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Effects of thermal treatments on the storage stability of whole pearl millet flour

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Abstract
The grains of pearl millet after treatments *viz*. hot water blanching and dry heating were milled into flour and then stored in polyethylene bags at prevailing room temperature (32±3 ºC) and refrigerated condition (4-7 ºC) for 35 days. Lipids in the pearl millet showed a rapid hydrolytic decomposition during storage. The magnitude of such degradation was influenced significantly by the nature of the storage condition, the temperature and heat treatment given to the seeds. The flour was analyzed periodically (0, 7, 14, 21, 28 and 35 days) for keeping quality by chemical methods. The hydrolytic breakdown of lipids was significantly low (about 2-3 folds) in the meals stored at refrigerated condition. The dry heating of seeds was found to be most effective in minimizing the undesirable changes in lipids of the meal as it decreased the production of peroxides and free fatty acids during storage. Sensory evaluation was also done of the chapattis made from the flour of differentially treated grains. The chapatti of control (untreated) showed acceptable flavor and taste only upto 7 days whereas chapatti prepared from the blanched and dry heated seeds exhibited acceptability more than a week.

Keywords: Pearl millet, heat treatment, shelf-life, lipids

1. Introduction
Pearl millet (*Pennisetum glaucum*) also popularly known as Bajra in India is an important food crop of semi-arid areas of the world and is termed as crop of food security because of its sustainability under adverse agro-climatic conditions. India is the largest producer of pearl millet both in terms of area and production with an average productivity of 780 Kg/hectare during the last five years (Zakari *et al.*, 2010) [14]. It is also termed as 'neutricereal' in view of its good nutritional specialties such as complex carbohydrates (67.5%), high proportion of dietary fibres and of other phyto-chemicals with neutraceutical qualities (Sumathi *et al.*, 2007) [12]. The nutritive value of Pearl millet is comparable to other cereals with regard to protein, fat and mineral content (Singh and Sehgal, 2008) [11].
The pearl millet grain is small but has a proportionally larger germ than all other cereal grains, except perhaps maize (Taylor, 2004) [13]. Hence, pearl millet tends to contain a higher content of triglycerides. These are rich in unsaturated fatty acids (Rooney, 1978; Lai and Varriano-Marston, 1980a; Kapoor and Kapoor, 1990) [6, 9]. When pearl millet is reduced into flour, the resulting flour is noted as having poor keeping quality especially under conditions of moderately high moisture and oxygen exposure (Chaudhary and Kapoor, 1984) [7]. This is attributed to the deterioration of its triglycerides through lipolysis and subsequent oxidation of de-esterified unsaturated fatty acids (Lai and Varriano-Marston, 1980b) [6]. These chemical changes manifest themselves as off-odors and/or off-taste of the flour or in products made from the flour. Thus it becomes unpleasant to eat.
Traditionally, pearl millet is manually pounded, generally using pestle and mortar, into flour in an amount that is just enough for a few days of household use. Households can only store pearl millet flour for short periods because it quickly goes rancid and becomes unpleasant to eat. There is also a demand for processing value-added, traditional, convenient food products made from locally grown raw materials such as pearl millet especially in urban areas. These factors therefore require a longer storage life for pearl millet flour. There is, however, scant information on how to improve the shelf life of pearl millet flour.
This research project, therefore, was designed to investigate the effects of different thermal treatments to prevent the development of rancidity in order to produce shelf stable flour and value-added products.

Materials and Methods

Grains of cultivar HC-20 Kharif 2008 of pearl millet (*Pennisetum glaucam*) were procured from the Bajra Department, CCS Haryana Agricultural University, Hisar. All the reagents used were of analytical grade.

Peroxide Value

Peroxide value of bajra samples was determined by Lea method (Ranganna 2004) [8]. 1 gm of the sample was weighed in a stoppered conical flask. 25 ml of solvent (chloroform +glacial acetic acid) was added in the ratio of 2:3.1 ml of potassium iodide solution was added. It was kept in dark for 5 minutes. 30 ml of distilled water was added followed by 1 ml starch indicator. Then, it was titrated against 0.01 N sodium thiosulphate until a colorless end point was obtained. The same procedure was repeated for the blank.

