STANDARDISATION OF THERMAL PROCESSING OF CUTTLEFISH NIDAMENTAL GLAND

By

BIKASH KUMAR PATI

THESIS

Submitted in partial fulfillment of the requirement for the degree of

MASTER OF FISHERIES SCIENCE

Faculty of Fisheries

Kerala Agricultural University

2007

DEPARTMENT OF PROCESSING TECHNOLOGY

COLLEGE OF FISHERIES
PANANGAD, COCHIN

DECLARATION

I hereby declare that this thesis entitled **"STANDARDISATION OF THERMAL PROCESSING OF CUTTLEFISH NIDAMENTAL GLAND"** is an authentic record of the work done by me and that no part thereof has been presented for the award of any degree, diploma, associateship, fellowship or any other similar title.

Bikash Kumar Pati

Panangad,
18/10/2007

Bikash Kumar Pati
2005-14-108

CERTIFICATE

Certified that this thesis entitled **"STANDARDISATION OF THERMAL PROCESSING OF CUTTLEFISH NIDAMENTAL GLAND"** is a record of research work done independently by Mr. Bikash Kumar Pati under my guidance and supervision and that it has not previously formed the basis for award of any degree, fellowship or associateship to him.

Place : Panangad
Date : 18/10/2007

Dr. P. M. Sherief
(Chairperson, Advisory Committee),
Professor & Head of Dept.
Department of Processing Technology
College of Fisheries,
Panangad, Kochi.

NAME AND DESIGNATION OF THE MEMBERS OF THEADVISORY COMMITTEE/ EXAMINATION COMMITTEE

CHAIRPERSON **Signature**

Dr. P. M. SHERIEF
PROFESSOR & HEAD OF DEPT.
DEPARTMENT OF PROCESSING TECHNOLOGY,
COLLEGE OF FISHERIES,
PANANGAD, KOCHI.

MEMBERS

Dr. SAJAN GEORGE
PROFESSOR,
DEPARTMENT OF PROCESSING TECHNOLOGY,
COLLEGE OF FISHERIES,
PANANGAD, KOCHI.

Dr. M. C. GEORGE
ASSOCIATE PROFESSOR,
DEPARTMENT OF PROCESSING TECHNOLOGY,
COLLEGE OF FISHERIES,
PANANGAD, KOCHI.

Smt. MALIKA V.
ASSISTANT PROFESSOR (SELECTION GRADE),
DEPARTMENT OF MANAGEMENT STUDIES,
COLLEGE OF FISHERIES,
PANANGAD, KOCHI.

EXTERNAL EXAMINER 19.12.07

ACKNOWLEDGEMENT

I wish to express my heartfelt gratitude and deepest sense of indebtedness to my guide and Major Advisor Dr. P. M. Sherief, Professor, Department of Processing Technology, College of Fisheries, Panangad, for his keen interest, guidance, encouragement and valuable discussion in every step of this piece of research work.

I express my humble gratitude to Dr. D. D. Nambudiri, Dean, College of Fisheries, Panangad, for providing facilities to do this piece of work. I am also thankful to ICAR, Delhi, for providing me with scholarship and contingency grant through out the course of the study.

I feel great pleasure in expressing my regards and profound indebtedness to my Advisor committee member Dr. Sajan George, Professor, Department of Processing Technology, College of Fisheries, Panangad, for his valuable counseling, guidance and support during the research work and writing of thesis.

I acknowledge my sincerest gratitude and profound indebtedness to my Advisor committee member Dr. M. C. George, Associate Professor, Department of Processing Technology, College of Fisheries, Panangad, for his enthusiastic guidance, suggestion and active support in carrying out the work.

My sincere thanks are also due to Smt. Malika V., Assistant Professor (Selection Grade), Department of Management Studies, College of Fisheries, Panangad, for the statistical planning of the experimental and also for her cordial and timely help for the preparation of the thesis.

I am grateful to Dr. T. K. Srinivasa Gopal, Principal Scientist, Fish Processing Division of Central Institute of Fisheries Technology, Cochin, for permitting me to use their canning facilities and constant guidance through out my work.

I acknowledge deeply my debts to Dr. Lizy Behanan, Professor, Mr. Krishna Kumar, Associate Professor, Department of Processing Technology, College of Fisheries, Panangad and Dr. C. N. Ravi Shankar, Senior Scientist, Fish Processing Division of Central Institute of Fisheries Technology, Cochin, for their generous help, support and guidance.

The assistance rendered by the library staff of College of Fisheries, Panangad and Central Institute of Fisheries Technology, Cochin is gratefully acknowledged.

I express my sincere appreciation and heart-felt thanks to Aruna bhai, Mohan sir, Laxmisha sir, Anu madam, Sindhu chechi, Rajamma chechi, Kareem cheta for their assistance through out my work.

In this opportunity I never forget to give thank to all of my wonderful classmates, Anju, Navya, Sridevi, Indu, Rajani, and Siji chechi for their love, help, cooperation, encouragement and support in my work and study.

I wish to extend my sincere thank to my friends, juniors and seniors Monoj sir, Samir, Mama, Siba, Samar, Ketan, Viral, Biak lun, Mathivanan, Jyoti bhai, Sushanta bhai, Vivek, Sunil, Darshi, Niraj, Bulu, Ankur for their critical remarks and constructive suggestions.

I am grateful to my brothers, sisters and relatives for their affection, support and understanding which enabled me to continue my study.

I express my reverence to GOD Almighty who has afforded his enormous strength during the present work and my years of study.

Bikash Kumar Pati

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INTRODUCTION

1. INTRODUCTION

Commercial food preservation methods are aimed at preventing undesirable changes in the wholesomeness, nutritive value and sensory quality of food, controlling growth of microorganisms and obviating contamination by adopting economic methods. Thermal processing for long- term preservation of foods came into existence in the early 19th century and the French man Nicholas Appert in 1811 is credited as the inventor of this technology. The growth of canning industry was phenomenal in the west. Billions of food cans are processed annually and the number of death attributable to food poisoning from improper processing has been reduced to single digit figures.

Containers are an important part of this preservation method. Appert's pioneering work, which established thermal process as a method of preservation, was conducted using glass jars sealed with cork. Glass containers are still used when product display is desired. The disadvantages of glass are its liability to thermal shock during cooling, fragile nature during handling and relative heaviness. Introduction of open top sanitary (OTS) cans was an important development in canning industry. It has a cylindrical body with soldered lock seam and unsoldered double seam ends. No flux or solder can come into contact with food stuff packed and the easiness to clean make the can sanitary. Tin plate cans are most popular in use. It is in fact a sheet of iron plate coated on either side with tin giving a final composition of 98 % steel and 2 % tin. The thickness of the steel plate varies from 0.19 mm to 0.3 mm depending on the size of the can. Tin plate is now available in many specifications and the can maker has to make the correct choice depending upon the product to be processed and the can size needed. Generally a low metalloid content steel with a phosphorus content of 0.02 % called Type MR quality is used in the manufacture of fish cans. Tin plate cans can be 'three piece' cans or 'two piece' cans depending upon how they are made.

The main advantages of tin plate cans over other types of container are: strong enough to protect the contents adequately during shipment can be readily

fabricated to desired size, capable of being heated for sterilization and readily hermetically sealable at high speeds.

India is one of the largest producers of raw material for food processing industry in the world with the potential to be a dominant player in the international food market. Though the value addition is still very low, one of its major inabilities is lack of quality assurance and competitive innovative foods to meet international standards. The seafood processing industry in India is largely export oriented, confining mainly on block frozen sea foods. The traditional processing sector consists of mainly the salted and dried products, which cater to the need of lower and middle- income populations of the country. Canning as a method of food preservation is of minor importance at present and was at its peak in early 1970's, packing shrimp for export and slumped within a few years. The collapse of seafood canning in India was attributed to lack of diversification and dependence on export market only. Canning at present is mainly confined to very few species such as sardines, mackerels, tuna and crab meat. The quantity processed is almost insignificant and the market for canned product is limited to armed forces and a few Northeastern states. The non- profitability of canning in India as a method of seafood preservation is due to irregular supply of large quantity of fresh fish species at reasonable cost, high cost of containers resulting in high retail prices of canned foods, which do not match the prevailing purchasing power. Expensive manpower, infrastructure and lack of quality assurance programmes also added to its collapse. At present the quality of canned fish packed in India is inconsistent and does not match with the prescribed standards, as the canners usually depend on trial and error technique and rely more on previous experience. The common quality defects are gross underweight, the drained solid weight being less than 40 % of the net weight, whereas the prescribed quality requirements is observed with regard to vacuum, headspace, turbidity of the brine, texture and uniformity of size. Therefore, a lot of effort is required in order to revive the fish canning industry in India and gain consumer

confidence. This involves a scientific approach to standardisation of procedures and thermal process requirements.

The situation of seafood canning in India at present obviously points to the urgent need for improvement in all aspects of commercial canning. The necessity of undertaking investigation based on scientific principles in matters of formulating procedures and processes for fish species, need not be overemphasized. Systematic study and research on varieties of new products can provide a good foundation for the future of the canning industry in India. The seafood processing industry has been identified as a thrust area for development. This industry is included in the priority lending sector. Therefore a wide scope exists to attract entrepreneurs for domestic and export trade. The demand for processed seafoods is likely to increase significantly provided various aspects of quality assurance are followed. As a WTO member, Indian consumer will have a wide choice with imported commodities. Hence quality and cost are the determining factors. The prevailing low labour cost and availability of raw material can be advantageously used. In this direction the use of tin plate can is promising for thermally processed foods as an alternative to other containers.

In view of the above facts, the present investigation was taken up to standardise a procedure for the thermal processing of cuttlefish nidamental gland, packed in tin plate can. The advent of multi- day trawling in west coast of India has resulted in the availability of cephalopods, a major item in the seafood trade of the country, which forms around 7.14 % of our seafood export value. They are exported in different forms such as whole, whole cleaned, double skinned, strips, beaks, wing and roe. The seafood products exported under the name cuttlefish roe is, in fact, the accessory reproductive gland, the nidamental gland. It is a pair of flattened glands associated with the female reproductive system in cephalopods. The glands are extracted from mature specimen of female cuttlefish and exported as a frozen product. Members of European Union (EU) and Japan are the major importer of cuttlefish products from India. Thermal processing of nidamental gland has not so far been attempted. Hence thermal processing of cuttlefish

nidamental gland to produce a ready- to- eat product packed in tin plate can will definitely lead to realization of a high unit value.

In this investigation an attempt has been made to study the raw material characteristics and to arrive at a standard processing procedure and optimum thermal process for the product, packed in tin plate can. It is hoped that this study will be useful in attracting entrepreneurs to venture in producing a uniformly superior quality thermal processed products at affordable costs, revive the canning industry and generate employment potential.

REVIEW OF LITERATURE

2. REVIEW OF LITERATURE

Heat processed foods, though often referred as "canned foods" include heat processed foods in bottles, jars, pouches and metal cans. The heat treatment is applied with the objective of destroying specific, usually pathogenic organisms and also spoilage causing microorganisms, ignoring those which are non health hazard and not capable of causing spoilage. Thermal processing of foods constitutes a significant part of the world's food preservation technique. With the growing demand for convenience, the need for off the shelf, ready to cook and ready to eat packaged food is constantly on the rise. The industry has also to pay attention to the factors such as, the demand for portable ready to eat packaged food, which can be carried home, or to the work place, use of safe packaging material, use of recyclable and environment friendly biodegradable materials. Canning is one of the important methods of fish preservation for future use. Extensive information exists in all aspects of food canning in general like works of Baumgartner (1956), Ball and Olson (1957), Ross (1966), Lock (1969), Kramer and Twigg (1970), Stumbo (1973), Gilbert *et al.* (1982), Stumbo *et al.* (1983), Lopez (1987) and Larousse and Brown (1997).With the improved raw material availability, liberalized economic and trade policies of the government it is hoped that the seafood canning industry in the country can be revived.

2.1. PRINCIPLE OF THERMAL PROCESSING

The thermal processing is not designed to destroy all microorganisms in a packaged product. Such a process would result in low product quality due to the long heating required. Instead, the pathogenic microorganisms in a hermetically sealed container are destroyed and an environment is created inside the package, which does not support the growth of spoilage type microorganisms. In order to determine the extent of heat treatment, several factors must be known (Fellows, 1988): type and heat resistance of the target microorganism, spore or enzyme present in the food; pH of the food; heating conditions; thermo-physico properties of the food and the container shape and size, and storage conditions following the process.

Foods contain different microorganisms and/or enzymes that the thermal process is designed to destroy. In order to determine the type of microorganism on which the process should be based, several factors must be considered. In foods that are vacuum packed in hermetically sealed containers, low oxygen levels are intentionally achieved. Therefore, the prevailing conditions are not conducive to the growth of microorganisms that require oxygen (obligate aerobes) to create food spoilage or public health problems. Further, the spores of obligate aerobes are less heat resistant than the microbial spores that grow under anaerobic conditions (facultative or obligate anaerobes). The heat resistance of food spoilage microorganisms has been studied extensively and thermal resistance data are available for the more resistant organisms in a variety of products (Esty and Meyer, 1922). The heat tolerance of microorganisms is greatly influenced by pH or acidity. From a thermal processing standpoint, foods are divided into three pH groups: high- acid foods (pH < 3.7), acid or medium- acid foods (3.7- 4.5) and low- acid foods (pH > 4.5). With reference to thermal processing, the most important distinction in the pH classification is the dividing line between acid and low acid foods.

Sterilization or its commercial equivalent for the reduction of viable microbes to some predetermined level, forms the basis of a substantial class of food preservation operations, and is particularly important in canning (Kumar *et al.*, 2001). The main purpose of sterilization is the destruction of microorganisms by heating, which causes spoilage of food during preservation. The usually targeted microorganism in the sterilization of foods is the *Clostridium botulinum.* However, the use of non-pathogenic and more resistant species is preferred particularly *Clostridium sporogenes* (PA 3679) (Ranganna, 1986). The argument is that once these have been destroyed, all other less heat resistant spores can be safely assumed to be destroyed.

Most of the research work dealing with thermal processing devotes special attention to *Clostridium botulinum,* which is a highly heat resistant, rod- shaped, spore- forming, anaerobic pathogen that produces an extremely potent exotoxin

under favourable conditions, which leads to *'botulism'* in man. It has been generally accepted that *Clostridium botulinum* does not grow and produce toxins below a pH of 4.5 and is a potential health hazard only in foods with a pH above 4.5. Therefore, all low acid foods should receive a process that is adequate to destroy *Clostridium botulinum.* Generally canned foods receive a heat treatment that is more severe than that required to destroy *Clostridium botulinum* since several other species of microorganisms have a greater heat resistance. An order-of- the- process factor of 12D is used in the commercial heat processing of low acid foods that do not contain preservation levels of salt or other bacteriolabile or bacteriostatic chemicals (Gillespy, 1951).

2.2. PACKAGING MATERIAL FOR THERMAL PROCESSING

The new millennium is dominated by principal changes in the social, ecological and economical structure of the world population and its relation to food and nutrition. Contrasting situation exists in the world on post harvest sector *viz.*, excess supplies of good quality food in industrialized countries as against severe hunger in developing nations. To solve these problems there is an urgent need to use all the innovative potential of food science and technology. In this direction, the improvements in packaging technology have modernized the food industry world wide with food support from food process engineers in developing suitable processing and packaging material, machineries, process design and processing systems that perform predictably. Various kinds of packaging materials have been in use since the development of the canning technology.

