demineralizing water the exhausted, intimately mixed cation and anion resins are separated into layers by hydraulic classification and regenerated by appropriate treatment of each layer with solutions of acid or base, followed by rinsing and remixing (3).

In a catalytic process where considerable agitation is employed, attrition of the resins occurs, which probably prevents their successful re-classification for regeneration since their separation is governed by Stokes' law of settling. According to this law, the rate of settling of a spherical particle in a quiescent fluid medium is a function of the square of its diameter and of its density. The same principle applies to hydraulic classification. Therefore anion and cation resin particles, if equal or nearly equal in density and if the same size, should not be amenable to hydraulic classification. Even though cation and anion resins of a different size range were used initially, they would probably be inseparable or only partially separable after several re-uses on account of attrition.

In one experiment a mixture of anion and cation resins, recovered from the reaction mixture by centrifugation following a hydrolysis reaction, was washed on a Buchner funnel with acetone, followed by eight washings with distilled water. When these resins were used to catalyze a hydrolysis reaction without further treatment, 84% phosphorus removal from a nondegummed soybean oil was effected in 30 min. During this treatment the free fatty-acid content increased to 0.86%.

In another test the mixed resins were regenerated by washing with acetone followed by one wash of 50 ml. of one normal hydrochloric acid, three washes with distilled water, one wash with 25 ml. of one normal sodium hydroxide, and five washes with distilled water. The regenerated mixed resins catalyzed the hydrolysis of 71% of the phosphorus from nondegummed soybean oil in 30 min. at 121°C. The final free fatty-acid content of the oil was 0.19%.

A sample of the nondegummed soybean oil used in the tests and a sample of soybean oil recovered from a hydrolysis reaction, using mixed resins, were alkali-refined with 0.2% excess of 10% aqueous sodium hydroxide. The refined oils were analyzed for mono-, di-, and triglycerides by the adsorption method of Quinlan and Weiser (5). The refined oil contained 3.1% monoglycerides, 13.1% diglycerides, and 80.8% triglycerides while the hydrolyzed, refined oil contained 4.0% monoglycerides, 14.7% diglycerides, and 78.5% triglycerides. These data indicate that mono- and diglycerides are formed by the hydrolysis treatment although the unexpectedly high amounts of mono- and diglycerides in both samples may have been caused by oxidized glycerides which were adsorbed and eluted from the silica gel column together with the mono- and diglycerides.

Summary

A rapid method is described for treating nondegummed soybean oil with water and a mixture of cation and anion exchange resins at elevated temperatures. In less than 30 min. 85% of phosphatide phosphorus is converted to a water-soluble, petroleum ether-insoluble form. Very little free or combined fatty acids are associated with the phosphorus thus split off from the phosphatide, and only a moderate increase in free fatty acid in the oil phase occurs. It is proposed that the phosphatides are largely converted to diglycerides by the selective hydrolytic treatment.

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Storage of Cottonseed

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LOSSES on account of cottonseed deterioration during storage have been estimated at around $1 per ton. This figure may be considerably higher or lower in a given locality or during any specific season. Storage losses are caused primarily by two factors. a) Enzymes or microorganisms (1, 2) break down glycerides with the formation of free fatty acids and glycerol. This hydrolysis is accelerated in the presence of high moisture, high acid, or damaged seed. It is also accelerated by increased temperature up to the point where the biologically active agents are destroyed. b) Heat developed (from the biological activity) affects the seed pigments so that they are not satisfactorily removed during subsequent refining and bleaching.

Despite more than 50 years of commercial operation, in many respects cottonseed storage is still an "art," without sufficient scientific background. Some of the most interesting and intensive work done to date has yielded negative results in that no chemical inhibition of hydrolysis caused by enzymes or microorganisms has proven practical on a commercial scale. The inhibitors are either toxic or not sufficiently active (1).

