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Full Length Research Paper

Development of model equation for oil extraction from maize seed by factorial analysis

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ABSTRACT

This paper intends to develop a mathematical model that fits extraction process of vegetable oil from maize seed by factorial analysis: statistical method. The result of the studies of the extraction of vegetable oil from maize seed by leaching using normal hexane as solvent in a laboratory size soxhlet apparatus serves as the data for this work. In factorial analysis a number of factors that are responsible for the oil extraction that have been experimentally investigated are selected. Three factors- mass of solid, size of solid particles and volume of solvent are selectively compared. Two out of three factors were fixed while one factor was varied in eight degrees of freedom in a 54 run experiment. The physical model gives a numerical advantage of the optimum qualities of variables to be manipulated, as well as values for time and energy commitment, in the recovery of oil in commercial quantity. This oil will be a ready source energy and vitality for continuous existence of life.

Keywords: Vegetable oil, maize, hexane, factorial analysis

INTRODUCTION

For many years maize that is a major agricultural produce in Nigeria serves as a source of carbohydrate or starch whereas vegetable oil in commercial qualities can be recovered from it. Theoretically 10% w/w oil is said to be present but experiment further confirmed that 14% w/w of oil is recoverable from as little as 0.0015kg of seed.

Maize is a carbohydrate agricultural produce commercially produced in the northern part of Nigeria. The extraction of oil from maize is accomplished through a unit operation of chemical engineering known as leaching.

Factors Controlling and Influencing the Rate of Extraction

The selection of equipment for extraction process depends on controlling and influencing factors, which are responsible for limiting the rate of extraction. If the diffuse of the solute through the porous structure of the residual solids is the controlling factor, the material should be of small size so that the distance the solute has to travel is small. On the other hand, if diffusion of the solute from the surface of the particles to the bulk is the controlling factor a high degree of agitation of the fluid is required there are four important factors to be considered as follows:

Particle Size

Particles size influences the extraction rate in a number of ways. The smaller the particles size, the greater is the interfacial area between the solid and liquid, and therefore the higher is the rate of transfer of material and the smaller is the distance the solute must diffuse within the solid. It is desirable that the range of particle size should be small so that each particle requires approximately the same time for extraction, and in particular, the production of a large amount of fine material should be avoided as this may wedge in the interstices of the larger particles and impede the flow of the solvent.

Solvent

The liquid chosen should be a good selective solvent and its viscosity should be sufficiently low for it to circulate freely. Generally, a relative pure solvent will be used initially, but as the extraction proceeds the concentration of solutes will increase and the rate of extraction will progressively decrease, first because the concentration gradient will be reduced, and secondly because the solution will generally become more viscous.

Temperature

In most cases, the solubility of the material, which has been extracted, will increase with temperature to give a higher rate of extraction. In most cases, the upper limit of temperature is determined by secondary considerations, such as, for example the necessity to avoid enzyme action.

Agitation of the fluid

Agitation of the solvent is important because this increases the eddy diffusion and therefore the transfer of material from the surface of the particles to the bulk of the solution. Further agitation also prevents sedimentation and more effective use is made of the interfacial surface.

Factorial Analysis Of The Experiment

Factorial analysis is a methodological design plan followed to conduct the experiment. In factorial experiments some factors are varied while others are kept constant which is followed by the real factorial analysis i.e. the selective comparison of the variable factors (independent factors) to develop the response (dependent term) of the process (it's mathematical representation that provides the relationship of these factors to one another and also the effect of these factors on the factors on the overall rate of a process.)

In most cases, the factors affecting a process are obtained from literature, however, they can be predicted or assumed by considering the relationship between the physical or chemical properties of the system understudy.

Methods of Statistical Analysis

Statistical analysis is a body of techniques for deriving or organizing statistics, and for determining their essential significance. There are two types of data to which statistical methods may be applied: variable and attribute. Data are of the variable type when they can be considered, from a practical standpoint. As having some continuously measurable characteristics. Attributes on the other hand, are non-variable classifications in that they are in the form of counts or number or things called enumerations).

The types of statistical analysis, which can apply to either variable or attribute data, are the following

1. To test a given hypothesis concerning some observed characteristics.

2. To determine a reliable estimate of some factual value.

3. To represent a physical situation functionally.

The reason for such an analysis is the fact that all data are to some extent, one way or the other, subject to enhance error. These chance errors may arise whether the problem involves estimation – the test of a hypothesis- or the development of a reliable model.

As a means of testing a hypothesis or determining the reliability of some factual valve, a statistically designed experiment should use. Basically, these designed experiments enable the analyst to determine, with a pre-designed degree of confidence, the degree of variation in the experimental determinations, which is due to chance and that which is the result of some possible known or unknown influence. In addition, a statistical experiment is designed from the standpoint of been able to make a given number of reliable generalization from a minimum number of experiments. It is for this reason that in modern design of experiment, the statistical approach is needed from the beginning.