2.2.1. Glass

Glass is a hard, amorphous, inorganic, transparent, brittle, substance made by fusing silicates, some times borates and phosphates with certain basic oxides and then rapidly cooled to prevent crystallization. It is a senior member of the family of packaging materials and glass bottles are widely used in food packaging. Nicholas Appert, the father of canning, successfully developed the preservation methods for the first time in a glass bottle.

Glass containers have limited use as a container for heat processing of foods, despite the advantages of glass is being pure, easy to clean, corrosion free, leak proof, recyclable and transparent (Gray, 1950). The major problems with glass containers are the breakage problem and pressure cooling. Breakage can be reduced by careful handling and by avoiding scratches. The blowing of lids during cooling under insufficient pressure may be counteracted by careful, preferably automatic regulation of the pressure during cooling (Anon, 1952) or by applying a special spray cooling (Powers *et al.*, 1951). Bramsnaes and Ramussen (1953) found that glass jars require longer processing time than tin plate cans of similar size. Glass jars are mainly used for home canning purposes and a new processing recommendation for home canned smoked fish in glass jars was made by Raab and Hilberbrad (1993). Marketkar (1998) described the different types of tests required for container. The adequacy of process time is important, as glass cannot be processed at high temperatures safely.

2.2.2. Aluminium

Although, the canning as a method of food preservation was started in glass jars, the rigid metal containers became very popular. The traditional tin plate cans have progressively been replaced by aluminium cans. Aluminium containers were used for packing meat and fish products as early as 1918.

In 1930, A/s Norsk Aluminium Co. carried out extensive investigation on the use of aluminium sheets for making food cans (Howard, 1949). Lopez and Jimenez (1969) reviewed the use of aluminium cans for canning fruits and vegetable products. Griffin, Jr. and Sacharow (1972) suggested a suitable food grade lacquer coating for interior corrosion resistance. Naresh *et al.* (1988) studied the corrosion behavior of aluminium cans by electrochemical studies and found that corrosion reaction is faster in plain aluminium cans as compared to lacquered ones. Lahiri (1992) described the suitability of different aluminium alloys for various food products. He also reported on the corrosion behavior of aluminium cans. Lakhsminarayan (1992) reported that aluminium containers are

100 % recyclable and biodegradable. Srivatsa *et al.* (1993) studied the suitability of indigenously prepared aluminium cans for canning different food products. The standard aluminium of 99.5- 99.7 % purity is obtained by addition of one or more elements like magnesium, silicon, manganese, zinc, copper etc. (Mahadeviah and Gowramma, 1996). Ranau and Oehlenschlaeger (1997) studied the aluminium content in fish and fishery products and concluded that aluminium content of seafood does not present a significant health hazard. Balachandran *et al.* (1998) reported that the best promising alternative to tin plate has been considered as aluminium alloyed with manganese and magnesium. The advantages and disadvantages of aluminium alloys have been described by Balachandran (2001). Ranau *et al.* (2001) studied the changes in aluminium concentration of canned herring fillets in tomato sauce and curry sauce.

2.2.3. Tin- free steel cans (TFS Cans)

This was developed in Japan with the objective of providing a material, which did not require tin as surface coating materials. TFS is an important alternative to tin can. TFS has a steel base with chromium/ chromium oxide coating on the surface replacing the tin in conventional cans.

Barbeiri *et al.* (1970) studied the suitability of various type of chromium-coated steel against tinned steel for packaging food product. The various TFS material differs mainly with respect to surface treatments applied to the steel and the resulting differences in corrosion resistance, appearance and enamel adhesion (Anon, 1974). Tin free steel cans are well suited for canning fish. Investigations carried out showed that the quality of fish (mackerel) in brine with respect to organoleptic quality was quite acceptable when packed in TFS cans for up to 12 months of storage at 37°C. Naresh *et al.* (1989) have reviewed on the chromium coated steel plate as an alternative to tin plate for canning food products. Morries (1993) reported about the different packaging materials using polyester as a coating material for processing different food products. Mathews *et al.* (1998) studied groundnut oil packed in tin- free steel and tin plate container. The recent

development in India is the introduction of polyester coated tin free steel cans suitable for canning fish and fish products. Mallick (2003) studied the suitability of polyester coated tin free steel cans for the thermal processing of rohu curry.

2.2.4. Retort pouches

Retortable pouches, as the name implies, are pouches capable of withstanding retorting operation, are the latest development in the canning industry. The retort pouch is a rectangular type package usually made up of three-layer lamination. Some manufacturers give additional layer for better barrier properties. It is usually made up of outer polyester, middle aluminium foil and inner polypropylene layer. The outer laminate, which is polyester, provides atmospheric barrier properties as well as mechanical strength. The aluminium ply provides protection from gas, light, moisture and ensures a better shelf life. The inner polypropylene layer provides the best heat- sealing medium.

One of the early studies on the use of pouch was by Hu *et al.* (1955). He reported the feasibility of using plastic film packages for heat- processed foods. Lampi (1967) reported the microbiological problems faced in foods packed in retortable pouch. Ishitani *et al.* (1980) reported the effect of light and oxygen on the quality changes of retortable pouch packed foods. Madhwaraj *et al.* (1992) reported that spoilage in the flexible pouch is due to contamination of seal area. Vijayan *et al.* (1998) reported that fish curry remained sterile throughout the storage period of more than one year at ambient temperature and retained acceptable sensory characteristics. Gopal *et al.* (2001) reported that traditional Kerala style fish curry packed in indigenous retort pouch and processed to a F_o (accumulated lethality) of 8.43 gave a better texture.

2.2.5. Tin plate can

Tin is the most ideal metal for thermal processing and have a lion's share as far as packaging of food product is concerned. Tin plate containers made their appearance in 1810. The tin plate can is made of about 98 % steel and 2 % tin

coating on either side. The base steel used for making can is referred to as CMQ or Can Making Quality steel. Corrosion behavior, strength and durability of the tin plate depend upon the chemical composition of the steel base. Depending upon the degree of workability, strength and corrosion resistance required in the case of tin plates four types of steel are specified. They are type L, type MR, type MC and type M. First three are produced by cold reduction process. Type M is similar to type MC in composition but produced by hot reduction process.

The can body protects the contents against the entry of microorganisms, insects, air, light and moisture. They are light in weight and can be handled with ease. A very important advantage is that they can be sterilized at high temperature and pressure. Its unique advantage lies in the combination of the strength of the steel with the protective properties and the gloss of the tin layer. Regarding its corrosion resistance and staining properties, the steel plate may be considered to be covered on both sides with 4 layers; alloy, tin, protective oxide and oil (Hoare, 1950). The trend towards reduced tin coating necessissted the development of enamels or lacquers as reinforcement. Many such protective lacquers have been developed (Midwood, 1954). These can be adapted to any type of canned food and to any canning procedure (Flugg, 1951). Now a days cans are coated by inside spraying, which is more expensive but avoids damaging of the coatings during manufacture of the can. The sulphur resistant lacquer is especially needed as coating for species containing a large amount of trimethyl amine oxide (TMAO) (Anon, 1953). Epoxifide lacquers are the next addition; they are both acid and sulphur resistant and thus have a wide range of application. The non- toxicity of can linings has been stressed by Ives and Dack (1957). The corrosion of tin in contact with acidic fruit juices is attributed to the reversal of polarity of tin and thus tin becomes anodic to iron in acidic medium, thereby dissolving the latter (Albu- Yaron, 1992; Gowramma *et al.*, 1981).

Tin plate containers are considered as the ideal packaging material for preservation of food products due to their many advantages compared to other packaging material (Kapoor, 2001; Joshi, 2001).

2.3. STUDIES ON THE USE OF TIN PLATE CAN FOR SEAFOODS

Canning procedures for three species of sardines landed on the east coast of India have been worked out by Srinivasan *et al.* (1966). An attempt to can Indian style fish curry in tin cans was made by Rai *et al.* (1971). A study was carried out by Choudhari *et al.* (1978) for the prediction of drained weight of canned prawn under laboratory condition in tin cans. Saralaya and Nagaraj (1980) standardised the methods for canning of skinless mackerel fillets in quarterdingly cans, adopting deskinning by lye peeling. The method consists of dipping of fillets in boiling 1 % NaOH solution for one minute. George *et al.* (1985) found out that fresh frozen sardines were found to be suitable for canning in tin cans and can be stored up to 10- 24 weeks depending upon the season and initial quality. Later Vijayan and Balachandran (1986) developed sardine fish curry in tin cans. Canning studies with tin cans were done with freshwater fish rohu (Balachandran and Vijayan, 1988). They studied the effect of citric acid and calcium chloride in the brining solution to improve the texture.

2.4. THERMAL PROCESS TIME FOR CANNED FOODS

The thermal processing is regarded as the critical control point in canning. It is essential for the establishment of safety and stability of end products. Establishment of scheduled processes is important for successful canning. The maintenance of minimum initial temperature, process time- temperature, overpressure, venting of steam, are regarded as critical in canning. The first and foremost attempt to determine the thermal process time for canned foods was done by Bigelow *et al.* (1920). The method introduced by Bigelow and co-researchers is usually referred as 'general method' in canning industry. This method involves the graphical integration of the total lethal effect for the time-temperature combinations, which the 'cold spot' experiences during a thermal process. The main drawbacks of this method is that it can be applied only when the required processing conditions, such as retort temperature, initial temperature and can- size are identical with those of the given heat penetration data.

Ball (1923) developed a 'formula or analytical method'. This is simpler but many mathematical and empirical assumptions have to be made. The symbol 'F_o' introduced by Ball permitted a direct comparison of the relative sterilizing capacities of different heat processes. Olson and Stevens (1939) introduced the nomographic method applicable to canned foods exhibiting straight line semi-logarithmic heat curves. Stumbo (1948, 1949 and 1953) showed the relation between microbiological aspects and process calculations and described new procedures for evaluating thermal process for foods in cylindrical containers. Based on general method, Patashnik (1953) gave a simple method of process calculation. Ball and Olson (1957) improved Ball's original procedure by introducing two parameters which were related to the sterilizing values of heating and cooling phases of heat processes. Stumbo and Longley (1966) have given tables of the parameters 'fh/U' and 'g'. Rao and Prabhu (1971) calculated the F_o value by graphical and formula methods for the standard wet pack of medium grade cooked shrimps.

2.5. HEAT PENETRATION AND THERMAL PROCESS EVALUATION

For the attainment of microbiologically stable state, the amount of heat received by the product during the thermal processing need to be verified. This is determined by the temperature profile within the product during both heating and cooling phases. Extensive reviews are available on the fundamental aspects of heat transfer and the means by which it can be quantified. Heat penetration studies are indispensable to develop a thermal process for new products and to evaluate the effect of thermal process on sensitivity of nutrients and on organoleptic qualities of the product. The most authoritative reviews focusing the thermal death times of microorganisms and the time- temperature relationship within the can are available (Ball and Olson, 1957; Stumbo, 1973; Vinters *et al.*, 1975; Hersom and Hulland, 1980; Scott, 1992). Very little work has been carried out in India on this aspect in canned fishery products. The notable work on standardisation of Saralaya and Bhandary (1978), George (1987), Pujar (1988) and Parshwanath (1989).

The factors influencing the rate of heat penetration such as the characteristics of the container, contents, retorts and mode of heating have been studied very extensively and lot of literature exists on each of these factors. Some of the important reviews related to present study are those by Gillespy (1951), Stumbo (1953), Ball and Olson (1957), Hayakawa (1969), Bhowmik and Tandon (1987), Berry and Buch (1988) and Tung *et al.* (1988). The thermal conductivity values of various food stuffs are given by Woodams and Nowrey (1968).

Determination of the time- temperature history of processed food has practical and safety implications. Navankasattusas and Lund (1978) have discussed methods for time- temperature profile evaluation and measurement of lethality in processed foods. Data on thermal process schedules, which indicate the F_o value, time and temperature of the processing, are available in the literature (Lopez, 1996). Time- temperature histories may be derived by using direct measurements or by mathematical modeling (Tucker and Holdsworth, 1991).

2.6. EFFECTS OF THERMAL PROCESSING ON NUTRIENTS IN SEAFOODS

Fish is mainly a valuable source of protein, iodine, B- complex, fat soluble vitamins and poly unsaturated fatty acids (PUFA) (Borgstrom, 1965). Although the thermal processing makes microorganisms and spores inactive, it may cause destruction of essential nutrients that leads to deterioration of product quality. Much attention has been given to maximizing quality retention for a specified reduction in undesirable microorganism (Holdsworth, 1985; Silva *et al.*, 1992). However, since the degradation of heat- sensitive vitamins and other quality factors such as colour and texture will take place along with the microbial reduction, the optimum processing time and temperature must be utilized. Because of these safety and quality factors, care must be taken to avoid either over processing or under processing (Bichier *et al.*, 1995). It has been reported that moderate heating of fish has not affected the nutritive quality, while overheating leads to loss of nutrients. Reseachers have studied the effect of

thermal processing on the nutritive value of food. Bender (1972) has given the effect of heat on proteins in fish and the implication of heat damage on protein is revealed by Ford (1973). The reduction in lysine content during thermal processing may go up to 25 % (Tooley and Lowrie, 1974). The fish lipids are sensitive to change during heating. An increase in peroxide value has been observed in canned sea foods (Taguchi *et al.*, 1982; Taneka and Taguchi, 1985). The effect of thermal processing on these nutrients has received increasing attention as processors tend to overcook the product to achieve product safety, and the nutritional and sensory changes in fish canning has been reported by (George, 1987). A decrease in TBA value, TMA and vitamin B_1 (thiamine) for shrimp, rainbow trout and Alaska pollock have been reported. Ma *et al.* (1983) reported a toughening during the initial stages of heating and softening during the later stage of processing for shrimps and mussels. Taneka and Taguchi (1985) and George (1987) reported about the changes in nutritional and sensory characteristics in canned fishery products. A decrease in TMAO was observed in cooked squids (Kolodziejska *et al.*, 1994). Mochizuki *et al.* (1995) have analyzed the texture of cooked squid meat at different temperatures. Fellows (1990) has reported that there will be reduction of amino acids in canned products to the extent of about 10- 20 %. The effects of thermal processing in shrimp has revealed that the purine content in shrimp, mainly the adenine and hypoxanthine, decrease during thermal processing (Lou, 1997).

2.7. STATUS OF SEAFOOD CANNING INDUSTRY IN INDIA

Durability and large variety makes canned fish a widely accepted product utilizing 14 % of world seafood production in spite of high cost. The seafood canning industry in India is operating on a relatively small scale. Commercial fish canning in India flourished between 1970- 73, on the west coast with nearly 60-70 canneries operating and majority of which were situated in Kerala. All the canneries packed only shrimp meant for export. In the next few years the seafood canning industry was devastated and the situation has remained the same till now. Several reasons have been attributed to this debacle, such as dependence on one

raw material (shrimp) and a single market (export) (Saralaya, 1976), along with uneven supply of fish and day to day fluctuations in their prices, poor marketing effort and lack of Government support (Mascarenhas and Saralaya, 1986). Inconsistent quality of canned products and lack of process control resulting is not conforming to the standards are the major reasons for the lack of buyers for Indian canned fish. Many alternatives have been suggested, like use of indigenously produced tin plate and diversification of canned product (Anon, 1983; Perovic, 1983; Pillai and George, 1984; Mahadeviah, 1985; 1990). There is an urgent need for improvement, in all aspects, of commercial food canning in India.

Recently, market for processed foods has been growing at a greater rate due to change in life style and food habits of the people. The normal packing media like brine and oil are more suited to the western taste.

