DEVELOPMENT OF RAPID METHODS FOR THE ESTIMATION OF THE OIL CONTENT OF SINGLE COTTONSEEDS

Part IV.—Investigation of an Electrical Circuit for Measuring the Electrical Response of One Cottonseed at 1,000 Cycles/Second

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(Received June 5, 1960)

In an effort to develop further the electrical method or non-destructive estimation of oil in a single cottonseed, an extension circuit has been devised for increasing the sensitivity of the Marconi universal bridge, and its behaviour as well as the characteristics of the detector arrangement are studied for various percentages of imbalance. In this way, a sensitivity of almost the required order is attained, and some observations have been made with one to five kernels of M4 and L.S.S. cottonseeds. The effect of humidity is observed and the variation of dielectricconstant K of the cake with relative humidity has been measured. At theoretical calculation of the value of K for optimum discrimination between oil and cake is made, and the best value of relative humidity is deduced.

1. Introduction

In the previous communication on non-destructive techniques of oil estimation, a preliminary examination of the electrical methods was made,[1] and a tentative formula was developed for the estimation of oil in gramme-quantities of cottonseed kernels by capacitance measurements and by a combination of capacitance and conductance measurements. With the normal sensitivity obtainable with the Marconi universal bridge, (Type TF 868 A), several grammes of seeds were found to be necessary for a reliable estimation, and it is the purpose of the present investigation to study the possibilities of a simple extension of this technique to single cottonseeds.

For this, three factors need to be investigated: (1) the relation between the number of seeds and their capacity and conductance response, (2) the possibility of increasing the sensitivity of the bridge a hundred fold so as to obtain reliable estimations of oil with a single seed, and (3) the influence of atmospheric humidity on the electrical response. All these aspects are studied in the present paper, and the developments include a null-point capacitance balancing device and investigation of the optimum relative humidity and water content of the cottonseeds for the best results from this electrical method.

2. Preliminary Measurements with 5, 10, 15, etc. Cottonseeds

These measurements were made with the condenser described previously[1] (Fig. 1a), using weighed lots of five kernels, which were successively added to the insulated container between the condenser plates which were nearly 8 mm. apart. The weight of the kernels in the container was recorded together with the mean of three corresponding capacity and phase angle readings. The capacity readings were estimated to be reproducible to within 0.03 $\mu\mu$F and a plot of ΔC against weight of the kernels (in the container) is shown in (Fig. 1b) (hollow circles) for a temperature of 29°C. and a relative humidity of 65±5%. It is seen that a smooth graph, linear at the origin, can be drawn through the experimental points, none of which deviates the graph by more than the estimated error of 0.03$\mu\mu$F. The graph as a whole is non-linear and rises steeply when the number of kernels exceeds 15 (wt.$\simeq$0.6 g.), while the portion for less than 15 kernels can be considered as approximately linear.

The whole curve can be fitted by the equation

$$\Delta C = (0.14\ m + 0.21\ m^2)\ \mu\mu\ F, \qquad 1(a)$$

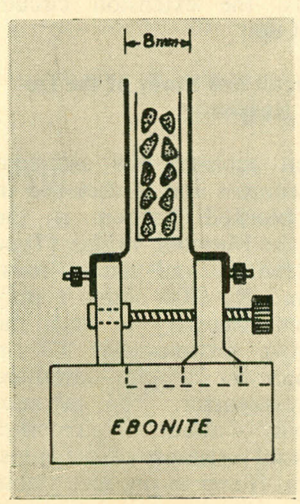

Fig. 1.—Diagrammatic sketch of measuring condenser with adjustable square plates (2.5 cm. side) and insulated container for holding the cottonseed kernels.

