

Separation by Dielectric Distribution: Theory¹

B. C. BLACK and E. G. HAMMOND,

Department of Dairy and Food Industry, Iowa State University of Science and Technology, Ames, Iowa

Abstract

Dielectric distribution may be defined as the distribution that mobile materials with different constants will assume in a nonuniform electric field. The material moves under the influence of dipoles induced by the electric field. If the suspended material is more polar than the medium in which it is suspended, it will move toward the region of highest field intensity, and if the suspended material is less polar than the medium, it will move toward the region of lowest field intensity.

Dielectric distribution will give useful separations. The dielectric distributions of phosphatide micells in petroleum ether and of a mineral oil emulsion in water were studied in a nonuniform radial field produced by a cylindrical condenser. The central and peripheral portions of the cylinder could be drained separately, and the concentrations in the two portions could be determined. The effect of time, voltage, and concentration on the distribution was studied. A theory was proposed to predict the distribution of material in a cylindrical condenser at equilibrium.

Introduction

SMALL PARTICLES of a given dielectric constant suspended in a medium of different dielectric constant will migrate in a nonuniform electric field. This movement, which has been termed dielectrophoresis, leads to a redistribution of the suspended particles in the field. We call this the dielectric distribution. Unlike electrophoresis, no net permanent charge need exist. The particles move under the influence of permanent dipoles or dipoles induced by the field. If the particles are more polar than the medium in which they are suspended, they will move toward the region of highest field intensity. Conversely, if the particles are less polar than the medium, they will move toward the region of lowest field intensity.

Separation processes apparently based upon dielectric distribution have been known for a long time. Lowden (1) in 1891 patented an invention for removing metal particles from used lubricating oils. This process probably depends on dielectric distribution. The Cottrell precipitator patented in 1911 was adapted to the removal of emulsions of water from crude petroleum (2,3). The droplets of water are collected by dielectric distribution, although this principle was not completely understood (4,5). Bates (6) also used a dielectric distribution process for refining petroleum.

The idea that a dielectric will move in a nonuniform electric field goes back at least as far as the fourth edition of a treatise on electricity by Föppl and Abraham in 1912 (7). The idea was rediscovered independently by Muller (8) and Pohl (9-14). Loesche and Hultschig (15) extended the theory. These theoretical developments were apparently completely divorced from the practical applications of the principle in the petroleum industry. However, dielectric

behavior can still not be completely predicted. The theoretical treatments have been incomplete. Disturbing effects, such as the leakage of current that always occurs at the voltage necessary to observe dielectrophoresis, are very poorly understood.

In this paper we have derived equations to predict the equilibrium distribution of suspended particles in the field of a cylindrical condenser. The results obtained for suspensions of phosphatide micells in soybean miscella and for suspensions of mineral oil in water are compared with the theoretical predictions.

Experimental

A diagram of the batch dielectric distribution apparatus is shown in Figure 1. It consisted of a Pyrex glass cylinder of 1.31 cm. I.D. and 12 cm long with a center metal electrode insulated with Teflon tubing 0.41 mm thick so that the total diameter was 0.31 cm. Metal foil wrapped around the outside of the cylinder served as the peripheral electrode. A potential difference was applied between the outside and inside electrodes with a high voltage transformer (No. 969-001-395, Jefferson Electric Company, Bellwood, Illinois). The field strength increased from the peripheral to the central electrode, thus giving a nonuniform field. The primary of the high voltage transformer was connected to a variable transformer which could adjust the input into the high voltage transformer from 0 to 120 v at 60 cps. The variable transformer was calibrated by measuring the voltage from the high voltage transformer with a voltmeter.

- A INLET
- B CENTER ELECTRODE
- C INSULATION
- D PERIPHERAL ELECTRODE
- E TO TRANSFORMER
- F BARRIER
- G PERIPHERAL OUTLET
- H CENTER OUTLET

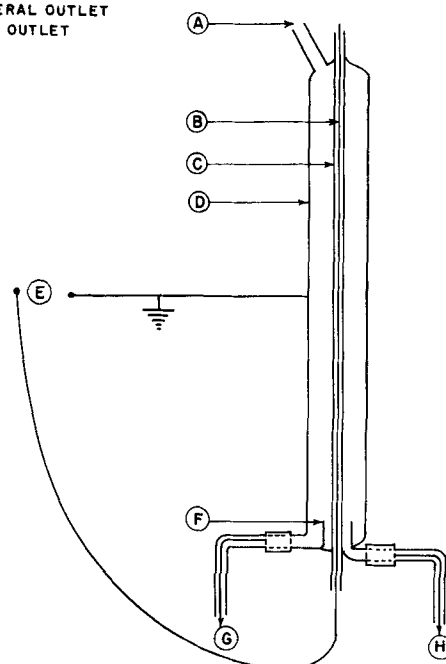


FIG. 1. The cylindrical condenser apparatus.

