and pH-meter (Acumet, ON, Canada) respectively. Water activity was measured using a hygrolab humidity temperature indicator (Aw-DIO, rotronic Inst. Corp.).

### **4.3.2 Thermal analysis**

For thermal analysis, mango puree samples were scanned in a differential scanning calorimeter (DSC) (TA Q100, TA Instruments, Newcastle, DE, USA) calibrated with indium for heat flow and temperature. The DSC was equipped with a refrigerated cooling system that efficiently monitored temperature up to -90°C. Nitrogen was used as purge gas at a flow rate of 50 mL/min. Hermetically sealed aluminum pans were used to avoid any moisture loss during the analysis. For glass transition temperature the samples of mango puree were sealed, cooled to -60°C, and then subjected to a programmed thermal scan at heating rate of 10°C/min to 100°C while the specific heat capacity was determined at temperature range of 10 to 100°C. A four axis robotic device on the system was used to automatically load samples and the reference pans to the DSC chambers. An empty aluminum pan was used as a reference. All DSC measurements were done in duplicate. DSC data were analyzed with the Universal Analysis Software (version 3.6C) for thermal analysis, which was provided with the instrument (TA Instruments, Newcastle, NJ). The glass transition temperature was obtained from the midpoint of onset and endpoint temperatures.

#### 4.3.3 Thermal conductivity

The thermal conductivity of mango puree sample was measured by T-type line heat source thermal probe. The probe was similar to the one described by Sweat (1975). The length of the probe was 50 mm and the diameter was 0.7 mm. The probe was calibrated using 0.4% agar gel.

The mango puree samples (approximately 80ml) was transferred to a 100ml glass container and equilibrated to pre-set temperature in a water bath prior to measurement. Fiberglass wool was added (1% by sample weight) to puree sample to prevent convection at elevated temperatures. The probe was completely immersed in the sample and kept for 2-3 minutes to stabilize with sample temperature. Mango puree samples were put inside the temperature controller to measure the thermal conductivity at 20, 40, 60 and 80°C. The power (3V, 200mA) was supplied to the probe and the time-temperature data were recorded each second using a computer controlled HP data logger (349708, Hewlett Packard, USA) for 30 seconds. The data were collected in triplicates for each concentration. A semi-log plot was made between time-temperature data and the resulted slope was used in equation (4.1) to evaluate thermal conductivity.

$$k = \frac{Q}{4\pi} \left( \frac{1}{slope} \right) \quad (4.1)$$

where k is the thermal conductivity ( $\text{Wm}^{-1}\text{C}^{-1}$ ) and Q is the supplied heat in J.

#### 4.3.4 Density and thermal diffusivity

A pycnometric method was used to measure the sample density. The thermal diffusivity was calculated using the equation (4.2) by using thermal conductivity and specific heat data obtained from the method described earlier. The average of three replicates was reported.

$$\alpha_T = \left( \frac{k}{\rho C_p} \right)_T \quad (4.2)$$

where  $\alpha$  is the thermal diffusivity ( $\text{m}^2\text{s}^{-1}$ );  $\rho$  is the density ( $\text{kg}(\text{m}^3)^{-1}$ ) and  $C_p$  is the specific heat in ( $\text{Jkg}^{-1}\text{C}^{-1}$ ).

#### 4.3.5 Rheological measurements

A controlled-stress rheometer (AR 2000, TA Instruments, New Castle, DE) equipped with a manufacturer supplied computer control software (Rheology Advantage Data Analysis Program, TA, New Castle, DE) was used to study both steady shear and dynamic oscillatory measurement of the mango puree sample. A 60 mm parallel plate attachment was used with a gap of 1000 microns. The AR 2000 was supplemented with an efficient Peltier temperature control system and the sample temperatures were precisely controlled and monitored. For each test, a measured volume (approximately 2 mL) of mixed sample was placed on the bottom plate of the rheometer. The test temperature was ranged between 20 and 80°C. The exposed sample perimeter was covered with metal trap to minimize evaporation at higher temperature. Dynamic oscillatory tests were carried out at a frequency sweep from 0.1 to 10 Hz. The oscillation stress was selected based on linear part of the viscoelastic range (0.2-0.3 Pa). Each time, a new sample was used for rheological measurement. All the rheological measurements were carried out in triplicate. Elastic modulus ( $G'$ ), viscous modulus ( $G''$ ), complex viscosity ( $\eta^*$ ), and phase angle ( $\tan \delta$ , ratio of loss modulus to storage modulus) were obtained directly from the software (Rheology Advantage, TA version 2.3).

For steady flow measurements, the rheometer was programmed for the set temperature and equilibrated for 2 minutes following a two-cycle programmed shear changing from 0.1 to 100  $\text{s}^{-1}$  and back to 0.1  $\text{s}^{-1}$  in 10 min. Rheological parameters (shear stress, apparent viscosity and shear rate) were obtained from the software.

Various rheological flow models based on shear stress-shear rate (Newtonian, Bingham, Casson, power law, Herschel Bulkley) were tested and the best fit model was selected on the basis of standard error, which is defined as:

$$\sum [(X_m - X_c)^2 / (n - 2)]^{0.5} / \text{Range} \times 1000 \quad (4.3)$$

where  $X_m$  is the measured value;  $X_c$  is the calculated value;  $n$  is the number of data points and range is the maximum value of  $X_m$  – the minimum value.

#### 4.3.6 Error estimation

The deviation between experimental and model values was represented by average percent error (E) using equation (4.4):

$$E = \sum \left( \left| q_{\text{exp}} - q_{\text{pred}} \right| / q_{\text{exp}} \right) \times 100 / n \quad (4.4)$$

#### 4.3.7 Statistical analysis

Statistical data analysis was done using SPSS (SPSS for windows, SPSS 8.0, SPSS Inc.). The significance of the data was considered at  $P \leq 0.05$  level.

## 4.4 Results and discussion

Water activity and pH of mango puree at different TSS are shown in table 4.1. Both pH and water activity decreased with the increase in total soluble solids.

**Table 4.1 Total soluble solids, pH and water activity of mango puree.**

Total soluble solids (°Brix)	pH	Water activity
15	3.86	0.977
20	3.82	0.970
30	3.78	0.942
40	3.76	0.935

### 4.4.1 Thermal conductivity

The thermal conductivity of mango puree varied with temperature and concentration. The values ranged between 0.388 and 0.592 W/m°C at the wider temperature and concentration range (Table 4.2). The thermal conductivity of mango puree sample was described by a regression equation (4.5) and the obtained parameters were compared with the model (4.6) developed by Choi & Okos (1986).

$$k = 0.557 + 1.59 \times 10^{-3} T - 5.4 \times 10^{-3} X_w \quad (4.5)$$

$$k = \sum_{i=1}^n k_i Y_i \quad (4.6)$$

where T is temperature (°C),  $X_w$  is the TSS (°Brix),  $k_i$  is the thermal conductivity of components and  $Y_i$  is the volume fraction of the component. Both models fit the data adequately ( $R^2 > 0.93$ ); however, the average percent error of equation (4.6) was much higher (10.15%) compared to the developed model ( $E = 3.09\%$ ). Figure 4.1a shows the

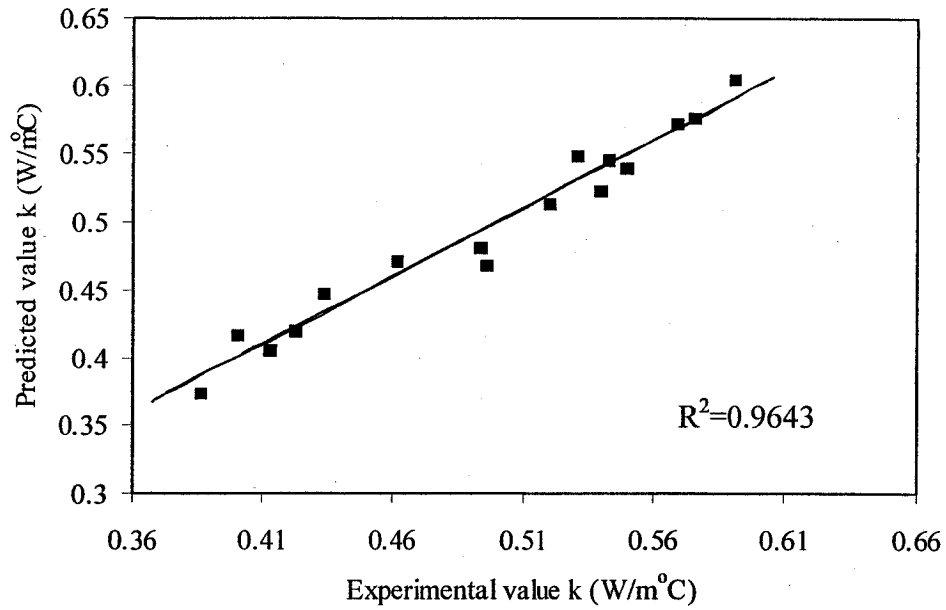
correlation between predicted and experimental values of thermal conductivity. Figure 4.1b illustrates thermal conductivity as function of concentration at 20 and 80°C.

**Table 4.2 Thermal properties of mango puree at selected temperature and concentrations.**

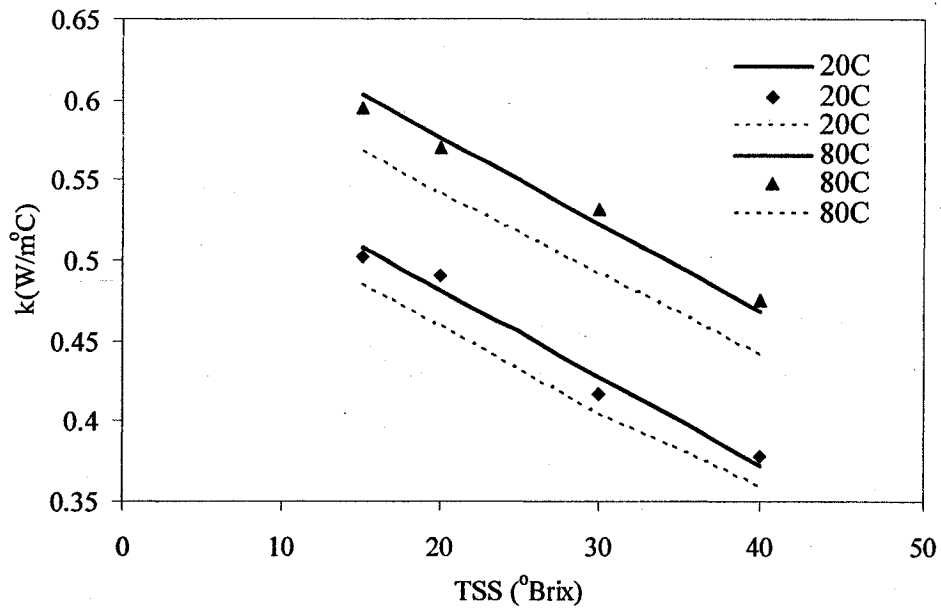
Concentration (°Brix)	Temperature (°C)	k (Wm <sup>-1</sup> °C <sup>-1</sup> )	C <sub>p</sub> (kJ kg <sup>-1</sup> °C <sup>-1</sup> )	Density (Kg(m <sup>3</sup> ) <sup>-1</sup> )	Diffusivity (m(s <sup>2</sup> ) <sup>-1</sup> x10 <sup>7</sup> )
15	20	0.5317	3.638	1075	1.359
	40	0.5505	3.720	1069	1.384
	60	0.5691	3.826	1057	1.407
	80	0.5917	3.811	1050	1.478
20	20	0.4940	3.451	1094	1.308
	40	0.5205	3.560	1089	1.342
	60	0.5439	3.654	1083	1.374
	80	0.5755	3.703	1076	1.444
30	20	0.4012	3.307	1135	1.068
	40	0.4233	3.439	1123	1.096
	60	0.4622	3.466	1116	1.195
	80	0.5411	3.492	1108	1.398
40	20	0.3876	3.113	1192	1.044
	40	0.4131	3.225	1185	1.081
	60	0.4343	3.327	1179	1.107
	80	0.4959	3.388	1172	1.249

The thermal conductivity values increased with temperature ( $P>0.05$ ) while a significant ( $P<0.05$ ) decreasing trend was noticed with concentration. Addition of sugar reduced the moisture content in the puree sample resulting decrease in thermal conductivity. Similar observations were earlier reported for different fruit juice concentrates (Romero et al., 1998; Shamsudin et al., 2004).





**Figure 4.1a Comparison of predicted and experimental values of thermal conductivity.**



**Figure 4.1b Effect of concentration on thermal conductivity of mango puree at 20 and 80°C (comparison with published models: — predicted values (Eq.4.5); ..... Choi & Okos model ; symbols experimental values)**

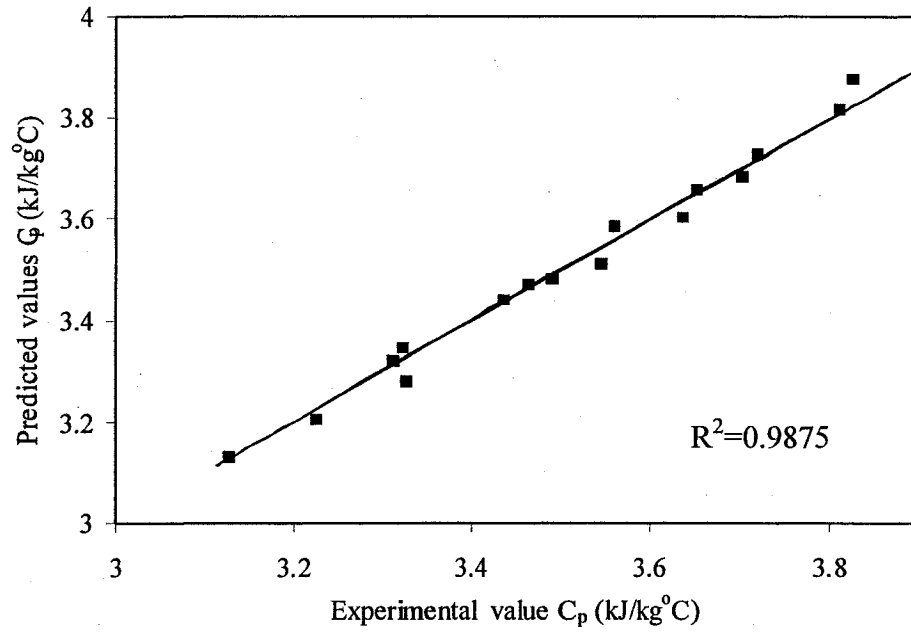
#### 4.4.2 Heat capacity

The variations of specific heat with respect to temperature and concentrations are also tabulated in Table 4.2. The specific heat of mango puree ranged between 3.11 and 3.81 kJkg<sup>-1</sup>°C<sup>-1</sup> while the temperature and concentration varied between 20 to 80°C and 15 to 40°Brix, respectively. The  $C_p$  was found to increase with temperature ( $p<0.05$ ) where as decrease with concentration ( $p<0.05$ ). Figure 4.2a compares the predicted and experimental values of heat capacity and 4.2b illustrates the effect of concentration at 20 and 80°C. The  $C_p$  of mango puree sample was found to follow a linear relationship with temperature and concentration, and represented by the following equation (4.7):