where m is the mass in g., and the non-linearity of the response was at first thought to be due to the weakening of the electrical field near the bottom edges of the condenser plates. This idea was examined by repeating the above measurements (i) with the bottom of the insulated container 6 mm. above the lower edge of the plates and (ii) with the same container placed between larger plates (4.5 cm. side as against 2.5 cm. previously). The results of (i) are shown by crosses in Fig. 1(b), and they exhibit no significant departure from the previous curve, while the results of (ii) are plotted in Fig. 1(c) and can be fitted by the equation

$$\Delta C=(0.19\ m + 0.19\ m^2)\ \mu\mu\ F \qquad (1b)$$

which indicates fair agreement with equation 1(a), thus showing that only a small part of the parabolic component can be attributed to the edge-effect. From the mean of the two equations 1(a) and 1(b), viz. $\Delta C = m\ (0.17+0.20\ m)\ \mu\mu\ F$, it follows that (a) for 1 to 2 g. of kernel, ΔC is of the order of 0.5 $\mu\mu$ F/g. , in agreement with the previously reported measurements, and (b) the capacity response for a small number of kernels will be nearly constant at about (0.19 ± 0.02) $\mu\mu$F per g., corresponding to a little less than 0.01 $\mu\mu$F per kernel of 40 mg. each.

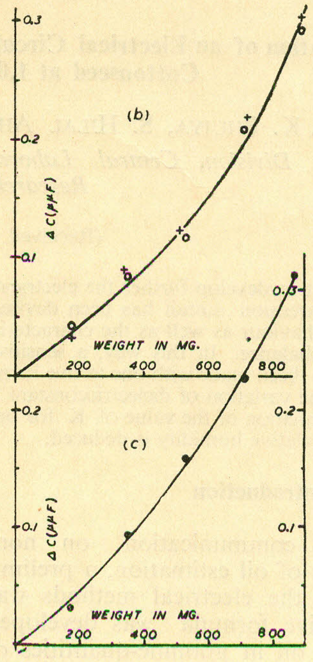

Fig. 1(b).-Measured response of 5 to 25 kernels, plotted as capacitance change against weight of kernels in the container: hollow circles, first set; crosses, second set with bottom container 6 mm. above the lower edge of the plates.

Fig. 1 (c).-Capacitance response for 5 to 25 kernels measured in a condenser with larger plates (4.5cm. side).

This is of the correct order of magnitude predicted by the previously derived[1] theoretical equation for kernels of mean dia. D, viz.

$$\Delta C=(1-1/k)+\frac{\text{Volume of sample}}{4\pi t^2} \div \{1-\alpha(1-1/K)\ D/t\}$$

$$\simeq(1-1/K)\left\{1+\frac{\alpha D}{t}\right\} \times \frac{\text{Volume of sample}}{4\pi t^2} \qquad (1c)$$

where $\alpha \simeq 0.9$ and t is distance between the conductor plates. For measuring such extremely small capacity changes, the extension circuit, described below, was set up.

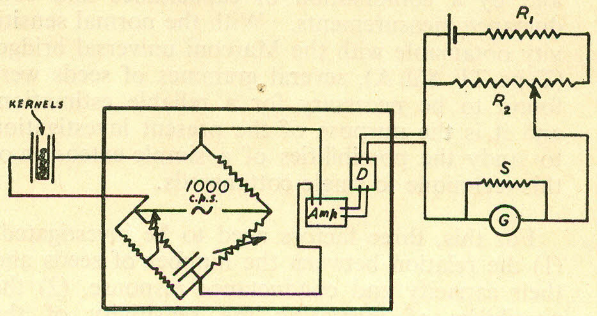

Fig. 2.—Sketch of extension circuit increasing the sensitiviy of the capacitance bridge.

3. The Extension Circuit and Study of the Detector Response

The extension circuit replaces the side-zero panel meter of the instrument and is sketched in Fig. 2, where the blocked portion is the normal circuitry inside the Marconi bridge (Type TF 868A.) The 6-volt battery and series resistances R_1 (250 ohms) and R_2 (200 ohms) supply the small steady current necessary to keep the deflection of the sensitive glavanometer 'G' on the scale, and 'S' is a shunt for damping unwanted oscillations of the glavanometer. The galvanometer used had a sensitivity of about 0.3 microamp. per division with an internal resistance of 208 ohms, and the above circuit was found to produce stable deflections of the pointer, which were nearly twelve times the corresponding deflections on the bridge panel meter.