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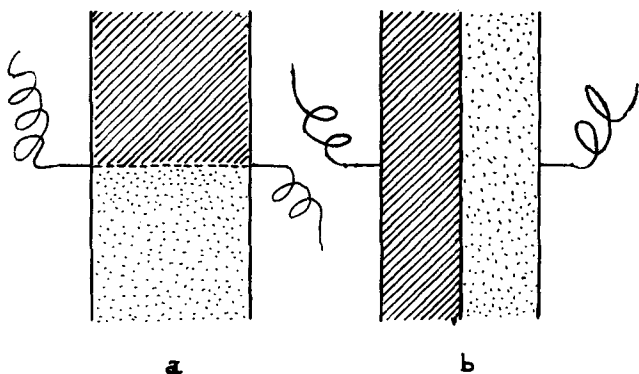


FIG. 2. Two modes of distributing materials in a condenser that give different capacities.

The material to be distributed was put in the apparatus at inlet A. After filling, the desired voltage was applied for a definite period of time. When equilibrium was attained, fractions were drained through the central and peripheral outlets, G and H. The barrier, F, 0.69 cm in diameter, divided the central fraction from the peripheral fraction. The velocity at which the two fractions were drained was in the ratio of the volumes on the inside and outside of the barrier so that mixing was minimized. The contents of both fractions were analyzed.

The soybean miscella was prepared from Iowa Hawkeye 1962 soybeans. The soybeans were crushed and then dried in an oven at 82°C for 60 min with occasional stirring (16). The soybean oil was extracted from the soybean meal with Skellysolve B, and the soybean miscella was filtered. The phosphorous content of the soybean miscella was determined by the method of Black and Hammond (17).

The oil in water emulsion was prepared from an oil with a specific gravity of one to avoid gravitational effects. The oil was a mixture of 4.05 ml of mineral oil (specific gravity 0.926) and 0.5 ml of 1,1,2,2-tetrachloroethane (specific gravity 1.6). The oil was emulsified with water in a Waring blender with Koldex (R. G. Moench and Co., Inc., 89 Terminal Ave., Clark, New Jersey) as an emulsifying agent. The weight of Koldex was 2% of the weight of the fat in the emul-

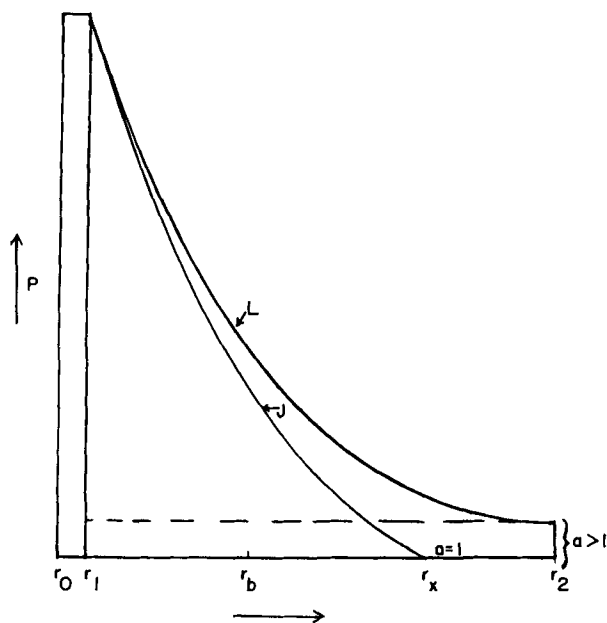


FIG. 3. Profiles of the fraction of polar particles, P , vs. radius, r , at equilibrium in a cylindrical condenser.

sion. The concentration of the oil suspension was determined turbidometrically by measuring the absorbance at 475 $m\mu$ with a Beckman DU Spectrophotometer.

