



Functional relationship between storage time and quality parameters of prawn pickle from *Macrobrachium dayanum* (Henderson, 1893)

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ABSTRACT

A study on quality changes of pickle from small freshwater prawn *Macrobrachium dayanum* (Henderson, 1893) under storage is reported in this communication. The fat content of the pickle decreased significantly ($p < 0.05$) from 21.62 to 18.33 g% during storage. No significant change was recorded in protein and ash contents. During storage, pH showed significant ($p < 0.01$) reduction from 4.63 to 4.00 with simultaneous increase in titrable acidity from 2.18 to 2.88. Peroxide value (PV), free fatty acid (FFA), total volatile bases (TVBN) and alpha amino nitrogen (AAN) increased with storage period. Functional forms of relationships established between storage time and each of the variables, found best in quadratic form for all parameters except PV, which is found to be best at linear form. Total microbial load increased from 4 log cfu g⁻¹ to 5 log cfu g⁻¹. During the entire storage period, the product was free from any visible fungal colonies. The overall acceptability of prawn pickle significantly ($p < 0.01$) decreased with storage days.

Keywords: Biochemical quality, Freshwater prawn, Prawn pickle, Sensory quality, Shelf life, Storage study

Introduction

Pickling of perishable foods in vinegar and edible oil with spices and condiments provide a ready-to-eat product stable at ambient temperature (Kumar, 1985). Besides being highly nutritious, pickles are also good appetisers. Low moisture and reduced pH are the major factors contributing to the stability of pickles (Frazier and Westhoff, 1984). Pickling also helps in improving the desirable characteristics like taste and flavour along with the preservative effect. Several workers have studied pickles prepared from meat of different fish and other aquatic animals in India such as freshwater fish (Chattopadhyay *et al.*, 1985), low cost marine fish (Vijayan *et al.*, 1989), blood clam (Gupta and Basu, 1985), clams (Yellappa and Chandrasekharan, 1989) and edible oyster (Sugumar *et al.*, 1994).

Though various traditional fish products such as dried, smoked and fermented fish are common in the north-eastern region of the country, pickling and consumption of pickled meat products is not very popular, especially among the rural population. Abraham and Shetty (1994) conducted a detailed study on the preparation of pickles from crustacean meat. Study by Karthikeyan *et al.* (2007) showed that many of the small indigenous

fish species and small freshwater prawns available in Tripura can be suitably utilised for production of pickle and other value added fish products. Small freshwater prawns such as *Macrobrachium dayanum* (Henderson, 1893) is available in plenty in the markets of Tripura. As the size is very small, it is prepared along with different vegetables and consumed with rice. Even though it is landed in large quantities, no concerted attempt has been made to study the utilisation of this resource as a source of protein for human consumption. In view of the good nutritive value and availability in north-eastern region of India, an attempt was made to prepare pickle from the meat of *M. dayanum* and its keeping quality was estimated to evaluate the shelf life at ambient temperature. Further, an attempt was made to establish functional relationships between storage time and other parameters like total volatile base nitrogen (TVBN), peroxide value (PV) and free fatty acids (FFA) contents.

Materials and methods

Experimental design

The study was based on evaluation of different quality parameters of prawn pickle prepared from the

freshwater prawn *M. dayanam*. Each of the parameters studied were analysed in triplicate. Functional relationships were established between storage time and each of the variables. The correlation of variation was recorded for scores obtained in each of the sensory attributes.

Preparation of prawn pickle

Freshly caught *M. dayanam* procured from the local markets of Tripura were brought under iced condition in insulated containers to the laboratory of College of Fisheries, Lembucherra, Tripura and used for pickle preparation. Standard recipe of Karthikeyan and Bahni (2006) was adopted for preparation of prawn pickle. Peeled and washed prawns (1000 g) were cut into small pieces (one inch) and marinated in half quantity of total salt (2.5%) and 0.5% turmeric powder. After about half an hour, the prawns were fried in 150 ml of oil and kept aside. Mustard powder (0.4%) and fenugreek (0.4%) were fried in rest of the oil. Chopped garlic (10%), ginger (2.5%) and green chilly (3%) were added and fried until characteristic fried smell emerged. Cumin seed (3%), chilly powder (3%), garam masala (1%), sufficient amount of salt and sugar (0.5%) were made into a slurry and added to the mixture and fried until golden brown colour and characteristic odour developed. The fried ingredients were cooled to slightly above room temperature and citric acid (0.5%), sodium benzoate (0.05%) and vinegar (250 ml) were added as preservatives. Prawn pieces were then mixed thoroughly and were kept overnight for maturation. Pickle was packed in clean dry, food grade polyester containers, layered with oil on top and then stored at room temperature (28 to 30°C). Proximate, biochemical, microbial and sensory quality parameters of the pickle were analysed by drawing samples at monthly intervals.