The procedure adopted was first to balance the bridge with the panel meter, and then to insert the jack plug, which (while disconnecting the panel meter) connects the extension circuit. R_2 was then adjusted to bring the galvanometer deflection near the middle of the scale and the bridge could then be balanced more accurately with the help of this galvanometer. Since the capacity balance is indicated by a "stationary" value of deflection of the galvanometer, it is clear that the limits of sensitivity will be determined by the response characteristics of the detector circuit, which rectifies the alternating out-of-balance voltages from the bridge proper and then feeds them to the indicating circuit. In the foregoing experiment it has been seen that when the distance between the plate is 8 mm. (giving a capacity of 2 to 3 μμF), the capacity change is nearly 0.01 μμ F per kernel, up to a total of 10 kernels. If the circuit is to measure the oil in a sample composed of only a few kernels, then it must give a clear indication for as small a capacity change as 0.01 μμ F or even less, if possible. Therefore it was considered desirable first to study the response of the set-up of Fig. 2 to capacity changes in the range of 0.01 to 0.2 μμF.

This was done by connecting the measuring condenser (Fig. 1(a))of capacity C_o, at about 3 μμF, in series with a variable condenser C of capacity 45 to 450 μμF, using stiff copper connections to avoid stray changes of capacity. By varying the large condenser, very small calculated changes of capacity in system can be produced, because the total capacity C_t is given by

$$C_t = 1/(\frac{1}{C_o} + \frac{1}{C}) = C_o/(1 + \frac{C_o}{C})$$
$$= C_o \left[1 - \frac{C_o}{C} + (\frac{C_o}{C})^2 \ldots\right]$$
$$= C_o - \frac{C_o^2}{C}\left[1 - \frac{C_o}{C} + \ldots\right]$$

Since $C_o/C < \frac{1}{15}$, we get, to a sufficient accuracy,

$$\Delta C_t = (C_t)_1 - (C_t)_2 = C_o^2/C_1^2 - C_o^2/C_2^2$$
$$= C_o^2 \Delta(1/C) \simeq C_o (C_o/C)(\Delta C/C) \qquad (2)$$

With the capacity C at 450 μμF, the bridge was balanced with the help of the extension circuit, which gave an estimated accuracy of ± 0.03 μμF i.e. ± 1% in the bridge dial setting. The galvanometer deflection was then noted for different settings of C (from which the values of ΔC were calculated as above) and the mean deflections for two sets of observations were plotted against ΔC_t (lowest graph of Fig. 3). The curve is seen to be parabolic and is well represented by the equation.

$$\text{deflection} = 280 \times (\Delta C_t)^2, \qquad (3)$$

which shows that the sensitivity as given by the slope, i.e. 560 ΔC_t divisions μμF, is proportional to ΔC_t and is very poor for $\Delta C_t < 0.05$ μμF.

The bridge dial setting was now increased a very little bit, the imbalance amounting to about 0.15 μμ F (i.e. 4%), and the galvanometer zero was set by adjusting the resistance R_2. Starting

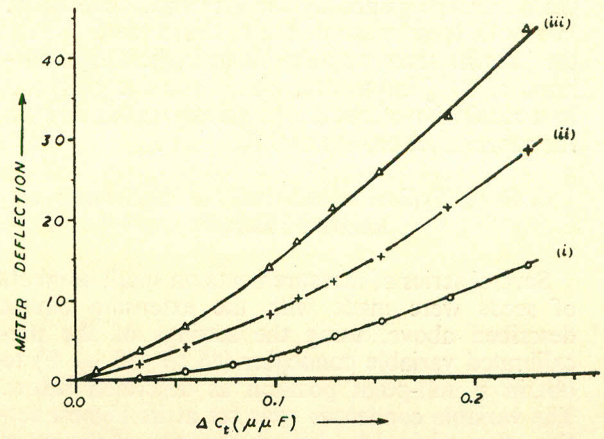

Fig. 3.—Graphs showing the measured response of the circuit arrangement of Fig. (2) for a given change ΔCt in the capacitance of the measuring condenser, starting from (i) exact balance, lowest curve, (ii) + 4% imbalance, middle curve (iii) + 7% imbalance, top curve.

