

Effect of Nitrogen Gas Enriched Packing on Quality and Storage Life of Pearl Millet Based Fried Snack

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Received: June 24, 2016; Revised: February 8, 2017; Accepted: February 21, 2017

Abstract

Purpose: The storage life of pearl millet-based, deep fried, ready-to-eat snacks, packaged in aluminum-laminated polyethylene having a thickness of 50 μm (with and without nitrogen) was evaluated under storage conditions of $38\pm 2^\circ\text{C}$ and 90% RH. **Methods:** The moisture content, free fatty acids (FFA), peroxide value, and crispness of the snack were evaluated throughout the storage period. The moisture content, FFA, and peroxide value increased with an increase in storage period, but the increase was less in packages flushed with nitrogen gas. The crispness decreased with an increase in the storage period, for snacks both with and without nitrogen packages. However, the decrease was less in nitrogen-flushed packages. FFA and peroxide values were strongly correlated with the moisture content of the snack. The storage life of the snack was found to be 60 and 45 days in packages with and without nitrogen respectively. **Conclusions:** The snack's predicted storage life, for snacks with and without nitrogen packages, was determined as 294 and 254 days respectively.

Keywords: Nitrogen flushing, Deep fried snacks, Shelf life prediction

Introduction

Food products undergo different types of deteriorative changes during storage. Hence, it is very important to know their storage life under specific environmental conditions, during which, no appreciable deterioration in quality and acceptability occurs. The various factors that affect food stability include humidity, oxygen, toxic vapors, physical contamination, light and the time-temperature history of the package (Khanna and Peppas, 1982). Two major deteriorative reactions viz., moisture absorption and oxidative rancidity govern the life of fat rich, dry products or fried snacks, during packaged storage. Dry food systems can lose their desired crispness during storage or upon opening of the package. Loss of crispness due to moisture uptake is a major cause of snack food rejection by consumers (Robertson, 2006). Puffed rice

cakes were reported to lose their crispness and become tough as water activity (a_w) increased through moisture absorption. Rice cakes with a_w between 0.2 and 0.4 have the best crispness and lowest hardness (Hsieh *et al.*, 1990).

Water activity also helps to predict safety and stability with respect to microbial growth, chemical and biochemical reaction rates, and physical properties. Therefore, by measuring and controlling water activity, it is possible to

- a) Predict which microorganisms will be potential sources of spoilage and infection
- b) maintain the chemical stability of products
- c) minimize non-enzymatic browning reactions and spontaneous autocatalytic lipid oxidation reactions
- d) prolong the activity of enzymes and vitamins
- e) optimize the physical properties of products such as moisture migration, texture, and shelf life.

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Thus, it can be concluded that the water activity may be used as a benchmark in deciding the cut off limit of storage duration of the products under study.

Various researchers have tried to model the deteriorative reactions of fresh or dry products during storage studies, based either on moisture migration (Gomes *et al.*, 2010; Siripatrawan and Jantawat, 2008; Roca *et al.*, 2008; Bourlieu *et al.*, 2008; Yan *et al.*, 2008) or lipid oxidation (Gomes *et al.*, 2010). Predicting the shelf life of a product based on both types of deteriorative reactions during storage, however, has only been attempted by very few researchers (Del Nobile, 2001; Achour, 2006). The present study was carried with the aim of predicting the storage life of pearl millet based fried snacks packed in aluminum laminated polyethylene (50 μm thickness) packages (Tiwari *et al.*, 2011; Balasubramanian *et al.*, 2014), with and without nitrogen flushing. The effect of packaging, environment and storage period was determined based on the quality of the snack stored under fixed conditions.

