Process optimisation for ready to eat Indian mackerel (*Rastrelliger kanagurta*) curry in high impact polypropylene (HIPP) containers using still water spray retort

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ABSTRACT
Mackerel curry is a traditional food which is considered as a delicacy in Kerala. Indian mackerel curry was prepared as per standard recipe and hot filled into high impact polypropylene (HIPP) containers. Dynopack sealing machine was used for top sealing using seethrough films comprising polyester, silicone dioxide coated nylon and cast polypropylene and processed in a still water spray retort at 121.1°C to different $F_0$ values of 6, 7 and 8 min. Based on the sensory and commercial sterility test, $F_0$ value of 8 min was found to be optimum for mackerel in curry medium. The fish curry processed to $F_0$ value of 8 was taken up for storage studies at ambient temperature (28±2°C) and changes in biochemical parameters like thiobarbituric acid (TBA), total volatile base nitrogen (TVBN), trimethylamine (TMA), instrumental colour and texture were analysed. Upon thermal processing, significant increase in chewiness as well as springiness and decrease in hardness was observed for mackerel at optimum $F_0$ value. The product was found to be commercially sterile throughout the period of storage and was found to have a shelf life of 75 days.

Keywords: $F_0$ value, HIPP containers, Indian mackerel, Instrumental colour, Texture profile analysis, Thermal processing, Water immersion retort

Introduction
Convenience food is a concept that is prevalent in the developed world since long, while its inception into the Indian market has been recent. With the changing socio-economic pattern of life and the increasing number of working couples, the concept is becoming popular in Indian market as it saves time and labour. This has resulted in increased demand for ready to eat food in recent years. Consumers are becoming increasingly aware of the nutritional content, toxic factor, microbiological quality and preservative free products while purchasing food. Preservation of food by thermal processing especially in retortable pouches will help in providing preservative free products with extended storage life as well as improved nutritional benefits compared to metal containers. Retortable pouches having three layer configuration of polyester/aluminium foil/cast polypropylene can perform the packaging function as that of metal cans and is free from disadvantages like poor barrier properties, pin holing and poor seal strength (Srinivasa Gopal et al., 1998; Vijayan et al., 1998). Fish curries packed in retortable pouches and processed in steam air mixture using over pressure autoclave were found to be in acceptable condition for more than one year at ambient storage (Srinivas Gopal et al., 2001; Sonaji et al., 2002; Manju et al., 2004; Ravishankar et al., 2002, 2008; Mohan et al., 2006, 2008, 2014, 2015). Compared to steam-air retorts, water immersion and still water spray retorts are found to be advantageous as the air pockets will not be formed inside the retort in latter types (Hardt-English, 2003). Faster heating rate and better nutrient retention is another advantage of using water spray and water immersion retorts (Mary et al., 2016). Of late, semi-rigid containers are gaining importance for thermal processing. Semi-rigid containers have the advantages that they can be formed in any shape *i.e.*, round, square, rectangle or hexagonal with varied depth for different volumes. Apart from this, they can also be used as eating bowl due to their rigidity. These containers are either made of multi-layer with many layers of polypropylene and ethylene vinyl alcohol or with high impact polypropylene (HIPP). Semi-rigid containers made of HIPP are found to be suitable for food contact applications and have good barrier properties (Mohan, 2008). These containers can also be made locally and are cheaper compared to many commercially available containers.

Fish curry is a traditional food which is considered a delicacy. Mackerel or sardine are most commonly
available fish species and is consumed in different forms all along the coastal belt of Kerala. They have high moisture content and have a limited storage life at ambient temperature. In order to ensure their distribution and long term storage, it is necessary to preserve them suitably. This will take care of food as well as nutritional security. No work has been carried out in India so far, on the use of semi rigid containers for thermal processing of fish curry using water immersion retort. Hence in this study, attempts have been made to standardise process parameters for the development of ready to eat mackerel curry in thermoformed containers which can be stored at ambient temperatures by subjecting the products to thermal processing using water immersion retort.

Materials and methods

Physical properties of the packaging materials

The high impact polypropylene trays were purchased from Kalyx Plasti Pack, Kannur, Kerala. Analyses of physicochemical properties of HIPP trays indicated its suitability for thermal processing. The thickness of the HIPP trays and the top sealing layer was measured as per ASTM (1964). Tensile strength and elongation at break were determined using Universal Testing Machine (Lloyd Instruments LRX plus, UK) and expressed as kg cm$^{-2}$ and percentage (%), respectively. Heat seal strength of the film used for sealing HIPP trays was determined as per ASTM-1434 (1973) and bond strength as per ASTM (1972). Water vapour transmission rate was determined according to ASTM-E-96-80 (1987) and gas transmission rate as per ASTM-D-1434 (1975). Suitability of the containers for food contact application was determined following IS: 9845 (1981).