with this new zero, the deflections were noted for several setting of the condenser, C, and the middle graph of Fig. 3 shows the new dependence of the deflection on the capacity change ΔC_t. The equation of this graph is approximately

$$52\,\Delta C_t + 300\,(\Delta C_t)^2,$$

whence the sensitivity is now

$$52 + 600\,\Delta C_t, \qquad 4(a)$$

which is 65 divisions / μμF for $\Delta C_t = 0.02$ μμF and 112 for $\Delta C_t = 0.1$ μμF. Relatively more uniform sensitivity can be obtained by increasing the off-balance setting, e.g. for an unbalance of about 7% (upper curve of Fig. 3). the sensitivity is

$$100 + 700 \times \Delta C_t, \qquad 4(b)$$

which varies between 100 and 170 divisions per μμF in the range of 0.00 to 0.10 μμF, so that a change of 0.01 μμ F would be clearly indicated as a galvanometer deflection of 1 division.

It would appear from the above measurements and discussion that the sensitivity can be increased as much as desired by increasing the amount of the imbalance. However, two factors militate against this; firstly, the automatic gain control in the amplifier becomes effective when the imbalance exceeds about 15% and secondly small fluctuations in the output from the amplifier are increased proportionally to the out-of-balance current, thus producing increased instability of the galvanometer indications, which cancels out the advantage gained by the increased deflections. For both these reasons, the optimum setting with the present setup appears to be in the neighbourhood of 10% imbalance, which would correspond to a sensitivity of about one galvanometer division for capacitance change of 0.005 $\mu\mu$ F.

4. Some Typical Results and the Importance of Relative Humidity

Several series of measurements on small numbers of seeds were made with the extension circuit described above, using the settings of the precalibrated variable condenser (45 to 450 $\mu\mu$ F) to obtain a null-point position as described below. The variable condenser was set at its highest setting, thus giving the lowest capacity of the series system, which was then balanced approximately with the bridge dial, after which the extension circuit was plugged in, the bridge dial set about 10% out of balance, and the rheostats adjusted to bring the (extension) galvanometer reading near the middle of the scale. The arrangement was then ready for use in estimating the capacity changes produced by putting small numbers of cottonseed kernels in the insulated container, already placed between the plates of the fixed condenser shown in Fig. 1 (a).

One weighed kernel was dropped into the container and the variable condenser adjusted to restore the initial galvanometer position, the new reading of the condenser scale being noted. Another weighed kernel was dropped in, and the initial galvanometer position again restored, and so on, until half a dozen seeds had been put into the container, which was finally weighed as a check. The total capacitance increase for each stage was calculated from equation (2), and was of the order of $0.08 \times (\Delta C\ C)$ $\mu\mu$ F.

Some typical results obtained with M 4 & L.S.S. seeds kept at the prevailing relative humidity of 85% are shown in Fig. 4 (a) where the total change in capacitance is plotted against the total weight of kernels in the container. Although the graphs still show a certain degree of non-linearity, of about the same order as that of the curves of Fig. 1, it is seen that the average scatter of the points about the curve is only 0.002 $\mu\mu$ F, i.e. about 5% of the plotted capacitance change, ΔC_t for 5 kernels. This indicates that an accuracy of this order or a little better is all that is attainable using 1 to 5 kernels in the present setup. This serves to point up the need for even more sensitive measuring apparatus. The curve for M 4 kernels was repeated at a time when the atmospheric humidity was 75%, i.e. 10% lower than for Fig. 4 (a) and this time values of the capacitance response about 15% lower were observed (Fig. 4 b). This indicated the need for investigation of the influence of atmospheric humidity and water-content of the kernels on their effective dielectric constant. Experiments in this direction were therefore undertaken using cottonseed cake in order to eliminate the effect of the oil.

