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Effect of different relative humidities on the oil extracted from stored cottonseed

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RESUMEN

Efecto de la humedad relativa de la semilla de algodón almacenada sobre el aceite extraído

Se ha estudiado el efecto de diferentes humedades relativas (ambiente, 10%, 46% y 97%) en aceites extraídos de semilla de algodón almacenada durante un año. Se utilizó como control el aceite extraído de semilla de algodón reciente no almacenada.

Se determinó la riqueza grasa y humedad en la semilla y el índice de acidez, índice de peróxidos y el porcentaje de gosipol en el aceite. Los resultados obtenidos para el aceite extraído a partir de la semilla almacenada bajo las condiciones de humedades relativas (ambiente, 10% y 46%) mostraron una ligera diferencia con los resultados obtenidos para el aceite extraído a partir de semilla de algodón no almacenada, mientras los resultados del aceite extraído de semilla de algodón almacenada al 97% de humedad relativa mostraron una diferencia considerable.

La composición en ácidos grasos y la relación ácidos grasos insaturados: saturados fue también evaluada en los aceites anteriores. Los ácidos grasos insaturados disminuyeron con el almacenamiento. Por consiguiente la relación ácidos grasos insaturados: saturados también disminuyó.

Se investigó también el efecto de la humedad relativa durante el almacenamiento de la semilla de algodón sobre la refinación y decoloración del aceite. El aceite de semilla de algodón almacenado bajo las condiciones de humedades relativas (ambiente, 10% y 46%) fue refinado y decolorado satisfactoriamente, dando aceites con buen color.

Mientras el aceite de semilla de algodón almacenado al 97% de humedad relativa no pudo ser refinado y decolorado satisfactoriamente. El aceite resultó de color muy oscuro y no respondió a la refinación y decoloración.

PALABRAS-CLAVE: Aceite – Almacenamiento – Decoloración – Humedad relativa (efecto de) – Refinación – Semilla de algodón.

SUMMARY

Effect of different relative humidities on the oil extracted from stored cottonseed

Effect of different relative humidities (room, 10%, 46% and 97% R. H.) on the oil extracted from cottonseed stored for one year was studied. The oil extracted from recent non-stored cottonseed was used as a control.

Moisture content and oil content in the seed and acid value, peroxide value and percentage gossypol in the oil were determined. The results obtained for the oil extracted from the seed stored under (room, 10% and 46% R. H.) showed a slight difference with the results obtained for the oil extracted from the non-stored conttonseed, while the results of the oil extracted from the seed stored under 97% R. H. showed a considerable difference.

Fatty acids composition and unsaturated: saturated fatty acids ratio (U:S) were also evaluated in previous oils. Unsaturated fatty acids were decreased by storage. Accordingly U:S was also decreased.

Effect of relative humidity during storage of the cottonseeds on refining and bleaching of the oil was also investigated. The oil of the cottonseed stored under (room, 10% and 46% R. H.) was satisfactorily refined and bleached, givining oils with good colour. While the oil of the cottonseed stored under 97% R. H. could not be satisfactorily refined and bleached. The oil was very dark in colour and did not response to refining and bleaching.

KEY-WORDS: Bleaching – Cottonseed – Oil – Refining – Relative humidity (effect of) –Sorage.

1. INTRODUCTION

Cottonseed from the time of its formation in the boll to the time of its processing in the mill is subjected to some types of damage. This damage affect the seed constituents e.g. the oil. Storage damage of cottonseed involves different enviromental factors. The primary cause of deterioration is the activity of enzymes within the seed, but as a deterioration progresses, the seeds probably become increasingly susceptible to infection by microorganisms (Bailey 1948). Damage can also be occurred in immature cottonseed and accordingly in its (Helmy *et al.,* 1994). Cottonseed meats stored for different lengths of time at various moisture levels were studied (Zaher *et al.,* 1988). Storage at high moisture contents or at high temperatures promotes the formation of free fatty acid (St. Angelo and Altshal 1964, Harris and Wamble 1967).

This study aims to investigate the effect of different relative humidities on some constituents and on refining and bleaching of the oil extracted from stored cottonseed.

Previous purpose is important to limit the proper relative humidity that should be used during ship transportation and storage of seed before processing.