Material and Methods

An expanded, ready-to-eat, extruded snack was developed using an extruder (model, company name, company located city, country) under optimized conditions: 13% moisture content (m.c.), 128°C, 470 rpm of extruder barrel speed (per ICMR guidelines), using pearl millet, QPM (Quality Protein Maize) and green gram (60:30:10). 25 g of developed snack samples were fried in hydrogenated vegetable oil (about 100g), in a frying pan at 155 \pm 5°C, for about 35s. After frying, the product was taken out from the oil. The fried sample was immediately seasoned (2 g dried seasoning powder in 50 g of snacks) with tomato seasoning (supplied by Symega, Ernakulam Kerala, India).

The seasoned snack was packed in Al-laminated polyethylene packages (50 μm thickness, having a water vapor permeability of $4.9 \times 10^{-7} \text{ kg.m}^{-2}\text{day}^{-1}\text{Pa}^{-1}$), with and without nitrogen flushing that were purchased from the local market. The water vapor permeability of the packages was measured using the method described by Jaya and Das (2005). The samples were stored under 38 \pm 2°C and 90% RH (storage condition) for the period of 60 days, until an off smell was perceived on package opening. The quality of the product was assessed every 15 days and analyzed for moisture, texture, FFA and

peroxide value.

Moisture content

The moisture content of the snack was determined using the hot air oven method (AOAC, 1984). A weighed amount (10 \pm 0.5 g) of samples was placed in clean, dried, and pre-weighed aluminum dishes. The contents were dried in an oven at 100 \pm 1°C for 4-5 h or until constant weight was obtained. Dried samples were transferred to desiccators, cooled and weighed. The percentage loss in weight was interpreted as the sample's moisture.

Free fatty acids

FFA content was determined using the AOAC (1984) method of sample analysis. About 30 to 40 g of snack sample was placed in a 250ml conical flask, where 150 to 200 ml of petroleum ether was added and oil from the sample was extracted by keeping it for about 60 min. with occasional shaking. Miscella was collected in another 100ml conical flask, 20ml of this miscella was placed on a pre-weighed, dried petri dish and kept in a hot air oven at 80°C for 2 h. The weight of the oil was calculated. Further, in 20 ml of the miscella, 10 ml of neutral ethanol was added and the mix was titrated against N/10 NaOH with phenolphthalein as an indicator. The FFA value was calculated using equation (1):

$$\begin{aligned} &\text{FFA (as \% oleic acid)} \\ &= \frac{\text{Titre value} \times \text{normality of NaOH} \times 0.282 \times 100}{\text{Weight of oil in miscella}} \quad (1) \end{aligned}$$

Peroxide value

For peroxide value, 1 g of oil extracted (supernatant) from the snack was taken in a conical flask, and 15 ml of solvent (acetic acid and chloroform 3:2) was added. Then, 1 ml of potassium iodide solution was added and allowed to stand for 5 min. This was followed by the addition of 35 ml of water, and the liberated iodine was titrated with 0.1N sodium thiosulphate solution, using starch as an indicator (AOAC, 1975).

Textural quality

Crispness is the crucial textural characteristics of extruded snack food products. The textural properties of extrudates were measured using a Texture Analyzer (Model TA+Di, Scientific Micro Systems, UK) attached

with 25 kg load cell. A 2 mm cylindrical probe (P2) was used to determine the hardness. The test was carried out using test speed of 0.5 mm/s and compression distance of 90% of the product size. A force–time curve was recorded and analyzed. Ten randomly collected samples were measured from replications. The crispness of the extrudate was determined by counting the number of positive peaks in the curve (Lazou and Krokida, 2010).

Storage life prediction

Moisture gain by dry or semidry foods can lead to several modes of deterioration, including microbial growth, loss of crispness, and loss of softness, hardening, and caking. The moisture gain or loss for a food held at constant temperature and exposed to a given external relative humidity can be predicted from simple engineering relationships based on water activity (Labuza 1980).

