FACTORS INFLUENCING DISTRIBUTION OF MICROINGREDIENTS IN MIXED FEEDS

by

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B. S., Kansas State University
of Agriculture and Applied Science, 1954

A THESIS

submitted in partial fulfillment of the

requirements for the degree

MASTER OF SCIENCE

Department of Flour and Feed Milling Industries

KANSAS STATE UNIVERSITY
OF AGRICULTURE AND APPLIED SCIENCE

1960

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INTRODUCTION

Feed microingredients have been defined as nutrients, drugs, stimulants, hormones and preservatives added to feeds at levels of no more than five pounds per ton or 0.25 per cent. Each has a special place in the efficiency of feed conversion and animal health. A typical broiler ration of 15 years ago contained four or five microingredients which supplemented a variety of natural feeds in order to satisfy the requirements for growth. Today 15 to 20 are commonly used to supply a fixed amounts of requirements, supplementing two or three natural feeds which supply protein and calories.

Most microingredients are available from several primary manufacturers. Although the chemical potency is defined and guaranteed, the physical form of the material varies with each supplier and often with each level of potency. Non-uniform distribution of microingredients in formula feeds has been known to occur and is often suspected. Many feed manufacturers have tried to overcome this occurrence by over-fortification. In some cases this has lead to toxicity and in all cases is expensive.

The problem presented to the feed manufacturer after the nutritionist has formulated a ration, is to randomly distribute all ingredients required by the formula and to maintain them uniformly throughout the processes of mixing, conveying, binning, bagging and transportation to the customer and even while being consumed by animals. It has often been proposed that the heterogeneous physical nature of microingredients such as particle size, shape, and density dissimilarities prevents their uniform distribution either during mixing or discharging from the mixer.

Therefore, the objective of this work was to study the distribution of microingredients when they differ in size and density from that of a carrier

of known size and density.

REVIEW OF LITERATURE

A study of the available literature on mixing dry solids indicates that most work has been of theoretical nature using sand, salt, coal, glass beads, or other material of reproducible physical nature. There are also a large number of publications pertaining to designs for mixing equipment. Little was found of direct application to feed materials either on the performance of mixers or the influence of particle size, shape, and density on mixing.

Mixing has long been recognized as an empirical operation, and although important to many industries, it is still more of an art than a science. In fact, an adequate definition of "mixing" or a "mixture", as applied to feed materials, is lacking. Perry (18) theorized that all samples taken from a batch of mixed materials should contain the same components. This indicates that an orderly arrangement is expected and for many years this was accepted as a guide to mixing goals. Only recently have investigators realized that mixing creates disorder rather than order.

Lacey (14) proposed that chance be allowed to determine position of particles in a mix; these chance effects accumulating until an equilibrium state is reached in which disorderliness is at a more or less stable maximum.

Physical Factors Effecting Mixing

Most research workers are aware that mixing and unmixing are continual operations and that an end-point is reached when further mixing or randomness is impossible. However, factors which cause a cycle of mixing and unmixing are likely to occur when mixtures are dissimilar in physical properties and

electrostatic charge (14, 8, 19, 24).

While describing the fundamental principles of segregation, Brown (7) indicated that the coefficient of friction, resilience of particles, shape and surface of the mixer must be considered as well as particle size, shape and density. Although offering no experimental evidence, he suggested that particle size is more important than particle density in causing segregation.

Considering the minute percentages in which some microingredients are added, for example, vitamin B-12 at 4 milligrams per ton, it becomes readily apparent that particle size must be decreased if the material is to be obtained in all samples. Bloom and Livsey (4) discussed the need of decreasing particle size in order to attain dispersion, however they fail to mention any apparent disadvantages that may occur in mixing small particles. While Wornick (25) agrees with the above authors findings, he warns that certain sensitive ingredients are unstable in the finely milled form and are likely to acquire electrostatic properties. This was verified when Danckwerts (12) found that electrostatic charges, roughness and stickiness became most important when small particles were mixed. It has also been noted that as particle size decreases, power consumption and time required to achieve uniformity increases. An example of this point is noted in the comparison of one liter of coffee beans to one liter of 350 mesh particles. When the former material is mixed, 1750 particles must be moved compared to seven billion of the latter (6). Therefore, some workers believe that mixing time is largely dependent upon two factors, particle friction and number to be rearranged.

Effects of Equipment on Mixing

Although dry blending equipment has been widely used in the chemical

and feed industry, the subject of equipment performance has few available published references. This has probably been due, at least in part, to the lack of quantitative methods for testing, which require certain theoretical and experimental tools (23). Various forms of blending apparatus have been used, such as double cones, horizontal mixers, twin shells and cylindrical drums in studying the rate and degree of mixing. A number of mixing rate equations have been developed (9, 15, 22, 23), but their applicability has been useful only for the specific mixer and materials employed. Consequently, while a number of studies have been useful as a guide in improving the performance of mixers, they have been inadequate in determining theoretical calculations of mixing times.

Lacey (15) has described how the rate of mixing is influenced by one of three mechanisms: (a) Convective mixing — transfer of groups of adjacent particles from one location in the mass to another, (b) diffusive mixing — distribution of particles over a freshly developed surface, and (c) shear mixing — the setting up of slipping planes within the mass. The occurrence of any of the above is mainly dependent upon the mixer used.

Yano, et al. (27), studied a number of mixing variables using the twin shell mixer. Employing two powders of -100/+200 Tyler mesh, they found that best mixing was obtained when the mixer was charged approximately a third full and rotated at 50-60 rpm for 2.8 - 3.5 minutes. It is interesting to note that these results were similar to those reported by Coulson and Maitra (9), although the mixer and blending mechanisms were quite different.

Except at the beginning of mixing, Yano, et al. (27) found that the volume ratios of the components mixed had no effect on either mixing rate or degree of mixing. This is of particular significance when one considers the

level of microingredient supplementation that is commonly being used in today's feeds.

Blumberg and Maritz (5) used a simple drum mixer rotated at 55 rpm at a 30° angle with the horizontal. In mixing equal quantities of dyed sand, they found that complete randomness could be attained in approximately 120 seconds, if the sands were of the same size and density.

Coulson and Maitra (9) used a mixing device similar to Blumberg's in studying a number of mixer and material variables on the rate of mixing and segregation tendencies. In studying speeds of rotations, they found 55 rpm to be optimum with poor mixing occurring at either higher or lower speeds. At high speeds, the whole mass rotated against the cylinder wall, while at low speeds the particles rolled down the inclined surface, after leaving their circular paths.

In using different sizes of coal and salt particles these same workers found that practically no mixing occurred if the mixer was first charged with the finer particles. However, if the coarser particles were added first, a state of randomness was obtained followed by unmixing at approximately the same rate. In contrast to these findings, Young and Snaddon (28) found in using an adjustable angle mixer that it was advisable to charge the mixer with the light material first, but the time required for perfect mixing would depend upon particle size, specific gravity and total quantity mixed.

In studying the effects of density differences on mixing, Coulson and Maitra (9) found that a well mixed batch of coal-salt particles segregated upon continued mixing. This was explained as due to differences in the rate at which two materials rolled down the surface as the drum rotated.