2. EXPERIMENTAL

Experimental was carried out as follows: Cottonseed (*Gossypium barbadense)* used in the present study was kindly supplied by the Cottonseed Research Institute of the Ministry of Agriculture. Cottonseed was divided into four portions, one kilogram each. The portions were packed in plastic nets. The four net packages of seeds were kept for one year under room, 10%, 46% and 97% relative humidity (R. H.). All samples were stored under room temperature.

The following was the treatments carried out on the samples: First sample (C_1) was the sample kept at room relative humidity. Second sample (C_2) was kept in a 5-litre closed glass container. The container was divided horizontally into two parts by a perforated disc. In the upper part, the seed package was placed over the disc. In the lower part, a super saturated salt solution of zinc chloride was set. This gave 10% relative humidity around the seed.

Third sample (C_3) was treated as the second sample except that, the super saturated salt solution used was potassium carbonate. It produced 46% relative humidity around the seed.

Fourth sample (C_4) was treated as C_2 and C_3 except using a super saturated salt solution of potassium sulphate to obtain 97% relative humidity around the seed.

The control sample (CS) was a sample of the cottonseed used without storage.

Relative humidity was controlled according to Buxton and Mellanby 1954.

Extraction of Cottonseed Oil: Oil was extracted from cottonseed after crushing and milling with commercial hexane in a Soxhlet apparatus. Samples were then desolventized under vacuum using a rotary evaporator.

Refining and Bleaching of Oil: Crude oil were refined and bleached according to the AOCS Official Methods 1980.

Refining: Fifty gm each of crude oils were placed in 100 ml beaker and heated to 60° C. The calculated amount of NaOH solution (18° Bé) was added to crude oil during stirring for 15 min., centrifuged at 3000 rpm, washed with warm water, recentrifuged, then decanted.

Bleaching: Fourty gm each of the refined oils was taken in 100 ml beaker and heated in an oil bath at 110° C. Bleaching earth «Thonsil» was added at 3% of the oil weight while stirring for 10 min. The clay was separated by centrifugation after cooling and the oil was recovered by decantation.

Spectrophotometric Analysis: Absorption spectra of crude, refined and bleached oils was measured in a Shimadzu UV-Visible Spectrophotometer, Model UV-240 Graphtcord (Tokyo, Japan). Wavelength range from 300-700 nm was used.

Moisture % and oil % in the cottonseed and acid value and peroxide value in the cottonseed oil were determined according to the AOCS Official Methods of Analysis 1980.

Gossypol % in the cottonseed oil was determined according to Pons *et al.* 1956.

Fatty Acids Composition: The samples were esterified by refluxing with methanol containing 3% concentrated sulphuric acid as described by Luddy *et al.* (1960). The formed fatty acid methyl esters were then extracted with diethyl ether in a separatory funnel, concentrated and kept in a stoppered bottles in a refrigerator till G. L. C. analysis.

Esterified samples were then analyzed using G. L.C. apparatus model Varian 3700.

The column was 12 feet long and 0.4 mm diameter. It was packed with 10% polyethylene glycol succinate on chromosorb W-HB. Programming temperature was from 70 to 90° C. Nitrogen was used as a carrier gas a flow rate of 30 ml/min. A flame ionization detector, was used in detection. The resulted peaks were compared with the retention time of authentic standard fatty acids. Percentage fatty acids were calculated in apparatus automatically according to the area of each fatty acid to the area of total fatty acids of sample.

3. RESULTS AND DISCUSSION

Results of the effect of different relative humidities (room, 10%, 46% and 97% R. H.) on the oil extracted from cottonseed stored for one year are illustrated in the Table I.

Table I

Changes in some constituents of the oil of cottonseed stored under room, 10%, 46% and 97% respectively, compared with the oil of non-stored cottonseed CS

Constituent	Room	10%	46%	97%	CS
% Moisture in seed	6.8	7.1	7.8	37.3	5.2
% Oil content	31.7	27.6	28.9	0.8	33.2
Acid value	8.4	6.8	18.0	44.0	5.6
Peroxide value	1.5	0.9	2.8	30.1	0.1
% Gossypol in oil	0.12	0.04	0.07	0.05	0.21

It can be noticed from the results obtained that storage of the seed under high relative humidity (97%) led to a very high increase in moisture content, where it increased from 5.2% in the non-stored cottonseed to 37.3% in the sample stored under 97% R. H. Whereas the samples stored under room, 10% and 46% R. H. showed a slight increase in their moisture content, it was 6.8%, 7.1% and 7.8% respectively.