Cottonseed is usually stored in seedhouses or silos. The seed houses may be of wood construction, but the modern types are made of steel with metal sides and roof. The roof is sloped at 45° to equal the angle of repose of seed. Cooling facilities are normally provided by large fans connected to ventilating ducts

TABLE I

Equilibrium Moisture-Points of Cottonseed at Various Relative Humidities

Material used as satd. sol.	% R.H. at 75°F.	Equil. moist.
CH_3COOK	20.0	5.5
$CaCl_2$	31.1	6.1
K_2CO_3	43.0	7.3
$Ca(NO_3)_2$	51.0	8.3
NaBr	62.0	8.8
$KNO_3 + NH_4Cl$	71.2	10.5
NaCl	75.0	11.5
$NaNO_2$	80.0	13.4
KBr	84.0	15.1
KCl	85.0	15.3
$BaCl_2 \cdot 2H_2O$	92.3	20.8
KNO_3	94.5	22.1
K_2SO_4	98.3	27.8
H_2O	100.0	29.0

located in the floor in such a way that air can be moved through the seed in any and all sections of the house. Silos are made of steel, or brick and tile. They are relatively small in diameter as compared to height. Cooling is obtained by ducts located in the floor.

Ordinarily cottonseed is unloaded into the available storage facilities and kept there until processed. Air is passed through the seed for cooling purposes whenever this appears desirable in the judgment of the mill superintendent. Seed temperatures are taken from time to time to warn of any build-up of temperature ("heating"). High acid, damaged, or other seed which is not thought to store satisfactorily is processed as received insofar as is possible.

The use of air to cool seed is, on the surface, a very simple process; however "blowing" with humid air may complicate matters considerably by actually increasing the moisture content of the stored seed as a result of the moisture equilibrium that is set up between the seed and the air in contact with it. Such an equilibrium was first shown in 1929 by Thornton and Briggs (3), later by several others (4, 5, and 6), and finally in greater detail by Karon (7, 8), who stored seed in desiccators at various relative humidities obtained through the use of salt solutions known to produce a definite relative humidity in the air above the solution. Thus one cannot "cool" with air without considering the effects on seed moisture.

There is ample evidence (1, 2) that, as seed increases in moisture, the development of FFA is markedly accelerated. If the seed is already field-damaged, this effect is even more pronounced. On the other hand, seed of low moisture can be stored over relatively long periods with little risk.

In connection with some other research we recently had an opportunity to study seed storage further, investigating in particular: the effect of air blowing on seed moistures and the feasibility of seed drying at room temperature and at elevated temperatures. A summary of our findings is reported here.

Seed Moisture Equilibrium

In our work we used uncleaned mixed seed as processed at oil mills at several locations. Varying relative humidities were obtained through the use of saturated salt solutions in desiccators kept at 75°F. The humidities were checked with an electric hygrometer. This was equipped with eight sensing elements covering a total relative humidity range of 5 to 99%. The hygrometer itself was checked daily against a sling psychrometer, using the relative humidity of the room as a check-point. The relative humidity of the salt solutions, as given in the literature (9, 10), agreed with the electric hygrometer readings except in the high humidity ranges (*i.e.*, over 58%). In these cases it was always found that some decomposition of the salt had occurred or that the solution was not completely saturated. Hence, when there was any disagreement, the relative humidity determined by the electric hygrometer was accepted as correct.

Weighed samples of cottonseed were placed in the desiccators and withdrawn at definite time-intervals for moisture analysis by the oven method. From these data the moisture equilibrium points were determined (Table I).

A graph of these findings (Figure 1) shows that they agree quite well with Karon (7) (dotted line) except in the high humidity regions. At these levels it is difficult to find the equilibrium moisture-point of cottonseed before decomposition takes place. For example, it was found that, after 11 days of storage, mold formation was great enough to give erroneous moisture results. This can be demonstrated by recalculating the original moisture basis, the gain in weight of the sample, and the weight after drying in the oven. Results are shown in Table II. The average of 7.9% for the original moisture can be calculated for all samples except the two stored for 11 days at 93 and 97% relative humidity. This increase shows that there was a loss in dried weight apparently because of decomposition.