2.8. RESEARCH ON SEAFOOD CANNING IN INDIA

The research of seafood canning in India started at the end of 1960's. Earlier studies were confined to shrimps (Nandakumaran *et al.*, 1969). Govindan (1972) has given an account on the possibility of utilizing various fish species for canning in India. Several researchers felt that the dependence of shrimp alone for canning is risky (Chidambaram, 1976; Saralaya, 1976). Work on standardisation for canning of oil sardine (Srinivasan *et al.*, 1966, Sen and Revankar, 1971, Madhavan *et al.*, 1974, Nigam, 1974), tuna (Madhavan and Balachandran, 1971; Balachandran *et al.*, 1982), sardine in natural pack (Nair *et al.*, 1974), lactarius (Balachandran and Madhavan, 1976), squids (Varma and Joseph, 1980; Raghunath and Solanki, 1986; Parshwanath, 1989), seer fish (Nasser, 1980), giant cat fish (Ranganath, 1981), edible oyster meat (Balachandran *et al.*, 1984), white sardine (Jeyasekaran, 1985), rohu (Balachandran and Vijayan, 1988), Kerala style fish curry (Vijayan *et al.*, 1998), mackerel curry (Gopal *et al.*, 1998), rohu curry (Sonaji *et al.*, 2002) and seer fish curry (Gopal *et al.*, 2002) have been reported.

Saralaya and Nagaraj (1978) standardised methods for canning of shell fishes, namely, clams in different media, such as oil, brine and masala. Several studies to generate supporting data for standardisation of canned products have been carried out among which the notable ones are studies on the effect of precooking in fish during canning (Joshi, 1978), studies on the factors influencing heat penetration and sterilization in canning (Venkatesha Murthy, 1981 and Pujar, 1988). Investigation on the effect of thermal process variation on the quality of canned mackerel was also done (George, 1987).

2.9. CEPHALOPODS

Cephalopods are exclusively marine molluscs and there are about 660 species in the world oceans, which are diverse in form, size and nature (Voss, 1977; Worms, 1983). Of these, less than a hundred species are of commercial importance. In Indian seas there are about 80 species of cephalopods of commercial and scientific importance (Oommen, 1977a; Sarvesan, 1974). In recent years, the cephalopods have gained great importance owing to the increasing demand, next to shrimp, in the export trade. Cephalopods were fished from the seas around India from very early times and at present, constitute one of the important exploited marine fishery resources of our country. About three-fourth of the present catch is landed on the west coast, with the three maritime states Kerala, Maharastra and Gujarat- accounting for the lion's share. The cuttlefish fishery in India is mainly constituted by two species, *viz.*, *Sepia pharaonis* and *Sepia aculeata*. *Loligo duvaucellii* is the single species that almost constitute the squid fishery of India. The squid and cuttlefish are noted for high yield and lack of bones. Almost the entire catch of Indian cephalopods is exported to the overseas market. Cephalopod consumption depends to a large degree on traditional habits and consumer tastes, again, consumer acceptability of any product depends upon the quality of the raw product and therefore, the quality of cephalopod raw material and finished products are to be ensured before they are processed and exported.

2.9.1. Proximate composition

Investigations of the proximate composition of the meat provide the basic data on the chemical properties of the raw material (Kreuzer, 1984). Japanese scientists, Matsumoto (1959) did fundamental research on chemical components of cephalopods, especially proteins, while Tanikawa *et al.* (1953) mainly carried out applied research.

2.9.1.1. Moisture

The cuttlefish, *Sepiella inermis*, was reported to have a moisture content of 74.7 % and squid, *Loligo indica*, 75.05 % (Suryanarayanan *et al.*, 1973). Joseph *et al.* (1977) reported an approximate moisture content of 83 % in squid (*Loligo spp.*). Cuttlefishes, *Sepia pharaonis* and *Sepia esculenta*, were found to have a moisture content of 76.4 % and 81.5 %, respectively (Suyama and Kobayashi, 1980). Raghunath (1984) has reported a moisture content of 78.33 % in the squid, *Loligo duvaucellii*. The cuttlefish, *Sepia aculeata*, was observed to have a moisture content of 76.85% (Joseph and Perigreen, 1988). Selvaraj *et al.* (1991) reported 82.9 % moisture content in squid, *Loligo duvaucellii*.

2.9.1.2. Protein

Muscle proteins together with non- protein nitrogenous compounds are referred to as crude protein and the percentage distribution varies in different food myosystems. The crude protein content in seafood depends on the species and variety, the state of nutrition, the stage of the reproductive cycle of the animal and on specific properties of the different parts of the organism (Sikorski, 1994a).

Migita and Matsumota (1954) found 55.5 % of water extractable protein in squid compared to 27.5 % in carps and 22.3 % in horse mackerel. The crude protein content on dry weight basis was found to be 80.12 % and 81.5 %, respectively in, *Sepia orientalis* and *Loligo vulgaris* (Pandit and Magar, 1972). In squid (*Loligo spp.*) an approximate protein content of 15-16 % was reported by

Joseph *et al.* (1977). Sastry and Srikar (1985) reported 3.13 g total nitrogen content/100 g in *Sepia aculeata.* The squid, *Loligo duvaucellii,* was reported to have 3.11 % total nitrogen, 1.41 % water soluble nitrogen and 0.72 % non protein nitrogen (Raghunath, 1984). Selvaraj *et al.* (1991) reported total nitrogen content of 2.8 % and salt soluble nitrogen of 0.6 %, in the squid, *Loligo duvaucellii.*

2.9.1.3. Lipid

The lipid in the body and in the diet constitute a concentrated form of energy for metabolism and storage purposes and may also serve important non-caloric metabolic functions (George and Berger, 1966).

Pandit and Magar (1972) reported a lipid content of 3.9 % for *Sepia orientalis* and 4 % for *Loligo vulgaris* on dry weight basis. The cephalopods, *Sepiella inermis* and *Loligo indica,* were reported to have a lipid content of 5.56 % and 5.4 %, respectively, on dry weight basis (Suryanarayanan et al., 1973). An approximate fat content of less than 1 % was found in squid (*Loligo spp.*) by Joseph *et al.* (1977). Joseph and Perigreen (1988) observed a fat content of 0.83 % in *Sepia aculeata,* on wet weight basis.

2.9.1.4. Minerals

Aquatic organisms absorb minerals from their diet and surrounding water and deposit them in skeletal tissue, muscle and different organs (Lall, 1989). The ash content of a particular animal indicates the amount of inorganic constituents present in the tissue. Minerals are important for aquatic animals because of the role they play in the formation of skeletal structure, maintenance of colloidal system and regulation of acid- base equilibrium besides being a key component of hormones, enzymes and enzyme activation (Lall, 1995). According to Kuhcau (1962) the consumption of trace elements rich- marine invertebrates is of particular value in every state of malnutrition especially when there is a lack of animal protein and an excess of starchy food, which are deficient in trace element. Minerals are significant because of their nutritive value, safety considerations and their influence of taste and flavour (Haard, 1992).

Most trace elements are however, found to be high in the viscera than in the muscle of marine invertebrates. Pandit and Magar (1972) reported the ash content in *Sepia orientalis* and *Loligo vulgaris* was 8.41 g/100 g and 7.4 g/100 g, respectively, on dry weight basis. Suryanarayan *et al.* (1973) reported an ash content of 13.42 % in *Sepiella inermis* and 12.5 % in *Loligo indica* on dry weight basis. Joseph and Perigreen (1988) reported 4.53 % ash content on wet weight basis in cuttlefish, *Sepia aculeata.* In *Loligo pealei* and *Illex illecebrosus* the ash content was found to range from 0.8 % to 2 % and 0.3 % to 2 %, respectively over a period of 2 years (Krzynowek *et al.*, 1989).

2.10. LOW TEMPERATURE PROCESSING OF CEPHALOPODS

Quality has been defined as aggregate of separate factors, each of which has some influence on acceptability (Howgate, 1978).

The keeping time in ice of the squid, *Nototodarus sloani gouldi*, caught off the south west coast of Tasmania, was investigated by Young *et al.* (1973). It was found that the material remained acceptable for up to eight days and the water content of squid increased significantly with time in ice or chilled seawater. Joseph *et al.* (1977) found that during iced storage of whole cleaned squid, the total nitrogen and non protein nitrogen content decreased considerably. Based on biochemical and organoleptic changes it was seen that squid meat could not be kept in ice, in prime condition, for more than two days. *Illex illecebrosus* and *Loligo pealei*, iced in boxes with a squid to ice ratio of 2:1, had an average total keeping time of 6.3 days (Learson and Ampola, 1977). Icing squid in bulk is not regarded an appropriate method for preserving high quality squid (Kreuzer, 1984). Ampola (1980) is of the opinion that, bulk icing cannot be considered a method suitable for maintaining squid in high quality on board the fishing vessels, in particular so far as texture and appearance are concerned, although spoilage is retarded, if properly, iced. Slabyj and True (1981) estimated the shelf life of whole squid (*Illex illecebrosus*), chilled in brine upto 5 days at 0.6°C. Soluble nitrogen in squid (*Loligo duvaucellii*) during storage in crushed ice and water in

the ratio of, 1:2:0.2 by weight, was studied by Raghunath (1984). Sastry and Srikar (1985) reported the ice stored cuttlefish meat as good only upto 2 days, fair upto 4 days and acceptable upto 6 days of storage in ice with reference to both texture and overall acceptance. Steady decrease in total nitrogen, non- protein nitrogen and salt soluble proteins were observed during storage. Nakamura *et al.* (1985) investigated that in the case of squid, k- value may not be a suitable indicator of freshness. Increase in k- value of common squid after catch is much faster than that of fish, because of differences in degradation pathways of adenosine triphosphate (ATP), and its related compounds. Yamanaka *et al.* (1987) reported agmatine as a potential index for freshness of common squid (*Todarodes pacificus*) stored at, 0°C, 3.5°C and 15°C. Park and Hur (1990) studied changes in freshness during iced storage of common European squid (*Loligo vulgaris*).

Freezing and frozen storage prolong the shelf life of seafood by retarding enzymatic and microbial degradation. But protein denaturation occurs during prolonged storage resulting in moisture loss and textural changes (Selvaraj *et al.*, 1992).

Joseph *et al.* (1977) studied frozen storage changes in squid, *Loligo duavaucellii*. Quality tests indicated that frozen squid stored at -18°C or lower can be kept for one year in good quality for direct consumption or for processing (Ke *et al.*, 1979a). L- sodium glutamate, was found effective in preventing denaturation of squid muscle protein by Iguchi *et al.* (1981). With regard to the frozen resistant nature of the squid, Stanley and Hultin (1982) found that frozen northern Atlantic squids, *Loligo pealei* and *Illex illecebrosus*, were significantly tougher than their fresh materials, which might be caused by protein cross- linking because of high levels of dimethyl amine (DMA) and formaldehyde. *Loligo vulgaris*, stored for 100 days at -20°C was found to show good sensory quality. Protein solubility also remained almost constant during the entire storage period; rancidity was also not detected (Borderias, 1982). Protein solubility, thiobarbituric acid (TBA) values and sensory tests were used as quality indices for frozen squid at the Instituto del Frio, Madrid (Kreuzer, 1984). Stanley and Smith (1984) also

reported that freezing produced a tendency for squid muscle fibres to lose their outer membranes. Effect of raw material quality on the shelf life of squid (*Loligo duvaucellii*) mantles, was studied by Joseph *et al.* (1985). Changes in freshness of Japanese common squid (*Todarodes pacificus*) during cold storage was studied by Nakamura *et al.* (1985). The freezing and cold storage characteristics, of cuttlefish fillets have been studied by Joseph and Perigreen (1988). Park and Hur (1990) studied the skin stripping, freezing and thawing conditions of common European squid (*Loligo vulgaris*). Selvaraj *et al.* (1991) studied the effect of ascorbic acid dip (0.5 % w/v for 10 min) treatment on frozen storage of squid (*Loligo duvaucellii*). Toughness of the mantles of cuttlefish (*Sepia pharaonis*) were found to increase when they were frozen stored at -10°C for 0, 0.5, 1, 2 and 4 months (Yuh and Chau, 1998). Sophia and Sherief (2003a) have reported the effect of different treatment on the iced storage shelf life of cuttlefish (*Sepia aculeata*) fillets. They have shown that dip treatment in 2 % citric acid solution improves the appearance and overall quality of cuttlefish fillets. The treated samples was organoleptically in good condition for 4 days and fair up to 6 days of iced storage and discolouration was noticed during this period. They also found that salt plus citric acid treatment of the cuttlefish fillets is effective in retaining the texture, physical appearance and overall quality of the frozen fillets. After 8 weeks of frozen storage, the untreated samples show yellow discolouration a maillard type of reaction was postulated as the possible yellow discolouration in frozen stored cuttlefish fillets and this reaction can be arrested by treatment with salt and citric acid (Sophia and Sherief, 2003b).

2.10.1. Low temperature processing of cuttlefish nidamental gland

All the entire catch of cuttlefish nidamental gland is processed as a frozen product in India. The frozen cuttlefish nidamental glands constitute about 0.03 % of marine products being exported from India. Italy, Belgium, Portugal and other European countries are the major importer. In Italy, around Venice, cuttlefish nidamental gland is eaten after boiling and with oils as in salads, may be cheaper substitute for oyesters. Santhosh kumar *et al.* (1999) have reported nutrient

composition of nidamental gland from the cuttlefish, *Sepia pharaonis*. They reported the frozen cuttlefish nidamental gland contains 75.83 % moisture, 11.30 % total nitrogen, 5.42 % crude fat and 5.5 % ash on dry weight basis. Besides that, they have also reported cuttlefish nidamental gland contains calcium (345.04 mg/100 g dry wt.), phosphorus (1308.00 mg/100 g dry wt.) and phospholipids (2.38 %).

2.11. THERMAL PROCESSING OF CEPHALOPODS

Cephalopods constitute about 2 % of marine landings in India and are mainly processed by freezing for export. The characteristic strong odour of squid during boiling was attributed to be due to a sulphur containing amine with piperidine nucleus, which gets easily decomposed when heated with acid or weak alkali (Yaminishi and Matsuzaka, 1955). Kimura *et al.* (1969) studied the collagen from, *Todarodes pacificus* and *Octopus vulgaris* together with collagen from other invertebrates. Its shrinkage temperature was found to be 49°C for both squid and octopus. The thermal stability of the collagen of cephalopods was found to correspond to that of warm water fishes. The biochemical composition in squid meat (Suryanarayanan *et al.*, 1973; Joseph *et al.*, 1977) shows that squids can be classified as high protein low fat material. It was found that approximately 25 % of the original protein content (on wet weight basis) was lost by the end of the first minute of cooking at 100°C (Otwell and Hamann, 1979a). Cooking the mantle to 100°C caused gross distortions in all mantle tissues (Otwell and Giddings, 1980). An excellent account on utilization of squids for human consumption is given by Kreuzer (1984). Works on canning of squids (Varma and Joseph, 1980; Parshwanath, 1989) indicated that the squids constitute a good raw material for heat processed foods due to their firm texture after blanching. Raghunath and Solanki (1986) have developed canning procedure for squid mantles in brine.

Value addition and diversification to meet the national and international market demands is a major challenge faced by the Indian fish processing industry.

Value addition is particularly important in fish processing industry as it fetches a high unit value. The need for off the shelf, ready- to- cook and ready- to- eat packaged food is constantly on the rise. Thermal processing of cuttlefish nidamental gland has not so far been attempted. Hence thermal processing of cuttlefish nidamental gland to produce a ready- to- eat product will definitely lead to realization of a high unit value.