As it will be seen, the high increase in moisture content of the seed led to undesirable changes in its oil.

Oil content decreased tremendously from 33.2% in the non-stored seed to 0.8% in the sample stored under 97% R. H. This might be due to a hydrolysis occurred in the oil as a result to high increase in moisture content and to activation of hydrolytic enzymes. On contrary, oil content decreased slightly to 31.7%, 27.6% and 28.9% in the seed stored under room, 10%, 46% and 97% R. H.

Acid value increased severely from 5.6 in the oil of non-stored cottonseed to 44° in the sample stored under 97% R. H., this might be due to hydrolysis occurred in the ester linkage of the oil glycerides, followed with liberation of free fatty acids. Increase in free fatty acids led to increase in refining loss, where they are transferred to soap by neutralization with alkali at refining step. Otherwise, the seeds stored under room and 10% R. H. gave oils with acid value near the non-stored sample (8.4 and 6.8 respectively), and noticeably higher in the sample stored under 46% R. H. (A.V. 18.0).

Peroxide value of the oil of the cottonseed stored under 97% R. H. was 30.1, against 0.1 for the non-stored sample. Peroxide value in the oil of the seed stored under room, 10% and 46% R. H. were 1.5, 0.9 and 2.8. The remarkable increase in the P. V. of oil of the sample stored under 97% R. H. might be due to increase in activation of oxidizing enzymes in the seed as a result to increase in moisture content of the seed and/or to a destroy occurred in natural antioxidants.

Percentage of gossypol in oil decreased from 0.21% in the oil of non-stored cottonseed to 0.12, 0.04, 0.07 and 0.05% in the oil of the cottonseed stored under room, 10%, 46% and 97% R. H. respectively. Decrease in gossypol might be attributed to a dissociation taken place in the pigment.

Fatty acids composition of the oil of the seed stored for one year under different relative humidities were also studied. Results was shown in Table II. Results revealed that:

– Total unsaturated fatty acids decreased from 70.6% in the oil of recent non-stored seed to 59.9, 63.5, 57.4 and 47.3% in the oil of the seed stored for one year under room, 10, 46 and 97% relative humidity (R. H.) respectively.

The decrease may due to the degradation occurred in insaturated fatty acids. The major unsaturated fatty acid was linoleic acid. It decreased considerably in the oil when the seeds were exposed to storage especially in the oil of the seed stored under 97% R. H., where it decreased from 47.1% in the oil of the recent non-stored seed to 27.8% in the oil of the seed stored under 97% R. H.

The second major unsaturated fatty acid was oleic acid, it was not significantly changed. It ranged between 19.2 and 22.5% in the oil of stored seed against 22.6% in the oil of non-stored seed. Previous results indicate that the main degradation and/or oxidation occurred in the highly unsaturated fatty acid i. e. linoleic acid here than the monounsaturated fatty acid.

Table II

Fatty acids (FA) composition of the oils extracted from cottonseeds stored one year under different relative humidities (RH) compared with recent non-stored cottonseed (CS)

FA	CS	Room (RH)	10% (RH)	46% (RH)	97% (RH)
Saturated: S					
$C_{12:0}$	–	–	0.1	0.3	–
$C_{14:0}$	0.6	1.1	1.0	1.4	1.9
$C_{15:0}$	–	2.0	1.3	2.6	2.5
$C_{16:0}$	26.7	29.1	28.2	29.3	38.5
$C_{17:0}$	–	3.3	2.7	4.0	3.7
$C_{18:0}$	2.0	4.6	4.1	4.8	6.1
$C_{20:0}$	0.1	–	0.1	0.2	–
Total	29.4	40.1	37.5	42.6	52.7
Unsaturated: U					
$C_{16:1}$	0.7	2.2	1.9	2.8	0.3
$C_{18:1}$	22.6	21.0	22.5	22.0	19.2
$C_{18:2}$	47.1	36.6	39.1	32.5	27.8
$C_{18:3}$	0.2	0.1	–	0.1	–
Total	70.6	59.9	63.5	57.4	47.3
U:S	2.4	1.5	1.7	1.4	0.9