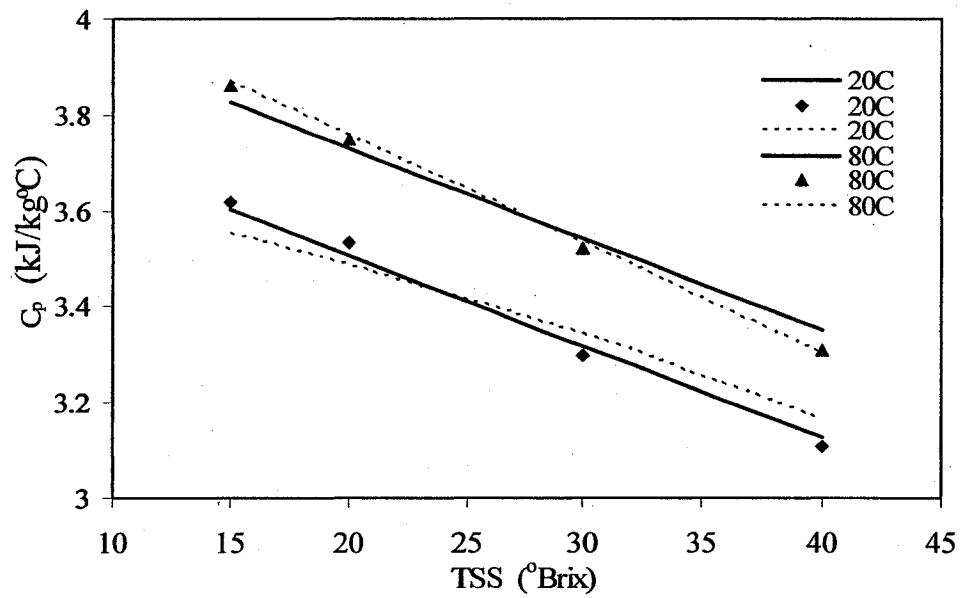
$$C_p = 3.81 + 3.73 \times 10^{-3} T - 1.9 \times 10^{-2} X_w \quad (4.7)$$

$$C_p = \sum C_{pi} X_i^w \quad (4.8)$$

The experimental data fitted well ( $R^2>0.96$ ). The above equation was compared with the model (4.8) described by Choi and Okos (1986) and higher average estimated error was found in the later model (2.36% for Choi and Okos compared to 0.90% for Eq.4.7). A similar trend was reported for other fruit juice/concentrates by Zainal et al., (2001) and Constenla et al. (1989).



**Figure 4.2a Comparison of predicted and experimental values of heat capacity.**

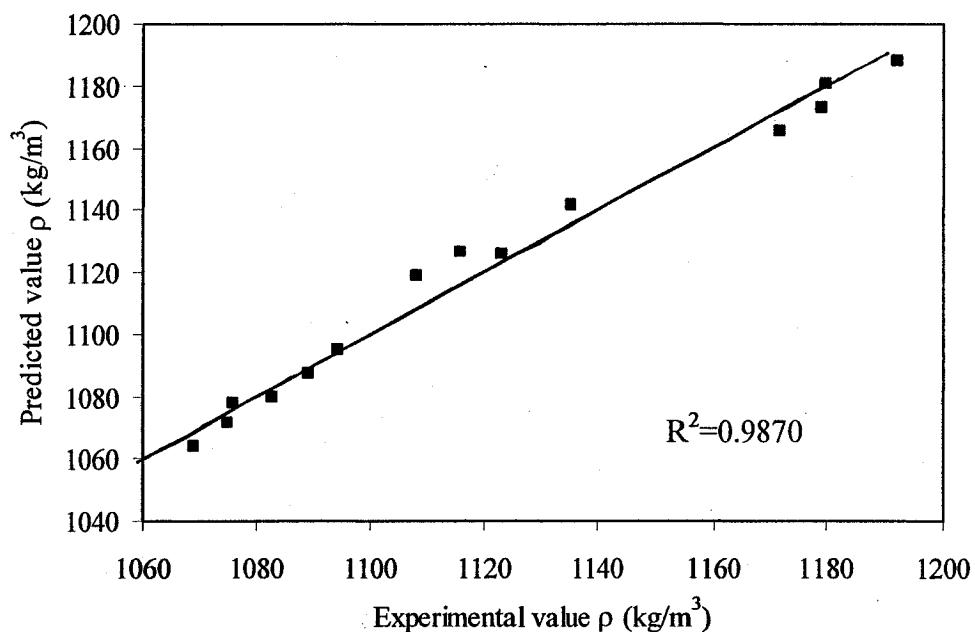


**Figure 4.2b Effect of TSS on specific heat of mango puree at 20 and 80°C**  
 [comparison with published models:— predicted value (Eq.4.7); ..... Choi & Okos model; symbols experimental values]

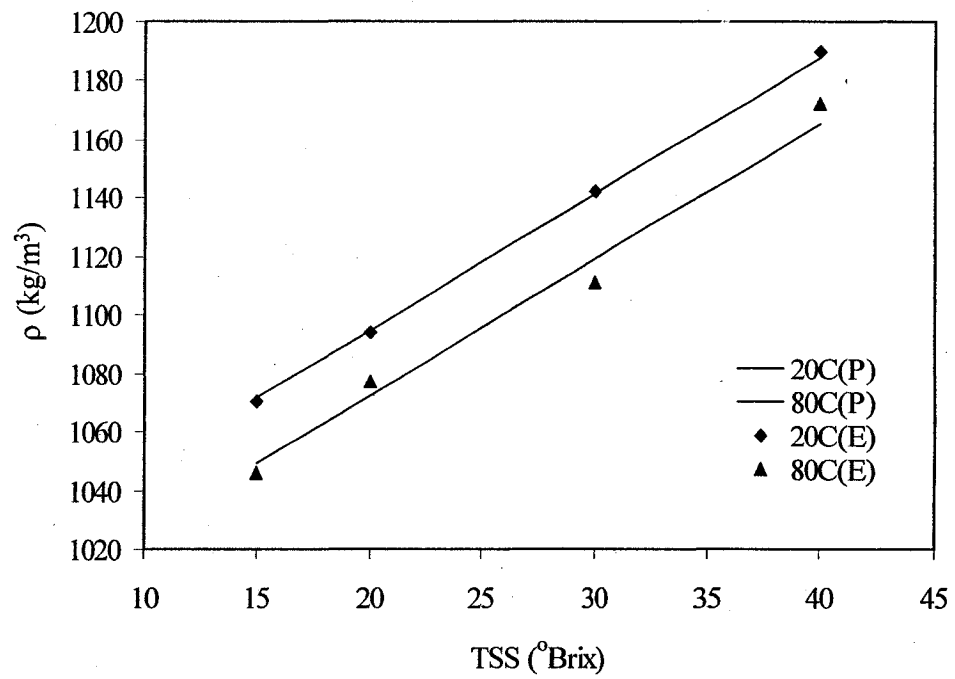
#### 4.4.3 Density

Figure 4.3a represents the correlation between experimental and predicted values where as variation of density with concentration at 20 and 80°C is shown in Figure 4.3b. Equation (4.9) represents ( $R^2 = 0.99$ ) the variation of density with concentration and temperature. Density of mango puree varied from 1050 to 1192  $\text{kg m}^{-3}$  at selected temperature and concentration ranges.

$$\rho = 1009.5 - 0.376T + 4.65X_w \quad (4.9)$$



**Figure 4.3a Correlation between experimental and predicted values of density of mango puree**



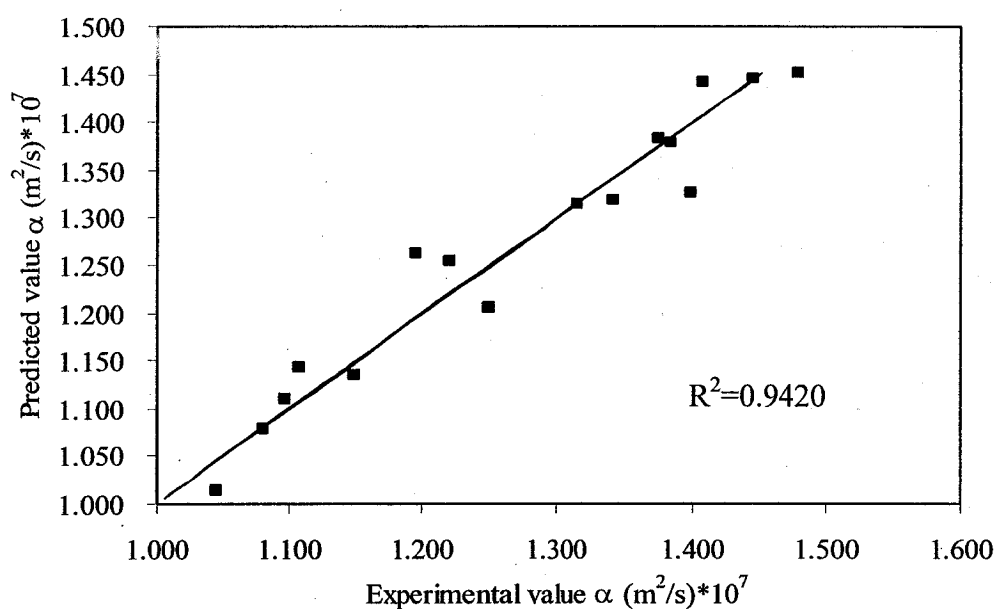
**Figure 4.3b Density of mango puree at selected concentration levels and 20 and 80°C ( —predicted values (Eq.4.9); symbols, experimental values)**

#### 4.4.4 Thermal diffusivity

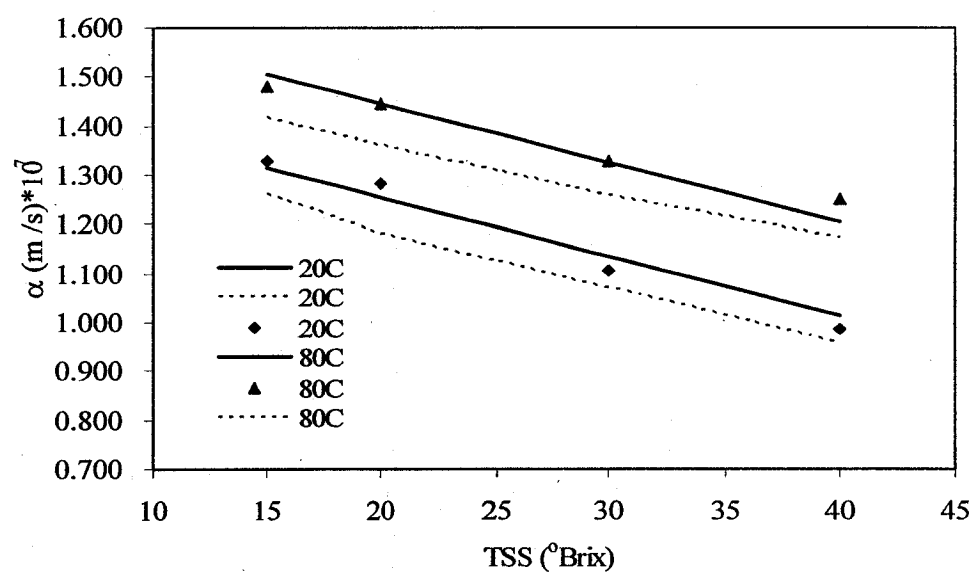
The thermal diffusivity ( $\alpha$ ) of mango puree as function of concentration and temperature are reported in Table 4.2. With increase of concentration  $\alpha$  decreased while an increasing trend was observed at higher temperatures (Figure 4.4b). This is obvious as thermal diffusivity depends on thermal conductivity and specific heat of sample. The variations of  $\alpha$  value followed a linear relationship ( $R^2>0.93$ ) (equation 4.10) with temperature and concentration. Figure 4.4a compares the predicted and experimental values of  $\alpha$ . Equation 4.10 was compared with model (4.11) predicted by Martens (1980) and higher average estimated error was found in the later model (4.05% compared to 9.11%). A similar trend was reported for other fruit juice/concentrates (Romero et. al., 1998).

$$\alpha = 1.43 \times 10^{-7} + 3.2 \times 10^{-10} T - 1.2 \times 10^{-9} X_w \quad (4.10)$$

$$\alpha = [0.057363 X_w + 0.000288 \times (T + 273)] \times 10^{-6} \quad (4.11)$$



**Figure 4.4a Comparison of predicted and experimental values of thermal diffusivity of mango puree.**



**Figure 4.4b Thermal diffusivity of mango puree as function of TSS at 20 and 80°C [comparison with published models: — predicted value Eq(4.10); ..... Martens model; symbols experimental values)**

#### 4.4.5 Glass transition temperature

The presence of the glass transition phase in the system has been identified on the thermograms by a change in heat capacity of mango puree. Typical thermograms for mango puree at different concentrations are shown in Fig 4.5. The glass transition temperatures (onset, mid and end point) and changes in heat capacity during thermal transition are shown in Table 4.3. The glass transition temperatures were quite stable.

The magnitudes of  $T_g$  decreased with an increase in soluble solids content resulting in a maximum glass transition temperature at 40°Brix. Jang et al. (2001) observed similar decreasing trend in  $T_g$  by addition of sucrose in wheat starch. Generally, water acts as plasticizer in the hygroscopic region while sugar-water has an anti-plasticizing effects resulting lowering the glass transition temperature by addition of sucrose (Slade and Levine, 1987). It is also reported that addition of sugar changes the storage stability of foods (Levine and Slade, 1986).

