

# The Effect of Dielectric Heating on Storage Quality of Cottonseed

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THE deterioration and resulting accumulation of free fatty acids in moist cottonseed during storage has been a major problem in the cottonseed industry for many years. Evidence that the basic cause lies in the metabolic processes which take place in the seed as a result of enzyme action has been presented by Altschul *et al.* (1). Bacteria appear to play only a minor role.

Various procedures have been employed in the mills to reduce losses from excessive formation of free fatty acids. Moist seed which are rapidly forming free fatty acids are often seed which become hot due to metabolic activity. The high temperatures attained result in extensive damage. A few mills are equipped for pre-drying before storage. The hard seed coat and the lint around it which serves as a good insulator make the practical application of ordinary drying systems to cottonseed rather difficult. A second way of attacking the problem and one which is in more general use is to blow or to suck air through the seed in the warehouse. This procedure has the double action of cooling the seed and of eliminating excess moisture. Altschul and coworkers (2, 3) have studied the use of chemicals for blocking the enzymes and hence preventing the formation of free fatty acids in the stored seed.

As a result of extensive research during the war period, the application of radio frequency electric power has found many industrial uses. Among these may be listed: the sterilization of packaged cereals (4), the processing of fruits and vegetables (5, 6), dehydration of hygroscopic materials (4), the bonding of plywood (4, 7), the plastic lamination of paper, asbestos, felt, cotton, etc. (4, 8), and the heating of material for plastic moulding (9).

When material which is a poor electrical conductor is placed between two metal plates and electric power of radio frequency is applied, the rapid reversal of electric charge results in molecular friction of the material. The dissipation of electric power in this manner results in the heating of the material; and the process is generally referred to as dielectric heating. An important characteristic of dielectric heating is that the heat is generated inside of the material. With most other types of heating the temperature of the inner material can only be raised by thermal conduction from the outside. Another important characteristic of dielectric heating is the evenness and extreme rapidity with which the temperature can be raised.

The purpose of the present investigation was to test the effectiveness of dielectric heating in destroying the enzymes of cottonseed and in this manner preventing the formation of free fatty acids during the storage of the moist seed. The rapid destruction of enzymes by radio frequency electric power has already been demonstrated by the adaptation of electronic heating to the blanching of vegetables by

Moyer and Stotz (6, 10). A study of the application of dielectric heating to the processing of cottonseed was made by Olschner (11).

## Experimental

*Dielectric Heating Equipment.* The dielectric heating unit used was a commercial machine intended for small industrial applications. The rated output of this machine was one kilowatt at a frequency of 14 megacycles per second. The unit consisted of a high frequency vacuum tube oscillator and a direct current rectifier type power supply which operated from a 110-volt A.C. line.

*Procedure for Treating and Testing Cottonseed.* Prime cottonseed were brought to the desired moisture content by adding the necessary amount of water with a spray. The seed were then placed in a sealed drum leaving enough space so that efficient mixing could be attained by rolling the drum. The seed were mixed in this manner each day for a week before the tests were started.

The seed to be subjected to the high frequency electric field were placed between two circular metal plates which served as the electrodes of the apparatus. These plates were 4.5 inches in diameter and were spaced 3 inches apart. Two hundred and twenty grams of seed completely filled the space between the electrodes. A perforated paper cylinder was used to hold the seed in position. Three sheets of filter paper were placed between the seed and each plate to absorb moisture that condensed on the cool plates from vapors coming from the seed. Each sample of seed consisted of 660 grams which was treated in three equal parts and then mixed before the storage period.

After the dielectric heating treatment the moisture content of the seed was determined. In some of the tests the samples were divided in half and water added with a spray to one half in order to readjust the moisture content to its original level. The samples were sealed in fruit jars and stored in an incubator maintained at 38.5°C. by thermostatic control. Instead of room temperature 38.5°C. was chosen in order to speed up the formation of free fatty acid and to make the test more rigorous. Free fatty acids in the seed were determined after the storage period by the method of the National Cottonseed Products Association.