A similar segregating phenomenon was observed by Weidenbaum and Bonilla

(22) when they mixed salt and sand particles of similar size and density in a horizontal rotating cylinder. They found after a long period of mixing that the sand concentration was higher in the center of the mixer than at the ends. This tendency was attributed to differences in tumbling properties between salt and sand and to the surface curvature near the end faces of the mixer. These workers believed the higher angle of repose of salt caused less movement of salt toward the center of the mixer. To correct this tendency, a mixer that throws particles was recommended for mixing two such materials.

Oyama and Ayaki (17) mixed different sizes of sand in a mixer similar to Weidenbaums and Bonilla (22) and discovered that radial mixing velocity was extremely rapid in comparison to longitudinal mixing. With uniformity about the radius occurring quickly, they point out that the time required for mixing is, therefore, mainly dependent upon the longitudinal mixing velocity.

Auch (2) has reported that it is not uncommon to find that certain mixtures have optimum mixing times. He has found that in mixing certain chemicals in a horizontal ribbon mixer, a cyclical movement of particles occurs if there is little or no tendency to form agglomerates. This allows the batch to approach uniformity and then recede again. Auch (2) has found this to be true in mixing baking powder containing light redried starch and denser sodium aluminum sulfate. But when light zinc stearate and heavier zinc oxide were mixed, this tendency was not noticed, and the mixture remained mixed regardless of mixing time.

Creger (10) made a study similar to Auchs (2) using sodium chloride and nitrophenide as tracers in a mixed feed and found these ingredients reached a maximum point of being mixed at different time intervals; decreasing from expected levels with prolonged mixing time.

Gray (13) made a number of mixer comparisons studying different materials and found it was difficult to compare the results of various mixers when the components mixed had segregation tendencies. When segregation tendencies were diminished, there were fewer differences among the various mixers. He found that segregation did not always occur, although there were obvious differences in physical properties of the ingredients mixed. For example, using the twin-cone mixer in mixing sand and ilmenite of equal size but with densities of 2.6 and 4.3 respectively, unmixing did not occur regardless of mixing time. However, aluminum and ilmenite of similar densities were incompletely mixed after 92 minutes. This was explained by the segregation tendencies of sand and ilmenite balancing out the mixing effect after a certain level of randomness was reached. But in the latter case, when segregation tendencies were not as great, mixing continued to improve at a slower rate.

Another interesting aspect of Gray's (13) work was the sampling technique employed. He used a probe containing a light and photocell for obtaining measurements related to components of the material adjacent to the photocell. The standard deviations of the reflectivity probe readings were used as a measure of the uniformity of composition of the mixture. Most other sampling techniques used (5, 9, 17, 22, 27) have consisted of using a probe, which has generally involved particle counting in order to test whether or not a batch was randomly mixed.

Adams and Baker (1), in evaluating various types of blending equipment, have found that sampling at the outlet of the blender at equally spaced intervals was most satisfactory. They reasoned that in judgingthe performance of blending equipment, the mixed state should be observed only as the material is discharged from the mixer. This was also one of the few studies conducted

in which the ratio of the quantities mixed were of unequal proportions. It is generally recognized that it is easier to attain uniformity if the substances involved are added in approximate equal quantities than when one component is present in small amounts (16).

Most authors in blending 50:50 mixes of materials have used either the binominal or the normal approximation to the binominal, however, Adams and Baker (1) found that the Poisson distribution was more appropriate for their experiment. Particle counts were made of each sample and illustrated by graphs on how 90, 95 and 99 per cent confidence limits could be drawn for a mean (M), and used in evaluating blender performance. This graphical method was successful in detecting trends in sample composition when the dyed particles were plotted against sample sequence. They stated that if seven successive results occurred on one side of the mean (M), the mixture was nonrandomly mixed.

Smith (20) in commenting on the size of sample indicated that if the particle size of the major constitutent is large, a large total sample must be taken to make the variance due to sample size small in comparison with the per cent additive used.

STATEMENT OF PROBLEM

A literature review indicated innumerable publications on theorizing mixing of solid particles but with little work in the area of mixed feeds. For the most part, the methods described were theortical, using 50:50 mixes of sands, glass beads, salt, or coal. Therefore, this investigation was conducted to study distribution of actual feed materials varying in particle size and density when added to a soybean oil meal carrier with fixed and known

limits of particle size and density.

EQUIPMENT

Mixer

The mixer used was a twin-cone "V" shaped stainless steel shell rotated by a one-eighth horsepower motor at 19 rpm, using gravity tumbling and dividing as principles of mixing (Plate I). A motor driven intensifer bar, which joined the axial supports across the mixer, was removed to permit uninterrupted action by gravity in combining and dividing the contents during rotation. The working capacity was rated as eight quarts.

Over-all height of the mixer was 21 inches with a width of 14 3/4 inches. Diameter of each shell was eight inches. The outlet opening was 2 15/16 inches in length and 1 9/16 inches in diameter with a discharge clearance of six inches.

The outlet plate was at the apex of the "V" hinged to slide away from a pivot position to allow emptying of the mixer. It was modified by enlarging to hold a threaded sampling cup. This was accomplished by boring and threading a three-fourths inch hole in the center of the outlet plate, which allowed a sampling cup to be centered under the outlet opening when the plate was in the closed position.

Sampling Cups

Two aluminum cups with approximate capacities of 2.5 and 32 grams were designed and used. The cups were light in weight and easily attached and detached from the outlet plate (Plate II).

EXPLANATION OF PLATE I

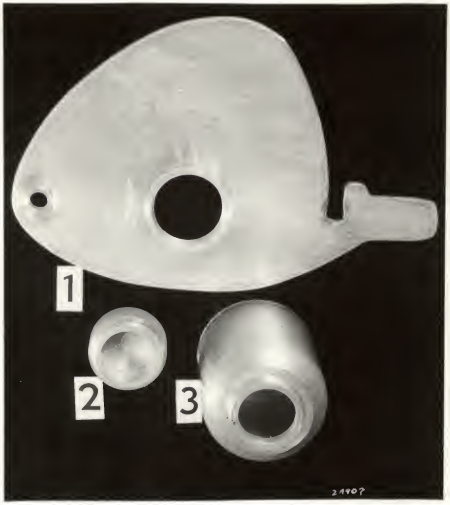
Twin-cone "V" shaped stainless steel mixer.

PLATE I

EXPLANATION OF PLATE II

Enlarged outlet plate (1) with sampling cups (2 and 3).

PLATE II



Sampling Probe

A stainless steel probe 14 inches long, consisting of a hollow tube with an opening at one end measuring three inches long and three-fourths inches wide was constructed. Inside the hollow tube was another tube or shell which was equipped with a round handle that could be rotated, thus allowing the opening to be closed, after the probe had filled. The capacity of the sampling probe was approximately 13 grams.

MATERIALS

Carrier and Size

Solvent process soybean oil meal was used as a carrier of the tracers[1] in these experiments. This material was selected because of its common use as a carrier of commercial feed additives. It is also included in most formula feeds as a protein source. Another important reason for its use as a carrier was because particles fairly uniform in size and shape could be obtained in large amounts by sieving. Other materials used as diluents in feeds, such as ground yellow corn or wheat middlings, allow a large proportion of bran-type particles of the same size but different in shape as compared with the kernel or endosperm portion.