Theory

The Efficiency of Separation

The amount of separation of the polar and nonpolar constituents in the cylindrical cell was expressed as percentage of efficiency. This term was derived as follows: Let the volume inside the barrier (refer to Figure 1) be compartment 1 and let the volume outside the barrier be compartment 2. Then P_1 is the volume fraction of polar material in compartment 1 at equilibrium, and, similarly, P_2 is the volume fraction of polar material in compartment 2 at equilibrium. The total polar material, P_T , is $P_1 + P_2$. Let V_1 be the volume fraction of polar material initially in compartment 1 and V_2 be the volume fraction of polar material in compartment 2.

Efficiency is defined as the actual fractional gain of polar material in compartment 1 divided by the maximum possible gain. The actual gain is $(P_1/[P_1 + P_2]) - V_1$, and the maximum possible gain is $1 - V_1$, therefore

$$\% E = \frac{P_1 - V_1}{\frac{P_1 + P_2}{V_2}} \times 100 \quad [11]$$

In the apparatus used, V_1 was 0.25 and V_2 was 0.75.

The efficiency was defined in this manner so that it would be 100% if all the polar material was inside the barrier and zero if the concentration of polar material was the same on both sides of the barrier.

Similarly an equation can be derived for the efficiency in terms of the nonpolar material so that it will be 100% if all of the nonpolar material is outside the barrier and zero if the nonpolar material concentration is the same on both sides of the barrier.

$$\% E = \frac{\frac{N_2}{N_1 + N_2} - V_2}{V_1} \times 100 \quad [12]$$

where N is the concentration of nonpolar material.

Derivation of General Distribution Equations

The force that a particle experiences in a nonuniform field is affected by the polarizability of the particle, a , the electric field intensity, F , and the field gradient, dF/dx . Therefore:

$$\text{Force} = aF (dF/dx) \quad [13]$$

Pohl (10) and Loesche and Hultschig (15) have derived equations for the velocity with which a particle moves when placed in a cylindrical condenser. They assumed that particles more polar than the suspending medium will tend to move toward the inner electrode until they reach equilibrium with the counter diffusional force which will arise from the concentration change. This is an oversimplification, because as the particles move, the field gradient will change if the particles contribute a significant fraction of the total dielectric value of the system. The particles move in a direction to reduce the field gradient, and when the gradient becomes zero, the particles experience a zero force according to Equation 4. Thus, equilibrium is reached when the field gradient is reduced to a value that balances the osmotic pressure at all points; however, osmotic effects are usually negligible for particles large enough to be separated by dielectrophoresis (13).

If there are enough particles, they will assume a distribution that makes the field gradient zero (i.e.,

the field constant) all the way across the condenser. If there are not enough particles to do this, then the equilibrium will be reached when the field gradient is zero as far out as possible from the electrode toward which the particles move. If the concentration of particles is high enough, pure particulate material should accumulate at the electrode towards which it moves. The field gradient will then be zero from the boundary of pure particulate material to the other electrode.

For a cylindrical condenser

$$F = Q/2\pi r l \epsilon \tag{4}$$

where F = field strength, Q = Charge on the condenser; l = length of the condenser; ϵ = dielectric constant; r = radius at which F is measured.

It can be shown easily that $dF/dr = 0$ if, and only if, $\epsilon = R/r$, where R is a constant.

For solutions, one may assume as a rough approximation that the dielectric constant of a binary mixture is a linear function of the volume fraction of the constituents (18) so

$$\epsilon = P\epsilon_p + (1 - P)\epsilon_n \tag{5}$$

where ϵ , ϵ_p , and ϵ_n are the dielectric constants of the mixture, more polar component, and more nonpolar component, respectively, and P is the volume fraction of the more polar component. This equation should be even better for a coarse suspension of solid particles where interaction between the solvent and suspended particle is limited to the surface. For a coarse suspension, however, the dielectric constant will depend on the distribution of the particles in the field. For example, it is easy to show that a condenser containing two dielectrics in the configuration of Figure 2a will have a lower energy for a given charge than the configuration of Figure 2b. It can also be shown that this energy will not change even if the dielectrics in Figures 2a or 2b are cut into fine sheets and interlarded, as long as the sheets maintain their original direction with respect to the field. Thus, suspended particles which are free to move should tend to take the lower energy configuration corresponding to Figure 2a and form chains between the condenser plates. Such chains have been observed in our work (19) and elsewhere (5,20). For configurations corresponding to Figure 2a, Equation 5 is still valid. In the case of the cylindrical condenser, the particles should form radial chains from the center toward the periphery. This will be compatible with the equilibrium condition that $\epsilon = R/r$, and the tendency to form chains will not be in conflict with the dielectrophoretic force as Pearce (5) assumed.