Variability

Statistical methods are predicted on the single concept of variability. Through it a basis is determined for experimental designed and analysis of data. In this sense statistical methods are concerned with deriving maximum information from a given set of data (analysis), and conversely minimizing the amount of the data (experimental design) to derive specific information.

However, before describing how statistics is used to answer these questions it is necessary to determine fundamental concepts, which are utilized. These includes

- 1. Standard Deviation
- 2. True value
- 3. Degrees of freedom
- 4. Normal frequency distribution
- 5. Theoretical model distribution

Standard Deviation

It is most efficient quantity for characterizing (the most reliable estimate) variability is called standard deviation (also called the root mean square). This is the square root of average squared difference between the individual observations and the average value, and is usually denoted by symbols i.e.

	Mass (g)		Vol. of Solvent (ml)	Particle Size (Um)	Time (min)	Temp. (⁰C)	Agitation
I-	1.	5	250	250	30	27 ⁰ c	-
	2.	5	250	500	30	-	-
	3.	5	250	710	30	-	-
II-	1.	5	300	250	30	-	-
	2.	5	300	500	30	-	-
	3.	5	300	710	30	-	-
111-	1.	10	250	250	30	-	-
	2.	10	250	500	30	-	-
	3.	10	250	710	30	-	-
IV-	1.	10	300	250	30	-	-
	2.	10	300	500	30	-	-
	3.	10	300	710	30	-	-
V-	1.	15	250	250	30	-	-
	2.	15	250	500	30	-	-
	3.	15	250	710	30	-	-
VI-	1.	15	300	250	30	-	-
	2.	15	300	500	30	-	-
	3.	15	300	710	30	-	-

$$S = \sqrt{\frac{1}{n-1}} \sum_{i=1}^{n} (X_i - x)^2$$
i. Variance or Mean square

It is the square of the standard deviation $S = \frac{1}{n-1} \sum_{i=1}^{n} (X_i - x)^2$ ------2 ii. Sum of squares

This is the sum of the squared differences before dividing by the number of observations

True Value

Because of analytical errors the response varies from sample to sample in homogenous system. However, it is intuitively obvious that, if an increasingly large sum of samples were taken (from the same homogenous batch system), the corresponding average yield (response) determination for this will approach some fixed value. This fixed value will be the true value of the system. This is statistically denoted by the Greek letter mu (u). And the estimated derivation S, between the yield, will also approach some fixed value, which is denoted by sigma E.

Degree of Freedom

In statistical analysis this quantity allows for a mathematical correction of data for constraints placed upon this data. The quantity n-1 in the above relations is the degree of freedom. It is stated that one degree of freedom is lost for the calculation of the standard deviation i.e. a constraint has been placed on the data.

Normal Frequency Distribution

When dealing with large numbers of values it is convenient to form an array of the data in such a way that the frequencies of occurrence of given values or range of

values are tabulated are grouped. These frequency arrays are essentially a grouping of data. This grouping is accomplished by the designation of ranges, which are called class intervals. where a sample is tabulated with a function of yield values and class intervals a distribution or frequency distribution tale is said formed. In many applications frequency data follows

Very closely a theoretical mathematical distribution called normal frequency distribution.

Theoretical Model Distribution

There are an appreciable number of statistical distributions, which have, for the most part, been derived from the normal frequency distribution. These are tabulated below.

Reliability of estimates

In analysing experimental data statistical theory as a powerful tool for determining with a reasonable degree of assurance whether certain observed differences might have been due to chance. In determining the degree of reliability associated with certain calculated characteristics the change range of difference between the true and estimated value (which vary by chance from sample to sample) can be determined. For instance in a laboratory experiment the average of the first ten (10) A determination is an estimate of the true mean (u) of the theoretical infinite number of such analysis. However, the question would be certainly be asked as to just how good this sample of ten (10) is in estimating **u**. If it is assumed that the differences between the x and u is only the result of chance and that the individual observations are normally distributed, then a measure of the reliability of the **x** in estimating **u** can be determined. This measure is

Distribution	Symbol	Туре	Symbolic form (Non-
		1	uiiieiisioiidi)
Normal	Z	Distribution of individual observations	$Z = \frac{x-u}{d}$
Student	Т	Distribution of sample means	$T = \frac{x - u}{s/n} -$
Chi-square	X ²	Distribution of sample variance	$x^2 = \frac{s^2}{\frac{d^2}{d} \cdot f}$
Inverted beta	F	Distribution of the ratio of two sample variances	$F = \frac{S^2}{S^2}$