Peroxide Value (meq/1000 g fat)=[(Sample Titer-Blank Titer) × Normality × 1000] ÷ Sample weight

Acid Value & % Free Fatty Acid Content (Ranganna, 2004) [8]: Approximately 1gm of the melted fat was added in the mixed neutral solvent and titrated with aqueous 0.1N sodium hydroxide shaking constantly until a pink color was obtained which persisted for about 15 seconds.

% Free Fatty Acids (Expressed in terms of linoleic acid) = [Titer Value × Normality×28] ÷ Sample weight

Acid Value (mg NaOH per g of fat) (expressed in terms of linoleic acid) = [Titer Value × Normality × 40] ÷ Sample weight

Processing and Storage of Pearl Millet Flour:

Two treatments *viz*: hot water blanching (dipped in water at a temperature of 98 ^{0}C/10 sec.) and dry heating (100^{0}C for 2 hours in an oven) were given to pearl millet grains. Seeds were ground to obtain flour. Physicochemical changes in treated grain flour samples were compared with the untreated sample. Two storage conditions *viz*. room temperature (32±3 ^{0}C) and refrigerated temperature (5±2 ^{0}C) were provided. Then flours obtained from the differentially treated grains were stored in polyethylene bags under ambient conditions and refrigerated conditions for 5 weeks. Fat was extracted from the samples using solvent extraction method (*n*-hexane). This extracted fat was used to estimate the peroxide value, acid value and % free fatty acid content of the samples after each week starting from 0 day of storage to determine the degree of lipolytic deterioration of stored flours.

Sensory Analysis

Sensory evaluation was carried out for chapattis made from treated and untreated samples. Chapattis were analyzed for their acceptability and desirability influenced by storage period. Sensory analysis was done by a panel of 5 judges. Chapattis were rated on hedonic scale from 1 to 9 points where,1 was for extremely disliked sample and 9 was for extremely liked ones.

Results & Discussion

Effect of Treatments on peroxide value

It was found that initially, the peroxide value of control (untreated) sample kept at room temperature was very low (1.960) because less peroxides were formed and then there was a sharp increase in peroxide value in 7 days (13.375). At refrigerated temperature, a continuous increase in peroxide value was observed upto 21 days from 1.839 at 0 day to 7.468 on 21 days storage (as shown in Figure 1). Further decrease in peroxide value was observed with storage time (as shown in Table 1) because the peroxides already present reacted to form super oxides which do not contribute to peroxide value. Decreased peroxide value after a certain incubation period 21 days was well correlated with the findings of Kadlag *et al*. (1993) [3].

Results showed that the initial peroxide value of blanched samples (6.454) were higher than the initial peroxide value of control sample due to the release of peroxides induced by thermal treatment. Peroxide value of the blanched sample stored at ambient conditions decreased 2 folds from 6.454 at 0 day to 3.375 at 7 days. Further, increase in peroxide value was observed in the second week of storage from 3.375 (7 days) to 4.744 (14 days) in case of blanched sample because peroxidation occurs at an accelerated rate in presence of moisture. In case of blanched samples, the effect of inactivation of lipases was very high and super oxides were formed at a higher rate. Therefore, peroxide value decreased during first week of storage but again increased in the second week due to the effect of high moisture content of the blanched sample (Shetty *et al*., 2002) [10] and then, again it started decreasing during subsequent period of storage due to formation of super oxides. Rate of lipolytic degradation was slower in case of refrigerated blanched samples than the samples stored at ambient conditions.

It was found that initially, the peroxide value of dry heated samples was comparatively higher as compared to blanched and untreated samples due to peroxide formation induced by high temperature.

A continuous decrease was observed in peroxide value of dry heated samples stored at room temperature as well as those stored under refrigerated conditions (Kapoor & Kapoor, 1990) [5] due to the formation of super oxides which do not contribute to peroxide value (Kadlag *et al*., 1993) [3], but the decrease in peroxide value for the refrigerated dry heated samples was 2-3 folds faster than the dry heated samples stored at ambient conditions. This was due to the reason that rate of lipolytic degradation is slower at low temperature storage (Kadlag *et al*., 1993) [3].