Figure 3 shows the profile that the fraction of polar material, P , makes when it assumes dielectric distribution in a nonuniform field. The radii of the inner electrode, outer electrode, and cell barrier are r_1 , r_2 and r_b , respectively. The center of the apparatus is at r_0 . The point r_x is the point at which this curve intersects the r -axis and the fraction of P is zero. It can vary from r_1 to r_2 according to the quantity of polar material as shown in curves J and L. Values of $r_x > r_2$ are outside the apparatus and are meaningless. If the quantity of polar material is great enough it may have a value greater than zero at r_2 , and the value of the dielectric constant of the solution will be greater than that of the medium. Let us relate these two values by a parameter, a so that $\epsilon = \epsilon_n a$ at r_2 .

At equilibrium, a general equation for any point from r_1 to r_x may be written:

$$\epsilon = \frac{\alpha \epsilon_n r_x}{r} \tag{6}$$

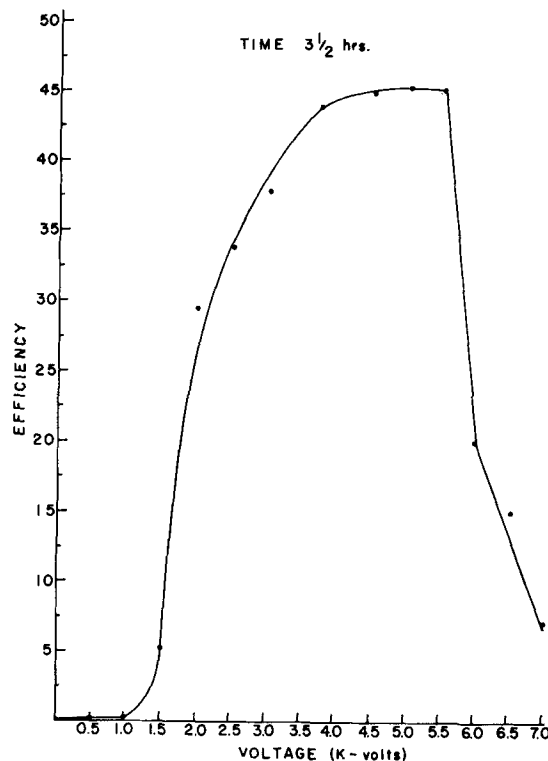


FIG. 4. Efficiency vs. voltage at constant time.

Then by definition if $r_x \leq r_2$, a must be 1. In cases where $r_x > r_2$ to use this equation, we set $r_x = r_2$ and the $a \geq 1$. The value of r_x or a may be obtained as follows: The concentration surface of the polar material in three dimensions can be obtained by rotating the curves in Figure 3 around the r_0 -axis 360° . The integral under this surface will represent the total amount of polar material, P_T , after correction for the volume of the central electrode.

$$P_T = \frac{P_1}{P_x} \int \pi r^2 dP + \pi r_2^2 P_2 - \pi r_1^2 P \tag{7}$$

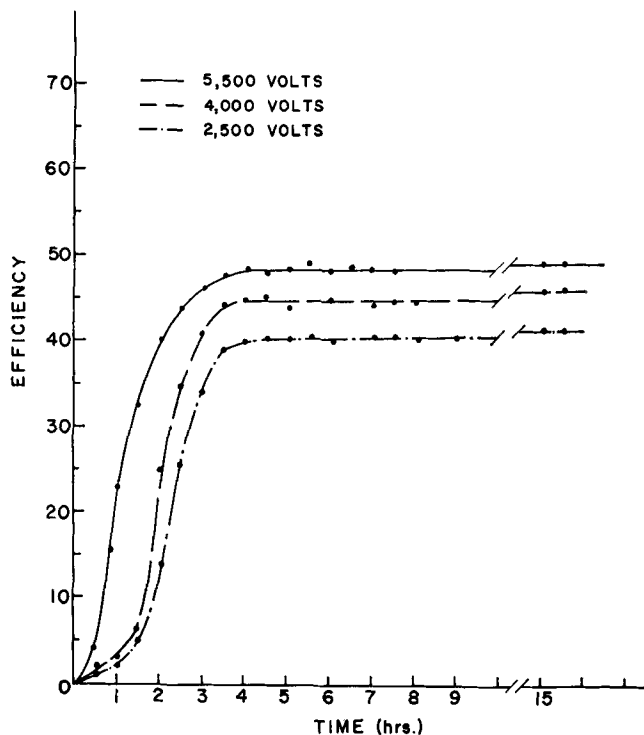


FIG. 5. Efficiency vs. time at constant voltages.