Table 1. Theoretical Model Distribution Table

	iment
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No of	X ₀	X 1	X 2	X 3	X1X2	X1X3	X2X3	X1X2X3	Y
Run									Grams
1	+	+	+	+	+	+	+	+	0.33
2	+	-	+	+	-	-	+	-	0.48
3	+	+	-	+	-	+	-	-	0.88
4	+	-	-	+	+	-	-	+	1.12
5	+	+	+	-	+	-	-	-	0.40
6	+	-	+	-	-	+	-	+	0.56
7	+	+	-	-	-	-	+	+	0.39
8	+	-	-	-	+	+	+	-	0.44

Table 3. Factorial analysis table for 2nd experiment

No of	Xo	X 1	X 2	X 3	X1X2	X1X3	X2X3	X1X2X3	Y
Run									Grams
1	+	+	+	+	+	+	+	+	0.89
2	+	-	+	+	-	-	+	-	1.92
3	+	+	-	+	-	+	-	-	1.86
4	+	-	-	+	+	-	-	+	2.11
5	+	+	+	-	+	-	-	-	0.39
6	+	-	+	-	-	+	-	+	0.56
7	+	+	-	-	-	-	+	+	0.40
8	+	-	-	-	+	+	+	-	0.48

referred to as a **confidence interval**. In particular a sample of a randomly selected observations might be obtained from which x would be calculated. However, what is derived in a range of values that will include, with reasonable specified probability, the true value of \mathbf{u} , i.e. we would be confident (corresponding to the selected probability) that the value of u lies within this interval.

Design of Experiment

Experimental designs are particularly applied to the study

of process variables and how they affect the product. The experiments employ regression analysis, i.e. their quantitative effects, to determine the effect of the variables. Data, which serve as the basis for regression analysis, can be obtained from the recorded experimental data. A selected procedure was employed to develop a controlled combination of variables so as to determine a reliable analysis. Three basic types of experiments are frequently used in the chemical industries. These are

1. Factorial

No of	X ₀	X 1	X 2	X 3	X1X2	X1X3	X2X3	X1X2X3	Y
Run									Grams
1	+	+	+	+	+	+	+	+	1.67
2	+	-	+	+	-	-	+	-	2.09
3	+	+	-	+	-	+	-	-	1.11
4	+	-	-	+	+	-	-	+	2.88
5	+	+	+	-	+	-	-	-	0.45
6	+	-	+	-	-	+	-	+	0.61
7	+	+	-	-	-	-	+	+	0.41
8	+	-	-	-	+	+	+	-	0.62

Table 4. Factorial analysis table for 3rd experiment

The dependent variable was obtained from the experiment data which the signs

- Means minimum value, and + Means maximum value

Table 5. Yield Description Table

Serial No. Of Run	X1	X2	X3	Mean Value Yield (g)	of	Percentage Yield (%)	Rate of Yield/ Extraction (%min)
1	250	250	5	0.77		15.4	0.513
2	250	300	5	0.58		11.6	0.387
3	500	250	5	0.48		9.6	0.320
4	500	300	5	0.47		9.4	0.313
5	710	250	5	0.40		8.0	0.267
6	710	300	5	0.41		8.2	0.273
7	250	250	10	0.88		8.8	0.293
8	250	300	10	0.77		7.7	0.257
9	500	250	10	0.78		7.8	0.260
10	500	300	10	1.63		16.3	0.643
11	710	250	10	0.73		7.3	0.243
12	710	300	10	0.77		7.7	0.257
13	250	250	15	2.04		13.6	0.453
14	250	300	15	1.50		10.0	0.333
15	500	250	15	1.63		10.9	0.363
16	500	300	15	1.23		8.2	0.273
17	710	250	15	1.28		8.5	0.283
18	710	300	15	0.96		6.4	0.213

Note: The serial number of run was obtained from the table of factorials as averages for the three (3) replicate experiments. From the above table the following tables are formed for selective comparison of the variable factors.

 Table 5. Results Interpretation

S/N	Factors Fixed/value	Factor Varied	Percentage Yield (%)
1	Mass / 15g Solvent / 250ml	Particle Size	13.6
2	Particle Size / 710um	Mass	8.2
3	Particle Size / 710um	Solvent	7.7

2. Fractional factorial

Box-Wilson

For a process application, which includes the study of three variables, say, particle size, volume of solvent and mass of solids on the rate of extraction a factorial experiment could be effectively used. For this a reasonable number of operating levels of sizes, volume and mass would be selected and with the selected levels for each the factorial experiment would require all possible combinations. In the case where levels were specified this would include 27 sets of tests. These test result would be interpreted by a functional representation (regression analysis) and an analysis of variance. For process application including more than three variables the number of test becomes excessive with a factorial experiment. Therefore, the fractional factorial or the Box-Wilson experiments can be effectively used.

Regression Analysis

Most statistical techniques known are primarily concerned with the testing of hypothesis. A more important and useful area of statistical analysis in engineering is the development of mathematical models to represent physical situations.