MATERIALS AND METHODS

3. MATERIALS AND METHODS

3.1. MATERIALS

3.1.1. Cuttlefish nidamental gland

Mature female specimens of the cuttlefish, *Sepia pharaonis* were procured from fishing harbour, Kochi and transported under iced condition to the College of Fisheries, Panangad. The animals were dissected (Plate No. 1) to collect the nidamental gland (Plate No. 2) which were washed with potable water to remove blood and other adhering tissues.

3.1.2. Can

Three- piece drawn tin plate cans (4½ oz fluid capacity) of the size 301×203 (trade dimension) were used in the present study. They were internally lined with sulphur resistant lacquer coating.

3.1.3. Brine

Common salt was used for preparation of brine having a concentration of 2 %.

3.1.4. Chemicals and glasswares

The chemicals used were purchased from Merck Specialities Pvt. Ltd., Mumbai, India. All chemicals used were of analytical grade. The glasswares manufactured by Borosil, were used for the study.

3.2. EQUIPMENTS AND ACCESSORIES

3.2.1. Overpressure autoclave

The pilot scale retort of model 24 rotary retorting systems (John Fraser and

sons Ltd, Model.no. 5682, Newcastle- upon- Tyne, UK) was used for the study (Plate No. 3). The retort is constructed of mild steel and it can withstand a working pressure of 50 psig.

3.2.2. Double seaming machine

Metal Box Domestic can seamer (Metal Box Company, Calcutta, India) was used in the study for sealing the cans.

3.2.3. Thermocouple probe

The probe used for the experiments was copper/cupronickel thermocouples of Ellab SSA-12050-G700-TS stainless steel electrode (Ellab Co., Roedovre, Denmark) with a length of 50 mm, diameter 1.2 mm.

3.2.4. Packing glands and accessories

Ellab GKM-13009-CXXX packing glands (Ellab A/s, Roedovre, Denmark) with washers were used for the experiments.

3.2.5. Can punch

Ellab TC 89 can punch for rigid containers were used for making holes to fit thermocouple glands into the can.

3.2.6. Ellab recorder

Ellab CTF 9008 (Ellab A/s, Roedovre, Denmark) was used to record core temperature, retort temperature, F_0 value and cook value at a specific time interval of one minute. Temperature range of the instrument is –100.0 to +350.0°C with resolution of 0.1°C. The F_0 constants are programmed at T=121.1°C, Z=10°C and Cook value constants at T=100°C.

3.2.7. Spectrophotometer

Jasco's model No.V- 530 spectrophotometer was used in the present study.

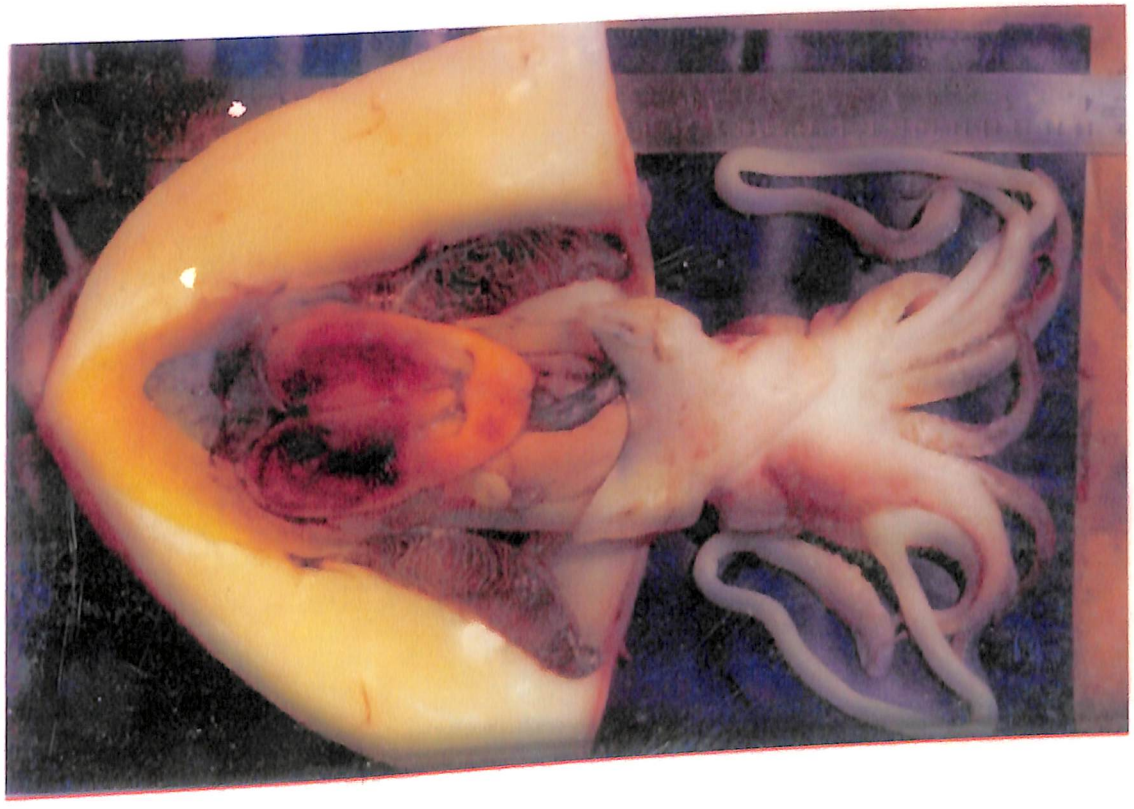

Plate No. 1 Dissected cuttlefish

Plate No. 2 Raw cuttlefish nidamental gland

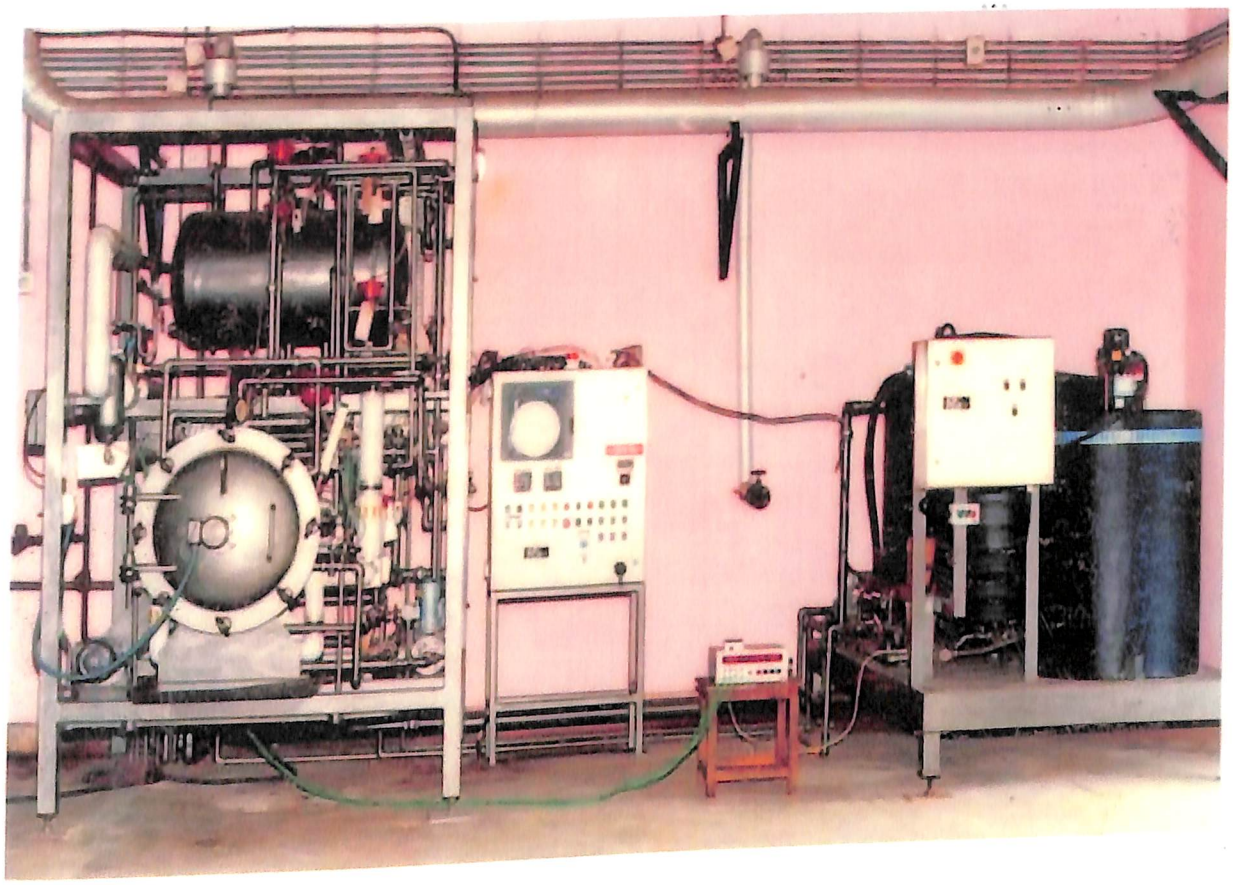

Plate No. 3 Overpressure autoclave

3.3. METHODS

3.3.1. Proximate composition

3.3.1.1. Moisture

The moisture content was determined by the AOAC (2000) oven drying method.

A known weight of sample (10 g) was weighed in a pre-weighed clean Petridish on an electronic balance. Dishes were placed in hot air oven at 100 ± 1°C for 16 hours, cooled in a desiccator and weighed, until a constant weight was obtained and moisture content was calculated and expressed as percentage.

3.3.1.2. Ash Content

Ash content was determined by the method of AOAC (2000).

About 1-2 g of the sample was transferred into a pre weighed silica crucible. The sample was carbonized by burning at low red heat and was placed in a muffle furnace at 550°C for about 4 hours until a white ash was obtained. Crucibles were weighed after cooling in a desiccator and percentage ash content was calculated.

3.3.1.3. Crude fat

Crude fat content was determined following the method of Radin (1981).

About 1 g of minced meat was taken in a mortar and homogenised with 18 ml of extraction solvent (hexane: isopropanol :: 3:2 v/v). It was then filtered into pre- weighed beaker. The residue was washed two or three times with minimum volume of solvent mixture. The solvent was evaporated off on boiling water bath, then cooled to room temperature in a desiccator and weighed. From the difference in weight the percentage lipid content was calculated.

3.3.1.4. Crude protein

Protein content was estimated by the Microkjeldahl's method (AOAC, 2000).

The principle of the method is as follows: The sample is digested with concentrated sulphuric acid in presence of a suitable catalyst, so that the protein nitrogen is converted to ammonium sulphate. The ammonium sulphate formed is, distilled with alkali and ammonia evolved is absorbed in boric acid containing Tachirho's indicator. The ammonia absorbed is then titrated against *N*/70 H_2SO_4. From the titre value, the percentage of the nitrogen in the sample is calculated. Since proteins on an average contain 16 % nitrogen, the crude protein content of the sample is obtained by multiplying the percentage of total nitrogen by 6.25.

About 0.5-1 g of the well-minced sample was transferred into a Kjeldahl flask of 100 ml capacity. A few glass beads and a pinch of digestion mixture (8 parts K_2SO_4 & 1 part $CuSO_4$) and 10 ml concentrated sulphuric acid were also added. It was digested over a burner until the solution turned colourless.

To the digested and cooled solution distilled water was added in small quantities with intermittent shaking and cooling until the addition of water did not generate heat. It was transferred quantitatively into a 100 ml standard flask and made up to the volume. With a 2 ml pipette made-up solution was transferred to the reaction chamber of the Micro-Kjeldahl distillation apparatus (Kjel Plus-Model: Distil- M of Pelican Equipments, Chennai). Two drops of phenolphthalein indicator and 40 % sodium hydroxide were added till the indicator changed to pink. Distillation was done for 4 minutes and ammonia liberated was absorbed into 2 % boric acid containing a drop of Tachirho's indicator. The amount of ammonia liberated was determined by titrating with 0.01 *N* standard sulphuric acid. Crude protein content was calculated by multiplying total nitrogen content with conversion factor of 6.25 and expressed as percentage.

3.3.2. Standardisation of procedure for the thermal processing of cuttlefish nidamental gland

The mature female specimen of the cuttlefish, *Sepia pharaonis* were procured from fishing harbour, Kochi and transported under iced condition to the lab. The animals were dissected to collect the nidamental gland and washed with chlorinated water to remove blood and other adhering tissues. The flow diagram is given in the Fig. 1.

3.3.2.1. Standardisation of blanching conditions

The washed glands were blanched in boiling 5 % brine for 2 min, 5 min, 10 min and 15 min. After blanching samples were subjected to sensory evaluation to find out the optimum period of blanching based on acceptability. The sensory evaluation sheet format is given in **Annexure I.**

3.3.2.2. Standardisation of retorting temperature

About 128 ± 2 g blanched glands were packed along with 68 ± 2 ml of 2 % hot brine into 301 × 203 size tin plate cans to maintain a pack weight of about 197 ± 2 g. Utmost care was taken to avoid the contamination of seal area in the container. Steam exhausting was done for 10 min to remove the air present in the containers. Immediately after the exhausting, cans were closed using the double seaming machine. The cans were then retorted at two different temperature, *viz.*, 115°C for 1 hour and 121.1°C for 30 min. The products were then subjected to sensory evaluation to determine the optimum temperature.

3.3.2.3. Standardisation of process time (B)

The temperature selected was adopted for further thermal process studies at different time periods, such as 20 min, 25 min and 30 min to obtain the required F_0 value. The products were then subjected to sensory evaluation and sterility test. From the results the process time suitable for producing organoleptically acceptable sterile material was found out.

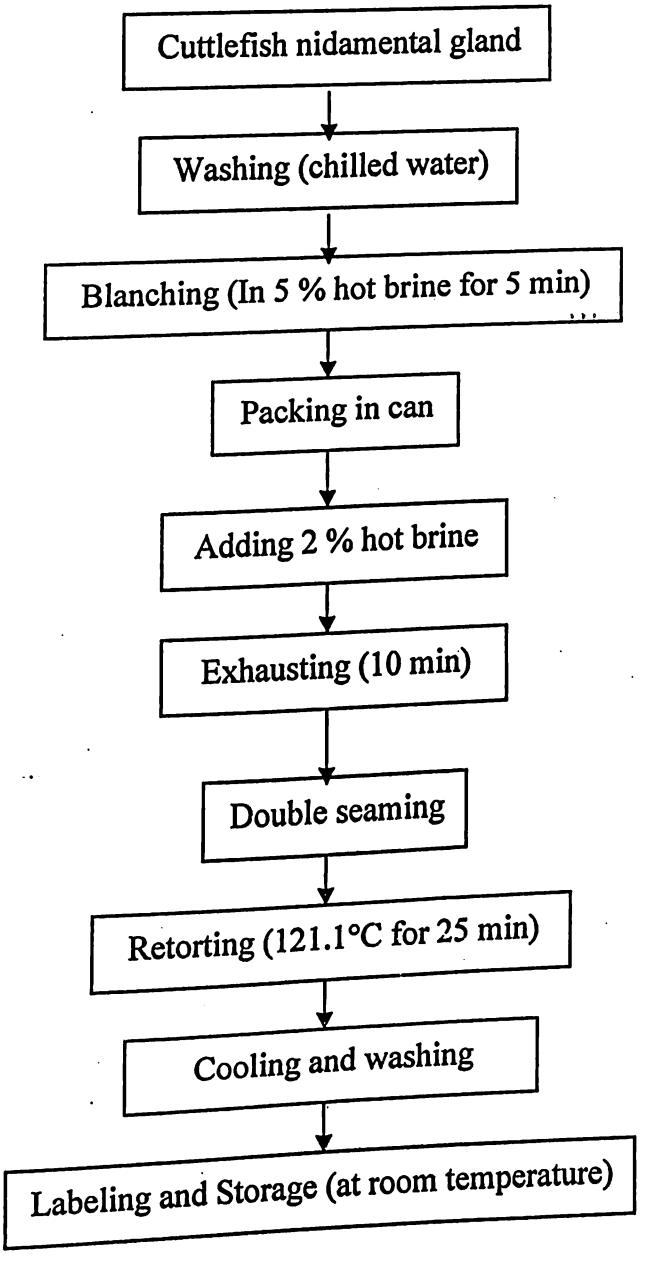

Fig. 1: Flow chart of tin plate can packed nidamental gland

3.3.3. Thermal process evaluation

The thermal processing aims at two fundamental objectives, *viz.*, safety from pathogenic bacteria and prevention of spoilage due to thermophilic spore survival. Regarding the first objective *Clostridium botulinum* is the target organism whose destruction is considered satisfying the safety from pathogenic bacteria.