Effect of Treatments on acid Value

Histochemical studies on kernels showed that that most lipids were concentrated in the germ and pericarp of the grain. Lipases located in the germ and surface layers of grain get mixed throughout the meal during milling and decompose the meal lipids into free fatty acids during storage. Therefore, acid value of flours was higher than those of the seeds. The acid value is a measure of the extent to which the glycerides in the oil have been decomposed by lipase action. It was found that the acid value showed a continuous and rapid increase in treated as well as the untreated samples. Initially, acid value was found to be higher in the dry heated samples (1.991) than that of blanched samples (0.856) and untreated samples (1.565) as shown in Table 2 due to release of free fatty acids stimulated by thermal induction (Kapoor & Sharma, 1997) [4]. Acid values of treated as well as the untreated samples increased throughout storage due to release of free fatty acids (Marston-Varriano *et al*., 1984), however, the rate of increase of these values was 3-4 fold lower in case of

blanched and dry heated samples compared to untreated samples (as shown in Figure 3), because of the reason that the lipases responsible for breakdown of lipids had been partially inactivated (Chavan *et al*., 1996) and relatively fewer number were present, which caused increase in acid value and % free fatty acid content during storage.

As the rate of increase in acid value was slower in case of dry heated sample (2.312 at 7 days and 4.896 at 14 days) compared to that in blanched samples (4.037 at 7 days and 6.025 at 14 days), therefore, dry heating treatment was found to be more effective for retarding the lipolytic degradation in pearl millet flour than hot water blanching treatment.

The results suggested that initial activity levels of lipases in grains might be having more important role in generating off-flavor in pearl millet flour during storage than the inherent fat content of the grain which merely acts as a substrate for lipid degrading and/or lipid oxidizing enzymes.

Effect of Treatments on % Fatty Acids Content

Linoleic, oleic and palmitic acids are the principal fatty acids in pearl flour (Kapoor & Kapoor, 1990) [5]. Small quantities of linoleic and stearic acids were found and traces of palmitoleic acid were also detected. It was found that initially, the percentage of free fatty acids was higher in case of dry heated samples (1.394) than that of the blanched samples (0.599) and untreated samples (1.095), all stored at ambient conditions (as shown in Table 3) due to the release of some free fatty acids induced by the high temperature given by dry heating.

However, the results showed that the rate of increase in %free fatty acids content was significantly lower in dry heated samples (1.394 at 0 days and 1.618 at 7 days) and blanched samples (0.599 at 0 day and 2.826 at 7 days) compared to untreated samples due to the partial inactivation of lipases responsible for hydrolysis of triglycerides (Pruthi, 1981) [7]. A smaller increase in % free fatty acid during 5 weeks of storage was observed in dry heated sample.

These results indicate that dry heating treatment is most effective for arresting the lipolytic deterioration of flour as it causes the acid value and % free fatty acid content to increase at comparatively slower rates than the other treatments. The application of dry heat to the grains effectively retarded the lipase activity and minimized lipid decomposition during storage. It is more convenient to heat seeds than meal. Such a heat treatment needs to be mild to avoid detrimental effects on other nutrients in the seeds. The use of hot water blanching treatment reduced the heating time to 10 seconds to obtain almost comparable results by dry heating treatment. Blanching treatment to dry seeds was simple, economical and adaptable for both domestic as well as large scale processing.

Effect of Storage Temperature

Storage temperature exhibited a significant impact on meal lipids. Hydrolytic and oxidative stability of pearl millet flour lipid was found to be greatly influenced by storage temperature. The lipids in the meal stored at ambient temperature were found to undergo a rapid hydrolytic breakdown during storage as evidenced by significant increased in acid value and % free fatty acid content in the extracted fat (Kadlag *et al*., 1993) [3]. Lipolytic deterioration measured in terms of peroxide value, %free fatty acid and acid value.