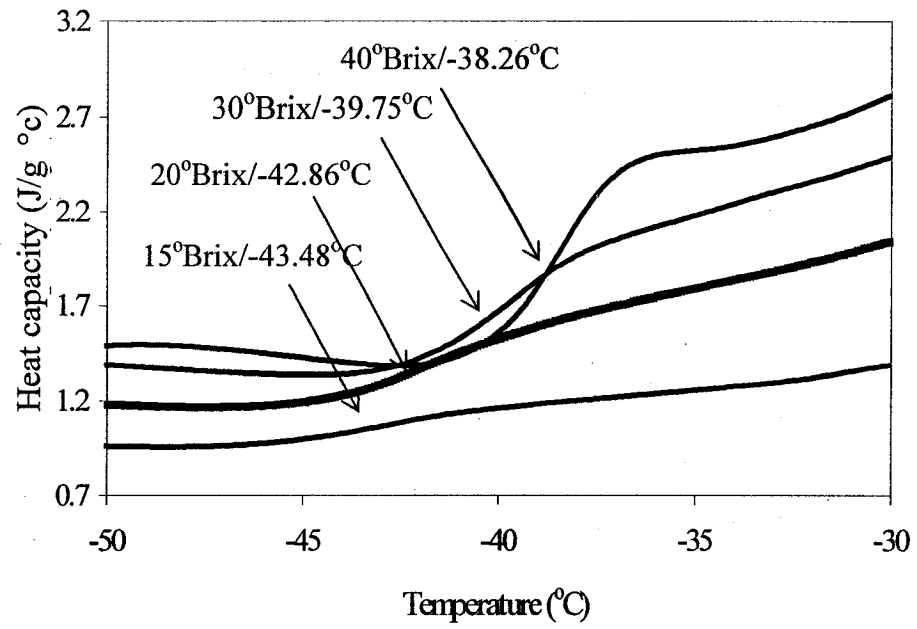
Effect of soluble solids content on  $T_g$  and  $\Delta C_p$  of mango puree followed a linear relationship and are described by the following equations:

$$T_g = 0.20TSS - 46.3 \quad (4.12)$$

$$\Delta C_p = 0.04TSS - 0.34 \quad (4.13)$$

Both these equations fitted well with coefficient of determinations ( $R^2$ ) greater than 0.99 while the standard errors ranged between 2 and 12%.





**Figure 4.5 Thermograms for mango puree at different concentration.**

**Table 4.3 Thermal transition parameters of mango puree at different concentrations**

Concentration (°Brix)	Onset point (°C)	Midpoint ( $T_g$ )	Endpoint (°C)	$\Delta C_p$ (kJ kg <sup>-1</sup> °C <sup>-1</sup> )
15	-44.97	-43.34	-41.66	0.20
20	-43.83	-42.19	-40.54	0.41
30	-41.86	-40.09	-38.32	0.73
40	-40.15	-38.26	-36.97	1.13

#### 4.4.6 Rheology

##### 4.4.6.1 Flow models

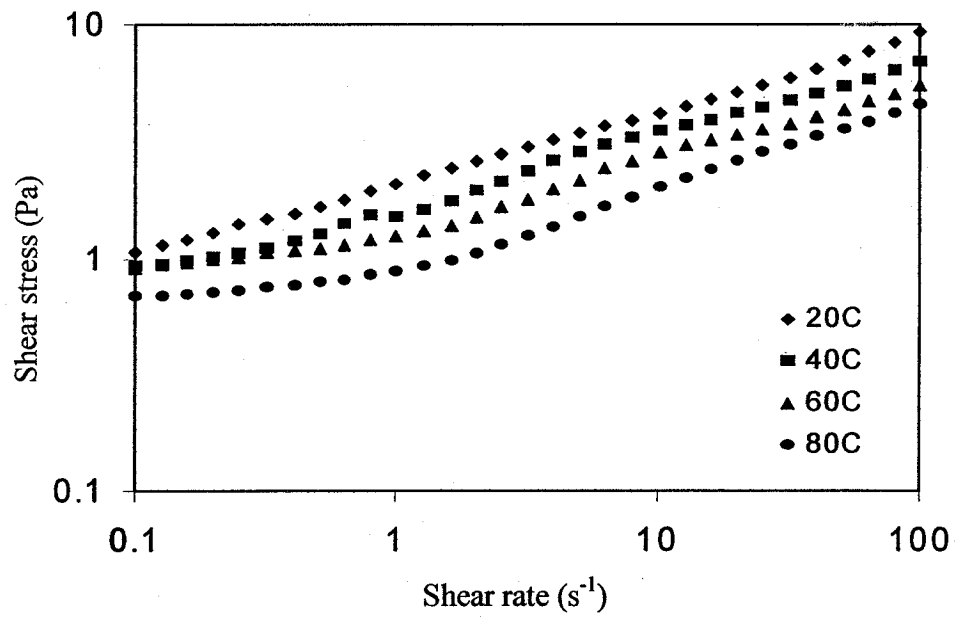
There were significant ( $p < 0.05$ ) variations in the up and downward curves of mango puree samples during shearing at the temperatures studied. Many researchers have observed similar nature of flow curves for various foods. The down curves (more consistent values) represent samples that have been subjected to prior shear history (during upward shear scan) and generally have reported to give more consistent data. These have been used in the present study to analyze the data. Figure 4.6 and 4.7 shows the flow curves of mango puree at different temperature and concentration.

The mango puree samples exhibited definite yield stress (0.19-3.8 Pa). However, the magnitude of yield was low compared to earlier reported values (Guerrero & Alzamora, 1998; Ahmed et al., 2005). At temperatures above 40°C yield was almost zero. The observed low yield stress was due to thermal processing apart from acidification and addition of sugar.

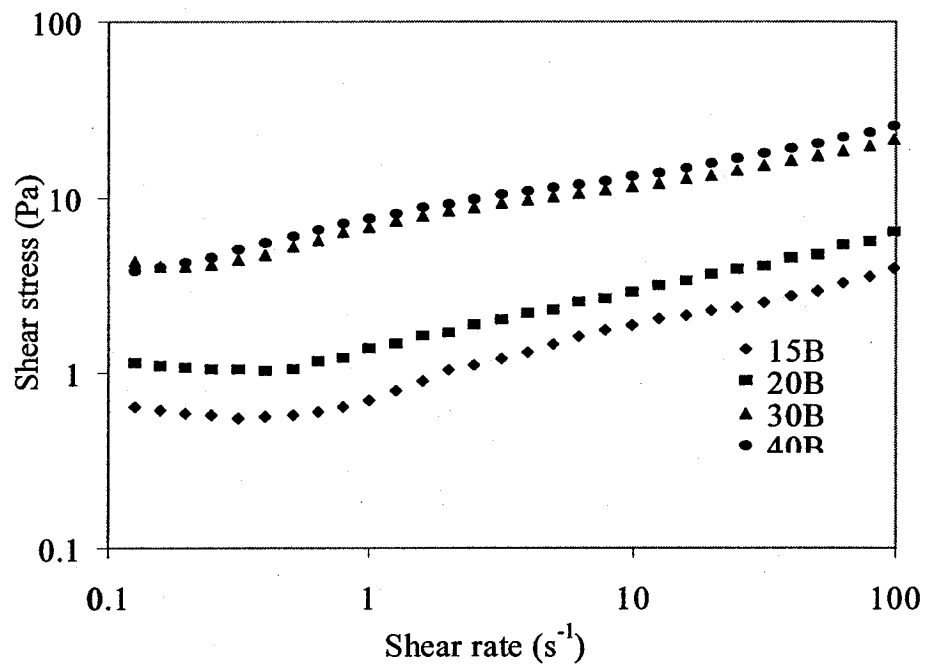
Shear stress-shear rate data of the thermal processed mango samples were verified for various rheological models and it was found that the power law model fitted the data adequately (Table 4.4). The power law model can be represented by the following equation:

$$\eta = K(\dot{\gamma})^{n-1} \quad (4.14)$$

where  $\tau$  is the shear stress (Pa),  $\dot{\gamma}$  is the shear rate ( $s^{-1}$ ), K is the consistency coefficient ( $Pa.s^n$ ), and n is the flow behavior index (dimensionless).



**Figure 4.6 Effect of Temperature on rheogram of mango puree at 20°Brix**



**Figure 4.7 Effect of concentration on rheogram of mango puree at 40°C**

**Table 4.4. Fitting of rheological models at 30°Brix at 20°C**

Model	$\tau_0$ (Pa)	K (Pa.s <sup>-n</sup> )	n (-)	SE
Newton	--	0.552	--	296
Power	--	7.904	0.275	5.46
Casson	6.61	0.125	--	27.3
Herschel-Bulkley	0.891	6.95	0.294	9.96
Bingham	8.025	0.332	--	96.4

**Table 4.5 Power law parameters at selected concentration and temperature**

Concentration (°Brix)	Temperature (°C)	K (Pa.s <sup>-n</sup> )	N (-)	R <sup>2</sup>
15	20	1.01	0.33	1.00
	40	0.82	0.34	0.99
	60	0.65	0.36	0.99
	80	0.52	0.35	0.99
20	20	2.10	0.38	0.99
	40	1.64	0.31	0.99
	60	1.43	0.28	0.99
	80	1.02	0.30	0.99
30	20	7.90	0.27	0.99
	40	5.48	0.31	0.99
	60	4.34	0.32	0.99
	80	2.58	0.34	0.99
40	20	8.94	0.27	0.99
	40	6.91	0.30	0.99
	60	4.52	0.32	0.99
	80	3.26	0.34	0.99

The power law parameters for mango puree at selected concentrations and temperatures are reported in Table 4.5. The flow behavior index ( $n$ ) varied between 0.27 and 0.38 indicating pseudoplastic shear-thinning nature of mango puree. However, there was no trend in  $n$  values with temperature. The consistency coefficient ( $K$ ) decreased systematically with temperature while increasing trend was noticed with concentration. Similar flow characteristics of pureed foods/concentrates are reported in the literature (Rao, 1977; Vitali & Rao, 1984; Cepeda & Villaran, 1999)

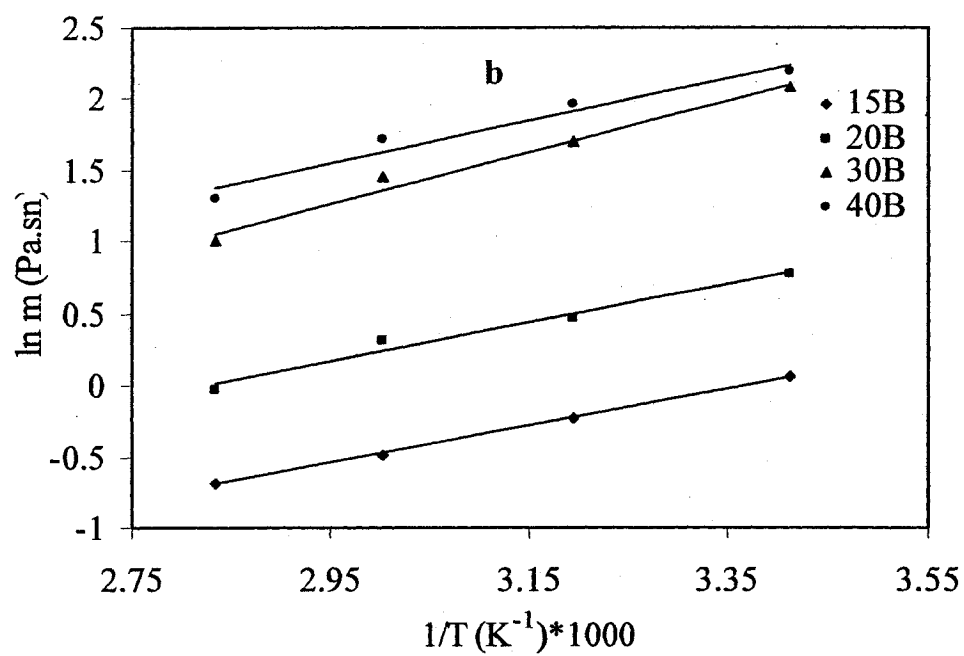
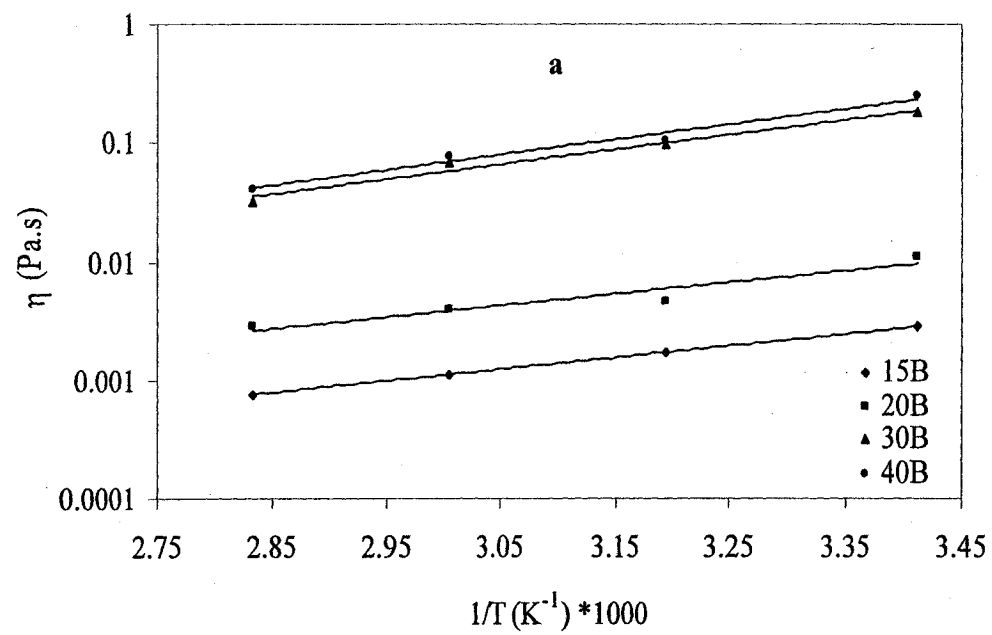
#### ***4.4.6.2 Effect of temperature on consistency coefficient and apparent viscosity***

Effect of temperature on rheological characteristics of mango puree is shown in Figure 4.8. Temperature dependency of consistency coefficient from shear stress-shear rate and apparent viscosity-shear rate data are expressed by Arrhenius relationship (Eqns. 4.14 and 4.15):

$$K = A_K \exp\left(\frac{E_K}{RT}\right) \quad (4.15)$$

$$\eta = A_\eta \exp\left(\frac{E_\eta}{RT}\right) \quad (4.16)$$

where  $K$  and  $\eta$  are the consistency coefficients based on shear stress and apparent viscosity at constant shear rate ( $50 \text{ s}^{-1}$ ) respectively,  $A_K$ ,  $A_\eta$  are the pre-exponential constants;  $E$  is the activation energy;  $R$  is the universal gas constant and  $T$  is absolute temperature.