To obtain the desired particle size of carrier, sieve separation tests were made using a Richmond Gyro-Lab continuous sifter. Soybean particles passing through a Tyler 28[2] mesh but retained on a Tyler 35 mesh were used in

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[1]Tracers as used in this study are feed materials that have been dyed and used at microingredient levels.

[2]Hereafter this process shall be denoted as -28/+35.

preliminary trials. This size particle range was selected because it was the size obtained in largest amount upon sieving a variety of poultry rations. After conducting several trials, it became apparent that a coarser carrier would have assisted in obtaining a more complete separation of tracer from carrier.

Carrier particles of -16/+20 were found more suitable because this size range allowed for a wider range of tracer particles to be used. Greater quantity of this size could also be obtained in a shorter sieving time.

Selection and Preparation of Tracers

Beet pulp, solvent processed soybean oil meal, di-calcium phosphate, and ground rock phosphate were selected as materials to be used as tracers. These feed materials were chosen for the following reasons: (a) availability, (b) a wide range of the desired particle sizes could be obtained by sieving, (c) their densities were representative of the wide range in densities of many microingredients currently being used in today's formula feeds. The physical properties of these materials are shown in Table 1.

Table 1. Physical properties of tested tracers.

Tracers	Beet pulp	Soybean oil meal	Di-cal phos.	Gr. rock phos.
Density (gr/cc)	1.43	1.41	2.33	3.00
Bulk density (lbs/ft^3)	15.52	36.80	41.40	77.04
Particles/gram	2200	2290	1620	940
Angle of repose(.)	40.1	25	30.2	31.3
Particle shape	long-spiral	elongated	spherical	spherical

To obtain the desired particle size, sieve separation tests were made using the Tyler ro-tap. Homogenity of particle size was obtained by sieving the material two or three times. However, uniformity of tracer varied according to size and density of the material used. For example, more uniform shaped particles were obtained when either di-calcium phosphate or ground rock phosphate were used. Beet pulp and soybean oil meal tended to be irregular in shape, especially as particle size increased.

The procedure used in preparing the tracer material was to place the sieved material on a paper and spray with a colored dye. By drying and re-sieving this, a trace material of the desired size was obtained. At first, a red vegetable dye, soluble only in water was used. This dye was found unsuitable however, because of clumping and sticking of dyed particles and time required for drying. A crystal violet dye, soluble in most organic solvents, was found more suitable. A small amount of the powdery material was dissolved in acetone, added to a hand sprayer, and then sprayed on the sieved material. Since acetone is a non-wetting volatile solvent, little drying was necessary.

FIXED VARIABLES

Total Quantity Mixed

Total quantity mixed was 1000 grams. This amount was used because it occupied approximately one-third of the total working capacity of the mixer, thus allowing for maximum tumbling and dividing action of the material mixed. This capacity was also in agreement with recent findings by Yano, et al. (27) which indicate best blending can be expected when the mixer is approximately 30 per cent charged.

Per Cent Tracer Used

Preliminary investigations of this research involved conducting a number of trials using various levels of tracer. Tracers were added at 1 per cent, 0.5 per cent, and 0.25 per cent. When sampled, no differences were observed in pattern of distribution or total percentage recovered. Therefore, to meet the normal requirement of a formula feed and to lessen the tedious task of counting the tracer present in a sample, 0.25 per cent tracer was used as a standard addition in the subsequent trials conducted.

Mixing Time

Since this study was begun by using standardized size of particles, total weights mixed, and RPM of the mixer, a mixing time was selected to assure maximum effect of the mixer to attain randomness under these conditions. Randomness was tested by sampling with probe in a number of selected spots, after the mixer had revolved a number of turns. This procedure was employed at 50, 100, 150, and 200 revolutions. An erratic recovery of tracer was obtained when mixed less than 50 turns. Mixing longer than 100 turns did not improve distribution of tracer. Therefore, 100 turns, equivalent to five minutes plus five revolutions, was established as a standard mixing time. This was also in agreement with recent results obtained with the twin-shell blender by Adams and Baker (1). They indicated the mixed state was reached in 100 turns and that 300 turns did not improve uniformity.

PROCEDURE

The procedure followed in conducting a trial with the mixer was to accurately weigh sieved soybean oil meal on a gram-scale. This weighed material,

which served as a carrier, was then added to the mixer through one of the inlet openings. Tracer materials were weighed on an analytical balance and added in a clump on top of the carrier in center of the mixer. When the mixer was closed tightly, it was rotated 100 revolutions, stopped in discharge position and sampled by removing all of the material in a fixed sequence via the use of sampling cups.

Sampling was done by moving the discharge plate to the right, attaching a sampling cup, then slowly moving the discharge plate back to its original position. After the cup had filled, the plate was again moved to the right and the cup removed and emptied on a watch glass. The first and third samples thereafter, until a total of ten, were used to measure mixing. Approximately 10 per cent of the material remained in the mixer, after ten samples were withdrawn.

A larger sampling cup was employed when the tracer particles were smaller than the carrier. Larger samples were necessary in order to obtain tracer in sufficient amounts to be weighed accurately after separating by sieving. When the tracer and carrier were the same size or larger, separation of tracer by sieving was impossible and tracer was counted by inspection. Small samples were used to lessen the tedious task of counting recovered material.

In an effort to obtain samples in the same approximate sequence with the smaller cup as with the larger, a modification was made in the sampling sequence. This was done by removing the first sample with the smaller cup, removing and discarding the next three samples with the larger cup. A repetition of this process was employed for all succeeding samples until ten were withdrawn.

After obtaining the required number of samples, each sample was then weighed to the third decimal place and tracer either counted and expressed as

dyed particles per sample or separated from carrier by sieving and expressed as per cent tracer per sample. In the latter case, samples were placed on a Tyler sieve which allowed tracer to be separated when the sieve was gently rotated by hand. After tracer was removed from carrier, it was again weighed to the third decimal place and per cent tracer calculated for each sample.

ANALYTICAL METHODS

Determination of Bulk Density

The standard procedure and apparatus as prescribed in the Handbook of Official Grain Standards of the United States for determining bushel weight was used in bulk density determinations. The weight per bushel was then converted to pounds per cubic foot.

Determination of True Density

The specific gravity of beet pulp, di-calcium phosphate, and ground rock phosphate were determined by the State Highway Commission using the Le Chatlier Method. Since many different lots of soybean oil meal were used, no attempt was made to calculate the density of this ingredient. The true density value for soybean oil meal indicated in Table 1 was quoted from data described by Creswell (11).

Determination of Angle of Repose

The following procedure was used in determining the angle of repose. The material to be tested was placed in a plastic funnel, approximately 7 1/2 inches high and 6 inches inside diameter, with an outlet diameter of 3/4 of an inch. The material was then allowed to flow from the funnel onto

a wooden board at an approximate height of six inches. It formed a cone whose base and height were then measured. Angle of repose, $\theta = \tan^{-1} \frac{\text{ht.}}{\text{base}/2}$.