Setting Equation 5 equal to Equation 6.

$$P(\epsilon_p - \epsilon_n) + \epsilon_n = \frac{a\epsilon_n r_x}{r} \quad [8]$$

This equation may be solved for P_1 , P_2 , P_x , or r , and these values may be substituted into Equation 7. (In solving for P_2 we must set $r_x = r_2$.)

$$P_T = \frac{\pi a \epsilon_n r_x}{\epsilon_p - \epsilon_n} (r_x - r_1) + \frac{\pi r_2^2 (a - 1) \epsilon_n}{\epsilon_p - \epsilon_n} - \frac{\pi r_1^2 \epsilon_n (a r_x - r_1)}{(\epsilon_p - \epsilon_n) r_1} \quad [9]$$

By definition.

$$P_T = P_1 \pi (r_2^2 - r_1^2) \quad [10]$$

where P_1 is the initial concentration of polar material before dielectric distribution. Setting Equations 10 and 11 equal, we may solve for either r_x or a by imposing the conditions $a = 1$ if $r_x \leq r_2$ and $r_x = r_2$ when $r_x \geq r_2$

$$a = \frac{r_2^2 - r_1^2}{2(r_2^2 - r_1 r_2)} \left[1 + \frac{(\epsilon_p - \epsilon_n) P_1}{\epsilon_n} \right] \quad [11]$$

$$r_x = r_1 + \left[\frac{P_1 (r_2^2 - r_1^2) (\epsilon_p - \epsilon_n)}{\epsilon_n} \right]^{1/2} \quad [12]$$

The efficiency E may be related to P_1 as follows: Let P_1 be the amount of polar material in compartment 1 of the apparatus. If we consider the case where $r_x < r_2$ and $a = 1$

$$P_1 = \frac{P_b}{P_b} \int \pi r^2 dP + \pi r_b^2 P_b - \pi r_1^2 P_1 \quad [13]$$

where r_b is the radius of the barrier dividing the two compartments. We may solve for P_1 as before and

$$P_1 = \frac{\pi \epsilon_n}{\epsilon_p - \epsilon_n} \left[2r_x (r_b - r_1) + r_1^2 - r_b^2 \right] \quad [14]$$

and the value of P_1 and the value of P_T from Equation 10 may be substituted into Equation 1 to yield

$$E = \left(\frac{\epsilon_n}{\epsilon_p - \epsilon_n} \right) \frac{2r_x (r_b - r_1) + r_1^2 - r_b^2}{P_1 (r_2^2 - r_b^2)} \frac{r_b^2 - r_1^2}{r_2^2 - r_b^2} \quad [15]$$

r_x may be obtained from Equation 12. By a similar process when $r_x \geq r_2$ and $a \geq 1$

$$E = \left(\frac{\epsilon_n}{\epsilon_p - \epsilon_n} \right) \frac{2a r_2 (r_b - r_1) + r_1^2}{P_1 (r_2^2 - r_b^2)} \frac{r_b^2 - r_1^2}{r_2^2 - r_b^2} \quad [16]$$

where a may be obtained from Equation 11. Thus, the efficiency of the separation may be predicted from the original concentration of particles, the dielectric constants, and the dimensions of the apparatus. Similarly, one can obtain equations for the distribution for the other cases that have been discussed. The only other case used in this paper is that where the particles are less polar than the suspending medium and accumulate at the periphery of the cylindrical condenser. Then we find:

$$E = \left(\frac{r_2^2}{\epsilon_n - \epsilon_p} \right) \frac{2a \epsilon_n \left(1 - \frac{r_b^2}{r_b r_2} \right) - \frac{\epsilon_p (r_2^2 - r_b^2)}{r_2^2}}{N_1 (r_b^2 - r_1^2)} \frac{r_2^2 - r_b^2}{r_b^2 - r_1^2} \quad [17]$$

and

$$a = \frac{\epsilon_p}{\epsilon_n} - \frac{\epsilon_p}{\epsilon_n} \left(\frac{N_1 (\epsilon_p - \epsilon_n) (r_2^2 - r_1^2)}{\epsilon_p - r_2^2} \right) \quad [18]$$

Results and Discussion

Crude soybean miscella was used to study the phenomenon of dielectric distribution. The crude oil is composed of phosphatides and neutral fat. When phosphatides are put into a nonpolar solvent, such as petroleum ether, a phosphatide micell structure is formed, while the neutral fat forms a true solution. The phosphatide micell is a more polar material than soybean oil or petroleum ether and should be concentrated in the place of high field strength.