The filled and sealed tin plate cans were heat processed in an overpressure autoclave at optimum temperature for selected process time. The process data was taken by inserting thermocouple needles into the center of the cold spot (positioned 1/3rd from the bottom). Thermocouple outputs were measured by using an Ellab CTF 9008 data recorder. The recorded data were analysed using a computer. The heat penetration data were plotted on a semi logarithm paper with temperature deficit (RT-CT) in logarithmic scale on Y-axis against time in linear scale on X-axis. Lag factor for heating (J_h), lag factor for cooling (J_c), slope of the heating curve (f_h) and time in minutes for sterilization at retort temperature (U) were determined. Total process time (T) was determined by adding process time (B) and 58 % of the come up time. The F_o was calculated by mathematical method (Stumbo, 1973).

3.4. SHELF LIFE STUDIES

The canned samples (Plate No. 4) were analysed at regular intervals of 30 days to determine their shelf life at room temperature. Analysis was done in triplicates for various physical, chemical, microbiological and organoleptic quality parameters.

3.4.1. Determination of pH

pH was determined as per IS 2168 (1971).

Plate No. 4. Canned cuttlefish nidamental gland

5 g of the sample was dispensed in 10 ml of distilled water and pH was measured by using pH meter.

3.4.2. Chemical parameters

The chemical parameters such as protein, lipid, moisture and ash were carried out for the canned product as in the section 3.3.1

3.4.2.1. Determination of Thiobarbutyric acid (TBA) value

Thiobarbutyric acid value was determined according to the method of Tarladgis *et al.* (1960).

10 g of sample was mixed with 100 ml 0.2 *N* HCI and homogenised to make slurry. Slurry was poured to a round bottom flask and connected to the TBA distillation apparatus. Distillation was done until 50 ml of the distillate was collected within 10 minutes. 5 ml of distillate was taken in a test tube; 5 ml TBA reagent was added and heated for 35 min. A blank was also done with distilled water. Colour developed was measured in a spectrophotometer at 538 nm and TBA value was determined and expressed as mg malonaldehyde/kg of sample.

3.4.2.2. Determination of salt content

Salt content was determined by the method of AOAC (2000).

2 g of sample was taken into conical flask (250 ml), 15 ml of HNO_3 and 25 ml of 0.1 *N* $AgNO_3$ were added with it and digested on sand bath until the solution was clear. Cooled and 50 ml of distilled water, 2 ml of Nitrobenzene, 1 ml of ferric indicator were added. Then the solution was titrated against 0.1 *N* of Ammonium thiocyanate until it becomes brick red.

3.4.3. Microbiological parameters

3.4.3.1. Total plate count

Total plate count of the sample was determined according to the method of Hitching *et al.* (1995).

10 g of the sample was weighed aseptically into a sterile sample dish and transferred into a sterile polythene pouch and soaked in 90 ml normal saline for 15 minutes, after which it was blended in a Stomacher blender (Stomacher 400 Circulator) for 60 seconds at normal speed. Using a sterile pipette, 1 ml of the supernatant was aseptically transferred into a 9 ml saline tube and mixed well using Vortex mixer. Similarly further dilutions were prepared for the inoculation.

1 ml each of the appropriate dilutions was pipetted to appropriately marked sterile petridishes taken in duplicates for each dilution. About 15-18 ml of molten plate count agar medium cooled to 45°C, was poured to each plate, mixed well with the inoculum and allowed to set for 30 minutes. The plates were incubated at 37°C for 48 hours in an inverted position. After the incubation period, the individual bacterial colonies were counted. The average counts of the triplicates were taken and the counts were calculated as cfu/g of the sample.

$$\text{TPC (cfu/g of sample)} = \frac{\text{No. of colonies} \times \text{Dilution factor}}{\text{Weight of the sample}}$$

3.4.3.2. Commercial sterility test

The thermal processed cans were tested for sterility as per IS 2168 (1971).

The thermally processed samples were incubated at 37°C for 15 days and 55°C for a minimum of 5 days. The incubated cans were aseptically opened and 1-2 gm of the samples were taken by a sterilized forceps and inoculated into the sterilized fluid thioglycolate broth in test tubes. A layer of sterilized liquid paraffin was put on to the top of the broth to create anaerobic condition and

incubated at 37°C for 48 hours and at 55° C for 5 days.

3.4.4. Sensory test

Sensory characteristics of samples were evaluated by 10 trained member panel on a ten- point scale as IS 6273 [II] (1971).

Sensory evaluation was based on characterization and differentiation of the various sensory characters such as appearance, texture, odour and flavour. Score was given based on a 10-point hedonic scale by trained taste panel members. Scores 9-10, 6-8, 4-5 and 1-3 were taken for excellent, good, fair and poor respectively for each of the sensory characteristic. The sensory evaluation sheet format is given in **Annexure II.**

3.4.5. Cut- out test

The cut- out test characteristics of the cans were determined by the method given by Saralaya (1978). The proforma is given in **Annexure III.**

3.4.6. Statistical analysis

The SPSS (2000) statistical package was used for analysis of the experimental results. Analysis of variance (ANOVA) was used to calculate significant difference ($P < 0.05$) during storage study. Mean separations were determined by the Duncan multiple range test.

RESULTS

4. RESULTS

4.1. CUTTLEFISH NIDAMENTAL GLAND

Cuttlefish nidamental gland was used as raw material in the present study for the preparation of the product, 'cuttlefish nidamental gland in brine' in tin plate can. The cuttlefish nidamental gland used was very fresh with a characteristic fresh odour and colour.

4.1.1. Proximate composition

The proximate composition of cuttlefish nidamental gland is presented in the Table 1. The fresh gland had moisture content of 74.64 %, crude protein 19.8 %, lipid 3.23 % and ash 1.59 %. There was a reduction in moisture content after blanching and the average percentage on wet weight basis of moisture, protein, lipid and ash were 65.21 %, 25.1 %, 5.38 % and 2.62 %, respectively. It was shown that there was increase in moisture content just after thermal processing and the average percentage on wet weight basis of moisture, protein, lipid and ash content were 76.9 %, 17.24 %, 2.32 % and 1.87 %, respectively. The statistical analysis showed a significant difference, at 5 % level of significance, among the proximate composition in the different stages of processing.

The microbiological quality of nidamental gland was evaluated using total plate count and was found to be 3.35×10^4 cfu/g.

Table. 1: Proximate composition of nidamental gland at different processing stages (mean ± SD, n =3).

Processing steps	Moisture (%)	Protein (%)	Lipid (%)	Ash (%)
Raw	74.64± 1.49^{b}	19.8±0.61^{b}	3.23±0.13^{b}	1.59±0.05^{c}
Blanched	65.21± 1.96^{c}	25.1±0.40^{a}	5.38±0.13^{a}	2.62±0.05^{a}
Thermally processed	76.9± 1.92^{a}	17.24±0.34^{c}	2.32±0.10^{c}	1.87±0.08^{b}

^{a,b,c,d} Means in a column with the same superscript letters are not significantly different (P>0.05).

4.2. STANDARDISATION OF BLANCHING CONDITIONS

The sensory evaluation score and salt concentration of blanched cuttlefish nidamental gland is given in the Table 2 and Fig. 2. Based on the value, 5 % brine for 5 minutes was selected for blanching. The statistical analysis showed a significant difference, at 5 % level of significance, among the sensory characteristics.

Table 2: Sensory evaluation score and salt concentration of the gland after blanching (mean ± SD, n=10).

Concentration of brine (%) /Time (min)	Texture*	Saltiness*	Overall* flavour	Salt (%)
5/2	4.94 ± 0.25^{c}	6.06 ± 0.07^{b}	5.31 ± 0.19^{c}	1.57 ± 0.16^{d}
5/5	$\mathbf{5.69\pm 0.31^{a}}$	$\mathbf{6.19\pm 0.19^{a}}$	$\mathbf{5.69\pm 0.17^{b}}$	$\mathbf{2.15\pm 0.15^{c}}$
5/10	5.63 ± 0.23^{b}	5.81 ± 0.07^{c}	6.13 ± 0.28^{a}	2.38 ± 0.17^{b}
5/15	4.94 ± 0.20^{c}	3.94 ± 0.11^{d}	5.00 ± 0.11^{d}	3.93 ± 0.15^{a}

* All traits measured on ten- point scale 1 being least and 10 being the most.

^{a,b,c,d} Means in a column with same superscript letters are not significantly different ($P>0.05$).

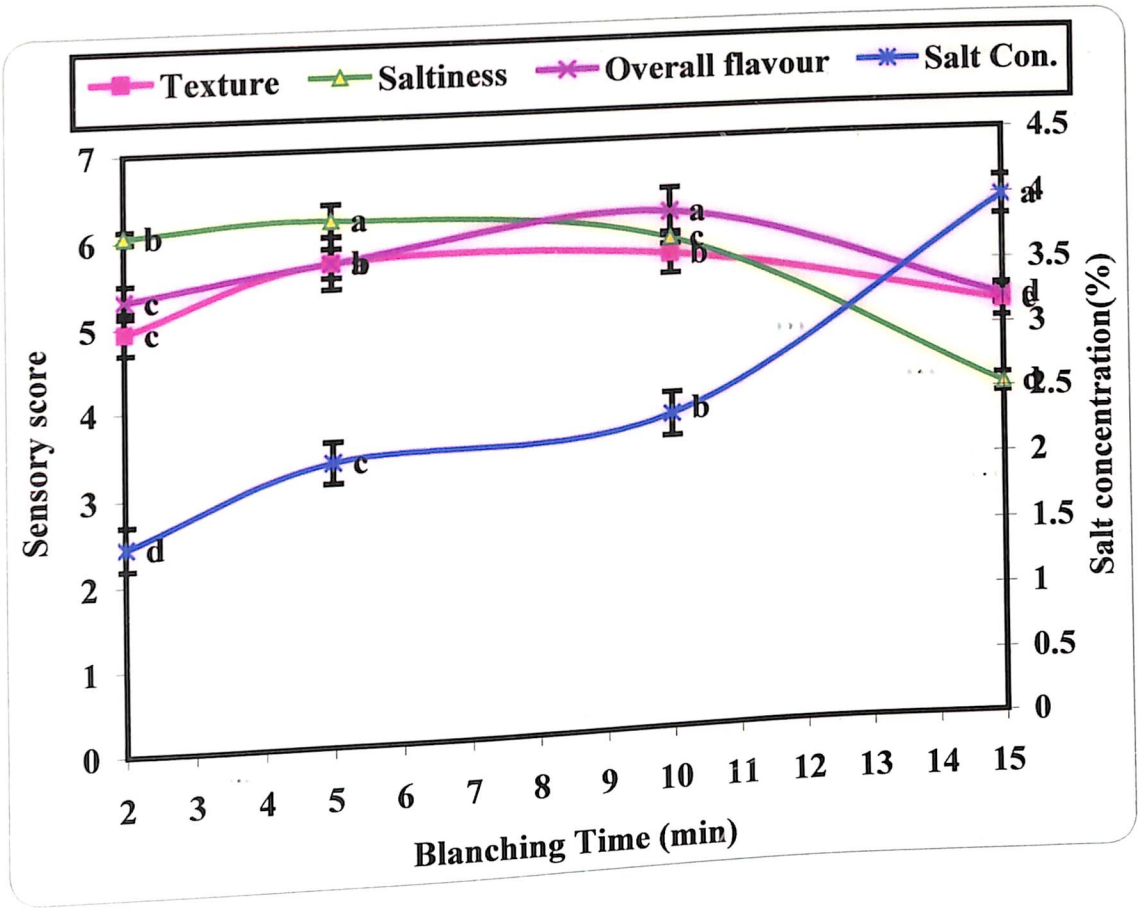

Fig. 2: Sensory evaluation score and salt concentration of the gland after blanching.

[a,b,c,d] Means in a line with the same letters are not significantly different (P>0.05).

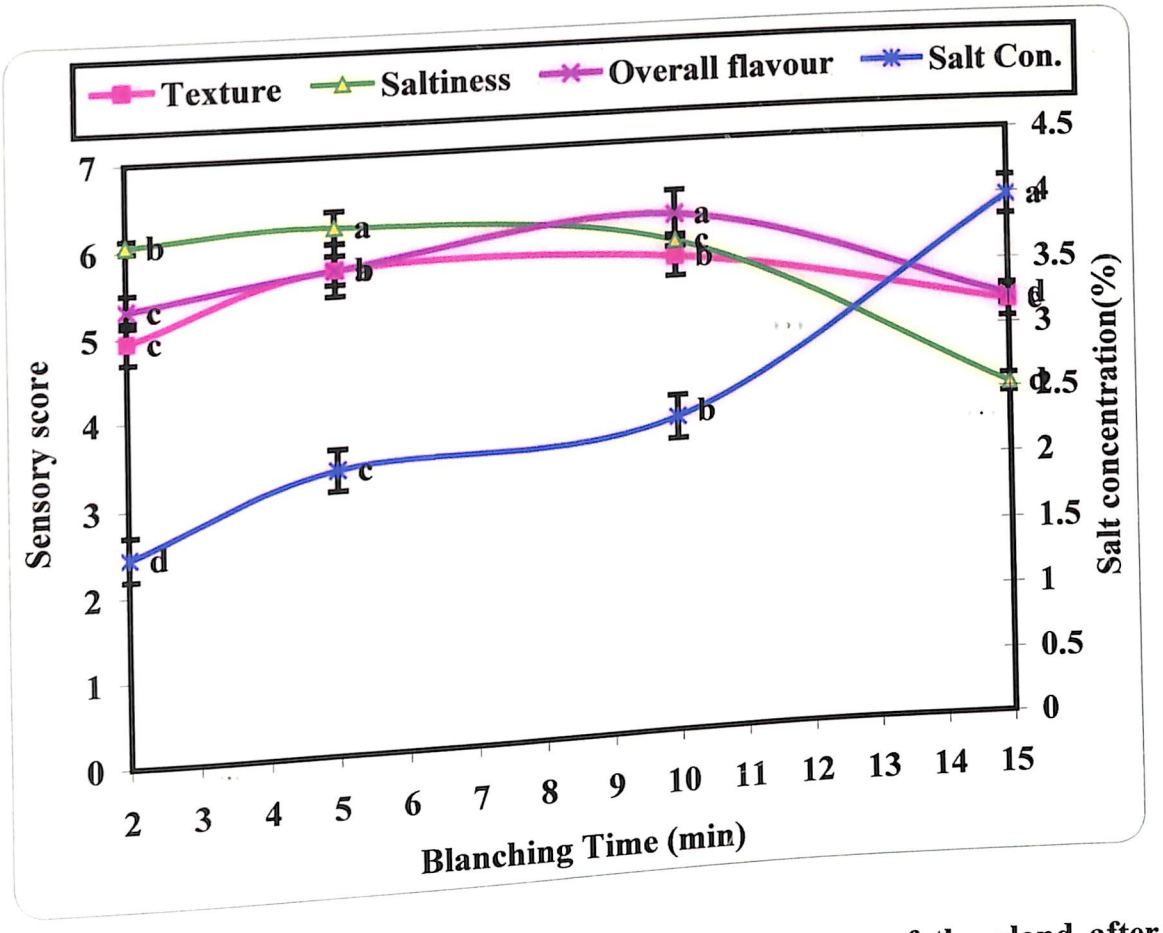

Fig. 2: Sensory evaluation score and salt concentration of the gland after blanching.