The water activity (a_w) of the seasoned snacks was first determined by Wink's weight equilibrium method using standard saturated salt solutions (Wink, 1946). The initial moisture content was determined by the oven drying method (AOAC, 1984). Approximately 5 g samples of known moisture content were weighed accurately in separate petri-dishes and kept in desiccators which were pre-equilibrated to different relative humidities (RH) having the salt solutions. Then samples were weighed regularly till their weight became constant. When samples equilibrated with environment, they were weighed (S) and their equilibrium moisture content (EMC) on dry weight basis was calculated by the following equation:

$$EMC (\% db) = \frac{(S - X)}{X} \times 100 \quad (2)$$

where S is the weight of the sample after equilibration and X is the dry weight of the sample.

Then, the time θ_w (days) required for the moisture content of the snacks to increase from an initial value of X_{pi} (kg water per kg dry solids) to its critical value X_{pc} (kg water per kg dry solids) can be obtained from Eq (2).

$$\theta_w = \frac{W_p}{p^* K_p A_p} \int_{X_{pi}}^{X_{pc}} \frac{dX_p}{R_h - a_w} \quad (3)$$

where W_p (kg) is the dry weight of the snacks kept inside

the packaging material, p^* (Pa) is the saturation vapor pressure of water at the temperature T_a ($^{\circ}C$) of storage, R_h (fraction) is the relative humidity of the storage environment, K_p ($kg\ m^{-2}\ day^{-1}\ Pa^{-1}$) water vapor permeability of the packaging material, A_p (m^2) surface area of the packaging material, a_w (fraction) water activity of snacks at moisture content X_p (kg water per kg dry solids) and temperature T_a ($^{\circ}C$).

In practice, researchers testing the shelf life of foods use a combination of higher humidity (percent RH) and temperature (T) than the food would normally be subjected to. Most food processors suggest, for dry foods, that the average temperature/humidity during distribution is 21 $^{\circ}C$ at 50-percent RH, and thus apply some factor by which the food shelf life under the adverse condition is multiplied by, to give the average shelf life. Using this method and equation 13, the shelf life of a food for which the mode of deterioration is moisture gain or loss, can be predicted if the external conditions of distribution and marketing are presumed to remain constant. However, in the real world, the humidity can vary as well as the temperature. Fluctuating temperature effects were discussed previously. In general, higher humidities are associated with higher temperature, but no exact pattern of correlation exists.

Statistical analysis

The data for each determination were collected in triplicate. The data were analyzed using the technique of analysis of variance (Panse and Sukhatme, 1978). The effects of moisture content, FFA, peroxide value, and textural quality on the basis of storage days were tested for significance at $P \leq 0.05$ using SPSS 16.0.

Results and Discussion

The most common tests recommended for assessing the quality of deep-fried snacks are moisture, acid-insoluble ash and fat contents, peroxide, and acid values of extracted fat (BIS 1989). The moisture content, FFA, peroxide value, and textural quality of the pearl millet based fried snack were evaluated during the storage period.

Moisture content

The moisture content of snacks in package without

nitrogen increased from 2.5 to 3.8%, whereas in the nitrogen flushed package, the increase was measured from 2.5 to 3.5%. The lesser increase in moisture during storage in a nitrogen flushed package was mainly due to the flushing of nitrogen that replaced the oxygen (air) and water vapor from the package.

The moisture content increased with an increase in storage period (Table 1). The mean percent moisture of the snacks at the beginning of storage was 2.5%, which increased significantly to 3.1% as the storage period approached 60 days. The gain in moisture content was due to the hygroscopic nature of the product, storage environment (temperature, relative humidity) as well as due to the nature of the packaging material.

The effect of packaging technique and storage on the moisture content of the snacks is shown in Table 1. Both packaging environment and storage period had significant effects on the moisture content of the snack. However, the interaction of packaging environment and storage period had non-significant ($P \leq 0.05$) effect on moisture content.