Peroxide value was found to be 2-3 folds lower in the meal stored under refrigerated conditions (4-7 ^{0}C) compared to the flour stored at ambient conditions (32±3 ^{0}C) as shown in Figure 1 & 2. Peroxide values of the blanched samples at refrigerated temperature were found to be 5.505, 2.804 and 1.690 at 0, 7 and 35 day respectively. On the other hand, the peroxide values of the blanched samples at room temperature were found to be 6.454, 3.375 and 2.683 at 0, 7 and 35 day respectively as shown in Table 1. Similarly, the peroxide values of the dry heated sample at refrigerated temperature were 10.128, 9.324 and 3.689 at 0,7 and 35 day, respectively, as compared to the peroxide of flour at room temperature which was found to be 10.423,9.663 and 5.000 at 0, 7 and 35 days, respectively. These values indicated that peroxide value got lowered significantly by 3-4 folds in refrigerated conditions as compared to the meal stored at room conditions.

The acid value was about 3 folds lower and the rate of increase in these values were also significantly lower for the samples stored in refrigerated conditions as compared to the samples stored at ambient conditions as shown in Figure 3 & 4. The acid value of the blanched samples at refrigerated temperature was 0.964, 2.871 and 7.701 at 0, 7 and 35 days, respectively. While at room temperature the values were found to be 0.856, 4.037 and 14.288 at 0, 7 and 35 days, respectively. Also, the acid value for dry heated sample at refrigerated condition was 1.931, 2.193 and 6,554 at 0, 7 and 35 day respectively. On the other hand, the values at room temperature were 1.991, 2.312 and 11.654 at 0, 7 and 35 days, respectively as shown in Table 2. These values indicated that rate of increase in acid value was almost double in case of sample that was kept at room temperature as compared to sample kept under refrigerated conditions.

The production of the free fatty acids is enhanced by the presence of high temperature. The values of % free fatty acids of blanched sample at refrigerated conditions were found to be 0.664, 2.009 and 5.391 at 0, 7 and 35 days, respectively. However, on the other hand, the production of free fatty acids at room temperature was 0.599, 2.826 and 10.001 at 0, 7 and 35 days, respectively. Similarly, the values of % free fatty acids of dry heated sample at refrigerated conditions were found to be 1.351, 1.535 and 4.588 at 0, 7 and 35 days, respectively. However, the production of free fatty acids at room temperature was 1.394, 1.618 and 8.158 at 0, 7 and 35 days, respectively. But in the untreated sample stored at refrigerated condition the production of free fatty acids was lower, 1.265 at 0 day and 9.152 at 35 day. On the other hand, for the sample stored at room temperature the production of free fatty acids was found to be 1.095 at 0 day and 19.642 at 35 day, which was significantly higher as shown in Table 3 and Figure 5 & Figure 6. These values signified that temperature has great influenced in the keeping quality of pearl millet flour. This behavior is because of the fact that rate of lipolytic breakdown is slower at low temperatures of storage (Kadlag *et al*., 1993) [3]

Sensory Evaluation of Chapattis made from Pearl Millet Flour: Chapattis were made from untreated, blanched and dry heated samples stored at 0 days and 7 days. The chapattis were analyzed by a panel of 5 judges. Chapattis were rated on hedonic scale from 1 to 9 points, where, 1 was for extremely disliked samples and 9 was for the extremely

liked samples.

The data of sensory analysis revealed that, not much significant difference existed in organoleptic properties (color, odor & taste) among the chapattis made from differentially treated flours at zero days of storage. The consumers preferred roasted flavor and sweet taste for chapattis and these chapattis scored more than 7 points on hedonic scale and were considered quite acceptable and desirable. The chapattis made from the control sample showed acceptable flavor and taste only upto a week of meal storage whereas, the shelf life of the chapattis of the treated samples was for more than a week. Chapattis made from all the samples stored for 7 days did not show any noticeable difference in color, however, the odor and taste of the chapattis made from untreated sample were found to be unacceptable due to enzymatic degradation favored by higher moisture content and not because of oxidative rancidity while those of the chapattis made from blanched and dry heated samples were quite acceptable as their moisture content was quite low for enzymatic degradation to take place (Hoseney *et al*., 1986) [2].