**Figure 4.8 Arrhenius model for mango puree concentrates a) apparent viscosity at  $50\text{ s}^{-1}$  and b) consistency coefficient.**

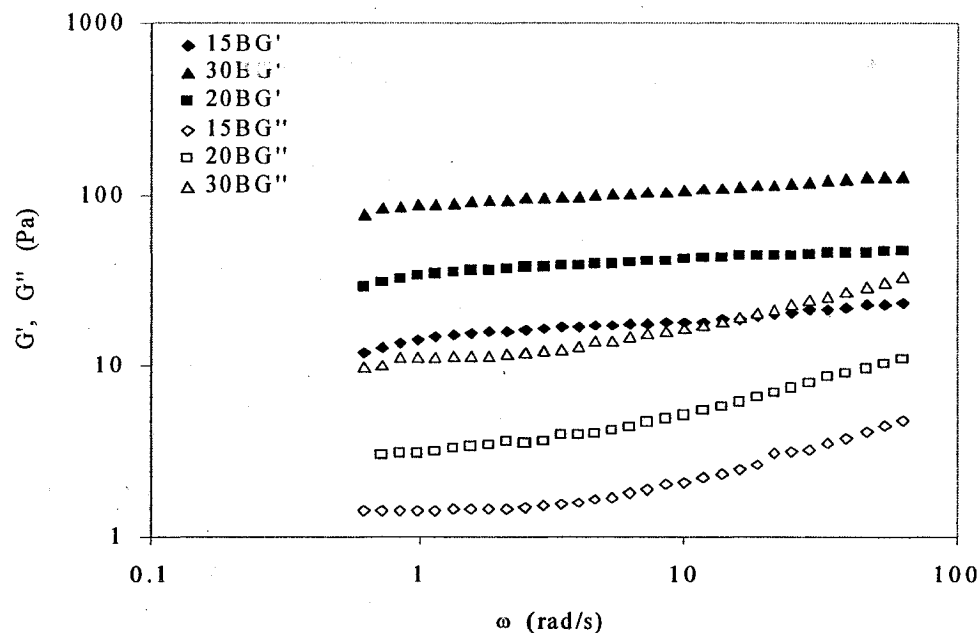
The coefficients of equations (4.14) and (4.15) were computed using the least square technique (Table 4.6). Magnitudes of the energy of activation relating to consistency coefficient and apparent viscosity were found to be 10.18-14.95 and 8.23-10.68 kJg<sup>-1</sup>mol<sup>-1</sup> respectively while the corresponding magnitudes of constants ( $A_K$ ,  $A_\eta$ ) were  $1.26 \times 10^{-2}$ - $5.64 \times 10^{-2}$  and  $2.6 \times 10^{-3}$ - $6.8 \times 10^{-3}$  respectively. The activation energy values for guava juice and banana puree were reported as 15.53 and  $18 \pm 1$  kJg<sup>-1</sup>mol<sup>-1</sup> respectively (Vitali and Rao, 1982; Guerrero and Alzamora, 1998). The difference in magnitudes could be attributed by fruit type, degree of thermal processing, addition of sucrose etc.

**Table 4.6 Arrhenius model parameters for consistency coefficient and apparent viscosity at 50 s<sup>-1</sup>**

Concentration (°Brix)	$A_K$	$E_{a(K)}$ (KJg <sup>-1</sup> mol <sup>-1</sup> )	$R^2$	$A_\eta$	$E_{a(\eta)}$ (KJg <sup>-1</sup> mol <sup>-1</sup> )	$R^2$
15	0.0126	10.18	0.99	0.0026	8.23	0.99
20	0.0239	10.99	0.97	0.0047	8.12	0.91
30	0.0176	14.95	0.97	0.0068	10.38	0.97
40	0.0564	12.46	0.95	0.0066	10.68	0.97

#### **4.4.6.3 Dynamic rheological characteristics**

Effect of concentrations on dynamic moduli of mango puree is shown in Figure 4.9. The values of elastic modulus ( $G'$ ) were higher than that of viscous components ( $G''$ ) through out the frequency range. Both  $G'$  and  $G''$  increased as function of concentration and the elastic components predominates over viscous components. It indicated the viscoelastic behavior of mango puree for all the concentrations. The phase angle ( $\delta$ ) represents ration of  $G''$  and  $G'$  that measure energy loss compared to energy stored in cyclic deformation. The phase angle values supported the viscoelasticity of mango puree.



**Figure 4.9 Effect of concentration on dynamic mechanical spectra of mango puree**

Dynamic frequency sweep tests were carried out in the limit of viscoelastic range to determine the frequency dependence of elastic and viscous modulus. The rheological moduli were modeled by equations of the following type (Rao & Cooley, 1992; Steffe, 1996):

$$G' = A\omega^b \quad (4.17)$$

$$G'' = C\omega^d \quad (4.18)$$

where  $\omega$  is the frequency and A, b, C and d are constants.

The regression coefficients of the above equations are reported in Table 4.7a and b. It is evident from the Table that both  $G'$ - $\omega$  and  $G''$ - $\omega$  data fitted adequately ( $R^2 > 0.89$ ) at selected temperature range. The slope of the equation (4.17) ranged between 0.008 and 0.272. However, the slope significantly ( $P < 0.05$ ) decreased with increase in concentration.



**Table 4.7a Regression parameters for elastic modulus as function of temperature and concentration**

Concentration (°Brix)	Temperature (°C)	A	B	R <sup>2</sup>
15	20	1.9787	0.2718	0.93
	40	2.1086	0.1113	0.95
	60	2.4097	0.1135	0.92
	80	2.9121	0.1527	0.90
20	20	3.3681	0.1353	0.98
	40	3.2002	0.1048	0.9
	60	3.4535	0.0895	0.98
	80	3.6726	0.0721	0.96
30	20	4.4674	0.085	0.99
	40	4.3696	0.0586	0.99
	60	4.4813	0.0414	0.99
	80	4.8087	0.0288	0.97
40	20	4.4923	0.0213	0.98
	40	4.4622	0.0134	0.99
	60	4.5722	0.0077	0.98
	80	4.9317	0.0096	0.98

**Table 4.7b Regression parameters for viscous modulus as function of temperature and concentration**

Concentration (°Brix)	Temperature (°C)	C	D	R <sup>2</sup>
15	20	0.529	0.745	0.99
	40	0.684	0.250	0.89
	60	0.440	0.176	0.94
	80	0.740	0.255	0.96
20	20	2.013	0.386	0.98
	40	1.700	0.332	0.92
	60	1.408	0.309	0.92
	80	1.622	0.247	0.95
30	20	3.142	0.335	0.96
	40	2.747	0.316	0.98
	60	2.593	0.281	0.98
	80	2.661	0.234	0.97
40	20	3.148	0.438	0.98
	40	3.084	0.328	0.95
	60	2.677	0.302	0.91
	80	2.790	0.221	0.92

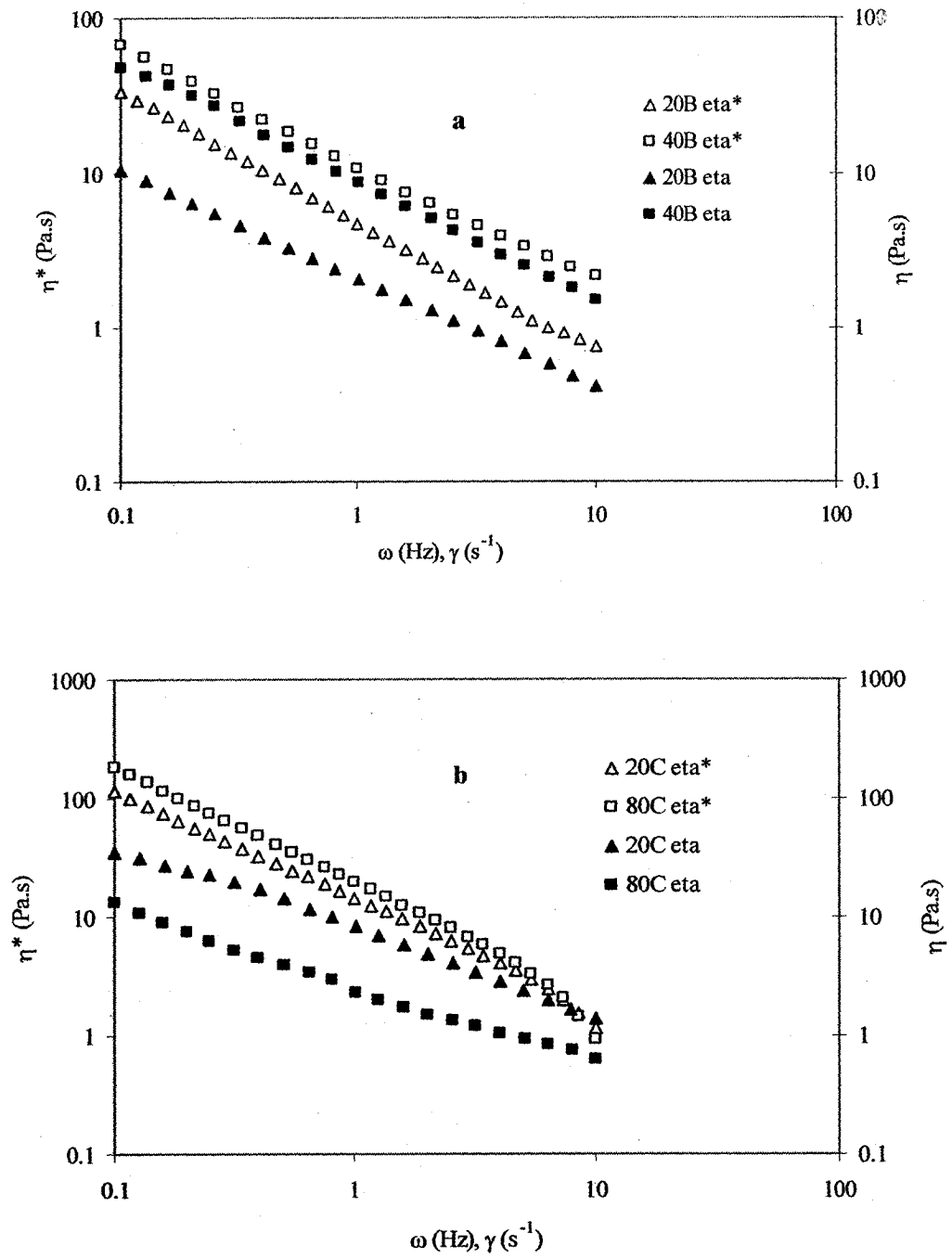
The product behaves like weak gel at higher temperature and concentrations (30-40°Brix). Elastic modulus showed no frequency dependence (slope <0.05) at those ranges (Rosalina and Bhattacharya, 2002). The slope of  $G''$ -  $\omega$ , showed strong frequency dependency (slope < 0.05) and the magnitudes decreased systematically with temperature while concentration did not show any trend.

#### 4.4.6.4 Application of Cox-Merz rule

The fluid rheology has been associated with internal structure of materials and the Cox-Merz rule provides such information on polymeric solution (Rao and Cooley, 1992). The Cox-Merz rule describes the superposition of shear rate dependence of steady-shear viscosity ( $\eta$ ) and frequency dependence of complex viscosity ( $\eta^*$ ) at equal values of angular frequency and shear rate:

$$\eta = \eta^* \Big|_{\omega = \dot{\gamma}} \quad (4.19)$$

Several researchers (Bistany and Kokini, 1983; Rao and Cooley, 1992; Tiziani and Vodovotz, 2005) reported that most of the food products do not follow the Cox-Merz rule. Mango puree samples did not follow the Cox-Merz rule at studied temperature and concentration range (Figure 4.10a and b). The failure of Cox-Merz rule has been explained on the basis of structural break down of food products beyond the yield point (Bistany and Kokini, 1983). The complex viscosity ( $\eta^*$ ) was found to be higher compared to steady shear viscosity ( $\eta$ ) and both increased with concentration (Figure 4.10a) while the curves found to be closer at higher concentration. It was found that  $\eta$  decreased with temperature while  $\eta^*$  did not follow the same trend and increased (Figure 4.10b). Differences between  $\eta$  and  $\eta^*$  at higher temperature could be due to structural changes of puree sample and/or starch components. Steady viscosity generally ruptures the texture during high shear rate while oscillation measurement retained the texture at its utmost.



**Figure 4.10 Applicability of Cox-Merz rule for mango puree sample a) effect of concentration and b) effect of temperature.**

A power type relationship can be used to correlate the steady shear viscosity ( $\eta$ ) and oscillatory viscosity ( $\eta^*$ ) by the following equation:

$$\eta = K^* (\eta^*)^{n^*} \quad (4.20)$$

where  $\eta$  is the steady shear viscosity,  $\eta^*$  is the complex viscosity and  $K^*$  and  $n^*$  are the consistency coefficient and flow behavior index considering oscillatory viscosity. The steady shear viscosity ( $\eta$ ) and oscillatory viscosity ( $\eta^*$ ) data fitted adequately ( $R^2 > 0.99$ ). The regression parameters are reported in Table 4.8. The  $K^*$  increased with both concentration and temperature while no systematic trend was observed for  $n^*$  values.

**Table 4.8 Power law parameters for dynamic and steady shear viscosity at selected temperature and concentration**

Concentration (°Brix)	Temperature (°C)	$K^*$ (Pa.s <sup><math>n^*</math></sup> )	$n^*(-)$	$R^2$
15	20	1.194	0.305	0.99
	40	1.781	0.507	0.99
	60	1.702	0.017	0.99
	80	2.567	0.011	0.99
20	20	4.810	0.154	0.99
	40	4.031	0.121	0.99
	60	4.739	0.132	0.99
	80	6.327	0.076	0.99
30	20	13.87	0.057	0.99
	40	14.19	0.032	0.99
	60	14.105	0.014	0.99
	80	18.56	0.013	0.98
40	20	11.27	0.240	0.99
	40	14.30	0.148	0.99
	60	11.68	0.127	0.99
	80	16.57	0.101	0.99

## 4.5 Conclusions

Effects of temperature (20, 40, 60 and 80°C) and concentration (15, 20, 30 and 40°Brix) on thermophysical and rheological properties of mango puree were investigated. Thermal conductivity, diffusivity and specific heat increased with temperature but decreased with concentration while a reverse trend was observed for density. Higher soluble solids content increased the glass transition temperature of mango puree. Thermal properties and glass transition temperature of mango puree were found to fit linear relationships with temperature and concentration ( $R^2 > 0.90$ ). Mango puree exhibited pseudoplasticity at steady shear measurement at shear rate of 0.1 to 100 s<sup>-1</sup>. Flow behavior index did not show any trend with temperature. Consistency coefficient increased with concentration while decreasing trend was observed with temperature. Dynamic oscillation shear measurement (0.1 to 10 Hz) revealed that mango puree behaved as weak gel at higher temperature and concentration. Superimposition of steady shear viscosity and complex viscosity at equal values of shear rate (0.1-10 s<sup>-1</sup>) and frequency (0.1-10 Hz) range were tested by the application of Cox-Merz rule and it was found that mango puree did not follow the Cox-Merz rule.