Free fatty acid (FFA)

After 60 days of storage, the FFA varied from an initial

value of 0.75% to 1.22%, for nitrogen flushed packages, and from 0.75% to 1.78% for flushed packages without nitrogen. During storage, the increase of free fatty acid (FFA) for snacks in nitrogen flushed packages there was slower, compared to the packages without nitrogen, on any given day of storage (Table 2). The slow rate of increase of FFA in nitrogen flushed packets could be ascribed to the lower concentration of oxygen combined with nitrogen being inert, causing the lipid oxidation rate to reduce while increasing the stability of the snacks in nitrogen flushed samples when compared to control (Paik *et al.*, 1994). Further, there was a gradual increase in FFA as the storage period increased. This increase in FFA content may be attributed to the increase of moisture content in the product, which contributed to fat hydrolysis during storage. Singh *et al.* (2000) have also reported an increase of free fatty acid in soy-fortified biscuits during the storage period.

The FFA on 45th and 60th days of stored snacks was observed to be 1.16% and 1.22%, respectively. However, on the 45th day of storage the snacks without nitrogen packing developed rancid smell, whereas snacks in nitrogen flushed packages the same smell was detected

Table 1. Effect of packaging environment and storage days on moisture content of snack

Storage days	Without nitrogen pack	Nitrogen flushed pack	Mean
0	2.50	2.50	2.50
15	2.70	2.60	2.65
30	3.00	2.90	2.95
45	3.46	3.30	3.40
60	3.80	3.50	3.65
Mean	3.10	2.96	
CD ($P \leq 0.05$)	Packaging environment (P) Storage days (S) Interaction (PxS)	0.092 0.145 NS	

Table 2. Effect of packaging materials and storage days on FFA (%) of extruded snacks

Storage days	Without nitrogen pack	Nitrogen flushed pack	Mean
0	0.75	0.75	0.75
15	0.88	0.85	0.86
30	1.04	0.91	0.97
45	1.16	1.04	1.10
60	1.78	1.22	1.50
Mean	1.12	0.95	
CD ($P \leq 0.05$)	Packaging environment (P) Storage days Interaction(PxS)	0.019 0.030 0.042	

on the 60th day of storage. Thus, where FFA is more than the critical level of 1.16% in snacks could be considered as not fit for consumption. Erickson and Frey (1994) and Tiwari *et al.* (2011) also reported that fried snacks with FFA > 1% are unfit for consumption.

Statistically, both the packaging environment and storage period had significant effect on the free fatty acid content in the tested snacks (Table 2).

Peroxide value

Peroxide values (PV) measure the content of hydroperoxides and are used as indicators of lipid oxidation (Gray, 1978). A variation from 0.55 to 5.41 meq/kg was observed in nitrogen flushed packages, and from 0.55 to 7.51 meq/kg in packages without nitrogen, during 60 days of storage. Lower values of PV in nitrogen flushed packages were due to low concentration of oxygen which retarded the oxidation reaction of oil/lipid during storage. PV was found to correlate strongly ($R^2=0.986$ to 0.996) with the moisture content in the sample (Figure 1). The increase in storage period led to an increase in moisture content

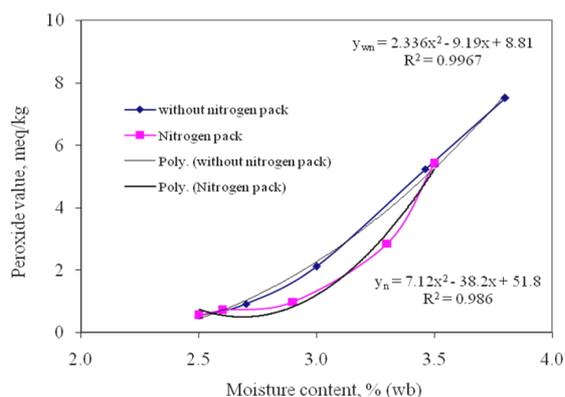


Figure 1. Effect of moisture content and packaging environment on peroxide value of the snack (where Y_{wm} is polynomial equation for without nitrogen and Y_n is with nitrogen).

and thereby to an increase in the value of peroxide. The critical levels of PV corresponding to critical levels of FFA and moisture content were observed to be 5.41 meq/kg in snacks with nitrogen flushed packages and 5.23 in snacks without nitrogen flushed packages. Tiwari *et al.* (2011) suggested that snacks with peroxide level <5 meq/kg of oil are fit for consumption.