STATISTICAL AND GRAPHICAL INTERPRETATION

The statistical model used by most workers in citing blending results of 50:50 mixes of materials has been the binominal distribution. However, in the present study of blending a small quantity of tracer particles with a larger number of carrier particles of equal size, the Poisson distribution is more useful. Since method of determining tracer present in a sample, when tracer was similar-sized or larger than carrier, consisted of counting number of tracer particles, the method of analysis was that of setting up control limits such as used in quality control work. The chance variable was the number of tracer particles per sample and it was assumed that the Poisson distribution approximated the distribution of this chance variable. For each trial, the value of the parameter (c) was approximated by the following formula:

(c) = (ave. wt. per sample) (no. of particles per gram) (per cent of tracer added)

The following control limits were generally used:

$$LCL^1 = c - 3\sqrt{c}$$

where $$UCL^1 = c + 3\sqrt{c}$$

$$\sigma = \sqrt{c}$$

so that there was approximately a 3 in 1000 chance of the particle count falling outside the control limits if the process produced random mixing and sampling.

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[1]Referring to Upper and Lower Control Limits.

Alternatively ($\bar{x}$), the average number of tracer particles per sample, can also be used as an estimate of (c), providing an adequate number of trials are conducted. This average as an estimate of (c) will not significantly effect the relationships presented in the following plates.

DESIGN OF EXPERIMENT

For each trial conducted, the questions of interest were: (a) Do the tracer particles distribute themselves in random fashion throughout the mix? (b) If the tracer particles are distributed randomly, does sampling from the point of discharge from the mixer upset the random distribution of particles? The design of the experiment was such that both questions could not be answered for each trial or a series of trials. Consequently, the two processes (a) mixing of material (b) emptying of the mixer, must, for the purpose of this study, be considered as a single process. Thus, for each experimental condition conducted, the hypothesis that, mixing and sampling produces a random distribution of tracer particles in a mix, was investigated.

While it is difficult to define what is meant by saying that particles are distributed in a random fashion, it can be noted that the occurrence of events where it appears that factors, other than the so-called chance variables are operative. The result is that the sample contains an unexpectedly large or small number of tracer particles.

It was also decided that a series of trials would be conducted for each tracer and size studied. This generally consisted of five to ten trials. This not only tended to present a better indication of any trends occurring, but also gave greater confidence in results obtained. The graphs and statistical analysis had as their purpose the presentation of such situations. In any

statistical work, it is generally desirable to have a graphical means of examining the data, since this often gives all the information that is required.

Initial studies involved the use of soybean oil meal tracer of the same density as soybean oil meal carrier. After conducting a thorough study of particles smaller, similar and larger in size than carrier, attention was diverted to inhibiting segregation tendencies, when tracer particle size was smaller than that of the carrier. This was accomplished by replacing a portion of the larger carrier (-16/+20) with equal amounts of smaller carrier material. Initially, 10 per cent (-32/+40) was used, while blending soybean tracer of (-80/+100) for several trials. Although distribution of tracer was greatly improved, segregating tendencies were still apparent. Therefore, 30 per cent (-40/+48) was used in blending soybean tracer of (-100/+115). A smaller-sized tracer was used in this case to facilitate complete separation of tracer from carrier upon sieving.

Further elements of this investigation involved blending of tracers slightly greater, much greater, and much less in bulk density than carrier. Di-calcium phosphate, ground rock phosphate, and beet pulp were the respective materials used in each of these instances. Reference is made in Table 1 to the densities of these ingredients. A study of these materials was limited to the study of particle sizes similar to (-16/+20) and much smaller than carrier (-80/+100). Thirty per cent of (-40/+48) was also used in study of ground rock phosphate, and it is expected similar complementary effects can be predicated while blending di-calcium phosphate, although this was not investigated.

During the study of beet pulp tracer (-80/+100), 20 trials were conducted.

The experimental procedure employed during the first 10 trials was the same as previously described, however, the last 10 trials comprised the use of a carrier that had been previously used. Upon the completion of each of these trials, the carrier was subjected to further sieving to remove all tracer and then used again in the next trial. Although this procedure was conducted with no planned significance, marked differences were obtained in distribution of the tracer.

The last phase of this work involved the addition of one per cent vegetable oil to the soybean oil meal carrier as an additional attempt to improve distribution of ground rock phosphate tracer. The oil was applied to the carrier by a small hand sprayer and then thoroughly mixed by hand, thereby obtaining an even distribution of the oil. Tracer was then added and the material mixed in the usual manner.

RESULTS AND DISCUSSION

Distribution of Soybean Oil Meal Tracer Similar, Larger and Smaller in Size than Carrier

Plate III shows the distribution of soybean oil meal as a tracer of the same size and material as the carrier. All samples contained particle counts within plus or minus three times the standard deviation ($\sqrt{c}$) of the theoretical mean. Therefore, there was no evidence to indicate non-random mixing.

The distribution of soybean oil meal as a tracer of larger particle size than the carrier is shown in Plates IV and V. Recovery of tracer particles (-20/+28), as shown in Plate IV, are all within three ($\sqrt{c}$) of the theoretical mean, indicating random distribution. Plate V shows distribution obtained when tracer is much larger (-14/+20) than carrier. No trend is discerned;

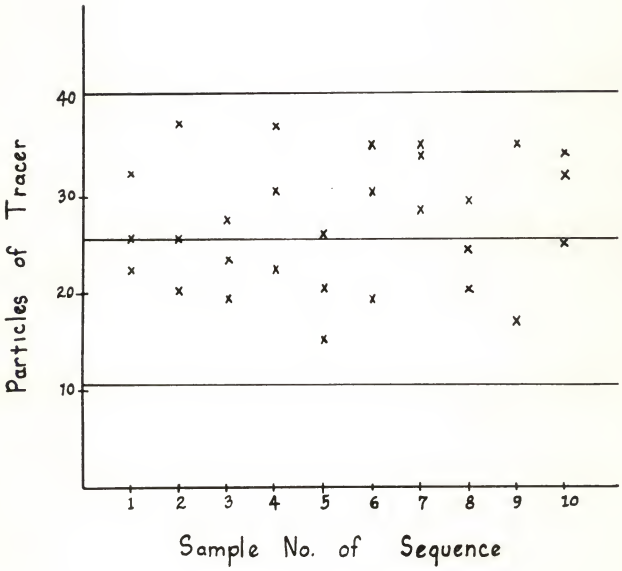
EXPLANATION OF PLATE III

Distribution of Soybean Oil Meal Tracer similar in size as carrier:

Carrier -28/+35

Tracer -28/+35

PLATE III


Particles of Tracer
40
30
20
10
1
2
3
4
5
6
7
8
9
10
Sample No. of Sequence

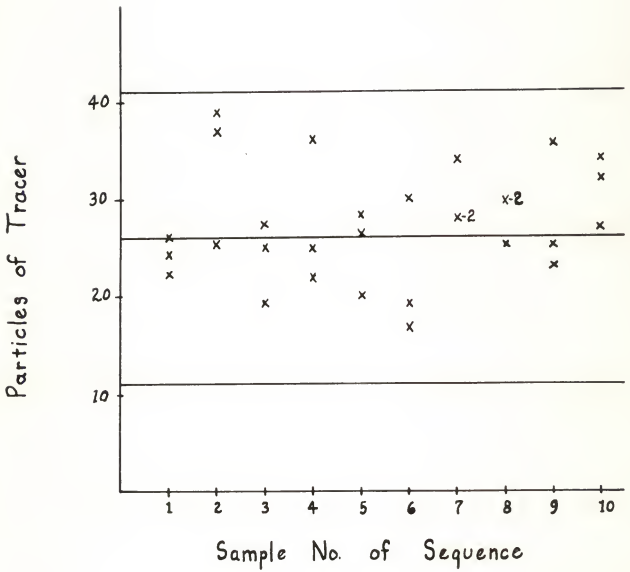
EXPLANATION OF PLATE IV

Distribution of Soybean Oil Meal Tracer larger in size than carrier.