## 4.5 References

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## CONNECTING STATEMENT

Part of Chapter V is prepared as a manuscript “Effect of soluble solids and high pressure treatment on rheological properties of protein enriched mango puree” for submission to a refereed journal and is authored by Gundurao, A. and Ramaswamy, H.S.

Gundurao is the candidate and the principal author of all papers. Ramaswamy is the thesis supervisor under whose guidance the research work was carried out.

Thermal processing is proven to be the most effective technique in preservation of fruits and its byproducts. But consumer requirements for foods are constantly changing. Today, consumer demands for foods that are fresh, natural and with high nutritional values. Hence, non-thermal processing like high pressure processing is attracting more attention from food scientists. High pressure processing not only has food preservation capabilities but also helps in achieving interesting functional effects. This chapter focuses on the effect of soluble solids and high pressure treatment on rheological and sensory properties of protein enriched mango puree.

The chapter is divided in to three parts

Part 1 describes the influence of HP on the rheological characteristics of protein enriched mango puree at selected concentrations of soluble solids

Part 2 describes the combined influence of soluble solids and protein content on the rheology of HP treated mango pulp. In this section the protein content and soluble solids were simultaneously varied keeping the total soluble solids concentration at 28% which is the level maintained in the canned products.

Part 3 deals with evaluation of sensory quality of HP treated protein enriched mango puree.

## **CHAPTER V**

### **EFFECT OF SOLUBLE SOLIDS AND HIGH PRESSURE TREATMENT ON RHEOLOGICAL PROPERTIES OF PROTEIN ENRICHED MANGO PUREE**

#### **5.1 Abstract**

Effect of HP treatment at different pressure levels and holding times on rheological properties of protein enriched mango puree were evaluated. In first part, the original soluble solids in mango puree (28°Brix) was lowered to 20, 23 and 26°Brix and each was supplemented with 2, 5 and 8% of whey protein. Pressure treatment resulted in a positive effect on elastic ( $G'$ ) and viscous moduli ( $G''$ ) as well as complex viscosity ( $\eta^*$ ). Values of  $G'$  were higher than  $G''$  demonstrating the product to behave more and more like a gel. The changes in rheological parameters at 500 MPa were modeled based on soluble solids and protein content. Additional rheological studies were carried out by simultaneously varying protein and soluble solids content by keeping the total solids at 28%. The effects of holding time and pressure level were similar to those observed previously in first part of this study. However, the positive effect of added protein was over dominated by negative effect of the simultaneous decrease in the soluble solids concentration, resulting in an overall decreasing effect as protein content increased (with a same magnitude decrease in soluble solids content). Developed models well predicted the combined influence of protein and soluble solids concentration on rheological parameters ( $R^2 > 0.85$ ).

Sensory evaluation of mango puree supplemented with protein (2, 5 and 8%) was performed before and after high pressure treatment (500MPa/3min) using a nine point hedonic scale. Sensory evaluation results indicated that protein enrichment up to 5% level did not adversely affect the sensory qualities.

## 5.2 Introduction

Mango is one of the highly consumed fruits of tropics with exotic flavor, delicious taste and great color. Both ripe and unripe mangoes are used for developing its by-products. Mango puree, nectar, juice, jams, jellies and leather are some of by-products of ripe mango where as pickles, mango powder, and salads are developed from raw mango. Mango puree is the main source of by-products that are manufactured from ripe mangoes. It is a rich source of  $\beta$ -carotene and ascorbic acid. It is a low source of fat and calories. It is also a low source of protein.

Globular proteins are commonly used as an ingredient in many food systems because of their ability to form gels and improve texture, flavor and stability of the product. Protein fraction in whey protein isolates (WPI) is typically greater than 90% and comprised of  $\beta$ -lactoglobulin,  $\alpha$ -lactalbumin, serum albumin and immunoglobulins (Chantrapornchai & McClements, 2002). Currently whey protein and its different components have been commercially used in various food and pharmaceutical applications for its health benefits. Food product development focuses both on nutritional aspects and sensory characteristics. A combination of sugar rich mango pulp with its exotic flavor along with a rich protein source like WPI could produce a nutritional beverage with fruit flavour.

Heat treatment has long been used for unfolding of globular proteins that promotes aggregation and gelatinization (Pelegrine & Gasparetto, 2005). Browning is the major quality problem during heat treatment of protein based beverages. In addition, sedimentation and insolubilization are other limitations in protein beverages during processing and storage (Iordache & Jelen, 2003). Some cases, thermal denaturation produces off flavors and/or results in toxic formation by destruction and cleavage of covalent bonds (Knorr et. al., 2002; Ngarize et. al., 2005). Therefore, there is a need to explore other processes to overcome those difficulties.

High pressure processing (HPP) has been experimented as one of the challenging technology in many food applications with better quality retention compared to thermal treatment. Pressure treated fruit products retain the original color, flavor and taste of the produce. HPP has been successfully applied to various fruit products like jams, jellies and

purees apart from commercially successful meat processing industries (Tedford et. al., 1999). The pressure level varies between 100-1200MPa for food applications.

The whey protein components ( $\alpha$ -lactalbumin and  $\beta$ -lactoglobulin) undergo irreversible denaturation during pressurization at 400MPa (Van Mil & Kromkamp, 1997; Hinrichs, 2000; Fertsch et. al., 2003). Pressure-induced whey protein gels mainly occur during pressure holding above 400MPa, which are essentially stabilized by intermolecular disulfide bonds, however, the structural stability should not be influenced by pressure release (Fertsch et. al., 2003). Increase in pressure holding time strengthens the gel, stimulating the formation of more intensive intermolecular interactions (Camp van & Huyghebaert, 1995a, 1996; Keim & Hinrichs, 2004). Since, HP does not affect covalent bonds, essential vitamins and nutrients are retained (Tedford et. al., 1999).

Rheological properties of protein foods have commercial interest by modifying the functional and structural properties from the original one. Heat is commonly used for the modification and numerous literatures are available of the subject. Currently, such modifications are carried out by high pressure processing with better acceptability. The cleavage of the desired chemical bonds is controlled by the pressure levels that result in desired texture and rheology of food products. Rheology of protein gels depends on solid contents, pH, temperature, time and ionic strength (van Kleef, 1986; Nagano et al., 1994; Renkema et al., 2002). The differences in rheological properties could partly be explained by the amount of protein incorporated into the network. The control of protein gels for various industrial applications requires a better understanding of the relationship between the structure of aggregates and the macroscopic properties of the gel. Dynamic rheological measurements in terms of elastic (solid-like characteristics) and viscous (liquid-like characteristics) moduli characterize the viscoelastic nature of protein foods during processing and gel formation. Little information is available on the addition of proteins on fruit puree during pressurization.

In view of potential of HPP in texture modification, it would be of great interest to study the rheological properties of WPI incorporated with mango pulp.

### **5.3 Material**

Canned mango pulp (Cv Alphanso, Cedar brand marketed by Phoenicia Products Inc. Montreal, Canada) was procured from a departmental store in Montreal. The total soluble solids (TSS) of the original pulp was 28°Brix. The experiments were conducted in two parts and the results are presented similarly. Sensory evaluation has been presented as part 3 of results.

### **5.4 Part 1: Mango puree with different protein concentrations**

#### **5.4.1 Material**

The original soluble solids level in mango puree was lowered to 20, 23 and 26°Brix by addition of distilled water and each was supplemented with WPI powder (BiPro, Davisco Foods Int., MN) at 2, 5 and 8%.

#### **5.4.2 Method**

Three pressure levels (425, 500, and 575 MPa) and holding times (0, 3 and 6min) were selected for pressure treatment.

##### ***5.4.2.1 High- Pressure equipment and treatment***

A semi-continuous type, pressure vessel (10cm diameter and 55cm height) with stainless steel cylindrical pressure chamber was used for this study (model ACIP 6500-5-12VB, ACB, Nantes, France). The maximum pressure tolerance level of the equipment was 650 MPa. The pressure come up time varied from 1 to 2 min depending on the applied pressure. Increase in pressure level increased the come up time. The depressurization time was less than 20s. Samples were submerged in pressure chamber containing water as the hydrostatic fluid medium. Temperature of fluid medium was maintained between 20-26°C during pressure treatment. The medium and sample temperature increased during the treatment as a result of adiabatic heating. Hence, the temperature of medium and sample were adjusted to a lower (than the desired target)

temperature before treatment. A thermocouple inside the chamber, attached to a temperature logger, was used to measure the temperature of the medium during pressure treatment.

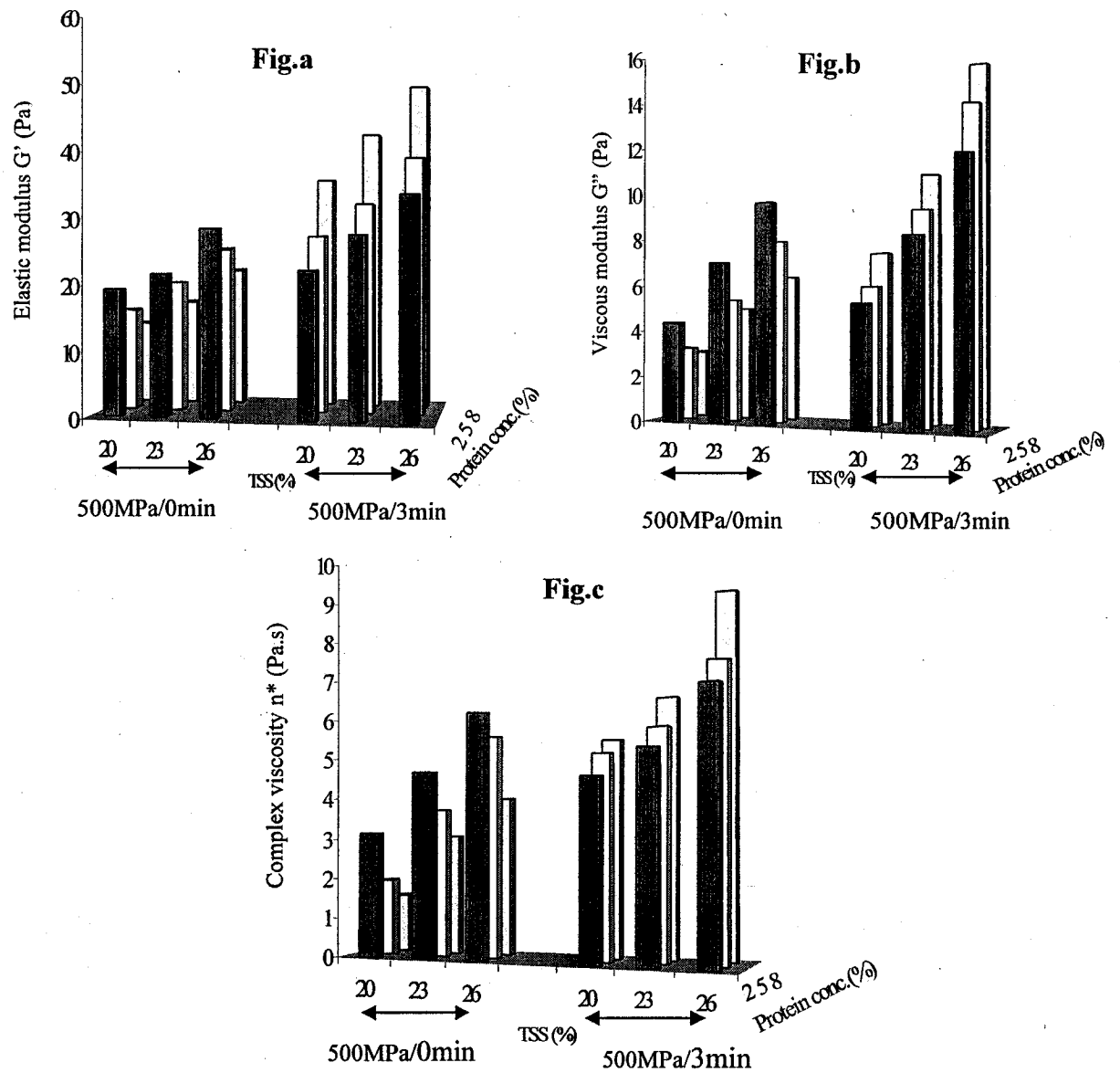
#### ***5.4.2.2 Rheological measurements***

A controlled-stress rheometer (AR 2000, TA Instruments, New Castle, DE) equipped with a computer control software (Rheology Advantage Data Analysis Program, TA, New Castle, DE) was used to study dynamic oscillatory measurement of the mango puree sample. A 60 mm parallel plate attachment was used with a gap of 1000 microns. The AR 2000 was supplemented with an efficient Peltier temperature control system and the sample temperatures were precisely controlled and monitored. For each test, a measured volume (approximately 2 mL) of mixed sample was placed on the bottom plate of the rheometer. The test temperature was maintained at 25°C. Dynamic oscillatory tests were carried out at a frequency sweep from 0.1 to 10 Hz. The oscillation stress was selected based on linear part of the viscoelastic range (0.1-0.2 Pa). Each time, a new sample was used for rheological measurement. Elastic modulus ( $G'$ ), viscous modulus ( $G''$ ) and complex viscosity ( $\eta^*$ ) were obtained directly from the software (Rheology Advantage, TA version 2.3).

### **5.4.3 Results and discussion**

#### ***5.4.3.1 Effect of time***

The selected rheological parameters namely  $G'$ ,  $G''$  and  $\eta^*$  were evaluated to monitor the possible changes after high-pressure treatment. Figures 5.1 a, b and c show the effect of holding time (0 and 3min) at 500MPa on dynamic moduli of mango puree. Increase in soluble solids content of mango puree increased the rheological parameters at each of the supplemented protein concentration levels.



**Figure 5.1** Effect of holding time (zero and 3 min) at 500 MPa on a) elastic modulus b) viscous modulus and c) complex viscosity of protein enriched mango puree.