Thermal processing

Indian mackerel (Rastrelliger kanagurta) (119±4.5 kg weight and 15.2±2.8 cm length) purchased from the local market was gutted, beheaded, washed thoroughly in potable water and then blanched in 10% brine for 30 min at 27°C and drained. Mackerel curry out of 1000 g of dressed fish was prepared using the following ingredients: tomato 500 g, onion 60 g, ginger 13 g, green chilly 25 g, turmeric powder 2.5 g, coriander powder 20 g, chilly powder 60 g, fenugreek 0.8 g, Malabar tamarind 50 g, oil 50 g, salt 16 g and water 500 ml. Onion and tomato were chopped and ground separately in a mixer. The onion slurry was heated in oil till the colour became light brown. At this stage, ground tomato was added and heating continued for 10 min. Chilly powder, coriander powder and turmeric powder were added to green chilly and ginger which were fried separately in oil and heated under a low flame. The fried ingredients were added to the boiling onion-tomato slurry and heating continued. Potable water was added to adjust the consistency of the curry. Ninety gram of fish and 60 g of curry was packed together in HIPP trays of size 12.5 x 9.2 x 2.5 cm (length x breadth x height) having 220 ml capacity, in order to maintain a 60:40 solid liquid ratio. The curry was filled under hot conditions to facilitate the removal of air in the head space. The trays were sealed on top with seethrough film made of three layers viz., outer polyester (PEST), middle nylon coated with silicon dioxide and inner layer of cast polypropylene (CPP) using heat sealing machine (Dynopack Industries, Hyderabad, India). Initially the cold point in the water immersion retort was standardised by placing thermocouples of Ellab SSA-12050-G700-TS stainless steel electrode (50 mm length; 1.2 mm dia) in different positions of the retort and heat penetration studies were carried out by monitoring the temperature of the product placed in the point. Minimum three containers were fixed with glands and thermocouples, the tips of which were inserted into a piece of fish. The filled and sealed trays were laid flat on the trays in a water immersion retort (Lakshmi Engineering Works, Chennai, Tamil Nadu, India). Processing media was water sprayed at a high pressure (28 psi) inside in a mist like formation, which was collected at the bottom and then directed to a circulation line. Steam is admitted into the circulation line and also directly inside the retort. The sprayed water absorbs the heat from incoming steam and transmits the same to the products. The thermocouple output was measured using an Ellab E-Val Flex, 14592 data recorder. The products were processed to different $F_0$ values of 6, 7 and 8 min at the retort temperature maintained at 121.1ºC.

Rapid cooling was done by cutting off the steam supply and recirculation of cooling water. The trays were unloaded from the retort when the core temperature reached around 30°C. The recorded data was analysed and heat penetration data were plotted on a log paper with temperature deficit (retort temperature-core temperature) against time. Lag factor for heating ($J_h$), slope of the heating curve ($f_{j_h}$), initial temperature deficit (I), final temperature deficit (g), time in minutes for sterilisation at retort temperature (U) and lag factor for cooling ($J_c$) were determined. The Ball’s process time ($B$) was calculated by Formula method (Ball, 1923). Total process time ($T$) was determined by adding Ball’s process time ($B$) and the effective heating period during come up time $i.e., 58\%$ of the come up time (Stumbo, 1973).

Ball’s process time ($B$) = $f_{j_h} \log (1xJ_h) - \log g$

Total process time ($T$) = $B + 58\%$ of come up time

To standardise the optimum process conditions for the thermal processing of mackerel fish curry, processing was done at three different $F_0$ values and tests for
commercial sterility, colour analysis, texture profile analysis and sensory evaluation were carried out.

Based on the results, an F₀ value was chosen according to which mackerel fish curry was prepared on a pilot scale for the evaluation of its shelf life at ambient temperature. Samples were taken at regular intervals of 15 days and were analysed for instrumental colour, texture profile, thiobarbituric acid (TBA), total volatile nitrogen (TVBN), trimethylamine (TMA), peroxide value (PV), free fatty acid (FFA) and sensory characteristics.

**Commercial sterility**

Commercial sterility of the processed samples were tested as per IS: 2168 (1971) to assess the adequacy of lethality.