Carrier -28/+35

Tracer -20/+28

PLATE IV


Particles of Tracer
40
30
20
10
1
2
3
4
5
6
7
8
9
10
x-2
x-2
Sample No. of Sequence

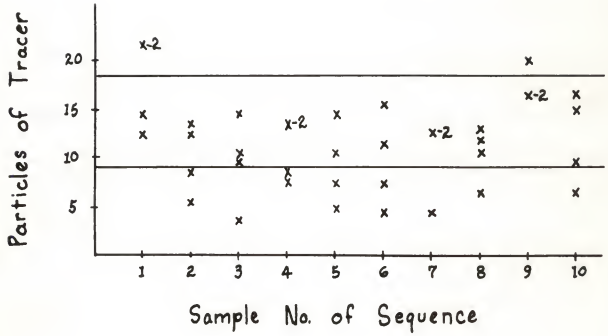
EXPLANATION OF PLATE V

Distribution of Soybean Oil Meal Tracer larger in size than carrier.

Carrier -28/+35

Tracer -14/+20

PLATE V


Particles of Tracer
20
15
10
5
x-2
1
2
3
4
5
6
7
8
9
10
Sample No. of Sequence

however, three counts were beyond the control limits, and the probability is .001 that those samples containing particles outside the control limits did not come from a perfect mix. This occurrence may be due in part to the large difference in size between carrier and tracer. Variance in particle shape was also apparent when this size tracer was blended, which could conceivably effect the flow properties of the material mixed.

A summary of results obtained in blending various sizes of soybean oil meal tracer smaller than carrier is shown in Plate VI. These observations were averaged and plotted as one because of their similarity of distribution. The graph shows that there is a definite trend toward a high concentration of tracer in the first few samples. This observation indicated that random distribution was inhibited during the mixing and sampling process of particles smaller than the carrier.

Since the design of the experiment included mixing and sampling of material as one process, an effort was made to test the mixture prior to removing cup samples by sampling with a probe. Areas of widely different concentrations of tracer were revealed by this sampling procedure. It was observed that this sampling device tended to push aside particles, disturbing material in closely adjacent areas and possibly allowing additional segregation to occur as material filled the probe reservoir.

Failure to obtain randomness of soybean oil meal tracer, when considerably smaller than carrier, indicated the possible importance of employing corrective measures to narrow the particle size differences between carrier and tracer. Therefore 10 per cent and 30 per cent of (-16/+20) carrier was replaced with equal amounts of (-32/+40) and (-40/+48) carrier material, respectively. The results obtained with ten trials each is shown graphically

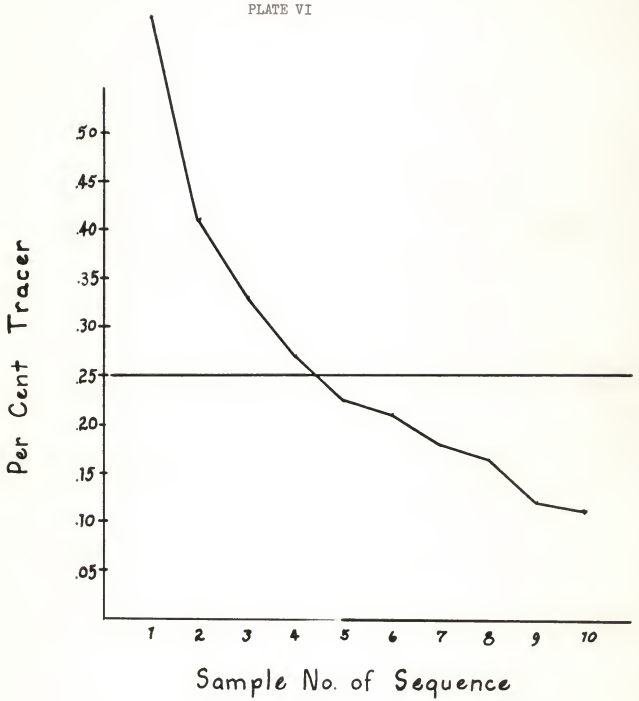
EXPLANATION OF PLATE VI

Distribution of Soybean Oil Meal Tracer smaller in size than carrier.

Carrier -16/+20

Tracer -48/+60; -60/+80; -80/+100

PLATE VI


Per Cent Tracer
.50
.45
.40
.35
.30
.25
.20
.15
.10
.05
1
2
3
4
5
6
7
8
9
10
Sample No. of Sequence

in Plate VII.

In the former case, using 10 per cent of (-32/+40) carrier, a trend is evident toward incomplete mixing of tracer and carrier, although a marked improvement is apparent for all samples tested. The variable position in the sample number of sequence within a trial showed a significant effect evidenced by an F value for which $P < .001$ under the null hypothesis (21). Using the L.S.D. (least significant difference) to further detect effect of position in the sampling sequence, one finds that the per cent tracer for the first sample removed was significantly higher than the second sample. However, the sample means for the second sample through the tenth sample showed no significant differences.

In the latter experiment virtually perfect blending was obtained, verified by non-significant F and L.S.D. values. Thus it can be stated in this experiment that the addition of a smaller-sized carrier had a marked influence on the distribution of tracer. The most logical explanation is that the smaller carrier filled air spaces between the larger particles, decreasing viscosity or friction between larger particles and allowing all particles to flow at the same rate during mixing and sampling. Thus tracer separation from carrier was inhibited. It was also likely that particle shape of the smaller carrier filler material was more near that of the tracer.

Distribution of Di-Calcium Phosphate Tracer

Plate VIII shows the dispersion of di-calcium phosphate tracer similar in size to the carrier. No segregation trend is evident although two counts are beyond the upper control limit. While no explanation is apparent, it may be noted that these occurrences were observed in the first and last samples

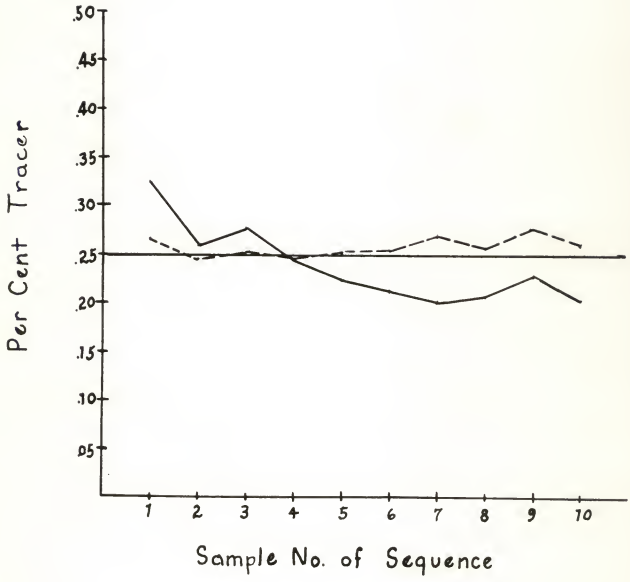
EXPLANATION OF PLATE VII

Distribution of Soybean Oil Meal Tracer smaller in size than carrier (-100/+115) when 10 per cent and 30 per cent (-16/+20) carrier is replaced with 10 per cent and 30 per cent of (-32/+40) and (-40/+48) carrier filler, respectively.