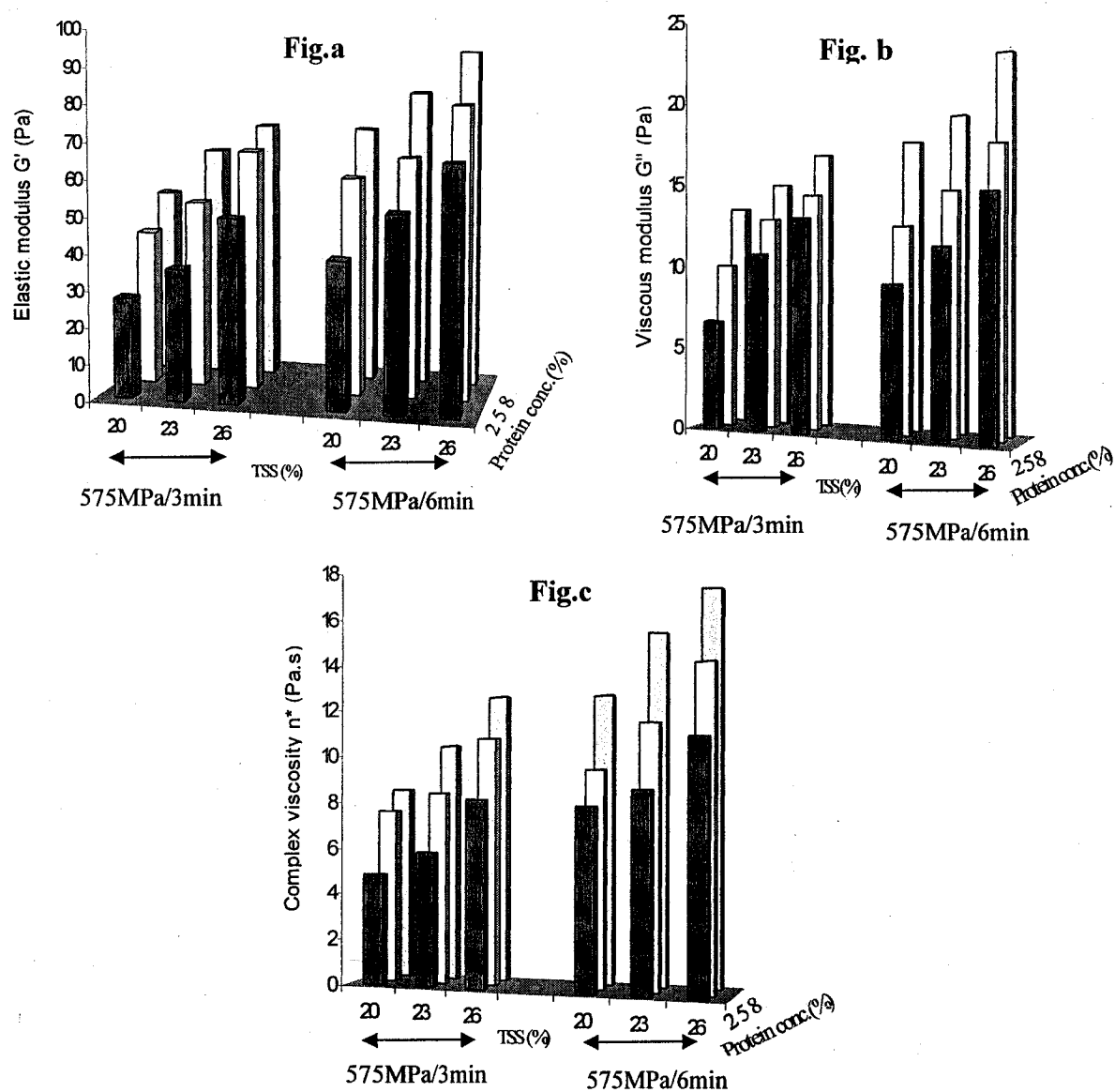
The pressure pulse (zero minute holding time) did not have any influence on the added protein and  $G'$ ,  $G''$  and  $\eta^*$  of the protein enriched samples were in fact decreased as result of increase in protein concentration level. Since the protein constituted an inactive bulk, higher protein concentrations resulted in lowering visco-elastic properties. However, with a 3 min holding time, the pressure treatment caused a significant ( $p<0.05$ ) increase in the viscoelastic property values at each soluble solids content level and at each protein concentration. The soluble solids level had a major effect than protein concentration in increasing the viscoelastic parametric values of pressure treated protein enriched mango puree. At any chosen pressure levels, a minimum holding time of 3 min was very essential to obtain desired textural properties of mango puree.

Figure 5.2. a, b, and c show the effect of holding time (3 and 6min) on  $G'$ ,  $G''$  and  $\eta^*$  at 575MPa. A clear increase in rheological parameters was observed at each protein concentration and soluble solids concentration levels as the holding time increased from 3 min to 6min. This supports the earlier results that a minimum holding time of 3 min was required at 500 MPa to initiate the denaturation or texturization process, the 6 min treatment further reinforcing the gel network formation.

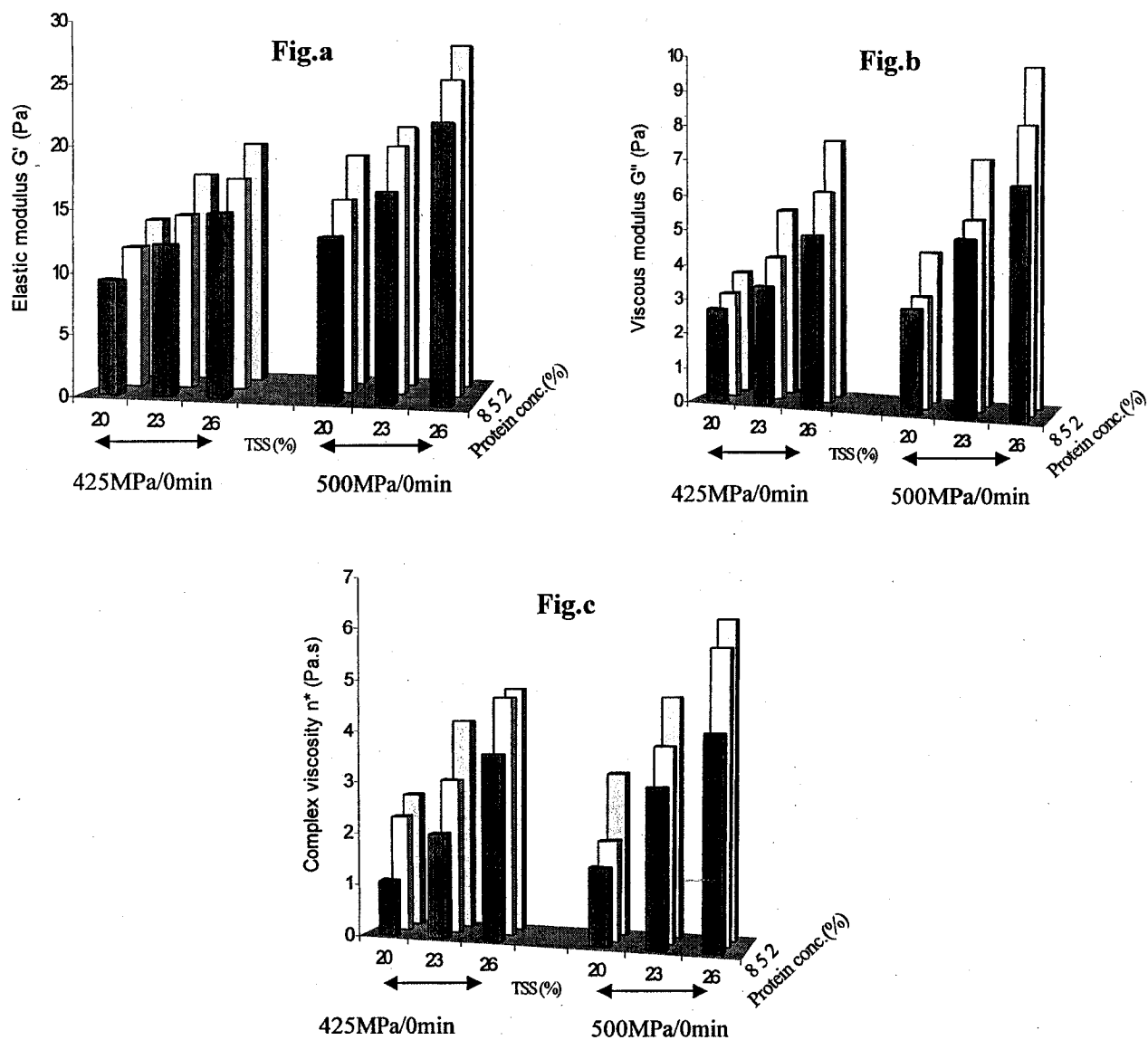
#### **5.4.3.2 Effect of pressure**

Effect of pressure level on rheological properties of protein added mango puree between 1) 425 and 500 MPa with a holding time of 0 min (Figure 5.3 a, b and c) and 2) 500 and 575 MPa with a holding time of 3min (Figure 5.4 a, b and c). The zero min holding time results at 425 and 500 MPa (Figure 5.3) were similar to the those previous observed at 500 MPa (Figure 5.1). The protein effect curves appear to be different, but it is actually caused by the reversal of pressure level scale. Results observed at 3 min holding time at pressure levels of 500 and 575 MPa (Figure 5.4) were again similar to those observed previously (Figure 5.2) with both soluble solids and protein concentration contributing to gel structure build up as result of HP treatment.  $G'$ ,  $G''$  and  $\eta^*$  values increased when pressure was increased from 425 to 500 to 575 MPa. The values of  $G'$  varied between 9.33 Pa at 425 MPa/0min and 93.3 at 575 MPa/6min,  $G''$  from 2.69 Pa and 22.8 whereas  $\eta^*$  varied from 1.061 and 17.2 Pa.s.

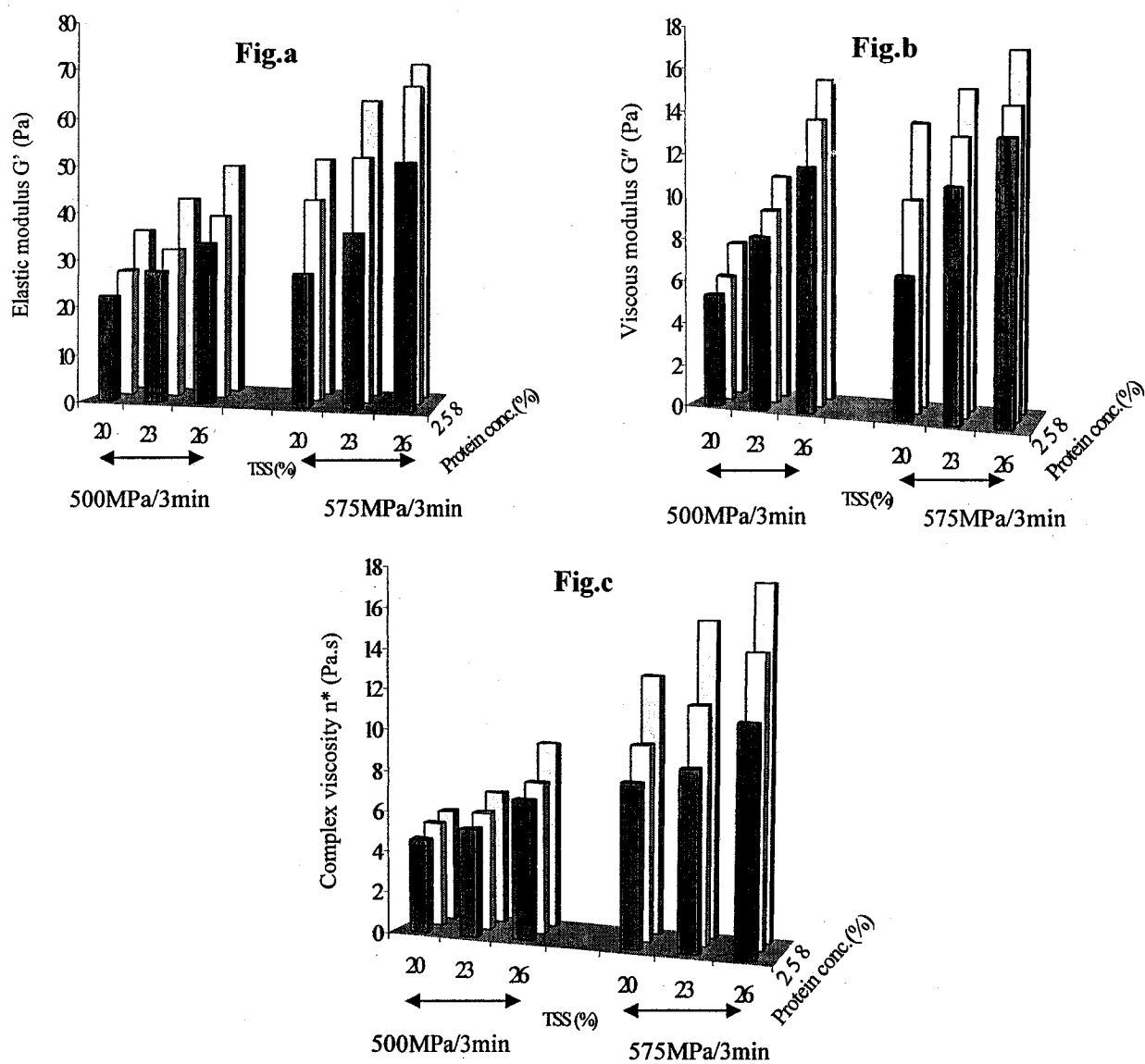




**Figure 5.2** Effect of holding time (3 and 6 min) at 575 MPa on a) elastic modulus b) viscous modulus and c) complex viscosity of protein added mango puree



**Figure 5.3** Effect of pressure (425 and 500 MPa) at zero min holding time on a) elastic modulus b) viscous modulus and c) complex viscosity of protein added mango puree.



**Figure 5.4 Effect of pressure (500 and 575 MPa) at 3min holding time on a) elastic modulus b) viscous modulus and c) complex viscosity.**

Holding time had a more significant effect ( $p < 0.05$ ) than applied pressure for formation of stabilizing interactions linking the network and to increase the gel strength. The values of elastic modulus ( $G'$ ) were higher than that of viscous components ( $G''$ ) through out the frequency range. This shows the gelling behavior of protein as an effect of both pressure and holding time greater than zero. The elastic component predominated over viscous component indicating the viscoelastic behavior of mango puree for all the protein concentrations.

## **5.5 Part 2: Combination effect of soluble solids and protein concentration**

Addition of protein increased TSS level and decreased the moisture present in mango puree. Thus, the focus of second part of experiment was to vary the protein level in mango puree maintaining the constant TSS level.

### **5.5.1 Material**

In the second part of the study, protein slurries were prepared with the same solids level (28%) as that of mango puree (28°Brix). The protein slurry and mango puree were then mixed in different proportions to yield differing amounts of soluble solids and whey protein. The protein content selected were 2, 3, 5, 7 and 8% P which simultaneously lowered the soluble solids level in mango puree to 26, 25, 23, 21, 20% thereby maintaining the final solids content at 28% (2%P/26%TSS, 3%P/25%TSS, 5%P/23%TSS, 7%P/21%TSS, 8%P/20%TSS). A Pearson square rule was used to prepare these combination protein enriched mango purees.

### **5.5.2 Methods**

Protein supplemented mango puree was subjected to five levels of pressure (425, 450, 500, 550, 575 MPa) and holding time (0, 1, 3, 5, 6 min). High pressure equipment and treatment and rheological measurements are as explained in part 1.