*Determination of Temperature Inside of the Seed.* A copper-constantan thermocouple, made of No. 28 B. & S. gauge wire, was placed in a small hole drilled through the center of a seed. The drill size was so selected that the wire almost filled the hole. This seed was placed between the plates of the apparatus in the center of a 220-gram sample. To prevent high frequency current from arcing to the leads, they were brought out through small glass tubes arranged in a plane parallel to the metal plates and extending several inches beyond the wall of the perforated paper container. These tubes tapered to an opening just large enough to allow the wires to pass out the

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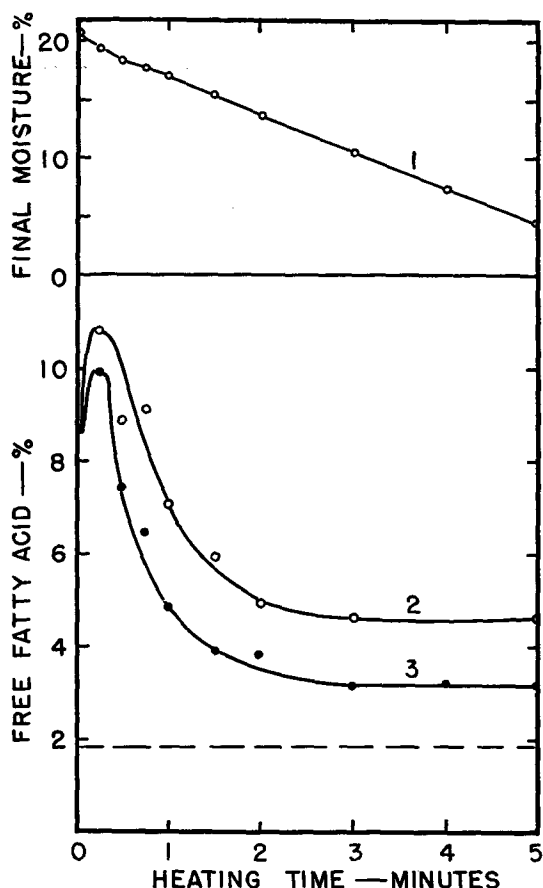


FIG. 1. Dielectric heating experiments with cottonseed containing 20 per cent moisture.

Curve 1, Moisture content; 2, Free fatty acid content after storage for 51 days at 38.5°C. Moisture content of each sample readjusted to 20 per cent before the storage period; 3, Same as 2, except that the moisture content was not readjusted.

end as they entered the seed. Clips were arranged so that the indicating millivoltmeter could be connected to the leads immediately after removing the high frequency power. It was necessary to use this procedure as the capacity between the meter and ground was sufficient to distort the field in the vicinity of the thermocouple and cause an arc through the seeds.

To obtain data for the temperature-time curve the power was applied for one-half minute, then removed for a few seconds while a temperature reading was taken. The power was again applied for a half minute before being removed for the next temperature reading. This was continued until the full six minutes of heating time was completed. It was found by observation that the temperature drop during the short period required for taking a reading was almost negligible. The temperature curve obtained in this way should represent very nearly the temperature obtained by a continuous heating period.

#### Results and Discussion

The results of a typical series of tests given in Table I show that the dielectric heating treatment was very effective in preventing the formation of free fatty acids. The seeds which were treated for five minutes before storage contained only about one-fifth as much free fatty acids as the untreated seed. The free fatty acid content of the seed used in these tests was 1.5% at the time the experiments were started.

It will be noticed that treatments lasting only one minute or less actually increased the formation of free fatty acids. Since this type of result was obtained in repeated tests, it appears that dosages of radio frequency power less than necessary to destroy the enzymes actually stimulate enzyme activity under certain conditions.

The blocking of free fatty acid formation shown in Table I is the result of two separate and distinct mechanisms. First, dielectric heating rapidly removes moisture from the seed, and it is well known that moisture is an important factor in the formation of free fatty acids in whole seed. Superimposed on this effect is the destruction of the enzymes. The data given in Table I gives no basis for evaluating the relative importance of the two mechanisms.