Table 1: Peroxide values of pearl millet flour samples (expressed in meq/100 g fat)

Samples	Days					
	0	7	14	21	28	35
Control RT	1.960	13.375	7.460	5.489	4.489	3.585
Control RFT	1.839	3.670	5.510	7.468	5.230	4.013
Blanched RT	6.454	3.375	4.744	4.049	2.652	2.683
Blanched RFT	5.505	2.804	3.759	2.845	2.397	1.690
Dry Heating RT	10.423	9.663	9.297	7.881	6.483	5.000
Dry Heating RFT	10.128	9.324	7.710	5.855	4.789	3.689

NOTE:
RT : Room Temperature (32±3^{0}C)
RFT: Refrigeration Temperature (4-7^{0}C)

Table 2: Acid values of pearl millet flour samples (Expressed in mg NaOH/g oil)

Samples	Days					
	0	7	14	21	28	35
Control RT	1.565	7.335	11.632	16.941	22.289	28.060
Control RFT	1.807	3.884	5.886	7.778	11.275	13.070
Blanched RT	0.856	4.037	6.025	8.344	11.310	14.288
Blanched RFT	0.964	2.871	3.351	3.866	5.416	7.701
Dry Heating RT	1.991	2.312	4.896	6.960	9.615	11.654
Dry Heating RFT	1.931	2.193	2.904	3.170	4.172	6.554

NOTE: RT : Room Temperature (32±3 ^{0}C)
RFT : Refrigeration Temperature (4-7^{0}C)

Table 3: % free fatty acid content of pearl millet flour samples (Expressed in terms of linoleic acid)

Samples	Days					
	0	7	14	21	28	35
Control RT	1.095	5.134	8.142	11.859	15.581	19.642
Control RFT	1.265	2.815	4.120	5.445	7.892	9.152
Blanched RT	0.599	2.826	4.217	5.841	7.988	10.001
Blanched RFT	0.664	2.009	2.346	2.678	3.791	5.391
Dry Heating RT	1.394	1.618	3.428	4.858	6.730	8.158
Dry Heating RFT	1.351	1.535	2.031	2.219	2.925	4.588

NOTE: RT : Room Temperature (32±3 ^{0}C)
RFT : Refrigeration Temperature (4-7^{0}C)

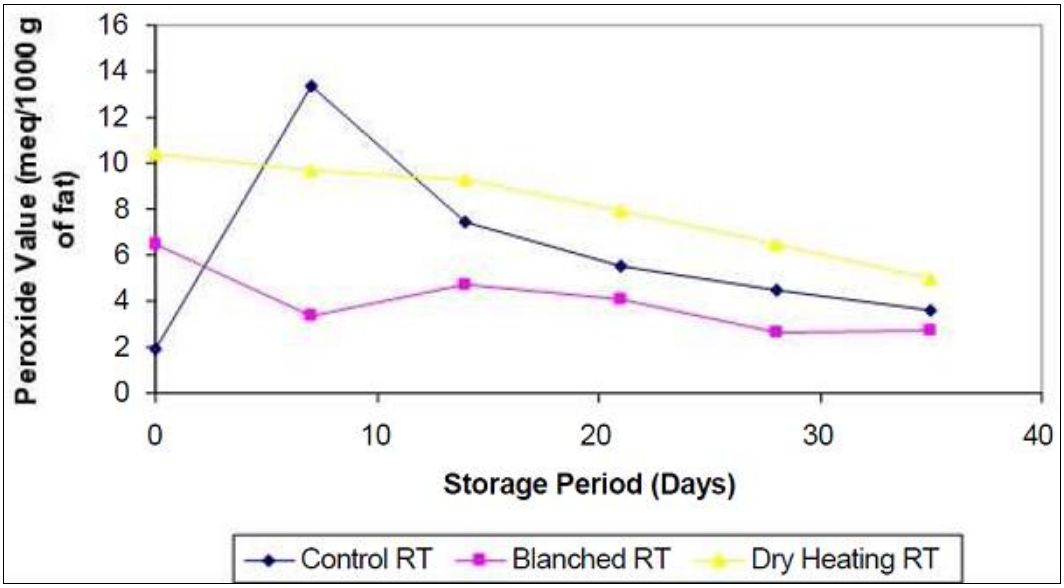

Fig 1: Effect of thermal treatment of peroxide value of sample stored under ambient conditions