Independent effects of protein and soluble solids on rheological parameters were evaluated by pressure treating pure mango puree (26, 25, 23, 21, 20% TSS) and protein enriched mango puree (2%P/26%TSS, 3%P/25%TSS, 5%P/23%TSS, 7%P/21%TSS, 8%P/20%TSS) at 500MPa and 3 min holding time.

### 5.5.3 Experimental design

A three level factorial design was selected to study the main effect and interaction effects. The experimental design adopted was a modification of Box's central composite design for three variables at five levels each. The three independent variables were protein concentration ( $X_1$ ), pressure ( $X_2$ ) and holding time ( $X_3$ ). The independent variable coded values were -1.5 (lowest level), -1, 0 (middle level), 1 and 1.5 (highest level). The pressure and soluble solids were co-variables (one determining the complementary level of the other, totaling to 28%). The actual values and the corresponding coded values of the three independent variables and responses of dependent variables are shown in Table 5.1. The correspondence between the coded and actual values can be obtained using the following formula:

$$Z = \left( \frac{X - X^o}{\Delta X} \right) \quad (5.1)$$

where Z is the coded value, X is the corresponding actual value,  $X^o$  is the actual value in the center of the domain, and  $\Delta X$  is the increment of X corresponding to 1 unit of Z.

The complete design consisted of 24 experimental points including six replications of the center point to estimate the pure error of the analysis and to predict the lack of fit of the models. It was assumed that the three independent variables affected each of three dependent Y variables (responses). Responses under observations were: elastic modulus ( $Y_1$ ), viscous modulus ( $Y_2$ ) and complex viscosity ( $Y_3$ ). Results were analyzed to compare experimental values with model predictions.

**Table 5.1 3-variable, 5-level central composite design and responses of dependent variables to the dynamic rheological properties of pressure treated protein enriched mango puree.**

Run	Independent variables <sup>a</sup> (coded and real values)			Dependent variables <sup>b</sup>		
	X <sub>1</sub>	X <sub>2</sub>	X <sub>3</sub>	Y <sub>1</sub>	Y <sub>2</sub>	Y <sub>3</sub>
1	3 (-1)	450 (-1)	1 (-1)	14.63	20.61	89.6
2	3 (-1)	450 (-1)	5 (1)	12.84	17.92	78.64
3	3 (-1)	550 (1)	1 (-1)	16.08	22.26	98.57
4	3 (-1)	550 (1)	5 (1)	12.56	19.06	76.61
5	7 (1)	450 (-1)	1 (-1)	7.453	11.74	45.33
6	7 (1)	450 (-1)	5 (1)	7.279	11.63	44.24
7	7 (1)	550 (1)	1 (-1)	7.484	11.83	45.51
8	7 (1)	550 (1)	5 (1)	12.19	17.22	74.66
9	5 (0)	500 (0)	0 (-1.5)	7.667	11.32	46.83
10	5 (0)	500 (0)	6 (1.5)	11.31	15.47	69.37
11	2 (-1.5)	500 (0)	3 (0)	16.24	22.59	99.51
12	2 (-1.5)	500 (0)	5 (1)	12.48	19.89	75.87
13	5 (0)	575 (1.5)	3 (0)	10.95	16.92	66.72
14	5 (0)	575 (1.5)	5 (1)	11.3	15.32	69.31
15	8 (1.5)	500 (0)	3 (0)	7.718	11.92	47.01
16	8 (1.5)	500 (0)	1 (1)	7.911	11.98	48.24
17	5 (0)	425 (-1.5)	3 (0)	9.811	14.17	60
18	5 (0)	425 (-1.5)	5 (1)	8.837	12.49	54.11
19(6X)	5 (0)	500 (0)	3 (0)	9.971	14.93	60.85

<sup>a</sup> X<sub>1</sub>, Protein concentration (%); X<sub>2</sub>, Pressure applied (MPa) and X<sub>3</sub>, Holding time (min).

<sup>b</sup> Y<sub>1</sub>, Complex viscosity (Pa.s); Y<sub>2</sub>, Viscous modulus (Pa), Y<sub>3</sub>, Elastic modulus (Pa)

#### 5.5.4 Statistical analyses

Experimental data were analyzed to fit the 2<sup>nd</sup> order polynomial equation to all dependant Y variables:

$$Y = a_0 + a_1X_1 + a_2X_2 + a_3X_3 + a_{12}X_1X_2 + a_{13}X_1X_3 + a_{23}X_2X_3 + a_{11}X_1^2 + a_{22}X_2^2 + a_{33}X_3^2 + a_{123}X_1X_2X_3 \quad (5.2)$$

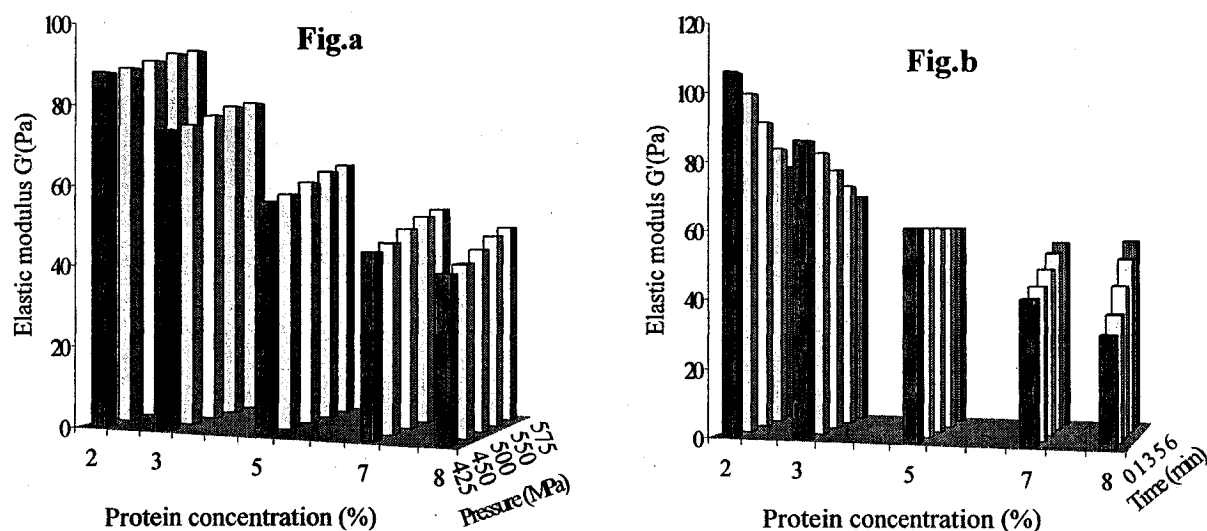
where a<sub>n</sub> are constant regression coefficients and X<sub>1</sub> (protein concentration), X<sub>2</sub> (pressure level), and X<sub>3</sub> (holding time) are coded independent variables.

SAS software (SAS Institute, 1999) was used to perform stepwise procedure to simplify the models and to obtain analyses of variance and regression coefficients.

### 5.5.5 Results and discussion

#### 5.5.5.1 Effect of protein concentration, pressure and time on $G'$ , $G''$ and $\eta^*$

In qualitative terms the oscillatory curves give a fingerprint of the state of the microstructure and thus characterize the viscoelastic properties of fluid foods. Figure 5.5a shows the effect of protein concentration and pressure and Figure 5.5b shows the effect of protein concentration and time on  $G'$  of mango puree.  $G'$  decreased with protein concentration (and the simultaneous decrease in soluble solids level in mango puree). Thus change in soluble solids content of mango puree had major effect on  $G'$  than protein concentration at all chosen pressure and time levels. Pressure had positive effect on elastic modulus but a decreasing trend was observed with holding time up to 5% protein concentration reversing to an increasing trend at 7 and 8%. This indicated that a minimum of 5% protein concentration was necessary to increase  $G'$  of mango puree with holding time.



**Figure 5.5. Elastic modulus as influenced by protein concentration and a) high pressure b) holding time.**

Viscous modulus did not show significant change with pressure at chosen protein concentration levels (Figure 5.6a) but increased with holding time (Figure 5.6 b).  $G''$  showed similar effects with protein concentration as  $G'$ .

Complex viscosity showed an increasing trend with pressure but decreased with protein concentration levels (Figure 5.7a). Similar to  $G'$ ,  $\eta^*$  decreased with holding time at 2 and 3% protein concentration but increased there after at 5, 7 and 8% (Figure 5.7b). So protein concentration >3% would increase the  $\eta^*$  of mango puree.

#### 5.5.5.2 Model development

1. A second order polynomial equation (5.2) was fitted to the experimental data (Table 5.1) of  $G'$ ,  $G''$  and  $\eta^*$  to evaluate the effect of protein concentration, pressure and time, using RSREG procedure of SAS. Best equation was chosen from stepwise selection testing the adequacy and fitness by analysis of variance (ANOVA). The ANOVA results for each of the dependent variables with coefficient of determinations ( $R^2$ ) are presented in Table 5.2. The models developed for  $G'$ ,  $G''$  and  $\eta^*$  seemed adequate with significant F values and non-significant lack of fit, but it showed  $R^2 < 0.80$  considering the low percentage of variability explained.

The polynomial equations for the responses are shown below:

$$G' = 62.2 - 15.9 X_1 + 6.6006 X_1 * X_2 + 8.16 X_1 * X_3 + 6.41 X_1 * X_2 * X_3 \quad (5.3)$$

$$G'' = 15.6 - 3.46 X_1 + 1.26 X_1 * X_3 \quad (5.4)$$

$$\eta^* = 10.2 - 2.601 X_1 + 1.0608 X_1 * X_2 + 1.32 X_1 * X_3 + 1.036 X_1 * X_2 * X_3 \quad (5.5)$$

where  $X_1$  is protein concentration,  $X_2$  is pressure and  $X_3$  is time.

2. Individual effect of protein concentration and soluble solids content in mango puree were evaluated as shown below. The effect of selected protein concentration levels at the central level of 23% soluble solids concentration of mango puree.



**Effect of 5%PC (Central level)** = Increase in value due to 5% protein addition at 23% soluble solids concentration.

**Effective contribution of 3%PC** at 23% soluble solids concentration = Increase in value due to 3% protein addition (and 25% soluble solids concentration) – increase in value due to a 2% increase in solute concentration from central level

**Effective contribution of 7%PC** at 23% soluble solids concentration = Increase in value due to 7% protein addition (and 21% soluble solids concentration) – decrease in value due to 2% dilution from central level

Similar approach can be used for 20, 21, 25 and 26%TSS with 8, 7, 3 and 2%PC as central level respectively.

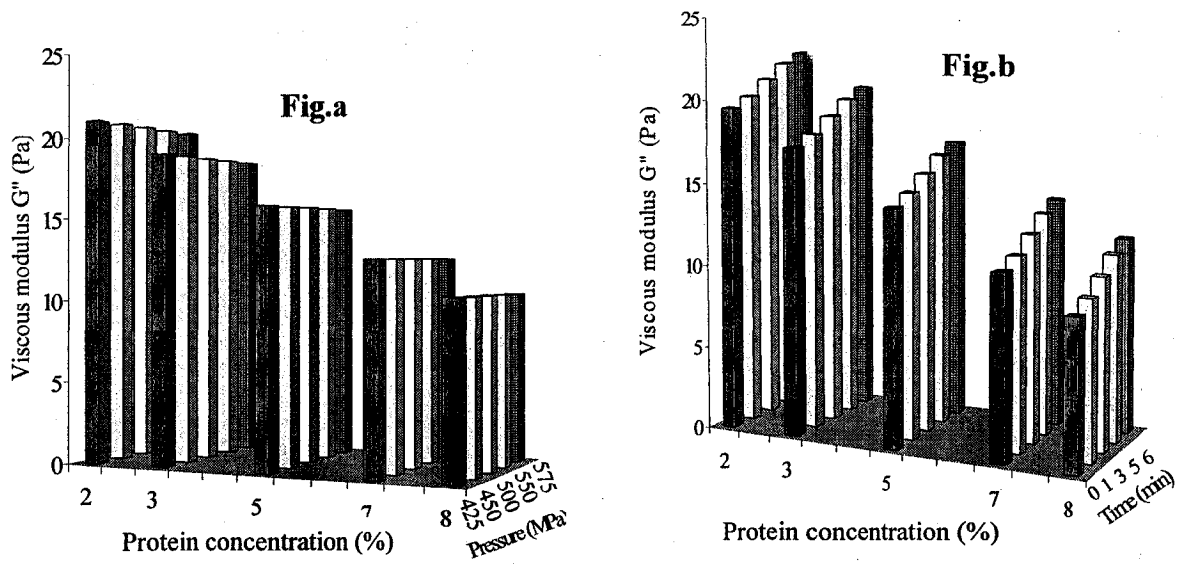
**Table 5.2 Regression coefficient and analysis of variance for six response variables**