[a,b,c,d] Means in a line with the same letters are not significantly different (P>0.05).

4.3. STANDARDISATION OF RETORTING TEMPERATURE

The sensory evaluation score for standardisation of retorting temperature is given in the Table 3. Based on the value, 121.1^{0}C was selected as retorting temperature, for thermal processing of cuttlefish nidamental gland. The statistical analysis showed a significant difference, at 5 % level of significance, among the sensory characteristics.

Table 3: Sensory evaluation score for standardisation of retorting temperature (mean ± SD, n=10).

Temperature (^{0}C)/ Time (min)	Appearance*	Colour*	Odour*	Taste*	Texture*	Overall*
115 /60	6.17±0.15	5.67±0.14	6.33±0.06	6.17±0.10	6.33±0.14	6.33±0.16
121.1/30	**6.38±0.12**	**6.00±0.08**	**5.75±0.12**	**6.75±0.08**	**6.56±0.12**	**7.19±0.18**

* All traits measured on ten- point scale with 1 being least and 10 being the most.

4.4. STANDARDISATION OF PROCESS TIME

The sensory evaluation score for standardisation of process time is shown in the Table 4. Based on the results, 25 minutes was selected as process time at the retort temperature of 121.1^{0}C for processing of cuttlefish nidamental gland. The statistical analysis showed a significant difference, at 5 % level of significance, among the sensory characteristics except appearance.

Table 4: Sensory evaluation score for standardisation of process time (mean ± SD, n=10).

Time (min)	Appearance*	Colour*	Odour*	Taste*	Texture*	Overall*
20	6.14±0.18^{a}	5.86±0.18^{a}	6.71±0.19^{b}	6.86±0.21^{b}	7.14±0.17^{a}	6.43±0.19^{c}
25	**6.14±0.21^{a}**	**5.71±0.17^{b}**	**6.86±0.21^{a}**	**7.00±0.18^{a}**	**6.93±0.21^{b}**	**6.71±0.22^{a}**
30	6.21±0.19^{a}	5.57±0.20^{c}	6.57±0.22^{c}	6.86±0.19^{b}	6.71±0.18^{c}	6.57±0.20^{b}

* All traits measured on ten- point scale with 1 being least and 10 being the most.

^{a,b,c} Means in a column with same superscript letters are not significantly different ($P>0.05$).

4.5. DETERMINATION OF F_0 VALUE

The rate of heat penetration was determined for nidamental gland in brine packed in tin plate can of standard net weight of 200 g at a retort temperature of 121.1^{0}C and process time of 25 min is given in Figs. 3 & 4. The process parameters and F_0 value calculated by formula method (Stumbo, 1973) are presented in the Table 5.

The processing was carried out at a temperature of 121.1^{0}C for 25 min. The come- up time was 10 min, the heating lag factor (J_h) was calculated to be 0.54 min and the cooling lag factor (J_c) was found to be 1.03 min. The f_h value was reported 12.50 min. Although the process time was targeted for 25 min, slight deviation in the targeted value was observed. The cook value obtained was 81.54 min. The total process time was determined by adding process time and 58 % of the come- up time. The value was calculated to be 30.54 min. The F_0 value was calculated using formula method (Stumbo, 1973) is presented in the Table 5. The F_0 value was calculated to be 11.25.

Table 5: Heat penetration data for thermally processed nidamental gland in brine in tin plate can.

Parameters	Value
Fo value (min)	**11.25**
J_h	0.54
J_c	1.03
f_h (min)	12.50
U	11.26
f_h /U	1.11
g (^{0}C)	0.37
B (min)	24.74
Come- up time (min)	10.00
Total process time (min)	30.54
Cook value (min)	81.54

Where, f_h= slope of heating curve, J_h = lag factor of heating, J_c = lag factor of cooling, U = time in minutes for sterilization at retort temperature, g = final temperature deficit, B = Ball's process time.

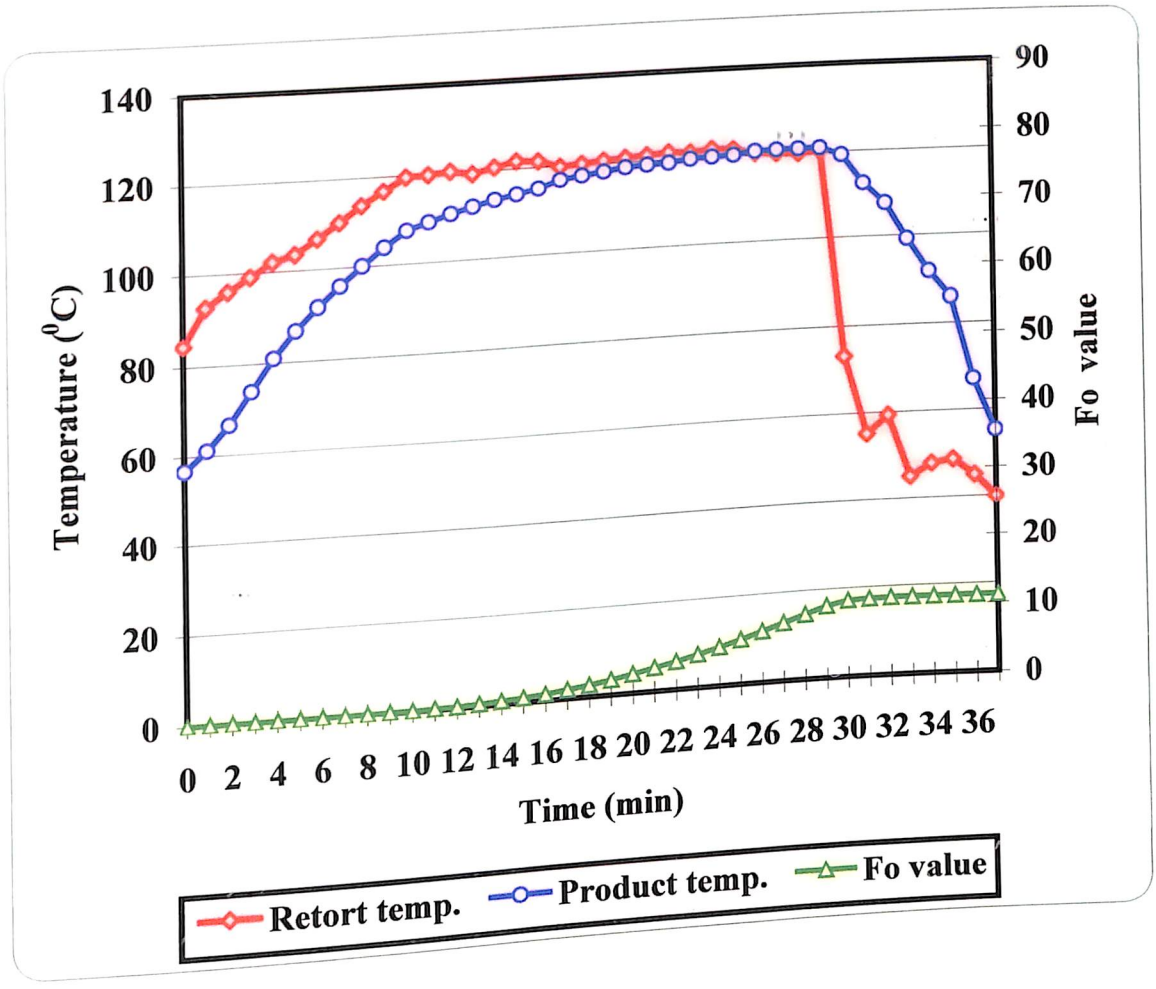

Fig. 3: Heat penetration characteristics of nidamental gland in brine medium with respect to F_0 value.

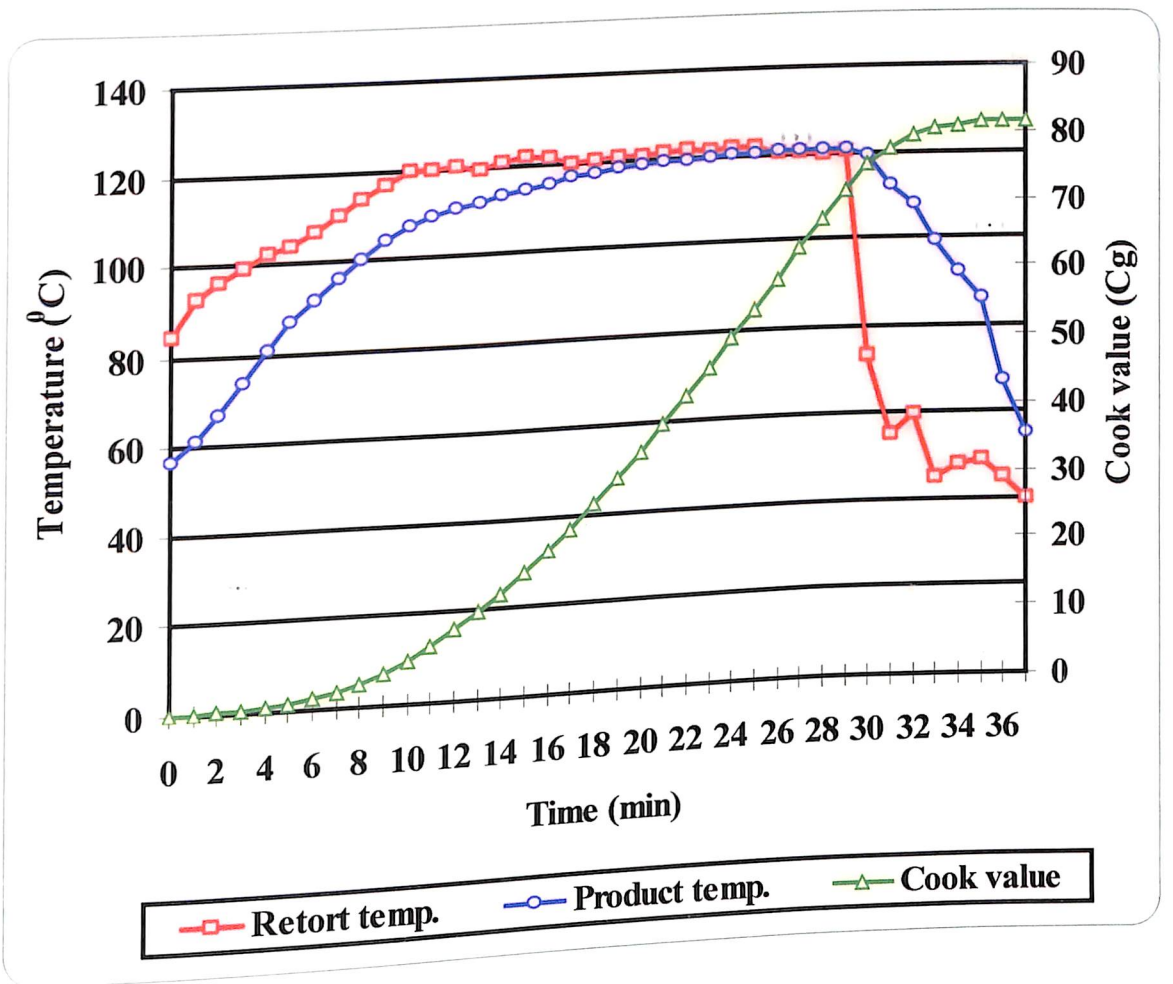

Fig. 4: Heat penetration characteristics of nidamental gland in brine medium with respect to cook value.

4.6. STORAGE STUDIES

The quality of the product packed in tin plate can was evaluated by conducting cut- out test. The organoleptic, chemical and biochemical characteristics were assessed soon after thermal processing and during storage at monthly intervals for three months.

4.6.1. Proximate composition

The variations in the moisture, protein, lipid and ash contents (on wet weight basis) of the processed nidamental gland during the storage period are given in Table 6.

During the storage period, the moisture content increased from an initial value of 76.90 % to 77.02 % on the 90th day. No significant difference was noted between the initial and final moisture levels.

The protein content showed a variation from 17.24 % to 16.97 % in 90 days, which is not statistically significant at 5 % level.

There was a slight variation in the lipid content from 2.38 % to 2.18 % which was also not statistically significant.

The ash content varied from 1.87 % to 1.91 % but was not statistically significant.

Table 6: Changes in proximate composition of the product during storage at room temperature (mean ± SD, n=3).

Storage period (days)	Moisture (%)	Protein (%)	Lipid (%)	Ash (%)
0	76.90 ± 2.38^{ab}	17.24 ± 0.56^{a}	2.38 ± 0.04^{a}	1.87 ± 0.06^{a}
30	77.50 ± 2.40^{a}	16.82 ± 0.49^{a}	2.16 ± 0.06^{a}	2.09 ± 0.05^{a}
60	76.28 ± 2.36^{b}	16.95 ± 0.33^{a}	2.28 ± 0.05^{a}	1.89 ± 0.07^{a}
90	77.02 ± 2.39^{ab}	16.97 ± 0.61^{a}	2.18 ± 0.07^{a}	1.91 ± 0.06^{a}

^{a,b} Means in a column with the same superscript letters are not significantly different ($P>0.05$).

4.6.2. Chemical parameters

The variations in the salt concentration, TBA value and pH of the processed cuttlefish nidamental gland during the storage period are given in Table 7.

The salt concentration values for the storage period are shown in Fig. 5. During the storage period the salt content increased from 1.62 % to 1.82 %. This change is found to be statistically significant.

The TBA values for the storage period are shown in Fig. 6. During the storage period the TBA value increased from 0.561 mg malonaldehyde/ kg to 1.092 mg malonaldehyde/ kg. This increase is found to be statistically significant.

The pH values for the storage period are shown in Fig. 7. During the storage period the pH decreased from 6.35 to 6.24. This change is found to be statistically significant.

Table 7: Changes in chemical parameters of processed nidamental gland during storage at room temperature (mean ± SD, n=3).

Storage period (days)	Salt (%)	TBA (mg malonaldehyde/kg)	pH
0	1.62 ± 0.07^{d}	0.561 ± 0.02^{d}	6.35 ± 0.14^{a}
30	2.06 ± 0.13^{a}	0.776 ± 0.05^{c}	6.32 ± 0.13^{ab}
60	1.73 ± 0.08^{c}	0.868 ± 0.03^{b}	6.16 ± 0.11^{c}
90	1.82 ± 0.11^{b}	1.092 ± 0.04^{a}	6.24 ± 0.07^{bc}

^{a,b,c,d} Means in a column with the same superscript letters are not significantly different ($P>0.05$).