——— 10 per cent (-32/+40)

– – – – – 30 per cent (-40/+48)

PLATE VII


.50
.45
.40
.35
.30
.25
.20
.15
.10
.05
Per Cent Tracer
1
2
3
4
5
6
7
8
9
10
Sample No. of Sequence

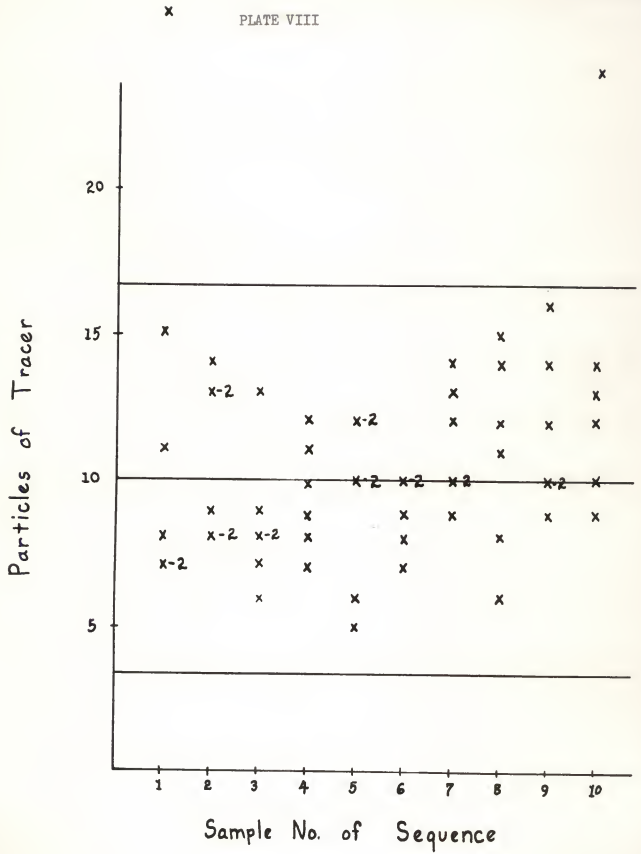
EXPLANATION OF PLATE VIII

Distribution of Di-Calcium Phosphate Tracer similar in size as carrier.

Carrier -16/+20

Tracer -16/+20

PLATE VIII


Particles of Tracer
20
15
10
5
1
2
3
4
5
6
7
8
9
10
Sample No. of Sequence

of the first trial conducted and may be due to experimental error. No similar observations were noted in samples of five additional trials.

It was also noted that there were differences between trials characterized by a significant F value for which $P < .001$ under the null hypothesis. Most of the trials were conducted on the same day so that differences due to atmospheric conditions or other external disturbances were minimized. This tendency may be explained as due to differences in density of individual particles. This was indicated when it was noticed that the number of particles in a gram of trace material often varied. Since tracer was added on a weight basis, this could allow total number of tracer particles to vary from one trial to another.

Plate IX shows a study of di-calcium phosphate tracer smaller (-80/+100) than the carrier. A segregating tendency similar to that observed in blending soybean oil meal tracer (Plate VI) was noted. Despite the fact that true and bulk density of di-calcium phosphate was greater than the carrier, apparently there was no additive segregation-effect in blending these materials. Although gravitational force is greater with greater particle density, size segregation occurs much more readily than density segregation (7).

Distribution of Ground Rock Phosphate Tracer

The graph in Plate X presents the distribution of ground rock phosphate tracer similar in size as the carrier. True and bulk densities were approximately twice that of the carrier (see FigureI). Although all but three counts are within the control limits, a definite trend was noted toward a low concentration of tracer in the first and last samples. However, in regard to samples two through seven, 29 counts were above the mean and only nine

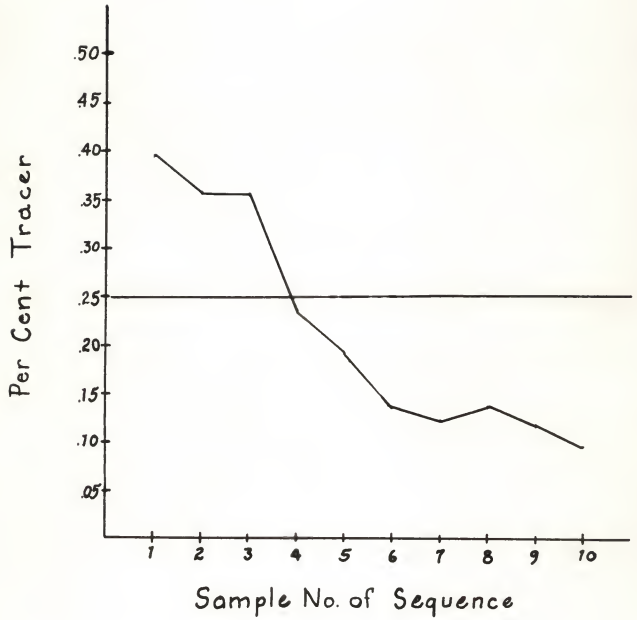
EXPLANATION OF PLATE IX

Distribution of Di-Calcium Phosphate Tracer smaller in size than carrier.

Carrier -16/+20

Tracer -80/+100

PLATE IX


Per Cent Tracer
.50
.45
.40
.35
.30
.25
.20
.15
.10
.05
1
2
3
4
5
6
7
8
9
10
Sample No. of Sequence

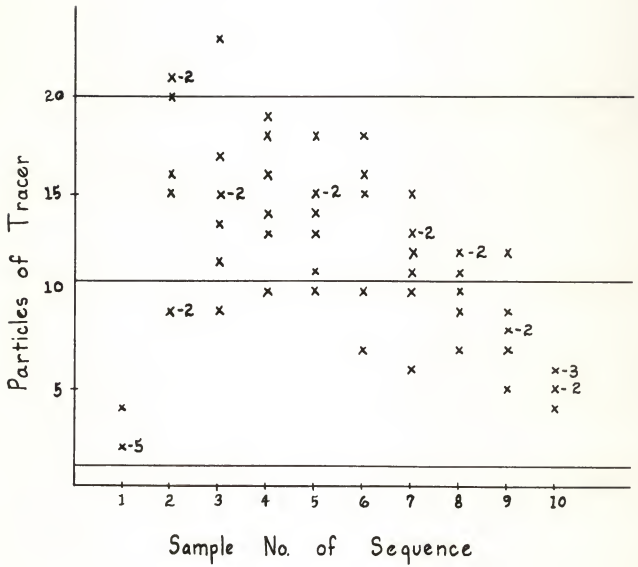
EXPLANATION OF PLATE X

Distribution of Ground Rock Phosphate Tracer similar in size as carrier.