Textural quality

Crispness, being the important sensory attribute of an expanded ready-to-eat fried snack, was evaluated throughout the storage period. It was observed that the crispness of the snack decreased with an increase in the storage period. This was due to an increase in the moisture content in the product. It was also found that the crispness of the snack was better retained in nitrogen flushed packages throughout the storage period due to a slower increase in moisture content. Although there was decrease in crispness, the product was still crisp at the end of storage period since the moisture content was limited to 3.5% (*i.e.*, a gain of 1% over initial moisture content). Nikolaou (2012) too found that the moisture content of ready to eat snacks should not be more than 4% to retain its crispy quality within acceptable levels. The result is also in line with the findings of Tiwari *et al.* (2011) who reported that the moisture gain >1.5% is not suitable for crispness in case of storage studies of dried snacks.

As revealed by critical difference values (Table 3), storage period significant affected crispness whereas packaging environment and the interaction of packaging environment and storage day had no significant effect.

Prediction of storage life of snacks

The Sorption characteristic of snack food was determined

Table 3. Effect of packaging materials and storage days on texture of extruded snacks

Storage days	Without nitrogen pack	Nitrogen flushed pack	Mean
0	481	481	481
15	475	479	477
30	463	467	465
45	458	460	459
60	445	458	451
Mean	464.4	469	
CD	Packaging environment (P)	NS	
($P \leq 0.05$)	Storage days (S)	8.929	
	Interaction (PxS)	NS	

Table 4. Predicted shelf life of the snack

Packaging environment	K_p ($\text{kg}\cdot\text{m}^{-2}\cdot\text{day}^{-1}\cdot\text{Pa}^{-1}$)	A_p (m^2)	P^* (Pa)	a_w	X_i	X_c	θ_a (days)	θ_p (days)
Without nitrogen	$4.9\cdot 10^{-7}$	0.0154	6539	0.201	0.025	0.034	45	254
With nitrogen				0.228	0.025	0.035	60	294

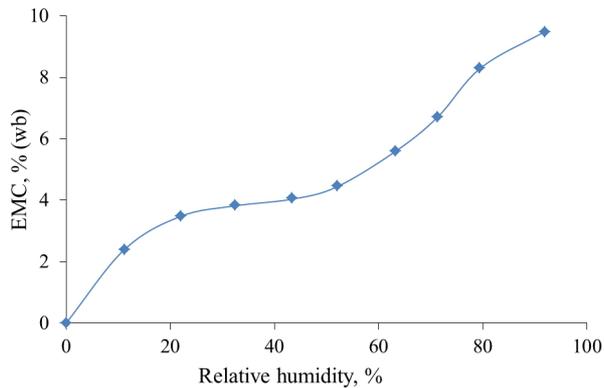


Figure 2. Sorption isotherm of snacks.

at varying relative humidity levels and is presented in Figure 2. Using the sorption curve (Figure 2), water activity of the snack corresponding to initial and critical moisture content was calculated at 0.219 and 0.228, respectively. The other parameters of Eq. (3), such as the water vapor permeability of the package, effective area of the package, saturated vapor pressure as well as initial and critical moisture content are given in Table 4. With these values of water activity, predicted storage life of snacks at ambient conditions was calculated using Eq. (3). The predicted storage life of snacks without nitrogen flushing was calculated as 254 days, whereas with nitrogen flushing it was found to be 294 days. However, the predicted life could not be validated through real time studies.

Conclusions

The storage life of snacks under storage conditions at 38°C and 90% RH, was determined as 60 and 45 days respectively, in packages with and without and nitrogen. The predicted storage life for packages with nitrogen was calculated at 294 days and at 254 days for packages without nitrogen. Peroxide value and FFA were found to be major contributing factors to snack deterioration. Both peroxide value and FFA correlated strongly with the moisture content in the snack.

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