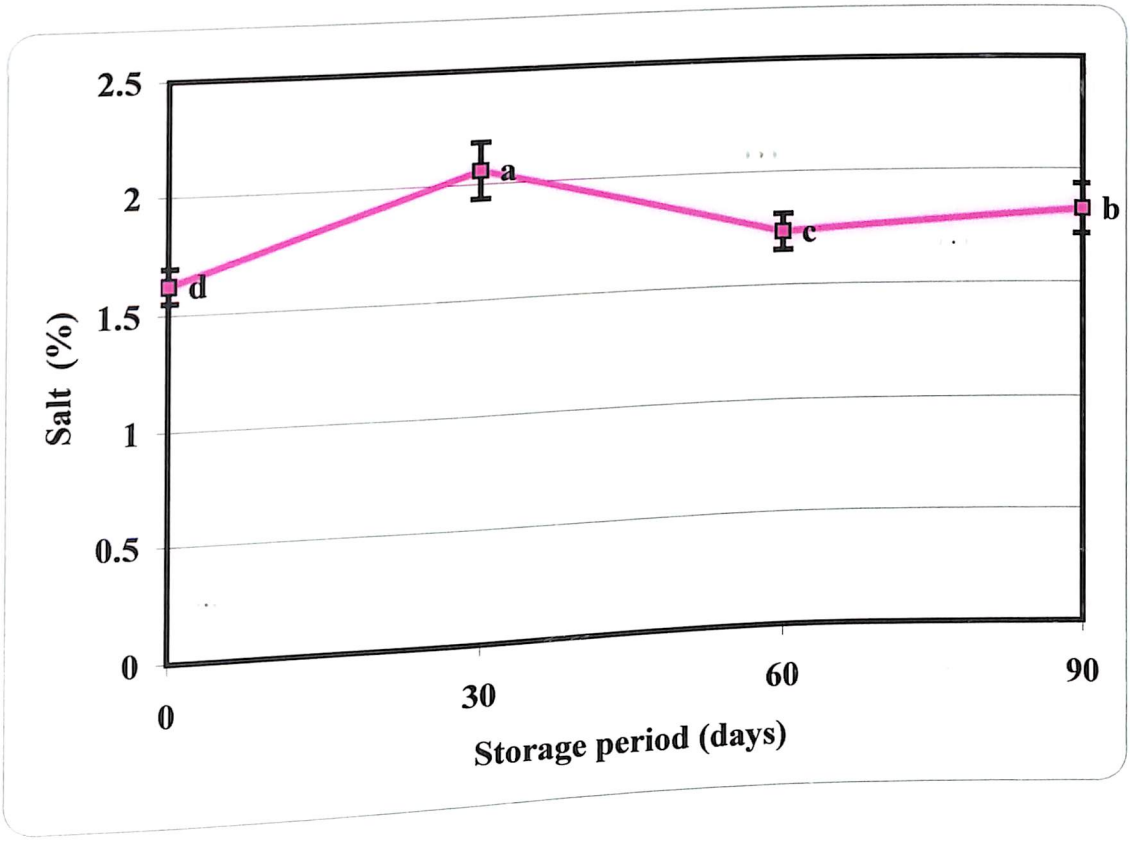

Fig. 5: Variations in the salt concentration of the product during storage at room temperature.

[a,b,c,d] Means in a line with the same letters are not significantly different (P>0.05).

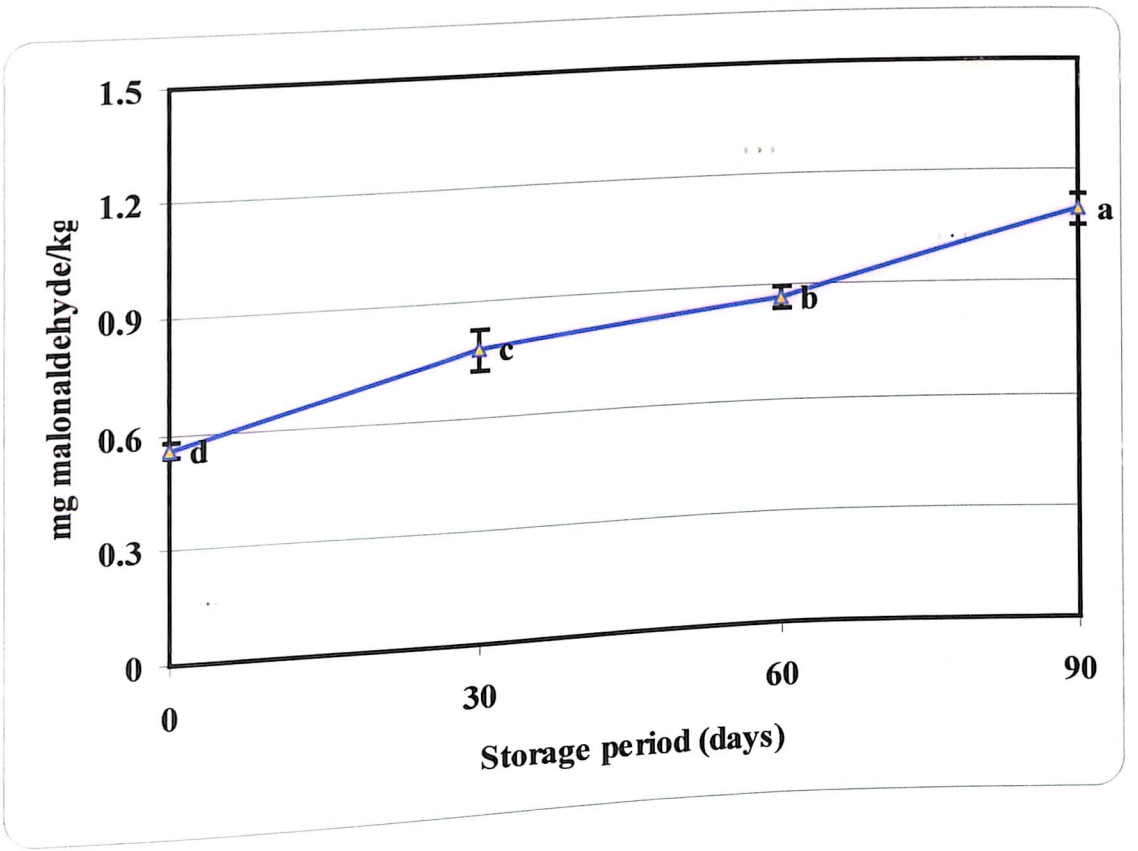

Fig. 6: Variations in the TBA values of the product during storage at room temperature.

[a,b,c,d] Means in a line with the same letters are not significantly different (P>0.05).

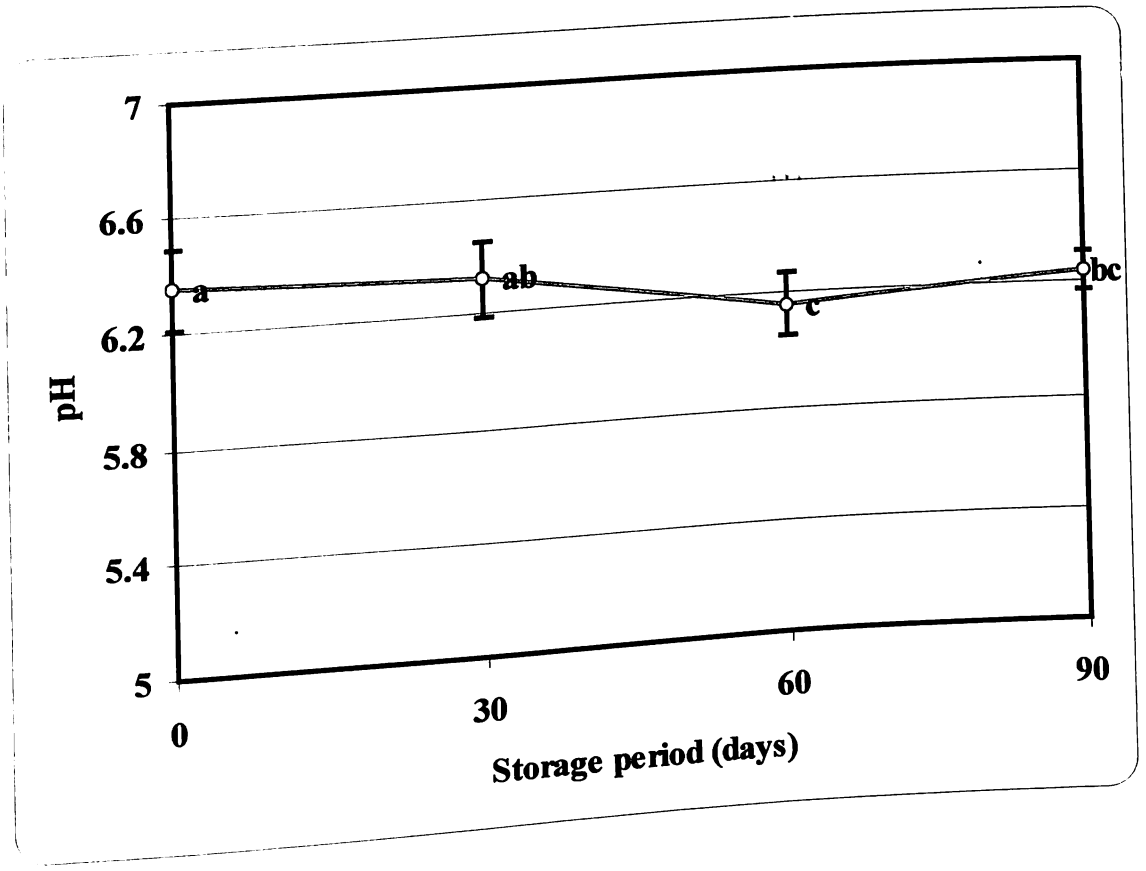

Fig. 7: Variations in the pH values of the product during storage at room temperature.

[a,b,c] Means in a line with the same letters are not significantly different (P>0.05).

4.6.3. Microbiological analysis

Sterility test was carried out for the product after processing and on the 90th day of storage period as mentioned in section (3.4.3.2). The results showed the product was sterile after the processing and throughout the storage period.

4.6.4. Sensory evaluation

Appearance, colour, odour, taste, texture as well as overall acceptability of the product were evaluated organoleptically. The means of the scores are given in Table 8 and Fig. 8. There were significant changes in the scores of the sensory parameters except overall acceptability. The overall acceptability of the product remained in 'good' condition (score>6) throughout the storage period.

Table 8: Sensory evaluation score of the product during storage at room temperature (mean ± SD, n=10).

Storage period (days)	Appearance*	Colour*	Odour*	Taste*	Texture*	Overall*
0	7.11±0.14^{a}	6.56± 0.26^{a}	7.33 ±0.22^{c}	6.78 ±0.14^{b}	6.61± 0.21^{a}	6.78 ±0.18^{a}
30	7.36± 0.08^{b}	6.71± 0.15^{c}	7.71± 0.15^{a}	7.14±0.23^{b}	7.00± 0.29^{b}	7.57 ±0.12^{a}
60	6.75 ±0.08^{a}	6.00± 0.13^{b}	7.10 ±0.28^{b}	7.40 ±0.24^{a}	6.95± 0.32^{a}	7.40 ±0.12^{a}
90	6.56± 0.33^{a}	5.94± 0.19^{b}	7.22±0.25^{b}	6.94±0.29^{a}	6.83± 0.29^{a}	6.83± 0.21^{a}

* All traits measured in ten- point scale 1 being least and 10 being the most.

^{a,b,c,d} Means in a column with same superscript letters are not significantly different (P>0.05).

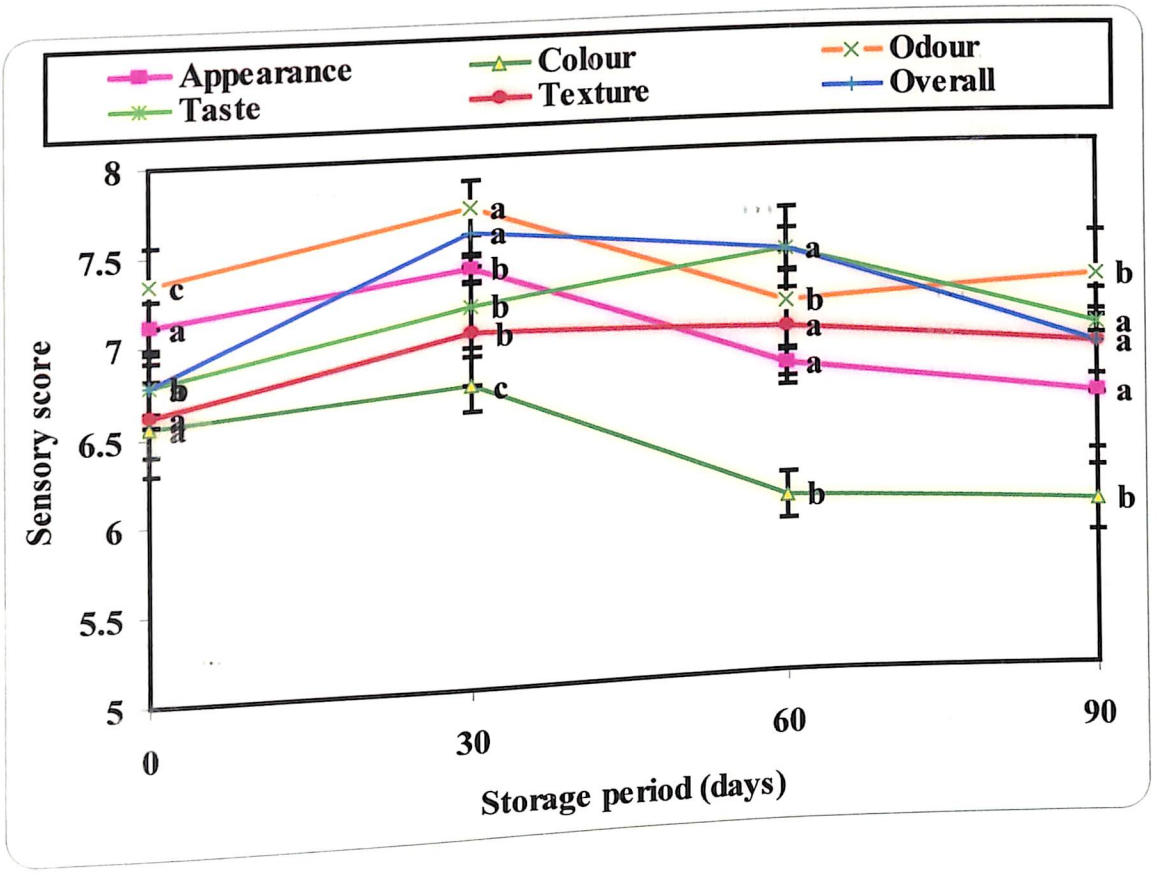

Fig. 8: Variation in the sensory parameters of the product during storage at room temperature.

[a,b,c] Means in a line with the same letters are not significantly different (P>0.05).

4.6.5. Cut- out characteristics

Thermal processed product quality is usually assessed by measuring various quantitative parameters which include physical measurements such as net weight, solid weight, solid- liquid ratio, condition of the pack etc. The cut- out characteristics of cuttlefish nidamental gland in brine is given in Table 9. There was little variation in net weight, i.e., 201 g to 205 g. Solid weight as percentage of net weight was found to be around 89 % and liquid content as percentage of net weight was around 11 %. The mean weight of empty can was around 44 g. The mean vacuum and mean gross head space were around 6.5 inch and 7.74 mm, respectively. The mean volume of liquid was around 23 ml and mean number of pieces was 6. Curds, foreign matter, lacquer peeling and blackening etc., were absent throughout the storage period. The liquid was slightly turbid and meat adhesion was slightly present.