First an efficiency vs. voltage study was made to determine the voltage at which the maximum efficiency could be obtained. This was done by filling the appa-

ratus with soybean miscella (5.4% soybean oil in Skellysolve B) and applying fixed voltages for 3½ hr. The results are shown in Figure 4.

The efficiency was very low at low voltages and increased rapidly with an increase in voltage until a plateau was reached. The efficiency remained fairly constant with further increases in voltage up to a certain voltage where there was a rapid decrease in efficiency. This voltage at which the efficiency decreases has been termed the slough-off voltage by Pohl and Schwar (11). They found that the slough-off voltage depended on the size of the center electrode, amount and type of insulation, the gap between electrodes, and the size of the particles. They attributed slough-off to the capture of net charges by the particles at the central electrode, thus causing the particles to repel each other.

The results of an efficiency vs. time study are shown in Figure 5. The three curves were constructed by using three different constant voltages, 2,500 v, 4,000 v and 5,500 v and a 5.4% solution of crude soybean oil in Skellysolve B.

There was an induction period (practically no efficiency) at first, then the efficiency rose rapidly and leveled off at a plateau value. The plateau should represent the equilibrium point at which the field gradient becomes zero. All three of the curves started to level off at about 4 hr. This does not agree with Pohl's equation (10) which states the velocity of motion is a function of the square of the voltage. The length of the induction period seemed to be an inverse function of the voltage. There was an increase in the height of the plateau with increase in voltage. This indicates some counterforce is opposing the establishment of equilibrium at $dF/dr = 0$. It seems unlikely that back diffusion caused by the higher concentration of particles near the inner electrode can account for the counterforce, because the phosphatide micells are too large to have much osmotic effect.

The induction period may represent a change in size of the particles. Pohl (10) calculated that the rate that the particles move in a cylindrical condenser should be proportional to the cube of their radius. According to our theory, the particles should tend to form chains under the influence of the field, and the induction period may represent the time required to form chains big enough to move at an appreciable rate. However, the velocity equations derived by previous workers have not considered the effect of the change in field gradient during dielectrophoresis. It may be that a more realistic derivation would indicate a lag period, even at constant particle size.

An efficiency vs. time study was made with two different concentrations of phosphatide, at 4,000 v. The more dilute solution gave the higher plateau efficiency. Also the more dilute solution showed a lower efficiency during the induction period than the more concentrated one. The increase in plateau efficiency with dilution can be accounted for by the theory derived earlier. The lower efficiency during the induction period could be accounted for by slower formation of the chains of phosphatide particles suggested above.

Efficiency vs. concentration experiments were conducted to test the theory of dielectric distribution presented earlier. The time and voltage in this study were constant at 3½ hr and 4,000 v, respectively. The results are shown in Figure 6. The dots represent the experimental data. Values for ϵ_p/ϵ_n were calculated from the efficiencies at different concentrations using Equation 15 and 16. The theoretical curve was plat-

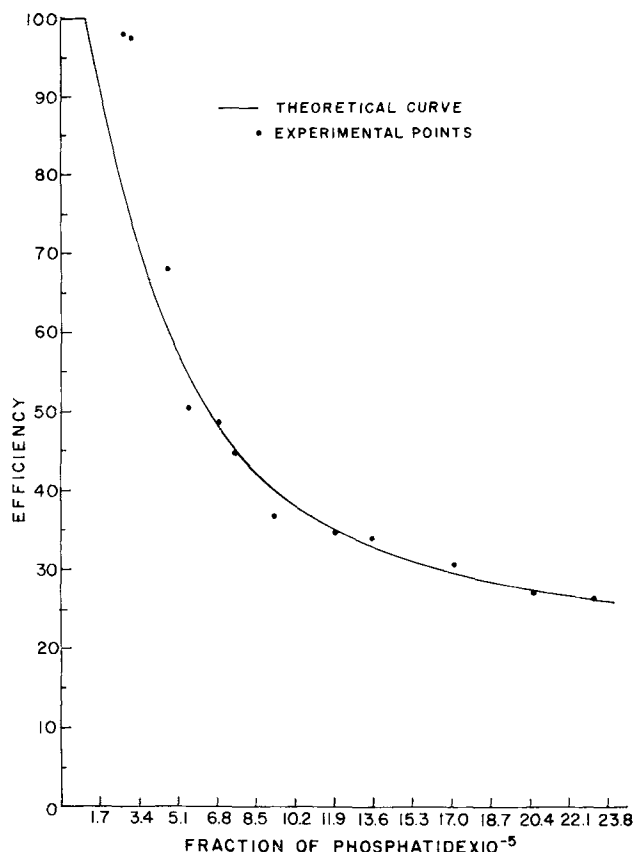


Fig. 6. Efficiency vs. the weight fraction phosphatide (assuming a mol wt 788) at 3.5 hr and 4,000 v.