Proximate and biochemical composition

The moisture, crude protein (Micro Kjeldhal N x 6.25), crude fat (solvent extraction) and ash content were analysed following AOAC (2000). The salt soluble nitrogen (SSN) was determined by Microkjeldahl's method (Dyer *et al.*, 1950), non-protein nitrogen (NPN) by AOAC (2000), total volatile base nitrogen (TVBN) by the method of Conway (1947) and alpha amino nitrogen (AAN) was determined using Pope and Stevens (1939) method. The peroxide value (PV) of the sample was estimated by the method of Jacobs (1958) and free fatty acid (FFA) as percentage of oleic acid as per the method of Olley and Loveren (1960). To determine pH, 5 g of sample was homogenised with 45 ml of distilled water and pH was measured using a pH meter (Sartorius Make,

Germany). Titrable acidity (% acetic acid) was measured using the method of Konecko (1979).

Microbial analysis

The microbial quality of the pickle was assessed by estimating total plate count (TPC) and halophilic bacterial count (APHA, 2001). The pickle was also observed for visible fungal colonies during storage. Total plate count was estimated by pour plate technique on nutrient agar plates. For enumeration of halophilic bacteria, spread plate method on nutrient agar plates with 3% additional salt was used.

Sensory analysis

Sensory characteristics of the pickle were evaluated at monthly intervals following the method of Siddaiah *et al.* (2001) with 10 nos. of trained taste panels using a 1-9 point hedonic scale. The sensory quality of the pickle was judged for attributes such as appearance, colour, texture, odour as well as overall acceptability and the mean of the scores given by the panelists were taken.

Statistical analysis

All the data reported are the average of triplicate observations. One-way analysis of variance (ANOVA) as well as correlation and regression analysis of the important physico-chemical and sensory parameters were performed as per the method described by Snedecor and Cochran (1967) using Statistical Package for Social Sciences (SPSS) version 17.0.

Results and discussion

Analysis of proximate composition of pickle from freshwater prawn *M. dayanam* (Table 1) indicated that, except protein, all other parameters showed decreasing trend during storage. There was an increase in moisture content from 51.05 to 58.53% up to 90 days of storage and there was decrease during further storage. A similar study by Suryanarayan *et al.* (1958) on pickled sardine revealed decrease in moisture content from 78% to 52.8% initially up to 70 days, which increased to 54.8% during further storage up to 125 days. Changes in protein and ash content with storage period were not significant, whereas fat content decreased significantly ($p \leq 0.05$) from 21.62 to 18.33 g% during storage. This might be attributed to the degradation of fat due to oxidation.

TVBN and AAN values of the prawn pickle showed an increase during storage. TVBN value increased from 6.39 to 32.13 mg%, however was within the acceptable limit for fish products. A similar result for

Table 1. Changes in proximate composition of prawn (*M. dayanum*) pickle during storage at ambient temperature

Parameters	Storage days						
	0	30	60	90	120	150	180
Moisture (%)	51.0±1.20 ^a	53.4±1.24 ^b	57.2±0.95 ^c	58.5±0.83 ^c	57.9±0.85 ^c	55.8±0.55 ^d	54.7±0.56 ^d
Crude protein (g%)	14.8±1.21 ^a	14.8±2.20 ^a	14.3±0.96 ^a	14.2±0.63 ^a	14.2±1.97 ^a	14.6±0.99 ^a	14.6±0.85 ^a
Crude fat (g%)	21.6±1.04 ^a	20.3±0.90 ^b	20.2±1.29 ^b	20.0±0.46 ^c	18.7±1.25 ^d	18.9±1.21 ^d	18.3±0.36 ^d
Ash (g%)	3.2±0.42 ^a	2.8±0.68 ^b	2.7±0.89 ^b	2.4±0.91 ^b	2.5±1.18 ^b	2.3±0.63 ^b	2.3±0.62 ^b

* Values are mean ± standard deviation (n = 3)

Values within the same row with different superscripts are significantly different ($p \leq 0.05$)

TVBN in fish pickle from *Nemipterus japonicus* during storage of 180 days was reported by Chandrashekar *et al.* (1978). Lower TVBN value indicates good quality of the products and high values of TVBN correlate with high bacterial activity as well as high rate of spoilage, which results in unacceptability of the product for human consumption (Joseph *et al.*, 1983). Functional relationships between storage days and other variables was established (Table 2) by fitting linear, quadratic and cubic curves. The relationship of storage days versus TVBN (Fig. 1a) shows that coefficient of determination (R^2) values of cubic form of relationship is best fitted between the variables ($R^2 = 0.98$) compared to linear and quadratic forms. This means that storage time alone can explain 98% variability of TVBN. But, linear fits are also satisfactory and significant.