Table 9: Cut- out characteristics of the product during storage at room temperature.

Sl. No.	Characteristics	0 day	30 days	60 days	90 days
1	Standard Net Weight (g)	200	200	200	200
2	Standard Solid Weight (g)	130	130	130	130
3	Vacuum (inch)	6	8	6	6
4	Gross Head Space (mm)	7.6	7.97	7.94	7.45
5	Gross Weight (g)	244	248	247	250
6	Solid + Can Weight (g)	220	221.5	226	232
7	Empty Can Weight (g)	40	47	45	45
8	Observed Net Weight (g)	204	201	202	205
9	Observed Solid Weight (g)	180	174.5	180	187
10	± Net Weight (g)	+ 4	+ 1	+ 2	+ 5
11	± Solid Weight (g)	+ 50	+ 44.5	+ 50	+ 57
12	Packed Weight (g)	135	135	135	135
13	Number of Pieces	6	6	6	6
14	Curds	Absent	Absent	Absent	Absent
15	Volume of Liquid (ml)	24	25	20	22
16	Turbidity of Liquid	Slightly Turbidity	Slightly Turbidity	Slightly Turbidity	Slightly Turbidity
17	Foreign Matter	Absent	Absent	Absent	Absent
18	Meat Adhesion	Slightly Present	Slightly Present	Slightly Present	Slightly Present
19	Lacquer Peeling	Absent	Absent	Absent	Absent
20	Blackening	Absent	Absent	Absent	Absent

DISCUSSION

5. DISCUSSION

The main objective of the present study is to standardise the procedure for thermal processing of cuttlefish nidamental gland in brine packed in tin plate can to produce a ready- to- eat product. The standardisation process essentially involved determination of process parameters, process time, optimum solid liquid ratio and process levels to produce a safe product and assessment of quality changes during storage at room temperature.

5.1. CUTTLEFISH NIDAMENTAL GLAND

Cuttlefish nidamental gland was used as the raw material in the present study. The raw material quality is one of the important determinants of the quality of the finished product. Rapid chilling of seafood and storing at 0^0C are important in distant water trawling operations (Jones and Disney, 1981). In the present study the cuttlefish was obtained from multi- day trawling operation, spreading up to 6 days. The catch was iced immediately onboard and stored in boxes.

5.1.1. Proximate composition

The moisture, protein, lipid and ash content of the gland were found to be 74.64 %, 19.8 %, 3.23 % and 1.59 % respectively which are more or less same as reported by Santhosh kumar *et al.* (1999). The change in proximate composition after blanching showed a decrease in moisture content to 65.21 %, with an increase in protein, fat and ash contents. This is due to the absorption of salt during blanching.

The microbiological load as observed from Total Plate Count in the raw material was within the limit of acceptability.

5.2. STANDARDISATION OF BLANCHING CONDITIONS

Blanching is a mild heat treatment used to inactivate the oxidative enzymes in shell fishes, and given prior to further processing, which otherwise will result in undesirable changes in colour, flavour and nutritive value of the product during handling and storage. It also removes the non- condensable tissue gases, increases the bulk temperature of the tissue, cleanses the tissue, and wilts the tissue to facilitate in packing. In addition to that, it has to be ensured that a salt content of about 2 % remains in the final product. In the present study the glands were blanched in boiling 5 % brine for 2 min, 5 min, 10 min and 15 min. After blanching samples were subjected to sensory evaluation. The result obtained was put to statistical analysis and 5 % brine for 5 min was selected as optimum blanching conditions. Again the salt content was found to be desirable for this combination. After blanching, the gland developed a firm texture. The same phenomenon has been reported by Varma and Joseph (1980), and Parshwanath (1989) while working on squids.

5.3. STANDARDISATION OF RETORTING TEMPERATURE

The cans were retorted at two process conditions, *viz.*, 115^{0}C for 1 hour and 121.1^{0}C for 30 min. After retorting the samples were subjected to sensory evaluation. The results obtained were put to statistical analysis and 121.1^{0}C for 30 min was selected as optimum retorting condition and gave a higher score than the other.

5.4. STANDARDISATION OF PROCESS TIME

At the retorting temperature of 121.1^{0}C, the glands were processed for 20 min, 25 min and 30 min. After processing the glands were subjected to sensory evaluation. The results obtained were put to statistical analysis and based on which a period of 25 min was selected as the process time for thermal processing. As it is a value added and ready- to- eat product, it is important to give maximum emphasis on sensory attributes. Ball and Olson (1957) suggested that 42 % of

come up time should be considered as process time at the retort temperature. Thermal process may slightly overcook the product and so industry can adopt different thermal process level based on the consumer preference.

5.5. DETERMINATION OF F_0 VALUE

Design, monitoring, control and recording of process time and temperature ensuring adequate commercial sterilization is imperative. Of the various critical control points in the production of heat processed foods, none is more important than thermal processing. It is the essential step in the establishment of safety and stability of the end product. Deviation of critical factors such as initial product temperature, processing time, contamination of the material may result in survival of bacterial spores. Codex (1989) has duly recognized its importance in formulating recommendations for low and medium acid canned foods.

The purpose of heat penetration test is to determine the heating and cooling behaviour of the product for the establishment of safe thermal process during commercial production. Therefore the design and accuracy of the test are critical. Using time- temperature data obtained from heat penetration runs, it is possible to:

1. develop thermal process for new food/container,
2. validate changes to product such as size of container, change of processing system that may affect the heating characteristics,
3. evaluate the effect of thermal processing on nutrients and sensory characteristics of food and
4. develop a mathematical model for process modification and control.

In the development of thermal process to achieve microbiological stability, the most thermally resistant microorganisms that represent health hazard on one hand and spoilage hazard on the other are selected as target of destruction.

Clostridium botulinum spores are the target organism for minimum thermal process requirement keeping in mind the safety of the consumer. For products which are expected to contain high heat resistant spores, *Bacillus stearothermophilus* may be the target organism and it requires a higher process time. To achieve product safety it is generally agreed that the thermal process should at least cause 12 logarithmic reductions in the spores of *Clostridium botulinum* (12- D concept). Other considerations are the nutrient value and organoleptic quality. Formulated foods containing many ingredients are likely to be the source of most heat resistant microorganisms.

The low acid canned foods are generally processed so that every particle of food is exposed to 121.1^{0}C for 2.5 to 3 min, which provides a considerable safety factor for expected levels of contamination (Bryan, 1974). The recommended F_0 value for meat products is minimum 6 min (Shapton and Shapton, 1997). Frott and Lewis (1994) recommended an F_0 in the range of 5 to 20 min for fish and fishery products. An F_0 of 8.34 min was recommended for mackerel (Gopal *et al.*, 1998), 8.79 min for rohu curry (Mallick, 2003) and 11.5 min for seer fish moilee (Manju *et al.*, 2004).

The heating behaviour of foods in addition to time and temperature sequence for the thermal process is affected by various factors, like the temperature difference between heating/cooling medium and the product, the factors that affect heat penetration such as the nature of the product, type of container and heating/cooling medium. The heat penetration tests conducted to generate considerable information should be kept constant for a given set of processing condition. The size, shape and weight of the solids, the consistency and viscosity of the product should also be kept constant. Consequently special care should be taken to identify and control specific variables when subjected to heating.

In the present study an F_0 value of 11.25 was calculated for the product which is well in between the recommended range. The relationship between the

heating/cooling medium temperature and the product temperature during the thermal process in an overpressure retort is given in Figs. 3 and 4. From this it is evident that lethality and cook rate increased with temperature. These factors are helpful in calculating the destruction levels of target organisms and retention of nutrients. The f_h value was found to be 0.54 min for the product. It is a constant for a given product and container size and changes only when any change occurs in characteristic container contents, heating agents, etc. It shows the rate of change of product temperature to reach the retort temperature in a logarithmic pattern. Corrections are applied to compensate the time required to bring the retort to the required temperature. As the temperature of heating medium increases, the rate of heat penetration varies.

5.6. STORAGE STUDIES

Canned product undergoes certain changes in the chemical, organoleptic and physical quality during storage. With a view to study the probable changes it undergoes, the product developed from cuttlefish nidamental gland was cut- open at regular intervals and evaluated for quality. The results obtained are discussed below.

5.6.1. Proximate composition

The moisture content showed marginal variation during the storage whereas protein, lipid and ash contents did not show significant variation during storage.

5.6.2. Chemical parameters

There was significant change in salt content during storage period. In the first month the increase was high, it might be due to the seasoning of the product. There was an increase in TBA value during storage period. It could be due to the oxidation of lipid in the gland. The pH of the canned product was 6.35, which indicates the nidamental gland as a low acid food. During storage period the pH

went on decreasing. This could be due to loss of moisture content and/or formation of free fatty acid by liposysis.

5.6.3. Microbiological analysis

The product showed no growth of microorganisms after processing and during the end of storage study, which indicates the process given was sufficient to attain sterility.

5.6.4. Sensory evaluation

The sensory evaluation for the cuttlefish nidamental gland was carried out after processing and during the storage period using a 10- point hedonic scale. After the thermal process, the sensory scores were marginally different except overall acceptability. Overall acceptability of the product was good even after 90 days of storage which shows the shelf life stability of the thermally processed product.

5.6.5. Cut- out characteristics

There was little variation in net weight and solid weight as percentage of net weight was found to be around 89 %. The mean volume of liquid was around 23 ml. The cut- out characteristics showed a marginal increase in solid weight in the product. This could be due to absorption of brine in to the solid matter during storage duration.

SUMMARY

6. SUMMARY

1. The objective of the study was to standardise the procedure for thermal processing of cuttlefish nidamental gland, packed in tin plate can, and to produce a ready- to- eat product. Brine was used as a packing medium and thermal process requirement of the product was evaluated by conducting heat penetration study.

2. The chemical composition of cuttlefish nidamental gland indicated that the moisture, protein, lipid and ash contents were 74.64 %, 19.8 %, 3.23 % and 1.59 %, respectively. The protein, lipid and ash contents after blanching were increased to 25.1 %, 5.38 % and 2.62 %, respectively, whereas moisture content decreased to 65.21 %.

3. Blanching in 5 % brine for 5 min was selected as optimum blanching condition based on value of sensory attributes and result of statistical analysis.

4. A temperature of 121.1^{0}C was selected as optimum retorting temperature based on sensory evaluation score and result of statistical analysis.

5. A period of 25 min was selected as optimum process time, at the retorting temperature of 121.1^{0}C, based on sensory evaluation score and result of statistical analysis.

6. The filled can was sealed and processed in overpressure retort at a temperature of 121.1^{0}C for 25 min. The time- temperature profile obtained during sterilization was used to calculate the heating characteristics of the product. It was found that the retort come- up time (CUT) was 10 min. The process time (including 42 % of come- up time) was deviated slightly from the target value to 24.74 min and total process time was found to be 30.54 min. The F_0 value was determined to be 11.25 min.

7. A storage study was carried out on the keeping quality of the product, for a period of 90 days. The quality changes were monitored based on the

following tests conducted periodically: proximate composition, salt, TBA, pH, sensory evaluation, cut- out and sterility.

8. There were no significant variations ($P>0.05$) in the protein, lipid and ash contents during storage. The moisture content was slightly increased.

9. There were significant variation ($P>0.05$) in the salt, TBA and pH values.

10. Though there were significant changes in the sensory quality parameters, the product was in acceptable condition throughout the storage period.

11. Cut- out test showed a marginal increase in solid weight and sterility test showed that the product was commercially sterile during the period of study.

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7. REFERENCES

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STANDARDISATION OF THERMAL PROCESSING OF CUTTLEFISH NIDAMENTAL GLAND

By

BIKASH KUMAR PATI

ABSTRACT OF THESIS

Submitted in partial fulfillment of the requirement for the degree of

MASTER OF FISHERIES SCIENCE

Faculty of Fisheries

Kerala Agricultural University

2007

DEPARTMENT OF PROCESSING TECHNOLOGY

COLLEGE OF FISHERIES
PANANGAD, COCHIN

ABSTRACT

Cuttlefish nidamental gland is a pair of flattened glands associated with the female reproductive system in cuttlefish. The glands are exported as a frozen product under the name cuttlefish roe. The procedure for thermal processing of cuttlefish nidamental gland in brine packed in tin plate can is standardised to produce a ready- to- eat product. The standardisation process essentially involved determination of optimum blanching conditions, retorting temperature, process time, processing parameters, optimum solid- liquid ratio and process levels to produce a safe product and assessment of quality changes during storage at room temperature.

Cuttlefish nidamental gland in brine was packed in tin plate cans in 65 : 35 ratio (gland : brine) and thermal processed in an overpressure retort. The optimum blanching condition was found to be 5 % brine for 5 min. The retorting temperature and process time were selected as 121.1^{0}C and 25 min, respectively. The F_0 value attained by the process was found to be 11.25 min with total process time of 30.54 min. The cook value meant to achieve tenderness of product was 81.54 min. The processed product was found to be commercially sterile. The product was acceptable even after 3 months of storage at room temperature with regard to all sensory attributes like appearance, colour, odour, taste, texture and overall acceptability. There were significant variation in the salt, pH and TBA values ($P<0.05$). The cut- out test showed a marginal increase in solid weight. The study showed that the cuttlefish nidamental gland can be used for processing ready to eat product, which can be stored at room temperature for long periods.

ANNEXURE - I

SENSORY EVALUATION OF BLANCHED 'CUTTLEFISH NIDAMENTAL GLAND

Assessor: _____ Date: _____

(Please score the sample characteristics by placing the relevant score)
An honest expression of your personal feeling will help us.

CHARACTERISTICS	A	B	C	D
Texture				
Saltiness				
Overall flavour				

(Please score the sample characteristics according to the following scale)

Quality grade description for texture, saltiness and overall flavour:

Quality grade description	**Score**
Excellent	9-10
Very good	7-8
Good	5-6
Fair	3-4
Borderline of acceptability	1-2
Poor/Unacceptable	0
Remarks if any	

Signature

ANNEXURE - II

SENSORY EVALUATION OF CANNED 'CUTTLEFISH NIDAMENTAL GLAND'

Assessor: _____ Date: _____

(Please score the sample characteristics by placing the relevant score)
An honest expression of your personal feeling will help us.

CHARACTERISTICS	A	B
Appearance		
Colour		
Odour		
Flavour		
Taste		
Texture		
Overall acceptability		

(Please score the sample characteristics according to the following scale)

Quality grade description for appearance, colour, flavour, taste and overall acceptability:

Quality grade description	Score
Excellent	9-10
Very good	7-8
Good	5-6
Fair	3-4
Borderline of acceptability	1-2
Poor/Unacceptable	0
Remarks if any	

Signature

ANNEXTURE - III

CUT- OUT TEST FOR CANNED FISHERY PRODUCTS

Observations: Record your observations in the following proforma

Can Size / Particulars	1	2	3	4	5
Product					
Code					
Manufacturer					
Date of production					
Date of testing					
Can- size and type					
Std. solid- wt./Net weight.					
Vacuum					
Gross weight					
Solid + can weight					
Empty can weight					
Solid weight					
Liquid weight					
Net weight					
Pack weight					
Colour					
Texture					
Flavour					
Style(Appearance)					
No. of pieces					
Salt/ Sugar degree					
Total sugar degree					
Turbidity					
Acidity					
pH					
Size of pieces					
Broken or flakes					
Adhesion					
Curds					
Remarks					

Note: Study I.S.I. Specifications for canned fishery products.

(Source: Laboratory manual for courses in canning- by K.V. Saralaya