A plot of say sizes of extraction indicates a possible cause-and-effect relationship between these variables. Conceptually, the statistical interpretation for this type of application is different from the others. For this it is more in informative to develop a mathematical model to represent the indicated relationship i.e.

 $\mathsf{Y} = \mathsf{f}(\mathsf{x})$

Where x = the independent variable

Y = the dependent variable

This type of relationship is called regression analysis and is concerned with the development of a

- 1. Selection of a model
- 2. Calculation of the coefficients

3. Statistical test of the model to represent the physical situation

4. Evaluation of the model to determine direction for improvement

METHODOLOGY

The procedure followed in conducting the extraction experiment, the method used in evaluating the compositions and material analysis and the instrument used in the course of the experiment provide the basis of the factorial method. A step-by-step procedure was followed in the preparation of the material (maize seed powder) so as to provide for desired particle size of moisture, organisms and foreign particles.

Experimental Plan

Factorial Explanation

This experiment was designed to take into consideration the factors, which are believed to influence the rate of extraction process. From the theoretical survey particle size, amount by weight of solid, volume of solvent, temperature, degree of agitation and time of extraction are the basis factors that affect the extraction rate. Therefore, in the course of this research work we intended to fix time, temperature and degree of agitation, whereas other factors are varied as described below

Statistical Analysis Used

As discussed above the statistical functions such as

mean, standard deviation, variance etc. were analysed so as to develop a reliable regression and mathematical model.

RESULT

Represent the yield of oil extract.

The selected model equation for the process is given as $Yu = b_0 + b_1x_1 + b_2x_2 + b_3x_3 + b_{12}x_1x_2 + b_{13}x_1x_3 + b_{23}x_2x_3 + b_{123}x_1x_2x_3$

Applying factorial method the regression coefficients were obtained as;

 $\begin{array}{l} b_0 = 0.9621 \\ b_1 = 0.1971 \\ b_2 = 0.0996 \\ b_3 = 0.4829 \\ b_{12} = 0.0229 \\ b_{13} = -0.1246 \\ b_{23} = -0.1154 \\ b_{123} = 0.0322 \\ for full factori$

for full factorial analysis errors in each regression coefficients is the same and is obtain as 0.7556 and the statistical significance for each coefficient is tested given a mean value t call = 1.65 for the eight points variables at P = 0.05 (i.e. 5% significance), this is compared with the table value table = 1.86 at P = 0.05. By inserting the values of the coefficients in the selected model for the eight point's factors the fitted performance relation is represented. The fitted model representing the physical process is obtained as

 $\begin{array}{rcl} Yu &=& 0.9621 & - & 0.1971X_1 & - & 0.0996X_2 & + & 0.4829X_3 & + \\ 0.0229X_1X_2 & - & 0.1246X_1X_3 & - & 0.1154X_2X_3 & + & 0.0322X_1X_2X_3 \end{array}$

This is the mathematical model equation for the extraction process predicted by factorial analysis for three variable factors. A further test for adequacy was carried out by calculating the dispersion of adequacy {S (ad)} for the 2^3 replicate experiments. Given S^2 (ad) = 0.000000041.

Now, comparing the dispersion of adequacy with the experimental error by applying Fisher's test the calculated F-distribution over the normal curve area of the model is 0.000000071. Since this value is less than the F-table value given as 4.44 at F 0.00,5,16 the fitted model is regarded adequate.

Determination of the Relationship Between the yield and the Variables

The mean yield of extract is tabulated over the average numbers of run for the 2^3 factorials. This is a qualitative determination of the relationship between the variables that describes the response of one factor to the yield when with the other factors fixed. It provides the efficient variable conditions for maximum oil recovery for the system under study.

DISCUSSION OF RESULTS

The results obtained from the experiment provide the basis for factorial analysis for the selective comparison of the three factors viz; particle size (X1), volume of solvent (X2) and mass of solids (X3). First a linear model relation is selected as a basis for determination of the linear response of the yield to the variation in the selected factors. The accepted fitted model equation is given below,

Therefore, this is the quantitative mathematical model determined statistically by factorial analysis method representing the physical mechanism. It provides for the effective recovery of oil from maize seed.

Therefore, the **linear**, model equation representing the leaching of vegetable oil from maize seed is feasible for as high as 14% oil recovery. It showed that the mass of solvent has more influence followed by the particles sizes and the volume of solvent used.

CONCLUSION

This work proves that a linear mathematical model equation can be developed to describe the physical process for as much as 14% oil recovery from 0.015kg (710um size) maize seed power using Hexane as solvent (300ml) at room temperature. This agrees with the theoretical value predicted in the literature survey. I.e. 10%. Therefore a suitable linear mathematical model equation that fitted the physical situation was developed.

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