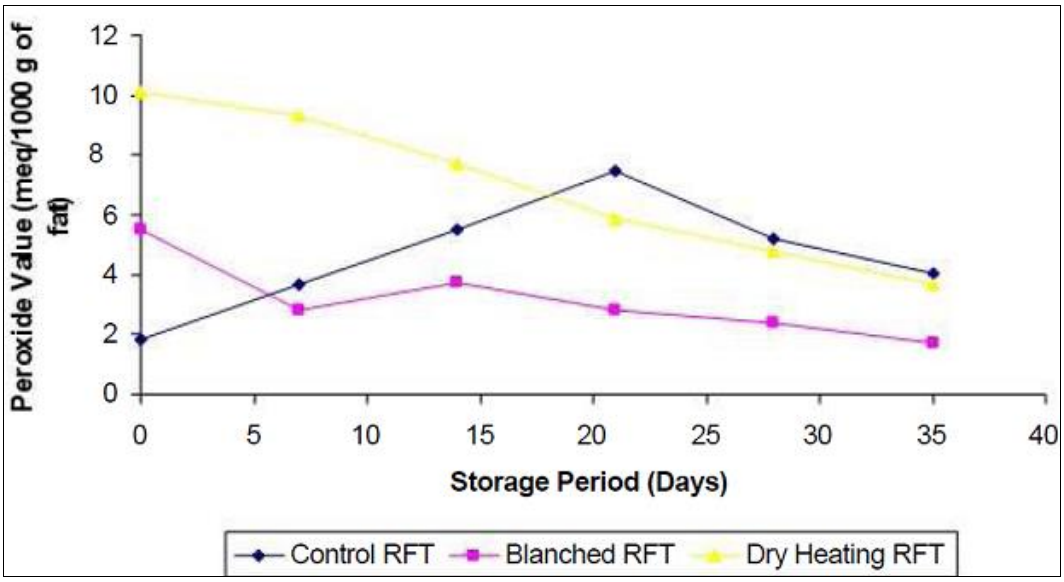

Fig 2: Effect of thermal treatments of peroxide value of sample stored under refrigerated conditions