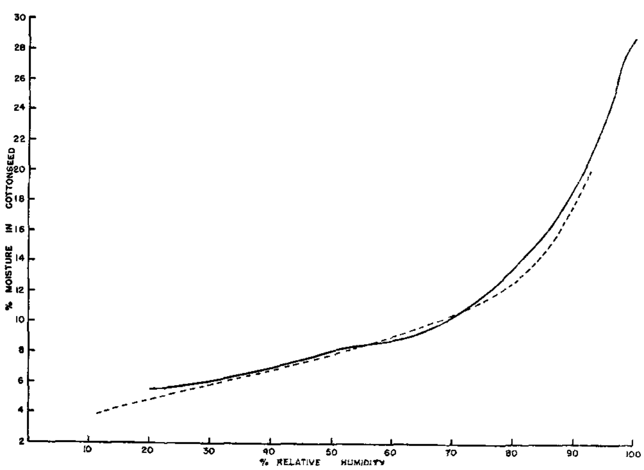

FIG. 1. Effect of R.H. on C.S. moisture.

The effect of temperature on equilibrium moisture was studied at 44, 77, and 100°F. It was found that increasing temperature merely accelerated the rate of moisture absorption, causing more rapid attainment of equilibrium. This confirms the work of Thornton and Bishop (4).

To check the effect of seed location on equilibrium moisture, seed from Cairo, Ill., Houston and Ft. Worth, Tex., Little Rock, Ark., and Harlingen, Tex., were stored at 31% relative humidity and found to reach essentially the same equilibrium moisture.

TABLE II

Change in Moisture Content Because of Decomposition

Days stored	Recalculated original moistures at		
	88% RH	93% RH	97% RH
3	8.0	7.9	7.9
6	7.9	7.8	8.0
11	7.9	8.9	10.0

The work reported here emphasizes the importance of taking air temperature and humidity into account when cooling seed by air since, if the air in contact with the seed is higher in relative humidity than that in equilibrium with seed of that moisture, the seed moisture will increase until the new equilibrium point is reached. In this way the moisture content of seed leaving the storage may be quite different from that in the seed initially.

It is not usually apparent what relative humidity in the outside air is maximum if seed moisture is not to be increased. This is because the final equilibrium depends on the relative humidity of the air in contact with the seed, and this may be quite different from the measured relative humidity of the outside air. As air increases in temperature, its moisture-carrying power also increases. Thus if cold outside air is passed through warmer seed, the air will have greater moisture-carrying power and may actually do some drying of the seed. On the other hand, if warm outside air is passed through relatively cool seed, the air will increase in relative humidity perhaps even to the point of becoming saturated and actually depositing water on the seed. Table III illustrates this point by showing what relative humidity in outside air at 80° would be required if one wanted the equivalent of 55% relative humidity in contact with seed of various temperatures. The cooler the seed, the lower the relative humidity required in the outside air since the moisture-carrying power of the air parallels the air temperature.

TABLE III

% Relative humidity	Seed temperature
28	60
35	65
40	70
47	75
55	80
63	85
75	90
88	95

To go one step farther, it will be seen that as air passes through seed, it may change in temperature and in relative humidity as it penetrates deeper and deeper into the pile. This will tend to build up a moisture gradient in the pile. If there are any warm spots, the air will tend to pick up moisture from these areas and translocate it to nearby cooler seed. Since increase in seed moisture tends to stimulate free fatty acid formation (1, 2), such moving of moisture in a pile is not desirable. This is one reason why "heating" seed or very moist seed should be kept separate. When this is done, cooling is facilitated and the seed may actually be dried considerably with air that would not be suitable for contacting drier seed (Figure 1).