**Analysis of colour**

Colour measurements were done using a Hunter Lab colourimeter (Miniscan XE Plus, Model No D/8-S) with geometry of diffuse/8° (sphere 8 mm view) and an illuminant of D65/10 deg. Fish meat was blended and the colour was measured to get the homogeneous colour of meat.

**Texture profile analysis**

Texture profile analysis were carried out using the food texture analyser (Lloyd Instruments, Model LRX plus F.T-39 No-2, UK) with the help of Nexygen software. Fish pieces with a sample size of 2 x 2 cm (length x breadth) and thickness of 1 cm was cut from the dorsal portion of fish and used for texture analysis. Cylindrical probe with 50 mm dia was used for texture analysis at a speed of 12 mm sec⁻¹ and trigger force of 1N using 500 N load cell. Compression of 40% was used.

**Biochemical analysis**

Proximate composition of fresh Indian mackerel was analysed following AOAC (2000). Storage studies were carried out at ambient temperature (28±2°C). The product was analysed at regular intervals of every 15 days for changes in their biochemical characteristics like pH, TBA, TVBN, TMA, PV and FFA. pH was determined using a glass electrode digital pH meter after homogenising the sample using distilled water (1:5w/v). TBA value was determined spectrophotometrically from an acidified distillate of the sample to assess the oxidation stability of the product (Tarladgis et al., 1960). TVBN and TMA were estimated by preparing a 10% tri-chloro acetic acid (TCA) extract of the sample using microdiffusion method (Conway, 1950). PV was estimated by preparing a chloroform extract of the curry sample followed by its iodometric titration (Yildiz et al., 2003). The chloroform extract was used for determination of FFA value as per AOAC (1989).

**Sensory evaluation**

Sensory evaluation based on characterisation and differentiation of the various sensory characters such as colour, flavour, texture and overall acceptability were evaluated by a panel of 10 trained judges on a 10-point scale (IS: 6273(II) 1971). The panelists were asked to assign a score of 1-10 as prescribed by Vijayan and Balachandran (1986). A sensory score of 4.0 was taken as the margin of acceptance.

**Statistical analysis**

A minimum of three containers were monitored for constant temperature data. Triplicate samples were used for all the biochemical and physical analyses. A minimum of ten samples were used for testing packaging materials. Analysis of variance (ANOVA) was used to define the significance of differences (p<0.05).

**Results and discussion**

**Physical properties of the packaging materials**

Physico-chemical properties of the packaging materials used for packing food provides useful information on their suitability for different processing conditions. The HIPP trays used for processing had a thickness of 996±1.2 µm. Tensile strength was 332±1.6 and 306±1.1 kg cm⁻² in the machine direction and cross direction respectively. The elongation at break for machine direction and cross direction was 92±3.54% and 98±2.25% respectively. Water vapour transmission rate was 0.76±0.05 g m⁻² 24 h⁻¹ at 37°C and 92% relative humidity (RH). Oxygen transmission rate was 9.5±0.14 cc m⁻² 24 h⁻¹ at 1 atm pressure. Overall migration residue test was carried out to assess the suitability for food contact applications and the results obtained were 5.4±0.3 mg l⁻¹ water extractives, 3.6±0.16 mg l⁻¹ n-heptane extractives and 2.14±0.21 mg l⁻¹ 3% acetic acid extractives. The results indicate that the overall migration residue for HIPP tray was <12 mg l⁻¹ which is well below the acceptable limit of 60 mg l⁻¹, indicating its suitability for food contact applications. The seethrough film comprising PEST, silicon dioxide coated nylon- and CPP, which was used as top sealing material had a thickness of 100±0.01 µm. Tensile strength was 782±0.01 and 615±0.01 kg cm⁻² in the machine direction and cross direction respectively. Heat seal strength for machine direction and cross direction was 562±1.1 and 439±1.3 kg cm⁻² respectively. Elongation at break for machine direction and cross direction was 82±0.02 and 96±0.03% respectively and the bond strength was 132±0.3 g per 10 mm. The water vapour transmission rate was 0.95±0.01 g m⁻² 24 h⁻¹ at 37°C and 92% RH and
oxygen transmission rate was 2.14±0.01 cc m⁻² 24 h⁻¹ at 1 atm pressure. Overall migration residue test was carried out to know the suitability for food contact applications and the results obtained were 4.5±0.16 mg l⁻¹ water extractives, 1.3±0.11 mg l⁻¹ n-heptane extractives and 1.88±0.23 mg l⁻¹ 3% acetic acid extractives. The film used for top sealing of the trays had an outer polyester layer, middle silicon dioxide coated nylon and an inner layer of cast polypropylene. Silicon dioxide is a nanoparticle, specially incorporated to provide good barrier properties. Analyses of the physical properties indicated that the packaging material can withstand thermal processing temperature of 121.1°C. The results also revealed that it can withstand a working pressure of 28 psig. The HIPP trays did not change shape during retorting and were found to be ideal for thermal processing.