Carrier -16/+20

Tracer -16/+20

PLATE X


Particles of Tracer
20
15
10
5
x-2
x-2
x-2
x-2
x-2
x-2
x-2
x-2
x-3
x-2
x-5
1
2
3
4
5
6
7
8
9
10
Sample No. of Sequence

counts were below the mean. While it is difficult to explain the first observation, the second event is doubtlessly due to the heavier particles settling toward the center of the mixer during sampling and thus being removed first. Similar results have been noted by Hall from an article by Adams and Baker (1) and Coulson and Maitra (9), but the mixing devices employed were quite different. The fact the first sample contained fewer tracer particles might be accredited to differences in particle shape of carrier and tracer. In other words, during mixing the more flaky, elongated-shaped carrier particles may retard the flow properties of the tracer, hence the first sample removed is low. But since design of the experiment included removing and discarding three large cups of material prior to removal of the second sample, this could allow sifting to occur between succeeding samples removed.

The graph in Plate XI demonstrates the effect of blending ground rock phosphate tracer with and without 30 per cent smaller material added as a filler. In the latter instance, the usual tendency to separate from the carrier was noted. This occurrence appeared to be slightly more pronounced than that observed in blending di-calcium phosphate or soybean oil meal tracer; especially the last five samples tested. Although the addition of the smaller carrier material largely minimized segregating tendencies, apparently when there are extreme differences in density coupled with large differences in particle size, segregation cannot be entirely curtailed by this method.

Distribution of Beet Pulp Tracer

Plate XII illustrates the dispersion of beet pulp tracer similar in size as carrier. The distribution of beet pulp showed a trend opposite that incurred while blending ground rock phosphate in Plate X. As indicated by

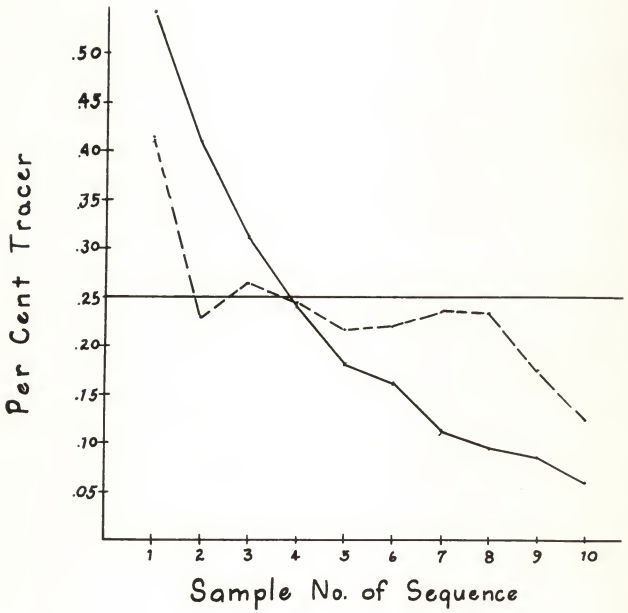
EXPLANATION OF PLATE XI

Distribution of Ground Rock Phosphate Tracer smaller in size than carrier (-80/+100) when blended with and without 30 per cent (-40/+48) carrier material added.

_____ Without 30 per cent

----- With 30 per cent

PLATE XI


Per Cent Tracer
.50
.45
.40
.35
.30
.25
.20
.15
.10
.05
1
2
3
4
5
6
7
8
9
10
Sample No. of Sequence

EXPLANATION OF PLATE XII

Distribution of Beet Pulp Tracer similar in size as carrier (-16/+20).

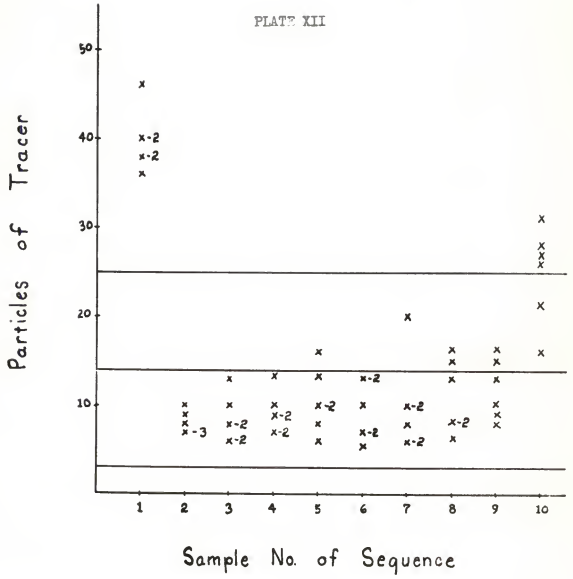


PLATE XII
Particles of Tracer
50
40
30
20
10
1
2
3
4
5
6
7
8
9
10
Sample No. of Sequence

the graph, the first and last samples were generally beyond the upper control limit, while in samples two through nine, 42 counts were below the mean and only eight appeared above. Apparently differences in particle shape and density between individual beet pulp particles were enough to allow segregation to occur. While some particles were similar in shape as carrier, others were fibrous and spirally-shaped. The fact that there were differences in density of individual particles became apparent during the determination of specific gravity. Approximately 1.5 per cent of the sample used in the determination would not sink in the kerosene solution used. This seems to imply that beet pulp of a given size may be composed of particles of various densities. If this reasoning is correct, one might then expect a wide variance in the density of the tracer present in the first and last samples. This, however, was not investigated.

Another possible explanation of the above pattern obtained may be the result of the greater inherent frictional forces characteristic of material (beet pulp) with higher angle of repose (Table 1). As a consequence during mixing, radial movement of the tracer was greater than longitudinal movement; thus resulting in tracer particles concentrating toward the apex of the "V" as the mixer rotated. Consequently, the first sample contained a high concentration of tracer. However, during the removal of succeeding samples, the tracer was again held back and thus caused a higher concentration of tracer in the last two samples.

Previous research by Weidenbaum (22) and Oyama (17), using a rotating horizontal cylinder, have likewise indicated that material characterized by a high angle of repose tends to affect the horizontal movement and tumbling properties of material blended.

Results obtained from the distribution of beet pulp tracer smaller than carrier (-80/+100) may be observed from two graphs in Plate XIII. Graph A represents the average results obtained after conducting ten trials; following the usual method of procedure. Effect of particle size on pattern of distribution is readily apparent and is substantiated statistically by a significant F value of 93.35, where $P < .001$ under the null hypothesis. Ten additional trials were then subsequently conducted using a lot of soybean oil meal that had been employed in previous tests. At the completion of each trial, the carrier was prepared for the next trial by separating the trace material from the carrier by sieving. As indicated by Graph B, a significant improvement in distribution of beet pulp tracer was attained using this method. Possibly there was a physical alteration in the carrier employed in Graph B, resulting from the continued sieving action or friction of beet pulp particles mixed. However, measurement of angle of repose coupled with microscopic inspection and chemical analysis of fat and moisture content revealed no significant differences in the two carriers.