An increase in AAN value from 7.12 to 47.5 mg% up to 120 days of storage followed by a sharp fall during further storage is attributed to the breakdown of protein molecules which might be responsible for typical flavour and odour of the product. Similar increase in AAN content of anchovy pickle with advancement of storage period was studied by Shiriskar *et al.* (2010). NPN values of the prawn pickle increased slightly from 0.62 to 0.76 g % whereas SSN values first decreased from 15.92 to 12.42 g% and then abruptly increased from 120 days till 180 days of storage. A consistent increase in NPN level with decrease in SSN level can be attributed to the denaturation of protein in prawn pickle during storage.

Both peroxide and free fatty acid values increased significantly ($p < 0.01$) from 26.63 to 77.35 meq O_2 kg^{-1} fat and 0.15 to 10.93% of oleic acid respectively during storage confirming the rancidity of fat. A similar increase in peroxide value of clam pickle during storage was

Table 2. Coefficient of determination (R^2) values for linear, quadratic and cubic fit between storage days and different quality parameters

Pair of variables	Linear	Quadratic	Cubic
Storage days vs TVBN ^a	0.912	0.979	0.980
Storage days vs PV ^b	0.956	0.957	0.957
Storage days vs FFA ^c	0.880	0.881	0.936
Storage days vs acidity	0.942	0.957	0.967

^aTVBN = Total volatile base nitrogen, ^bPV = Peroxide value,

^cFFA = Free fatty acid

reported by Gupta and Basu (1985) and an increase in FFA value of fish pickle during storage by Chandrashekar *et al.* (1978). Shiriskar *et al.* (2010) found an increase in lipid quality parameters *viz.*, PV and FFA accompanied by decrease in flavour as well as odour scores of anchovy pickle under storage. Relationship between storage vs PV (Fig. 1b) showed satisfactory fit by linear equation ($R^2 = 0.956$) whereas, that of storage vs FFA (Fig. 1c) is best represented by cubic form of relationship ($R^2 = 0.936$). This means storage time alone can explain 95.6% variability of PV and 93.6% of FFA.

The pH of the pickle decreased significantly ($p \leq 0.01$) from 4.63 to 4.00 during 180 days of storage, whereas the titrable acidity significantly increased ($p \leq 0.01$) during storage. This may be due to the production of acids in pickle by certain acid producing bacteria, which multiplied at lower pH value. The increased titrable acidity could also be attributed to loss of moisture and the effect of condiments mix. Tanuja and Hameed (1998) recorded a reduction in pH from 4.46 to 3.72 with an increase in acidity of squilla pickle for a storage period of 180 days. Sahu *et al.* (2012) reported a similar result for pH of about 4.99 and significant increase in titrable acidity in murrel pickle during 60 days of storage. Relationship established between storage and acidity (Fig. 1d) showed satisfactory fit in cubic form of relationship ($R^2 = 0.967$) which means storage time alone can explain 96.7% variability in acidity.

Meat products have been reported to get spoiled at a microbial load of more than 6 log cycles (Frazier and Westhoff, 1978). The TPC of the prawn pickle increased from 4 log cfu g^{-1} to 5 log cfu g^{-1} during storage of 180 days (Khanna *et al.*, 2004) and is well within the acceptable limit (ICMSE, 1986). The lower TPC values of the pickle may be due to the low pH of the product, which inhibited the multiplication of bacteria (Acton and Johnson, 1973). The halophilic counts of the samples were < 1 cfu g^{-1} during the entire storage period and no visible fungal colonies were observed during storage. The perceivable sensory attributes *viz.*, colour, appearance, feel, aroma, taste and texture are the deciding factors for consumer acceptance of any food. The sensory scores