**Heat penetration characteristics**

The water immersion retort showed uniform heating in all parts of the processing chamber with a variation of only ±1°C. The \( F_0 \) recommended for fish and fish products ranges from 5-20 min (Frott and Lewis, 1994). Mackerel curry in trays was heat processed for \( F_0 \) 6, 7 and 8. Curry processed at \( F_0 \) 8 using water spray in the water immersion retort was found to be sterile. Heat penetration characteristics of mackerel in curry medium processed at \( F_0 \) 8.1 are shown in Table 1. The come up time to attain 121.1°C was 9 min for mackerel in curry medium and it has been reported that the come up time should be kept as short as possible (NCA, 1968). The actual process time for mackerel curry to \( F_0 \) 8.1 was 40.55 min.

![Heat penetration and F₀ value of mackerel in curry medium](image1)

**Proximate composition of Indian mackerel**

Proximate composition of mackerel used for processing showed a moisture content of 77.63±0.01, ash content of 1.55±0, fat content of 1.59±0 and a protein content of 20.55±0.21%.

**Colour analysis**

Table 2 summarises the colour values for mackerel fish curry during the period of storage. The increase in \( L^* \) value of processed mackerel compared to that of raw mackerel is due to the leaching of muscle pigments during pre-cooking and thermal processing and leaching of white connective tissue containing collagen during heat processing (Mohan *et al.*, 2015). The \( a^* \) and \( b^* \) values showed increasing trend because of the absorption of spices like chilly powder and turmeric powder respectively and maillard reaction between sugar and amino acids.

**Texture profile analysis**

Table 3 shows the different parameters of texture profile analysis of mackerel curry processed to \( F_0 \) value
Table 2. Colour analysis of mackerel fish curry

| Days of storage | L’  | a’  | b’  |
|-----------------|-----|-----|-----|
| 0               | 38.87±0.09 | 5.01±0.05 | 10.77±0.08 |
| 15              | 57.12±0.08 | 6.66±0.08 | 24.25±0.08 |
| 30              | 56.90±0.06 | 6.79±0.04 | 25.58±0.02 |
| 45              | 54.62±0.09 | 6.97±0.05 | 26.39±0.22 |
| 60              | 52.96±0.13 | 7.65±0.1  | 27.42±0.09 |
| 75              | 52.39±0.07 | 7.93±0.04 | 27.87±0.05 |
| 90              | 51.28±0.04 | 7.98±0.01 | 27.89±0.01 |

Table 3. Texture profile analysis of mackerel fish curry

| Day   | Hardness 1 (N)  | Hardness 2 (N)  | Cohesiveness | Springiness (mm) | Gumminess (kg f) | Chewiness (kg f mm) |
|-------|-----------------|-----------------|--------------|-----------------|-----------------|---------------------|
| 0     | 41.02±0.07      | 38.28±0.3       | 0.09±0       | 0.67±0.02       | 0.25±0.03       | 3.76±0.15           |
| 30    | 30.69±0.53      | 21.72±0.61      | 0.11±0.01    | 2.54±0.02       | 0.35±0.03       | 3.70±0.04           |
| 60    | 25.60±0.16      | 19.57±0.21      | 0.26±0.02    | 4.71±0.12       | 0.71±0.01       | 3.20±0.06           |
| 90    | 22.73±0.10      | 17.41±0.13      | 0.24±0.02    | 4.92±0.04       | 0.84±0.03       | 3.27±0.07           |

of 8.21. Thermal processing led to significant decrease (p<0.05) in the hardness of the fish pieces in the curry which clearly indicated that cooking leads to softness. The main external factors affecting the texture of fish are temperature of cooking and presence of sodium chloride (Mohan et al., 2014, 2015). Hardness 2 values are always less than those values obtained at first compression. This is because non-compressed samples have a firm texture compared to compressed sample. Texture of fish meat is influenced by the collagen content of the meat as well. When core temperature reaches 92°C, collagen content of meat is denatured and converted to gelatin. The lowering of hardness values on thermal processing is due to the effect of temperature on collagen and resultant softening of the muscle. Cohesiveness refers to the visco-elasticity of the product. On the initial day, the cohesiveness was 0.09 and it increased to 0.24 on day 90 of storage. The faster heat penetration in HIPP trays led to the increase in cohesiveness (Ansar Ali et al., 2005). Springiness refers to elasticity of the sample, which was 0.67 mm on the initial day of storage and increased to 4.92 mm on day 90 of storage. The product showed elastic behaviour throughout the storage period. Gumminess also increased from 0.25 to 0.84 kgf. This is in agreement with the findings of Mohan et al. (2014, 2015) for thermal processed tuna in cans.