As far as oil color is concerned, it appears best to keep seed cool, even at the expense of some build-up in free fatty acid. Flakes which were allowed to increase in free fatty acid from 4.3 to 7.0% by merely standing on a laboratory bench in thin layers at room temperature (in this way avoiding any build-up of heat) did not show a significant increase in the color of the refined oil made therefrom although, in general, seed of higher free fatty acid would be expected to produce an oil darker in refined color.

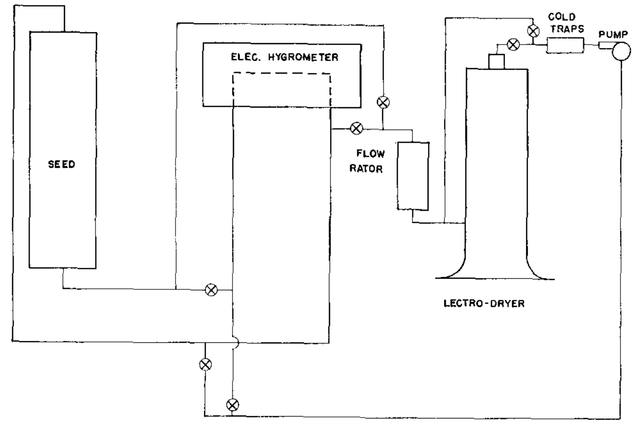

FIG. 2.

Seed Drying

Since the moisture content of cottonseed can be controlled by the relative humidity of the air in contact with it, theoretically at least, seed could be stored in a tight structure and then dried by means of dry air circulated through it. By suitable equipment the air could be re-dried before each pass.

Such a procedure would be feasible only under certain circumstances, one of which is that the drying rate would have to be sufficiently high to permit drying in a reasonable time. In most cases where solids are dried by gases, the drying rate proceeds fairly uniformly until a moisture level is reached, at which point the drying rate falls off considerably (11). This is the so-called "falling rate" period. If the "falling rate" occurs at too high a moisture level, drying time is excessive.

To obtain information on drying rates with the air of various humidities, a seed-drying system was constructed (Figure 2). The seed was contained in a glass column 2¼ in. in diameter by 38 in. long and was dried by blowing dehumidified air through it. The air was dehumidified by passing it through a set of traps containing ice or dry ice or by the use of a Lectrodryer, which removes moisture from air or other gases by adsorption, using activated alumina.

An electric hygrometer was connected to the system in such a way as to permit measurement of the relative humidity of the incoming and outgoing air at any desired time. The temperatures of the air were also recorded by an accessory of the same instrument. The air was continuously recycled in the system. For each run a 3-lb. batch of cottonseed was sprayed with enough water to bring the moisture content up to approximately 18 to 20%. The sample was allowed to equilibrate over-night in a cooler, and a moisture determination was run the next day prior to drying studies.

In the first run, dry-ice traps were the only devices used for removing moisture. Air was passed through the seed at a rate of 4,700 cc. per minute and 75 to 80°F., equivalent to a flow of 2.8 cu. ft. per square foot per minute. The results of this run indicate that the drying rate of cottonseed is relatively constant to about 7.3% moisture, which is considered low enough to store cottonseed safely.

The second run was made by using the Lectrodryer to produce air with a relative humidity of less than 5%. A flow rate of 2,350 cc. per minute at 80 to 90°F. was used until the moisture content dropped from

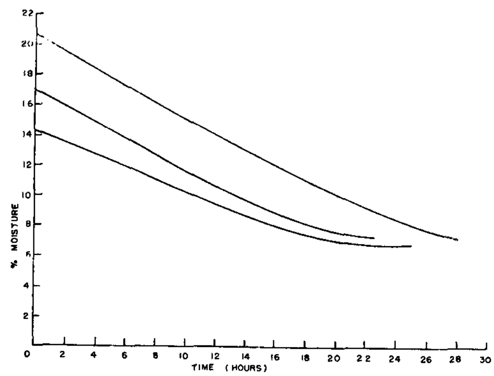

FIG. 3. Effect of drying time on C.S. moisture. Top curve—run number 3, middle curve—run number 1, bottom curve—run number 2.