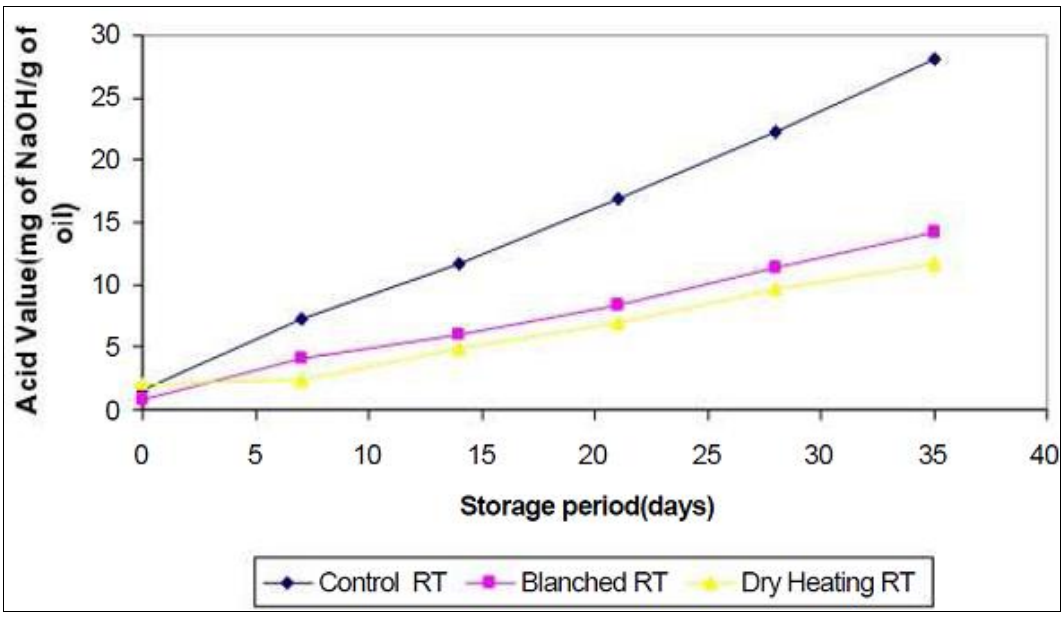

Fig 3: Effect of thermal treatment on acid value of sample stored under ambient conditions

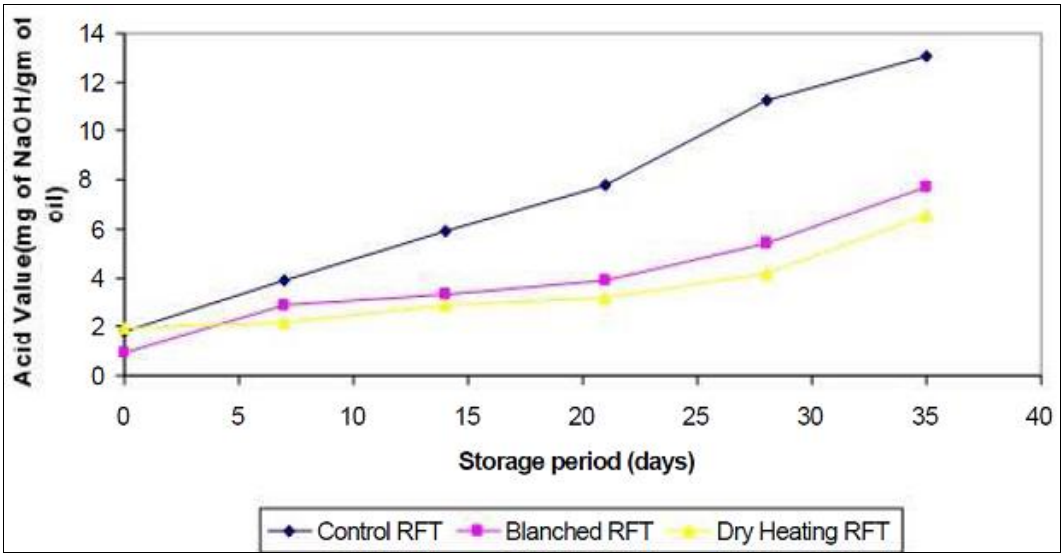

Fig 4: Effect of thermal treatments on acid value of sample stored under refrigerated conditions