Biochemical analysis

The changes that occurred in different biochemical parameters of the mackerel fish curry during the period of storage is given in Table 4.

Total volatile base nitrogen (TVBN) quantified a range of basic volatile compounds including ammonia, methyl amine, dimethyl amine and trimethylamine. The increase in TVBN content is due to thermal breakdown of trimethylamine N-oxide (TMA-O) during cooking and sterilisation steps. It reached a maximum limit of 31.5 mg after 3 months storage at room temperature. TVBN limit for acceptability is 30 mg. Sensory evaluation of samples stored for 90 days revealed a sensory score below 5.

Trimethylamine (TMA), a volatile component represents the majority of TVBN. The average TMA value of raw material was zero which indicates the sample used in the study was in fresh condition. A significant increase in TMA content was observed during the storage. TMA is produced from TMAO by thermal breakdown during the cooking and sterilisation process. Thermal processing exerted a higher effect on the TMA formation during storage.

TBA value, an index of secondary lipid oxidation showed an increasing trend during storage. The peroxides formed during storage may have decomposed to form compounds including aldehydes such as malonaldehyde which are responsible for increase in TBA value. TBA limit for acceptability is 2 mg. It reached more than 2 mg after 3 months storage.

FFA values of fish muscle gives an account of the degree of lipid hydrolysis that occurred during storage. FFA values of samples during storage showed a slight increase on day 15 of storage and decrease during further storage. Same trend was observed in case of peroxide value. There was not much lipid hydrolysis during storage.

Sensory evaluation

Sensory evaluation of the samples during storage revealed that the product were in acceptable condition up to 75 days at ambient temperature. The overall acceptability score which was obtained by adding all the attributes viz., appearance, colour, odour, flavour, taste and texture is given in Table 5. A final score of 3.8 was obtained on day 90 of storage.

The overall migration from the packaging material is within the limit indicating their suitability for food contact application. For mackerel in curry medium, processing up to F0 value of 8 min was appropriate. Process time in the water spray retort was 40.55 min for fish curry. The product was found to be commercially sterile during the entire period of storage. In accordance
Table 4. Biochemical parameters

| Days of storage | TBA (mg % malonaldehyde) | TVBN (mg %) | TMA (mg %) | PV (mEqO₂ kg sample⁻¹) | FFA (% oleic acid) |
|-----------------|--------------------------|-------------|------------|------------------------|-------------------|
| 0               | 0.29±0                   | 14±0        | 0±0        | 11.36±0                | 8.75±1.13         |
| 15              | 0.30±0                   | 16.1±0.99   | 1.4±0      | 13.75±0.88             | 9.63±1.24         |
| 30              | 0.48±0                   | 18.2±1.04   | 1.4±0      | 14.47±0.88             | 6.96±0.12         |
| 45              | 0.96±0                   | 21.7±0.99   | 5.6±0      | 10.78±0                | 5.56±0.10         |
| 60              | 1.16±0                   | 23.8±0.99   | 11.9±0.99  | 13.12±0.35             | 6.71±0.10         |
| 75              | 1.83±0                   | 28.7±0.99   | 20.3±0.99  | 10.64±0.35             | 6.79±0.10         |
| 90              | 2.19±0                   | 31.5±0.99   | 25.2±0.99  | 11.5±0.71              | 7.35±0.10         |

Table 5. Overall acceptability score obtained for the product during period of storage

| Days of storage | Overall acceptability |
|-----------------|-----------------------|
| 15              | 7.87±0.06             |
| 30              | 7.07±0.06             |
| 45              | 6.77±0.06             |
| 60              | 5.70±0.1              |
| 75              | 4.70±0.2              |
| 90              | 3.50±0.3              |

with the chemical parameters and the sensory score assigned by the panel members of sensory evaluation, the product was within the limit of acceptability for a period of 75 days at ambient temperature. The lower shelf life obtained could be attributed to the low barrier properties of the HIPPP trays towards oxygen and water vapour. An improvement in the barrier properties of the packaging material could further increase the shelf life of the product.

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