In order to obtain information concerning the significance of the enzyme destruction factor, experiments were carried out in which one-half of each sample of seed was rehumidified after the dielectric heating treatment but prior to the storage period. Water was added with a spray in the amount necessary to bring the moisture content back to the original value. Cottonseed containing 10, 16, and 20% moisture were used in these tests. The results are given in Figs. 1, 2, and 3. The dotted line represents the free fatty acid content of the seed at the beginning of the experiment. After the seed had been moistened and the free fatty acids determined, it was necessary to delay the dielectric heating treatment for

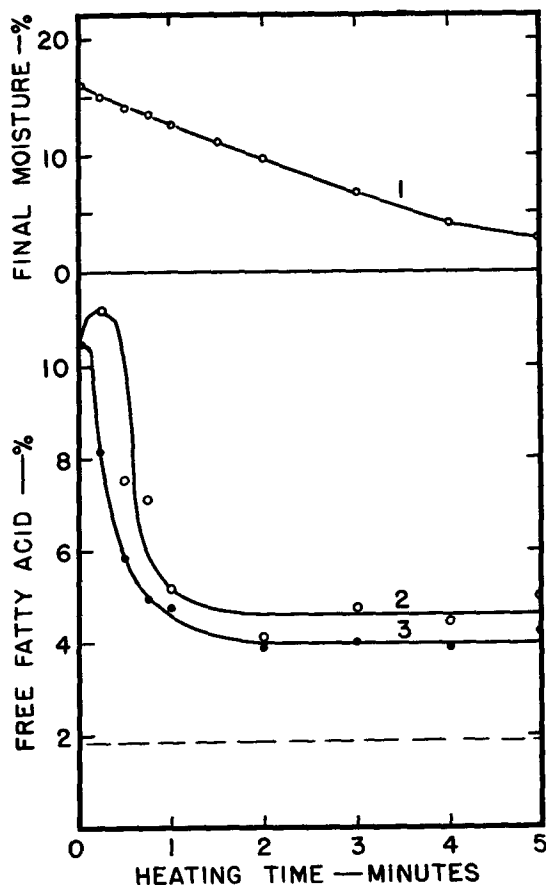


FIG. 2. Dielectric heating experiments with cottonseed containing 16 per cent moisture.

Curve 1, Moisture content; 2, Free fatty acid content after storage for 51 days at 38.5°C. Moisture content of each sample readjusted to 16 per cent before the storage period; 3, Same as 2, except that the moisture content was not readjusted.

TABLE I

Effect of Dielectric Heating on the Moisture Content and the Formation of Free Fatty Acids During the Storage of Cottonseed  
Storage time, 30 days; storage temperature, 38.5°C.

Dielectric heating time	Moisture content after heating	Free fatty acids after storage		
		Sample 1	Sample 2	Average
min.	per cent	per cent	per cent	per cent
0.....	20.62	15.63	15.64	15.64
0.5.....	19.18	20.12	20.43	20.28
1.....	17.82	18.58	20.11	19.35
2.....	14.47	4.88	4.70	4.79
3.....	9.59	4.72	4.78	4.75
4.....	10.48	4.47	4.62	4.55
5.....	4.59	3.08	3.37	3.23

two weeks. There may have been an appreciable increase in free fatty acids during this period.

The results emphasize the importance of the enzyme destruction factor and show that the formation of free fatty acids can be effectively reduced even though the seeds remain wet (20% moisture).

In considering the mechanism by which dielectric heating reduces the formation of free fatty acid, information concerning the temperatures produced by the treatment was desired. Since dielectric heating generates heat inside of the seed, a measurement of the temperature in the spaces between the seed would be of no particular significance. For this reason a procedure was worked out for determining the temperature inside of the seed. The results shown in Fig. 4 show that temperatures above the boiling point of water were reached in less than one minute. The