ted using Equations 15 and 16 and an average of the calculated ϵ_p/ϵ_n values. The experimental results agree well with the theoretical curve calculated in this way, particularly in the region of higher concentration.

The value for the dielectric constant for the phosphatide, $\epsilon_p = 9,321$ (assuming the phosphatide has a molecular weight of 788 and $\epsilon_n = 1.7$), appears to be unusually high for a molecule of this structure and size.

Next, experiments were carried out to test the validity of the case where the nonpolar particles are in limited quantity and accumulate at the periphery of the cylindrical condenser. This was done with an emulsion of mineral oil-tetrachloroethane in water.

Preliminary voltage studies indicated that 5,000 v was the most suitable voltage for the oil emulsion system. First the effect of time on the efficiency of separation was measured using a 1% oil solution. The efficiency was calculated using Equation 2. The efficiency leveled off between 2 and 3 hr similar to that in Figure 5. The plateau efficiency was not as flat as that for phosphatide which may indicate less uniform particle size.

An efficiency vs. concentration study was made at constant voltage. Two different times for equilibration were used. The results are shown in Figure 7. The two curves have the same shape as the one for phosphatide micells. Experimentally feasible concentrations did not give 100% efficiency. The longer time (17 hr) gave a higher efficiency for a given concentration.

Calculations were made to test the validity of the proposed theory. The general shape of the curve is that expected from the theory; however, no positive value of dielectric constants of the oil particles would give a curve which would fit the experimental data.

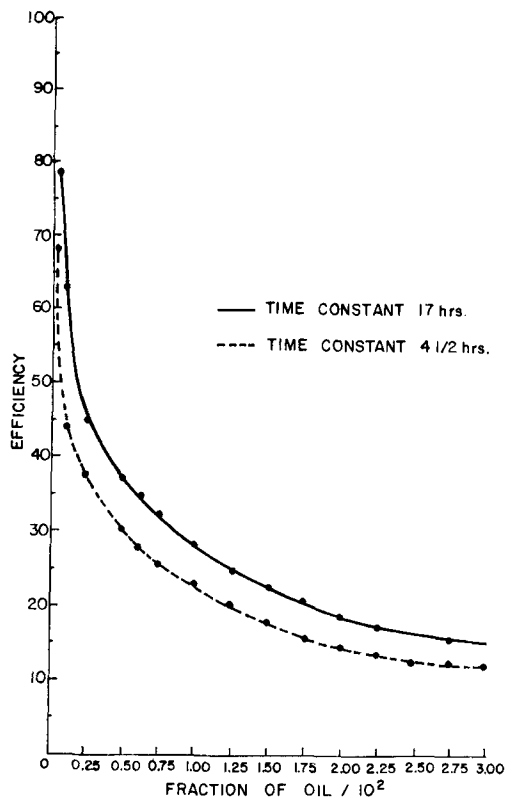


Fig. 7. Efficiency vs. the weight fraction of mineral oil-tetrachloroethane emulsion at 5,000 v and constant times.

The use of turbidity to measure the concentration changes in this system, where the particle size may be nonuniform, may introduce some error, but it is unlikely that it accounts entirely for the failure of the theory.

The dielectric distribution theory derived in this paper correctly predicts the general shape of the efficiency vs. concentration curves, but it fails to give an exact fit with reasonable values of the dielectric constants. The theory also fails to account for the change in equilibrium efficiency with voltage.

Pohl and Schwar (11) have proposed that the dielectrophoretic force is counterbalanced by the accumulation from the central electrode of net charges on the particles. This accumulation of net charges would not cause the particles to move appreciably in the rapidly alternating field, but it would cause the particles to repel each other. If the voltage were high enough, enough charge could accumulate to cause slough-off. Bacterial spore cells were found to form chains in parallel plate condensers (19) and showed no sign of repelling each other. A tendency to form chains could also be noted in a cylindrical condenser. When the voltage was high, the spore chains danced about on the central electrode and acted as if they were buffeted by some force emanating from the central electrode, but they did not repel each other and slough-off. It may be that spores are unusual in this respect, and the phosphatide and oil particles used in this study do accumulate charges and repel each other. The accumulation of net charges on the particles should lead to different distributions in direct and alternating current fields. This was not found to be the case by Pohl and Schwar (11).