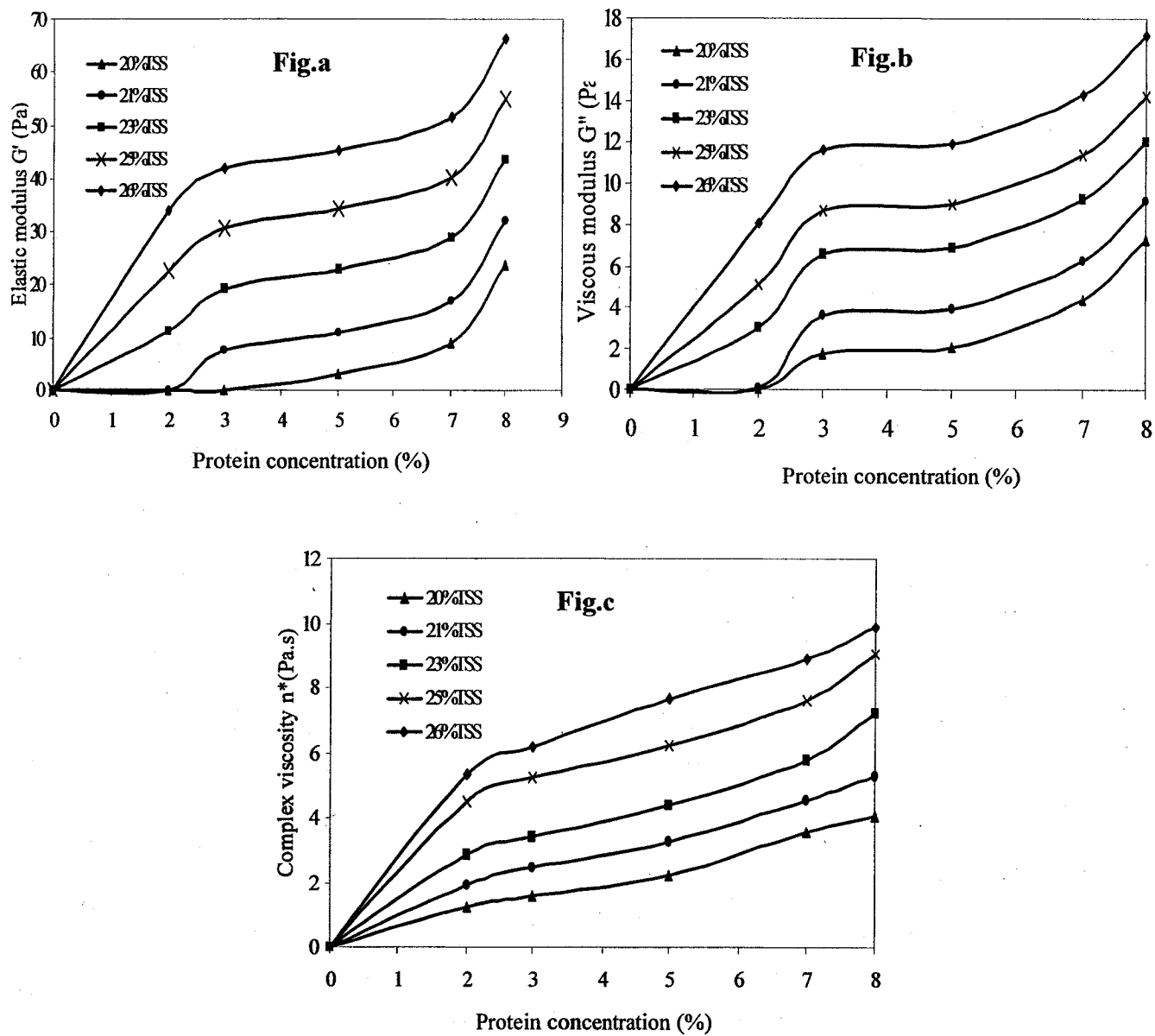
Coefficient	Complex viscosity	Viscous modulus	Elastic modulus
$a_0$	10.2***	15.6***	62.2***
<b>Linear</b>			
$a_1$	-2.601***	-3.46***	-15.9***
$a_2$	0.40609	0.423	3.29
$a_3$	0.512	0.4204	-0.24
<b>Interactions</b>			
$a_{12}$	1.0608**	0.361	6.6006**
$a_{13}$	1.32***	1.26*	8.16***
$a_{23}$	0.311	0.377	1.92
$a_{123}$	1.036*	0.391	6.41*
$a_{11}$	0.8024	0.4069	4.19
$a_{22}$	0.359	0.112	2.25
$a_{33}$	0.0516	0.0962	0.39
%Variability explained ( $R^2$ )	0.79	0.65	0.79
<b>F-value</b>			
Regression	18.08	21.10	18.04
Lack of fit	2.76	1.85	2.83
<b>Probability of F</b>			
Regression	<0.0001	0.0001	<0.0001
Lack of fit	N. S.	N. S.	N. S.

\*\*\* Highly significant ( $P < 0.01$ ), \*\* Significant ( $P < 0.05$ ), \* Borderline significant ( $0.05 - 0.1$ )

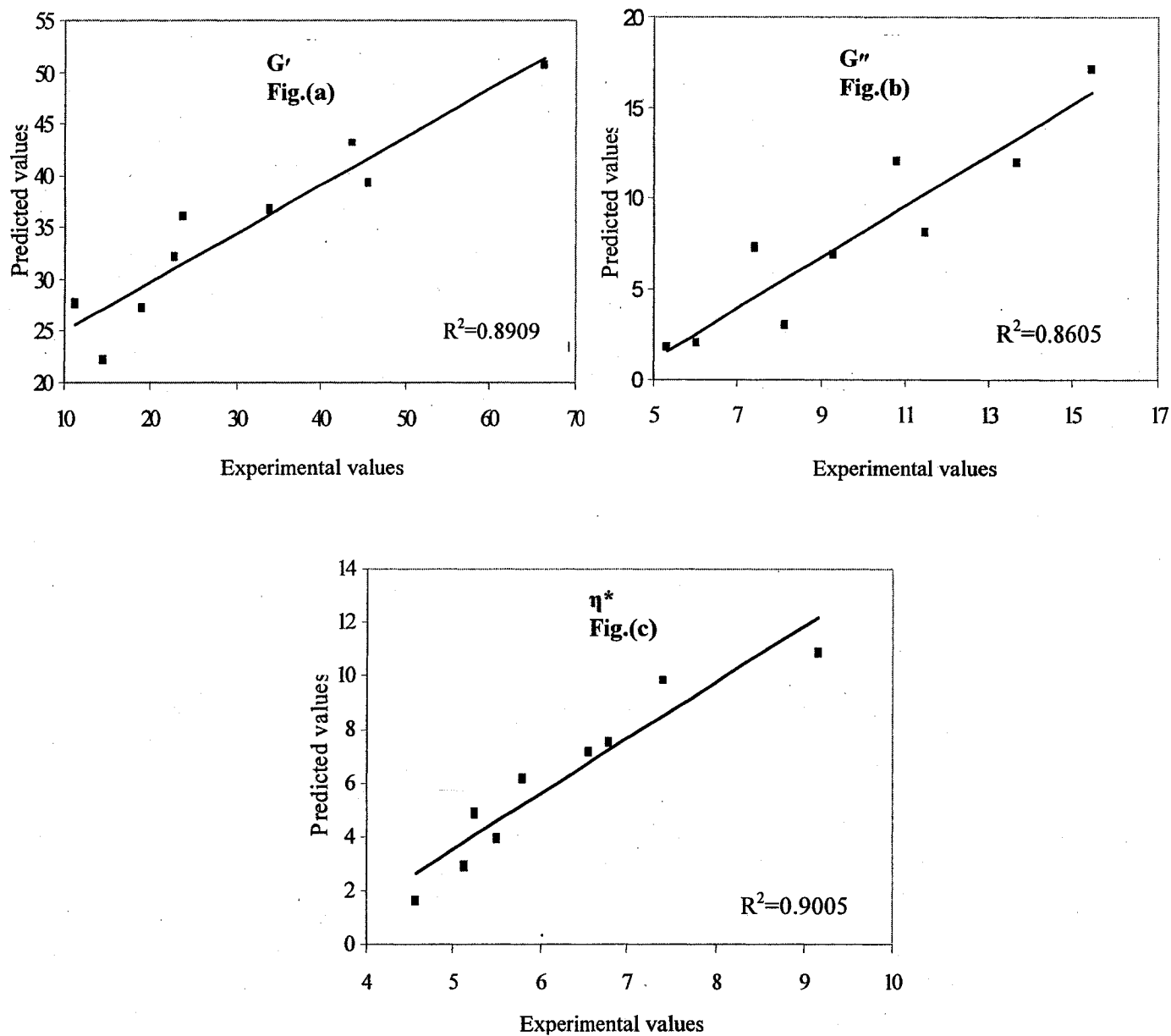
#### ***5.5.5.3 Individual effects of protein concentration and soluble solids***

Effect of protein concentration and soluble solids level in mango puree were isolated using the hypothetical model as explained in the previous section for pressure treatment at 500 MPa and 3 min holding time. An increase in rheological parameters was observed with the protein concentration at each soluble solids level (Figure 5.8 a, b and c). The soluble solids clearly over-dominated the effect of protein concentration. Developed models helped in predicting the effect of different ingredients on rheological parameters. A good correlation was found between the predicted values and combined effect of protein and soluble solids concentration ( $R^2 > 0.85$ ) as represented in Figures 5.9a, b and c.





**Figure 5.8 Predicted individual effects of soluble solids and protein concentration on a) elastic modulus, b) viscous modulus and c) complex viscosity at 500MPa and 3min holding time.**



**Figure 5.9. Predicted and experimental values of a) elastic modulus b) viscous modulus and c) complex viscosity at 500 MPa and 3min holding time.**

## **5.6 Part 3: Sensory evaluation of HP treated protein enriched mango puree**

### **5.6.1 Material and method**

The sensory evaluation of mango puree with different protein concentrations (2, 5 and 8%) before and after high pressure treatment (500MPa/3min) was performed using a nine point hedonic scale [10] by a panel of ten judges with equal ratio of men and women. The quality parameters chosen were color, sweetness, mouth feel, flavor, and acceptability. All samples were served in closed, opaque cups to retain flavor till the test is done. Samples were served in randomized order to avoid any effects based on the serving order. The judges were asked to compare protein added samples with each other and with pure untreated mango puree (control). The average hedonic scale ratings of the judges were as represented in Table 5.3.

### **5.6.2 Results**

#### ***5.6.2.1 Comparison of protein added samples with each other***

Significant ( $p < 0.05$ ) change in color was visible with sample enriched with 8% protein after pressure treatment whereas mango puree with 2% and 5% protein concentrations showed insignificant change. However, the color was acceptable before and after pressure treatment. Sweetness was not affected by pressure but 5% protein added sample was preferred to 2% and 8% protein concentration. Mouth feel also increased significantly ( $P < 0.05$ ) and better liked after pressure treatment. Flavor remained the same before and after pressure treatment. However, mango puree with 8% protein concentration showed slight off flavor and bitterness but not at 2 and 5%. Overall acceptance was better for 2 and 5% protein added sample than 8%.

**Table 5.3 Results of sensory evaluation of control and protein enriched mango puree before and after pressure treatment (higher values represent better acceptability) [B<sub>PT</sub>-before protein treatment; A<sub>PT</sub>-after protein treatment]**

	Control	2%PC		5%PC		8%PC	
		B <sub>PT</sub>	A <sub>PT</sub>	B <sub>PT</sub>	A <sub>PT</sub>	B <sub>PT</sub>	A <sub>PT</sub>
Color	8	8	8	8	7	8	5
Sweetness	7	7	7	8	8	5	5
Flavor	8	7	7	6	6	5	5
Mouth feel	7	7	8	6	8	5	7
Acceptability	8	8	8	7	6	5	5

#### **5.6.2.2 Comparison of control with protein added samples**

Insignificant ( $p < 0.05$ ) change in color between control and protein added mango puree was noticed before pressure treatment whereas change was significantly visible after treatment. Sweetness reduced with protein concentration and sample with 5% protein was preferred to 2%, 8% and control. Mouth feel was similar for control and protein added samples. Control had strong flavor and a significant change ( $P < 0.05$ ) was noticed only with 8% protein concentration.

## **5.7 Conclusions**

Soluble solids had major effect on rheological parameters than added protein. Effects of pressure and holding time on protein added mango puree were similar during the first and second part of experiments. Increase in pressure increased  $G'$ ,  $G''$  and  $\eta^*$ . Values of  $G'$  were higher than  $G''$  exhibiting the gelling properties of product. However, in the first part, soluble solids content of mango puree and protein concentration showed positive effect on  $G'$ ,  $G''$  and  $\eta^*$  except when the treatment was given as a pressure pulse. More work is needed to explain the pulse pressure effect on protein added mango puree. In the second part, effect of protein concentration was dominated by simultaneous decrease in soluble solids content resulting in overall decrease in rheological parameters with protein content.  $G''$  increased with added protein whereas, a minimum of 5% protein concentration was required to observe increasing trend of  $G'$  and  $\eta^*$ . Isolated effect of

protein from soluble solids content showed its gelling behavior. The models well predicted the rheological parameters influenced by soluble solids content and protein combination. Sensory evaluation results indicated that protein concentration up to 5% is more preferred than 8%. High pressure treatment did not help in reducing the off flavor at higher protein concentration.

## 5.8 References

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## CHAPTER VI

### GENERAL SUMMARY AND SUGGESTIONS FOR FUTURE STUDIES

#### 6.1 General summary

Thermo-physical and rheological characteristics of mango puree were investigated as function of temperature (20, 40, 60 and 80°C) and concentration (15, 20, 30 and 40°Brix). Both steady flow and oscillation dynamic shear were modeled by power type equation. Mango puree exhibited pseudoplasticity at steady shear measurement at shear rate of 0.1 to 100s<sup>-1</sup>. Flow behavior index decreased with concentration while an increasing trend was observed with temperature after 40°C. Consistency coefficient increased with concentration and decreased with temperature. Mango puree exhibited very low or negligible amount of yield stress. Arrhenius relationship well defined the temperature dependency of consistency coefficient from shear stress-shear rate and apparent viscosity-shear rate data. Dynamic oscillation shear measurement (0.1 to 10 Hz) revealed that mango puree behaved as weak gel at higher temperature and concentration. Cox-Merz rule was applied by superimposing steady shear viscosity and complex viscosity at equal values of shear rate (0.1-10 s<sup>-1</sup>) and frequency (0.1-10 Hz) range. It was found that mango puree did not follow the Cox-Merz rule. Inconsistence with the moisture content of the sample, thermal conductivity, thermal diffusivity and heat capacity increased with temperature but decreased with concentration while the reverse trend was observed for density. Addition of sucrose increased the glass transition temperature of mango puree indicating better stability. Thermal properties and glass transition temperature of mango puree were found to fit linear relationships with temperature and concentration ( $R^2 > 0.90$ ).

Soluble solids had major effect on rheological parameters than protein concentration. Effects of pressure and holding time were similar on mango puree when supplemented with solid whey protein or protein dispersion. Increase in pressure

increased elastic modulus, viscous modulus and complex viscosity. Higher value of elastic modulus than viscous modulus exhibited the gelling property of mango puree and protein combination. Soluble solids content of mango puree and solid whey protein concentration showed positive effect on rheological parameters except at pulse pressure effect. More work is needed to explain the pulse pressure effect on protein added mango puree. However, effect of different concentration of protein dispersion was dominated by simultaneous decrease in soluble solids content in mango puree resulting in overall decrease in rheological parameters. Viscous modulus increased with added protein whereas, a minimum of 5% protein concentration was required to observe increasing trend of elastic modulus and complex viscosity. Hypothetical models developed to isolate effect of protein from soluble solids content demonstrated the true effect of pressure on protein concentration. At applied pressure level, rheological parameters increased with protein concentration at all soluble solids level of mango puree. It also represented the gelling behavior of protein. However, soluble solids in mango puree over dominated the effect of protein concentration. The models well predicted the rheological parameters influenced by soluble solids content and protein combination. Sensory evaluation results indicated that protein concentration up to 5% had better acceptability than 8%. High pressure treatment did not help in reducing the off flavor at higher protein concentration.

## **6.2 Future research**

In view of application of high pressure in developing products with better nutritional and functional properties, it would be of great interest to

1. Study the affect of high pressure on rheological properties of fruits products supplemented with combination of different proteins (designer drinks)
2. Evaluate the shelf life and consumer acceptance of developed products.

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