– Total saturated fatty acids increased from 29.4% in the oil of non-stored cottonseed to 40.1, 37.5, 42.6 and 52.7% in the oil of the cottonseed stored under room, 10%, 46% and 97% R. H. respectively. The main saturated fatty acid in the oil of non-stored and stored cottonseed was palmitic acid. Fatty acids with 15 and 17 carbon atoms appeared also, they did'nt exceed 4% of total fatty acids. Appearance of these fatty acids may due to the activity of degradative enzymes during storage. These fatty acids did'nt appear in the oil of non-stored seed. Stearic acid increased from 2.0% in the oil of non-stored seed to 4.1-6.1 in the oil of stored seed. Increase of total saturated fatty acids is here due to the decrease occurred in the unsaturated fatty acids as a result to oxidation.

– Ratio of total unsaturated fatty acids to total saturated fatty acids (U:S) was calculated owing to its importance from the nutritional point of view. Increase in this value means increase in unsaturated fatty acids and essential fatty acids accordingly.

However U:S ratio was 2.4 in the oil of recent non-stored cottonseed. This ratio decreased in the oils of stored cottonseed to 0.9-1.7, owing to the decrease occurred in the unsaturated fatty acids.

Effect of different relative humidities (room, 10%, 46% and 97% R. H.) on refining and bleaching of the oil extracted from the seed stored for one year were illustrated in figures 1-3.

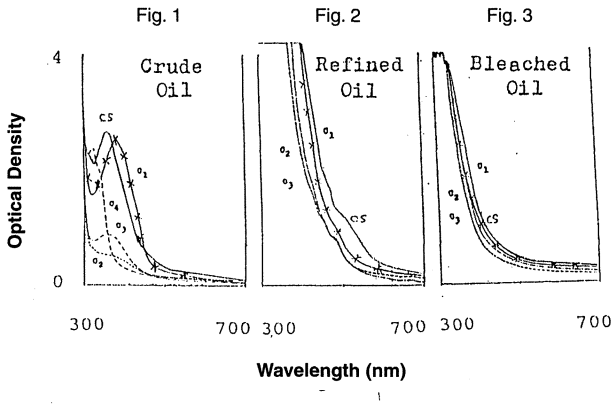

Figure 1, 2 and 3
Absorption spectra of the oil (C_1, C_2, C_3, C_4) extracted from the cottonseed stored under room 10%, 46% and 97% relative humidity respectively, compared with the oil of non-stored cottonseed (CS).

Figure 1 showed the absorption spectra of crude oils. The absorption spectrum of crude oil of the cottonseed stored at room relative humidity (sample C_1) showed absorption maxima of gossypol at 380nm. It has been noticed that the absorption maxima of the pigment shifted from 360nm in the oil of non-stored cottonseed CS, to 380nm in the oil of the sample C_1. This might be attributed to the effect of storage on gossypol. The absorption spectra of gossypol in oil decreased in the other two samples C_2 and C_3 (samples stored under 10% and 46% R. H.). Whereas absorption spectra of the sample stored under 97% R. H. (sample C_4) showed a small peak at 323nm and disappearance of gossypol, which might be attributed to formation of another compound from gossypol as a result to the increase occurred in moisture content of the seed, which accordingly caused rupturing in pigment glands and changing in gossypol of these glands.

Refining removed gossypol from the crude oil of the samples C_1, C_2, and C_3 (Fig. 2), whereas carotenoids appeared in minor humps in the region of 430 to 480 nm. Absorption spectra of the oil of sample CS remained higher than the absorption spectra of the oil of sample C_1, C_2 and C_3. Oil of sample C_4 was excluded from refining and bleaching, because it was unsuitable for these processes.

Although bleaching removed carotenoids from all the refined oil samples C_1, C_2, C_3 and CS, absorption spectra of the bleached oil of sample C_1 was higher than the absorption spectra of the processes.

Although bleaching removed carotenoids from all the refined oil samples C_1, C_2, C_3 and CS, absorption spectra of the bleached oil of samle C_1 was higher than the absorption spectra of the other oils (figure 3).

Results obtained previously, proved that relative humidity has an important effect on seed quality and accordingly on the oil extracted there from.

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