Addition of Oil to Carrier

Finally, as a result of these latter observations between carriers, an attempt was made to alter the surface texture of the carrier by adding one per cent vegetable oil. Ground rock phosphate tracer (-80/+100) blended with carrier containing one per cent added oil showed no tendency to segregate from the carrier in the first few samples (Plate XIV). Rather the converse of the tendency noted in Plate XI occurred, resulting in the last few samples containing higher quantities of tracer than those removed initially. The added fat doubtlessly affected the flow properties of the blended material

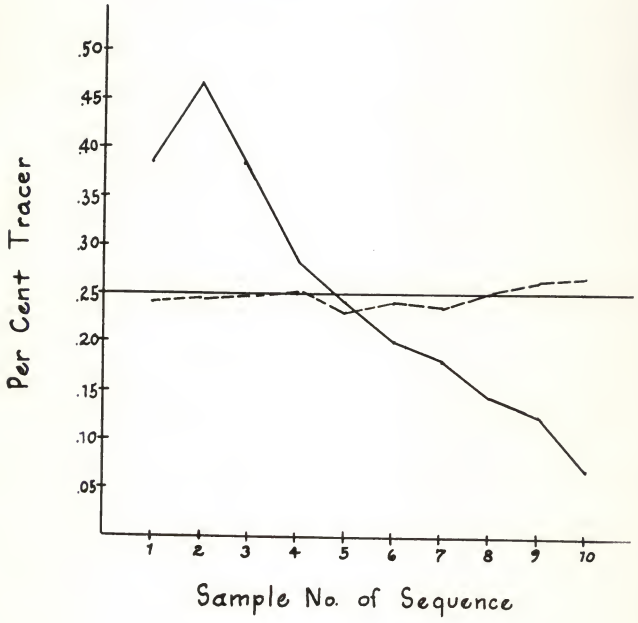
EXPLANATION OF PLATE XIII

Distribution of Beet Pulp Tracer smaller in size than carrier (-80/+100) when blended with two different lots of carrier.

_____ Graph A using a new lot of carrier

------ Graph B using a re-used lot of carrier

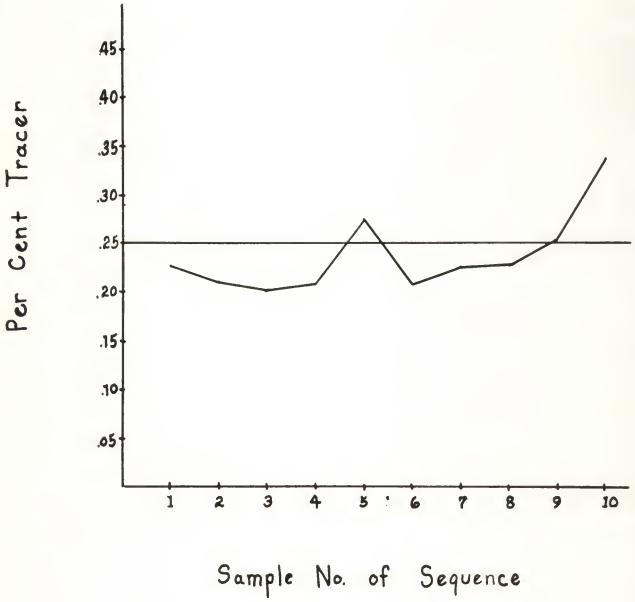
PLATE XIII


Per Cent Tracer
.50
.45
.40
.35
.30
.25
.20
.15
.10
.05
1
2
3
4
5
6
7
8
9
10
Sample No. of Sequence

EXPLANATION OF PLATE XIV

Distribution of Ground Rock Phosphate Tracer (-80/+100) with one per cent vegetable oil added.

PLATE XIV


Per Cent Tracer
.45
.40
.35
.30
.25
.20
.15
.10
.05
1
2
3
4
5
6
7
8
9
10
Sample No. of Sequence

during the sampling process, evidenced by material "caking" and breaking off in sections. It is also reasonable to assume that friction of particle against particle was increased by this procedure and hence retarded separation of tracer from carrier.

SUMMARY AND CONCLUSIONS

Using a standardized mixing and sampling procedure with a uniform carrier material and dyed particles of various materials added at microingredient levels, the following observations are reported:

1. When tracer was of the same material and of the same size or larger than the carrier, no clear-cut evidence was obtained to indicate non-random mixing.

2. Degree of mixing may be expressed as particle counts per sample, providing upper and lower control limits are established. This is possible if (c), the theoretical mean, is determined.

3. Blending tracer of the same material and density but smaller in size than carrier, a marked tendency to segregate in the first few samples was observed. A similar tendency was likewise observed in blending tracers of different densities but of smaller size than carrier.

4. Ten per cent and 30 per cent additive filler material of intermediate size between carrier and tracer was effective in improving the physical factors affecting segregation. Thirty per cent was more effective than ten per cent.

5. Particle size is likely the most important physical factor influencing segregation; however, density may become a limiting factor when there are extreme differences in material blended.

6. One per cent vegetable oil added to soybean oil meal carrier retarded separation of ground rock phosphate tracer from the carrier in the first few samples, allowing a higher concentration in the last few samples. This tendency may suggest that when friction of particle against particle is increased, separation due to differences in physical properties will be minimized.

7. A difference in the physical texture, was noted, between lots of carrier, which affected the distribution of beet pulp tracer.

ACKNOWLEDGMENTS

The writer wishes to extend sincere appreciation to Dr. Waldon Hastings for his untiring guidance and assistance in designing and writing the present study; to Dr. John A. Shellenberger for the provision of equipment and laboratory facilities; to Dr. Arlin M. Feyerhern for his assistance and supervision on the statistical aspects of this work; faculty members and students of the Department of Flour and Feed Milling Industries who either in the capacity of friends or advisers, contributed to the work with their stimulating conversation and suggestions.

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FACTORS INFLUENCING DISTRIBUTION OF MICROINGREDIENTS IN MIXED FEEDS

by

LA RUE BURLEIGH KABANCE

B. S., Kansas State University
of Agriculture and Applied Science, 1954

AN ABSTRACT OF A THESIS

submitted in partial fulfillment of the

requirements for the degree

MASTER OF SCIENCE

Department of Flour and Feed Milling Industries

KANSAS STATE UNIVERSITY
OF AGRICULTURE AND APPLIED SCIENCE

1960

The distribution of dyed feed materials varying in particle size and density, when added at microingredient levels to a soybean oil meal carrier of fixed size, was investigated by removing cup samples from the point of discharge of a laboratory twin shell mixer.

Charged volume of material blended, per cent tracer added, mixing time and RPM of the mixer employed were fixed at the following amounts, respectively: 100 grams; 0.25 per cent; 100 revolutions; 19 rpm.

A study involving the distribution of dyed tracer of the same material and of similar or slightly larger size than carrier gave no definite evidence of nonrandom mixing. The mixing and sampling process did not produce a random distribution of tracer material varying in particle density but of similar size as carrier.

Tracer material smaller in size and similar or varying in bulk and true density from that of the carrier, tended to separate from the carrier during the mixing and sampling process. However the addition of fixed quantities of smaller-sized filler material tended to retard the separation of tracer. Addition of vegetable oil to the soybean oil meal carrier also had an inhibiting effect on segregation of tracer.

Noted differences were observed in the physical texture between different lots of soybean oil meal carrier, which effected the distribution of tracer tested.