5. The Influence of Water-Content on the Dielectric Constant of Cottonseed Cakes

These experiments were carried using 6 cm. diameter pressed discs (thickness about 5 mm.) of cottonseed cake prepared in a small laboratory hydraulic press in the following way. A commercially available sample of cottonseed cake was washed with petrol, freed from bits of shell by sieving,

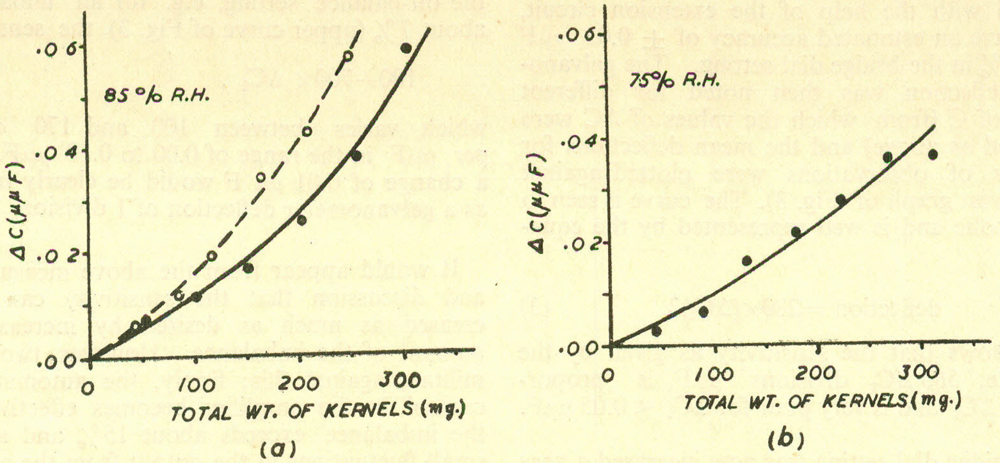

Fig. 4.—Typical results obtained with 1 to 5 cottonseeds, using the set up of Fig. 2 with 10% imbalance for (a) M4 and L.S.S. kernels at 85% atmospheric humidity; and (b) M4 kernels at 75% humidity.

TABLE 1.—DIELECTRIC CONSTANT OF PRESSED COTTONSEED CAKE MEASURED AT DIFFERENT RELATIVE HUMIDITIES.

Rel. humidity	100%	83%	54%	0%
Dielectric constant (K)	140±15	19.4±2	8.5±2	3.2±.3
1/K	$.007_1$	.052	.118	.313
Water content (%)	12.9	8.8	4.4	0.0

and then compacted by light pressure in a die at 100°C. under the hydraulic press. The dielectric constant of the disc of cottonseed cake was measured by placing it between the two plates of the condenser of Fig. 1(a) and adjusting the distance between the plates so that the sample disc was in contact with both plates. The dielectric constant 'K' was calculated as K=(Capacitance with sample/Capacitance with air), a small correction about 0.3 $\mu\mu$ F being necessary for the stray capacitance due to leads, etc. This correction was estimated from the configuration and dimensions of the condenser formed by these components.

The dielectric constant of the sample was first measured at the prevailing humidity, which was about 80%. The sample was then saturated with water vapour for about 20 hours, after which its dielectric constant was again measured. It was then put through one hydration→dehydration half cycle, and the corresponding values of 'K' for each value of the relative humidity were measured and are recorded in Table 1. A more accurate measure of wetness of the sample is its percentage water-content and this has also been recorded in every case by comparison with the mass of the completely dry sample, dried in a $CaCl_2$ desiccator for about 24 hours. (The samples were maintained at each value of relative humidity for this period in order to attain a satisfactory approach to equilibrium). It is seen that the dielectric constant increases rapidly with water constant, especially above 5% water, the maximum recorded value being about 140 for the fully saturated cake. Figures 5(a) and 5(b) show the graphs for 1/K against relative humidity and % water content, respectively.