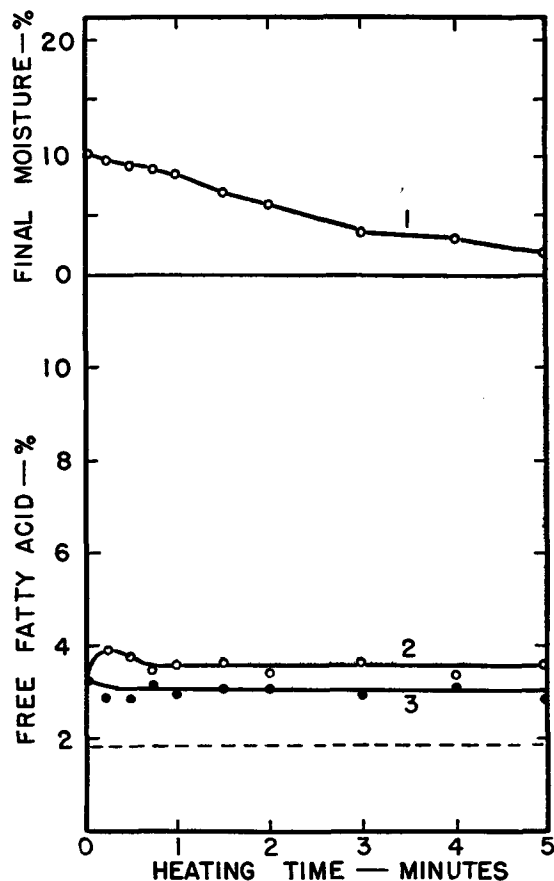


FIG. 3. Dielectric heating experiments with cottonseed containing 10 per cent moisture.

Curve 1, Moisture content; 2, Free fatty acid content after storage for 51 days at 38.5°C. Moisture content of each sample readjusted to 10 per cent before the storage period; 3, Same as 2, except that the moisture content was not readjusted.

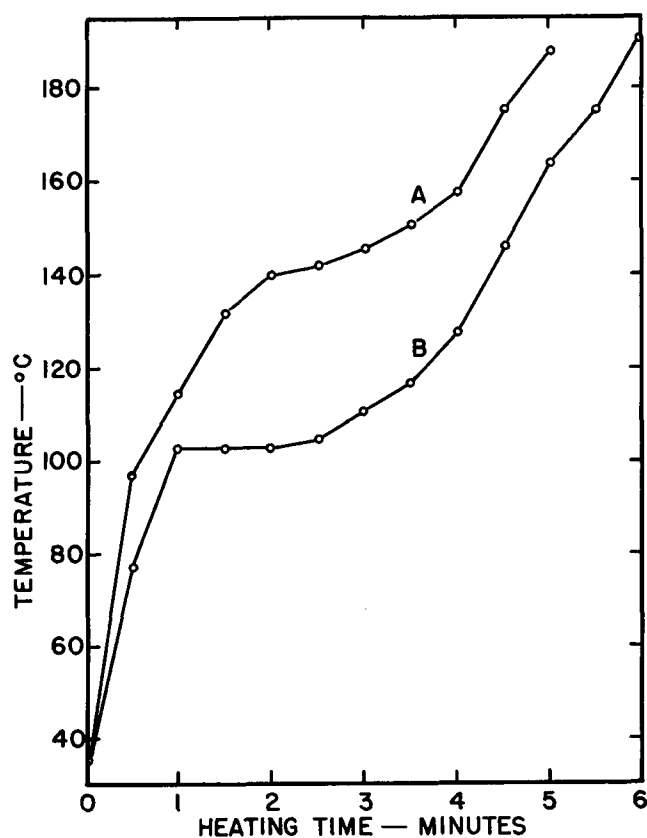


FIG. 4. The internal temperature of cottonseed subjected to dielectric heating. A, cottonseed containing 10 per cent moisture. B, cottonseed containing 20 per cent moisture.

flat portion of curve B obtained with seed containing 20% moisture is due to the heat required for the vaporization of water. After a considerable amount of moisture was lost (see Fig. 1) the temperature curve rises rapidly again. Although temperatures as high as 180°C. were reached in from four to five minutes, there was no indication that the seed were the least bit burned or scorched. Continuation of the treatment beyond five minutes did produce scorching.

It is not within the scope of this report to demonstrate the practicality of the industrial application of dielectric heating for the purpose indicated in this report. It would of course require only the simplest kind of adaptation to pass cottonseed continuously between two parallel plates. It is probable that the costs of installation and operation of dielectric heating equipment will have to be reduced before the process would be economically feasible. The numerous successful industrial applications of radio frequency electric power now in operation lead one to suspect that reductions in cost may be forthcoming.

#### Summary

It was found that the exposure of moist cottonseed to a radio frequency electric field for a period of from two to five minutes effectively reduced the formation of free fatty acids during subsequent storage of the seed. Two primary factors were involved in this action. First, moisture was rapidly removed from the seed and second, the enzymes responsible for free fatty acid formation were destroyed. Cottonseed containing 20% moisture accumulated free fatty acids only very slowly on storage at 38.5°C. after a brief dielectric heat treatment. The tem-

perature inside the cottonseed was measured during the dielectric heat treatment. Temperatures above 100°C. were attained in less than one minute.