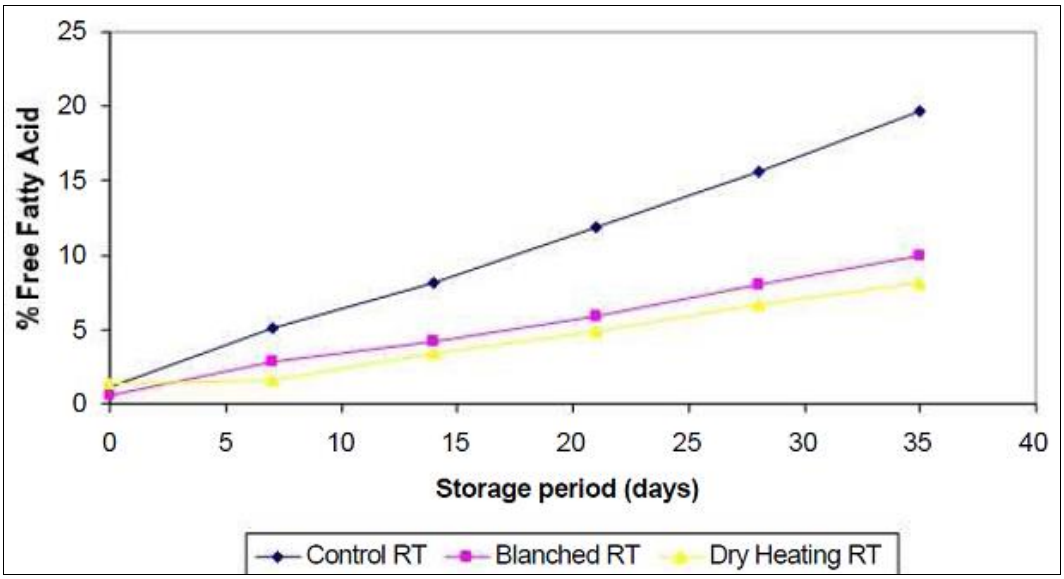

Fig 5: Effect of thermal treatments of % free fatty acid content of sample stored under ambient condition

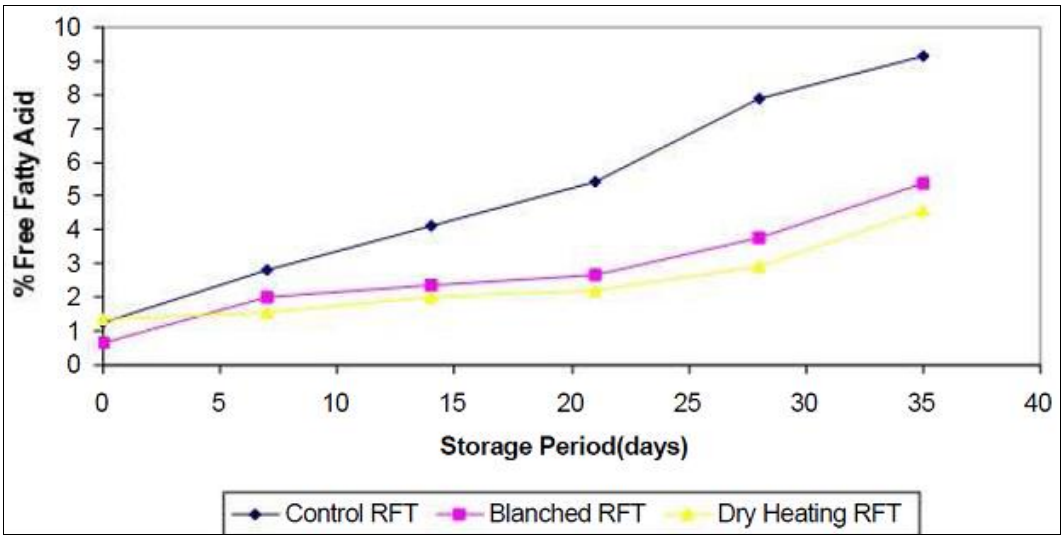

Fig 6: Effect of thermal treatments on % free fatty acid content of sample stored under refrigerated conditions

Conclusion

The results of the present study revealed that the storage of pearl millet flour under studied conditions resulted in rapid alterations in lipid components which increased with the increase in storage period. In the untreated sample, a rapid increase in acid value and % free fatty acid content of the

flour during storage can be attributed to the action of lipases located in the germ and on the surface layers of the seeds, which get mixed with the meal during milling and the meal lipids get decomposed into free fatty acid. Blanching and dry heating treatment were able to retard both hydrolytic and oxidative decomposition as was evident from lower values of acid values, % free fatty acid content and peroxide value of the sample. Thus, of all the treatment dry heating treatment was found to be the best followed by blanching in retarding the lipid degradation because it causes the acid value and %free fatty acid content to increase at slower rates than the other treatments. The application of dry heat to the grains effectively retarded the lipase activity and minimized lipid decomposition during storage. It is more convenient to heat seeds than meal. Such a heat treatment needs to be mild to avoid detrimental effects on other nutrients in the seeds. The use of hot water blanching treatment reduced the heating time to 10 seconds to obtain almost comparable results by dry heating treatment. Blanching treatment to dry seeds was simple, economical and adaptable for both domestic as well as large scale processing.

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