In view of the above wide variation in the value of K, it is desirable to investigate mathematically its influence on the ability of a capacitance measurement on a kernel to give an accurate estimate of the oil content. If the kernel contains x_o parts of oil of dielectric constant K_o and $(1-x_o)$ parts of proteinous matter of dielectric constant K_p, then the dielectric constant K_m of a homogenous mixture of the two should, according to the equation of Lichtenecker,[2] be given by

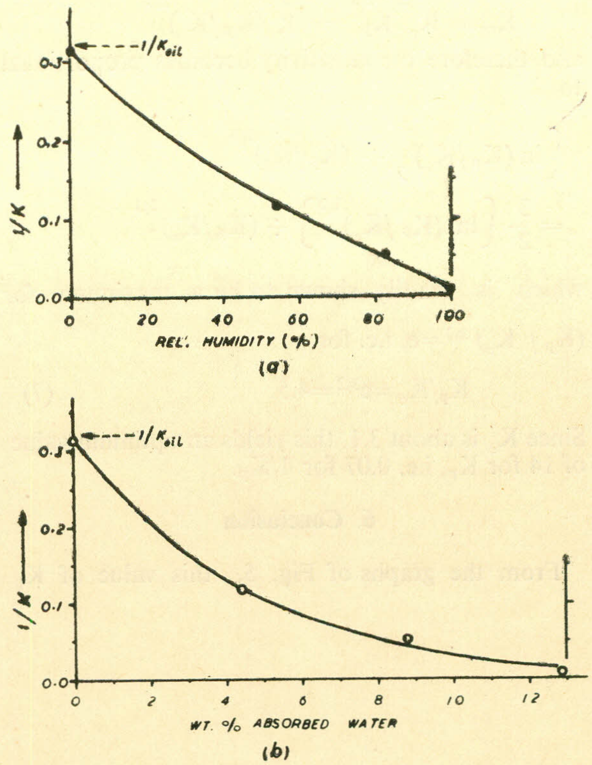

Fig.5.—Measured variation of dielectric constant K of a sample of cleaned cottonseed cake, plotted as (a) 1/K against relative humidity and (b) 1/K against % water absorbed by the cake.

$$\ln K_m = x_o \ln K_o + (1-x_o) \ln K \qquad (5)$$

whence for a small change δx_o in the value of x_o,

$$\delta K_m/K_m = \delta(\ln K_m) = \delta x_o \ln K_o - \delta x_o \ln K_p = \delta x_o \ln (K_o/K_p). \qquad (6)$$

Now, the capacity response due to the insertion of a single kernel is, from equation 1(c), approximately proportional to (1 — 1/K) so that the change in this response is proportional to $\delta(1/K_m) \div \delta x_o$, i.e. to

$$-(4\, K_m/K_m^2) \div \delta x_o = -(\delta K_m/K_m) \div (K_m\, \delta x_o) = \frac{1}{K_m} \ln (K_p/K_o)$$

from equation (6). Putting $x_o = 1/3$ and $(1-x_o) = 2/3$ as working approximation in equation (5), we get

$$K_m = K_o^{1/3} K_p^{2/3} = K_o(K_p/K_o)^{2/3}$$

and therefore the sensitivity becomes proportional to

$$\left\{ \ln (K_p/K_o) \right\} \div (K_p/K_o)^{2/3}$$

$$= \frac{3}{2} \left\{ \ln (K_p/K_o)^{2/3} \right\} \div (K_p/K_o)^{2/3}$$

which is readily shown to be a maximum for $(K_p / K_o)^{2/3} = e$, i.e. for

$$K_p/K_o = e^{3/2} = 4.5 \qquad (7)$$

Since K_o is about 3.1, this yields an optimum value of 14 for K_p, i.e. 0.07 for $1/K_p$.

6. Conclusion

From the graphs of Fig. 5, this value of K_p is seen to correspond to a water content of about 5% to 6% on the weight of cake and a relative humidity of nearly 70% to 80%. This corresponds to the conditions prevailing in the measurements plotted in Fig. 4, the results of which can therefore be taken as representative of the optimum performance of the present set up for single seeds, namely an accuracy of the order of 5% or perhaps a little better. While further efforts are being directed towards improving this accuracy, it should be noted that this figure is based on the experimental scatter of $\pm$ 0.002 $\mu\mu$ F, which is the smallest value obtainable at present with the best commercially available capacitance bridges.

References

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2. Lichtenecker, Phys. Zeits., **19**, 374 (1918).