#### Acknowledgment

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## Report of the Referee Board

FOR the year 1947-8 28 referee certificates were issued, including 26 renewals. The full list has been published in the *Journal of the American Oil Chemists' Society* except for the name of Dan L. Henry of Law and Company in Atlanta. There are also pending four new applications for appointment as referee chemist.

The only thing unusual in the experience of the Referee Board during the past year has been a large number of inquiries regarding new referee certificates and the fact that most of these are not resulting in actual filing of an application. Official analysis and grading of oil bearing seeds and derived products has become a very complicated subject. We have federal government supervision of seed grading, with a system for cottonseed which evolved from a system concerned with grading the cotton fiber, and with another system for soybeans which is in course of evolving from the grading system on grains. Then we have separate trade associations which appoint official chemists, each having its own routine for making appointments. Both the N.C.P.A. and the N.S.P.A. require official chemists to be referee chemists of our Society. Imagine the state of mind of a young chemist who first is urged by a soybean crusher to become an official chemist on soybean products, then writes to N.S.P.A. headquarters, is then referred to the chairman of the N.S.P.A. Technical Committee, is next referred to the chairman of the Referee Board of the A.O.C.S. (of which the young chemist may or may not be a member), and still faces the problem of dealing with the Department of Agriculture on beans even if he masters the intricacies of the A.O.C.S. and N.S.P.A. literature concerned with official analysis and grading of the oil and of meal.

The Referee Board is in a better position to observe than to correct this complicated situation. At present, we merely call it to the Society's attention.

G. W. AGEE	R. T. MILNER
J. P. HARRIS	A. S. RICHARDSON,
R. R. KING	chairman

## Report of the Cellulose Yield Committee, 1947-48

DURING the past year four sets of linter samples of three grades were sent out to 11 laboratories. One laboratory, number 4, reported only two sets of results so these results are omitted from the report. The average yield results for the three types of linters sent out are given below:

Lab. No.	No. Sets Samples Tested	Samples			Overall Average for Year
		A Linters	B Linters	C Fiber	
1.....	4	77.2	72.6	72.2	74.0
2.....	4	77.0	72.7	72.7	74.1
3.....	4	76.3	72.2	71.8	73.4
5.....	4	76.1	73.1	71.5	73.2
6.....	4	76.9	72.6	72.1	73.9
7.....	4	76.9	72.5	71.7	73.7
8.....	4	77.3	72.4	73.1	74.3
9.....	4	76.9	71.8	71.1	73.3
10.....	4	77.4	72.7	71.9	74.0
11.....	4	77.2	72.9	72.7	74.3
Average.....	....	76.9	72.5	72.1	73.8

As seen from the above table, the average results show very good agreement between laboratories.

#### Recommendations

It is recommended that samples be sent out to all laboratories which request the samples during the next year at least four times, as it has been shown that a frequent checking of the method and equipment is necessary for consistent agreement of results between laboratories.

E. C. AINSLIE	W. S. HUDE
M. G. BOULWARE	E. H. TENENT
C. H. COX	L. N. ROGERS,
	chairman

## Report of the Gossypol Analysis Committee, 1947-48

WHEN solvent extraction plants first began to process cottonseed in 1947, there was an urgent need for a quantitative analytical method for gossypol which would give concordant results when used by laboratory technicians and would require a minimum of time and cost. To meet these requirements this committee chose for study the established spectrophotometric method of F. H. Smith, *Industrial and Engineering Chemistry*, analytical edition, vol. 18, 43-45 (1946) and the modification, unpublished, proposed by W. T. Coleman. The latter uses a single mixture of isopropanol and water in place of the several ethanol mixtures required by the Smith method and is therefore simpler, quicker, and less expensive.

As a variety of photoelectric instruments were being used by the several committee members, it was considered advisable to have data on the instruments in use and the readings as found, as well as the translation of readings to per cent gossypol. An isopropanol extract of cottonseed meal and an aniline developed solution of the same were sent out to each committee member to be read on the same day. Re-