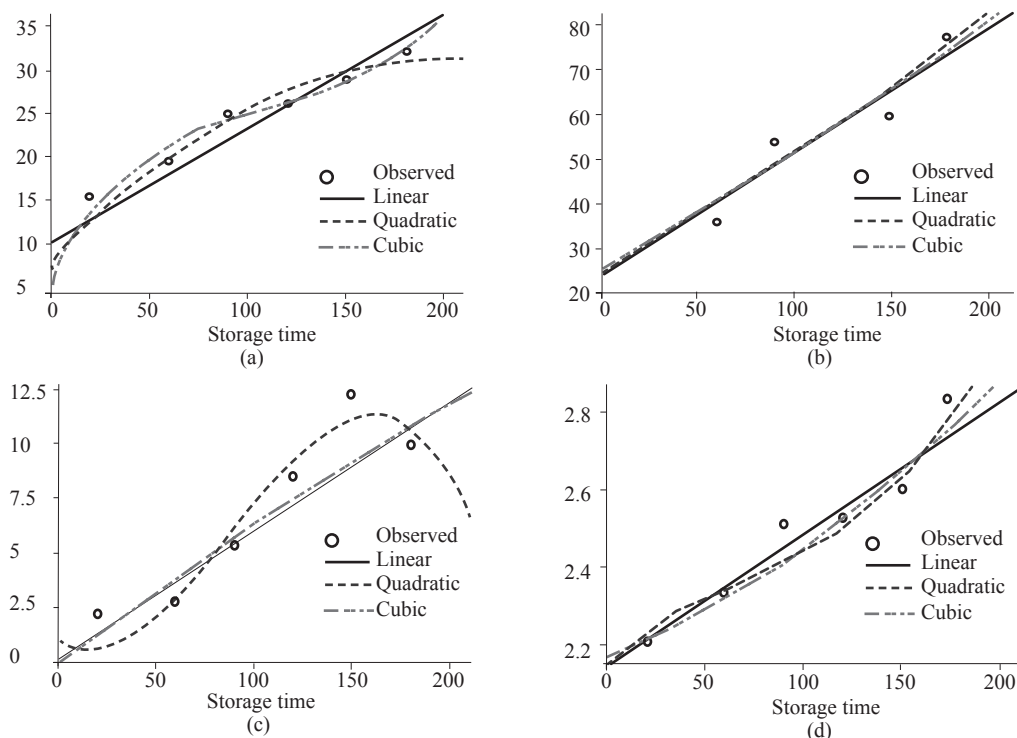


Fig. 1. Linear, quadratic and cubic curves showing relationships between storage time and (a) TVBN; (b) PV; (c) FFA; (d) Acidity values

of the prawn pickles decreased significantly ($p \leq 0.01$) during storage and based on overall acceptability scores, the pickle was found to have a shelf life up to 180 days at ambient temperature, retaining its quality. Shiriskar *et al.* (2010) reported that the overall acceptability and other individual sensory attributes of anchovy pickle decreased during storage and was kept in good condition up to 15 weeks.

Coefficient of variation (CV) can be taken as a measure of consistency of different sensory parameters of prawn pickle during storage (Table 3). Range of variation of different sensory parameters of prawn pickle with different storage period revealed that in terms of

appearance, colour and taste, the range of variations are 5.66 to 7.79, 2.85 to 7.9 and 9.0 to 13.64 respectively which is acceptable. But, in case of taste, texture and overall acceptability scores, the range of variations are higher *i.e.*, 6.83 to 17.38, 6.83 to 17.38 and 4.66 to 13.37% respectively. This also justifies the fact that the product is quite consistent in terms of appearance, colour and taste. On the other hand, the CV values of colour, texture, odour and acceptability are 33.12, 29.54, 28.44 and 29.20% respectively, though slightly on the higher side, are in the acceptable limit. The product was found non-defective as no visible fungal colonies appeared. The pickled product prepared in the present study can be considered acceptable as it retained good appearance, colour as

Table 3. Test of consistency of different sensory parameters of prawn pickle with storage days at ambient temperature

Storage days	Appearance	Colour	Taste	Texture	Odour	Overall acceptability
0	^a 8.6±0.67	8.4±0.67	8.2±0.87	8.1±0.83	8.1±1.14	8.4±0.69
30	8.30±0.47	8.2±0.60	8.0±0.77	8.0±0.77	7.9±0.94	8.1±0.94
60	8.1±0.60	7.8±0.44	7.3±1.00	7.7±1.41	7.8±0.83	7.5±1.01
90	7.5±0.53	7.3±0.76	6.1±0.69	6.5±0.53	6.5±1.13	6.4±0.79
120	6.8±0.50	7.0±0.50	5.5±0.58	5.5±0.58	5.2±0.50	6.0±0.58
150	7.0±0.50	7.0±0.50	5.0±0.50	5.7±0.58	4.7±0.57	5.7±0.58
180	6.5 ± 0.50	7.0±0.20	4.0±0.36	5.1± 0.58	4.1 ±0.28	4.5±0.21
Range	5.7– 7.8	2.9 – 8.0	9.0 – 13.6	8.1 – 18.4	6.8 -17.4	4.7 -13.4
^b CV (%)	9.9	33.1	14.1	29.5	28.4	29.2

^aEach observation is mean ± standard deviation of triplicates (n=3)

^bCV = Coefficient of variation

well as taste during the storage period of 180 days. The results of the study revealed that good quality pickle with good acceptability can be prepared from small freshwater prawns that are rich in protein and fat.

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Date of Receipt : 02.01.2014

Date of Acceptance : 26.06.2014