21.2 to 14.3%. Then the rate was increased to 3,000 cc. per minute at 80 to 90°F., equivalent to 1.78 cu. ft. per square foot per minute. Run number 2 (Figure 3) also showed a constant rate of drying down to moistures considered safe for storage.

The third room-temperature drying test was made by using only ice in the traps for drying. This was done to check the drying efficiency when using more humid air. An average flow rate of 3,950 cc. per minute at 80 to 90°F. was used, equivalent to 2.26 cu. ft. per square foot per minute. It is seen that seed can be readily dried by using 25% relative humidity air, with very little difference in drying rate as compared to air of less than 5% relative humidity (Figure 3).

A cursory investigation of the investment and operating cost involved in drying cottonseed with air at room temperature however showed that, although theoretically feasible, costs would be prohibitive. For this reason the drying of seed with hot air was investigated.

Drying of Cottonseed with Heated Air

In cooperation with the Louisville Dryer Division of the General American Transportation Corporation a series of tests were run to determine whether cottonseed with a 20% initial moisture could be dried successfully to an 8 to 10% level in commercial driers.

a) *Forced Air Drier.* Air at a velocity of 150 ft. per minute was passed through a steam heater, then concurrently over the cottonseed in a revolving 1 × 6-ft. tube. Three runs were made (Table IV). No lint damage was observed in any runs. However the desired 8 to 10% level was not obtained so another type of drier was tried.

b) *Finned Tube Drier.* Cottonseed was fed into a 1 × 8 ft. revolving drum, which had six finned steam tubes peripherally located inside and extending the length of the revolving drum. One test run was made but was unsuccessful because of insufficient openings at the discharge end. Also it was observed that free lint was gathering on the fins of the steam tubes. On account of the very slow discharge of the cottonseed it was dried to a moisture content of 3.9%, needlessly low for storage purposes.

c) *Smooth Tube Drier.* This drier operated in exactly the same manner as the finned tube type except that the heating tubes were smooth and the discharge openings were larger. The 7.3% moisture in run number 5 (Table IV) was satisfactory, but retention time was excessive. Larger discharge openings were installed on the drier and three additional tests were made. The same trouble was encountered in run number 8 as in run number 5. However run number 7 gave a final moisture content in the range of 8 to 10%, and the seed discharge was good.

TABLE IV
Summary of Drying Runs

Run No.	Feed rate (*lbs./hr.*)	Air temp. (°F.)	Moisture at start	Final	% Drop	Type of dryer
1	30	310	20.2	12.4	7.8	Forced air
2	30	325	20.2	12.2	8.0	Forced air
3	20	328	20.2	11.6	8.6	Forced air
4	45	320	20.2	12.3	7.9	Smooth tube steam dryer
5	30	330	20.2	7.3	12.9	Smooth tube steam dryer
6	30	330	20.2	11.4	8.8	Smooth tube steam dryer
7	30	350	20.2	9.7	10.5	Smooth tube steam dryer
8	45	350	20.2	7.4	12.8	Smooth tube steam dryer

These tests show that cottonseed can be successfully dried to an 8 to 10% level in commercial driers. Although this work was done on a pilot-plant scale where maximum feed rate was 45 lbs. per hour, driers are available which are capable of handling 100 to 300 tons of cottonseed per day.

Summary

The moisture content of stored cottonseed depends on the relative humidity of the air which is in contact with it. This is an important consideration in using air to cool seed in storage since the seed may be moistened or dried as a by-product of the cooling operation. Increasing the moisture tends to increase the FFA and refining loss and thus counteracts the effects of cooling.

Cottonseed can be dried satisfactorily with dry air at ambient temperature or by hot undried air. In either case the economics are not favorable unless a relatively large amount of seed must be dried every season. Equipment and installation costs are relatively high, making carrying charges on the investment of the same magnitude as the probable loss because of deterioration.

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