Our results indicate that particles accumulating at both the central and peripheral electrodes do not reach the concentration near the electrodes that theory predicts. These results could be accounted for by the

presence of net repulsive charges on the particles. This would also account for the change in equilibrium with voltage. A more sophisticated theory of dielectric distribution which would take this effect into account awaits future development.

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Separation by Dielectric Distribution: Application to the Isolation and Purification of Soybean Phosphatides and Bacterial Spores¹

B. C. BLACK and E. G. HAMMOND,

Department of Dairy and Food Industry, Iowa State University of Science and Technology, Ames, Iowa

Abstract

The redistribution of particle suspensions which occurs in a nonuniform electric field was used to separate soybean phosphatide micells from crude soybean miscella. Two arrangements of the non-uniform field were used. This method provides a convenient laboratory method for isolating and concentrating polar lipids. At present the method is too slow for commercial application but this may become feasible with improvements in apparatus design.

A simple apparatus was described that gave efficient separation of bacterial spores from vegetative cells.

Introduction

DIELECTRIC DISTRIBUTION may be defined as the distribution which mobile materials with different dielectric constants will assume in a nonuniform electric field (1). The more polar material moves toward regions of high field intensity, and less polar material moves toward regions of low field intensity. The application of dielectric distribution to analytical and preparative separations has received little attention in the past. Dielectrophoresis, the motion of particles in a nonuniform electric field, has been studied by several workers (2-8). They concluded the effect would not be appreciable for particles of molecular size, but for particles of 1 to 2 μ or larger, the effect would be quite large.

There are a number of methods which may be used to separate small molecules (chromatography, distillation, partition, etc.). As the particle size increases, these methods become impractical for various reasons. Density separation (centrifugation) and screening (filtration) are essentially the only methods available to work with large particles. Dielectric distribution should be useful in the same range of particle size where density separations and filtration are employed and should supplement these methods.

This paper reports the use of dielectric distributions

for the separation of soybean phosphatides from soybean miscella and for the separation of bacterial spores and vegetative cells.

Experimental

Cylindrical Condenser Apparatus

The cylindrical condenser that was used to separate the phosphatides from neutral fat was similar to that used previously (1). It consisted of a Pyrex glass cylinder 0.8 cm I.D. and 78 cm long. The barrier separating the inner and outer portions of the con-

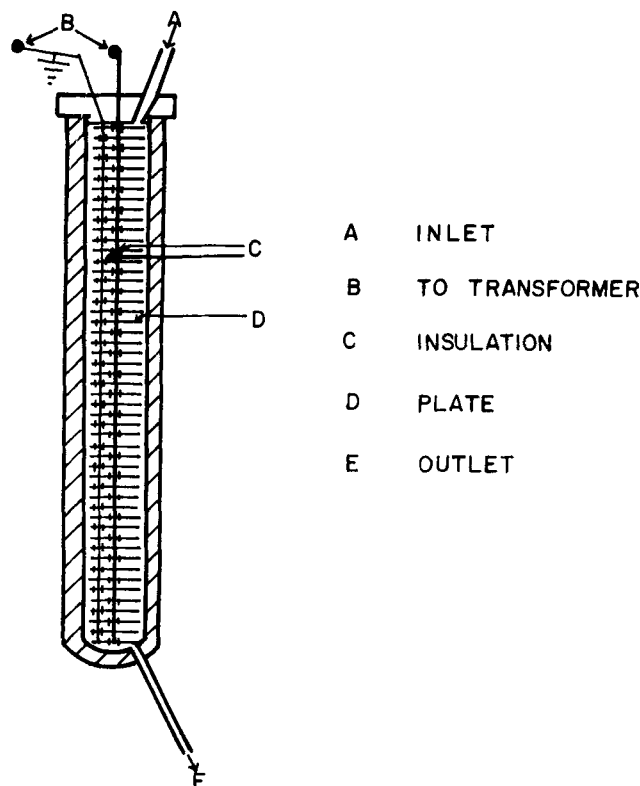


Fig. 1. The plate stack apparatus.

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