

Innovative Techniques in Hydrophobic Particulate Separation in the Food and Beer Industries

A.O. Ette;

Faculty of Engineering, University of Agriculture, Makurdi (Nigeria)

ABSTRACT

Oil is naturally hydrophobic but often forms an emulsion when finely dispersed in water. Such emulsion reduced the efficiency in the recovery of oil during extraction from vegetable oilseeds. This paper reviews some techniques of vegetable oil extraction in the food processing industry relative to some indigenous techniques. An extraction techniques based on modified indigenous technology is proposed. The principles and rheological background are discussed with respect to multiphase flow problem in equipment design/design parameters and operating characteristics. Results show water-air system to establish the flow dynamics will also be presented.

RÉSUMÉ

L'huile est par nature hydrophobique mais elle forme souvent une émulsion lors qu'elle est dispersée finement dans l'eau. Cette émulsion diminue l'efficacité de la récupération de l'huile pendant le travail d'extraction. Cette communication certaines techniques locales d'extraction de l'huile végétale dans les industries produisant des denrées alimentaires. proposé une technique d'extraction basée sur une technique locale mais modifiée. On a parlé des principes et de rhéologiques en ce qui concerne les problèmes complexes posés par les paramètres du modèle d'équipement ainsi que les spécifiques d'opération de ces derniers. Pour établir le courant de dynamique on a aussi présenté les résultats en système air-eau.

INTRODUCTION

Nigeria is blessed with abundance of vegetable oilseeds such as coconut, palm, palm kernel, groundnuts, soyabeans cottonseeds, sunflower seeds, maize, etc. The potential of Nigeria as a major producer of these crops has diminished due to neglect and was noted by Mielke (1985) who observed that the major production of palm oil has shifted from Nigeria to Malaysia. However, research efforts from our institutions have resulted in the development of improved cultivars for the above crops. Extraction of oil from these raw materials is still in its infancy in Nigeria and the development of appropriate processing technology is gaining some interest.

Presently, most of the available commercial technologies, in vegetable oil extraction, are built around solvent extraction using hexane extractant. The cost of these chemicals (solvents) has increased significantly in recent times resulting in reduction in production capacity or plant shut down. Increased awareness of the hazards of process chemicals in food products cause concern over the continued use of toxic chemicals in the extraction of oil. There is therefore need to develop alternative extraction/separation techniques which will incorporate the high extraction capacity of hexane and retain the product purity offered by mechanical press.

In rural areas, complete oil removal from oil seeds is accomplished by washing the digested seeds with copious amount of water. The local processor uses water which is a nontoxic solvent to extract edible oil. It is required that the extraction solvent be readily available and inexpensive. Water seems to meet these requirements but forms emulsion with fluid dispersed oil particles, thus reducing recovery and extraction efficiency. This paper discusses the steps necessary in solving the emulsion problem in the extraction of vegetable oil when water is used as a solvent. The principles of design of a method to treat the emulsion will be based on a multiphase flow analysis using water as the matrix.

BACKGROUND OF EXTRACTION TECHNIQUES

Commercial techniques

The recovery of oil from vegetable oil-seeds follows a general process route comprising:

- (a) Pre-extraction Preparation
- (b) Extraction, and
- (c) Refining.

The extent and complexity of each step depends on the physical characteristics of the individual raw materials (feed) and the available process technology. The processes described below are utilized in the palm oil industry but they are applicable to other vegetable oil seeds.

Pre-extraction Preparation

This is the first step in the extraction process which is designed to eliminate foreign materials, minimize contamination, reduce maintenance cost, and increase efficiency in oil recovery. The seeds are broken into small fragments to increase the available surface area. For palm kernel, Southworth (1985) reported that the seeds are reduced to about 0.25 and 0.4 mm diameter. The size reduction is followed by steam conditioning to provide:

- (1) appropriate moisture content in the meal.
- (2) rupturing of the cell-wall of the meal.
- (3) coagulation of protein.

The meal is cooked to 104-110°C at a moisture content of 3% by weight and it is then conveyed to the extraction circuit.

Extraction

The extraction phase is carried out using mechanical press or solvent extraction. Properly cooked meal is fed into a screw press and compressed to expel the oil. The de-oiled cake is discharged for use as fuel or formed into pellets for agricultural feed production. Extraction by solvent is carried out by using the meal prepared in the manner mentioned above. Two methods of meal-solvent contact are commonly practiced, i.e. percolation, and immersion techniques. In the percolation method, the meal is stored in specially designed vessel and the solvent, hexane, is percolated over the meal. The immersion technique is carried out by counter-current flow of the meal against the solvent/miscella in an extractor. The two methods are often combined for more efficient extraction. Solvent recovery from the de-oiled cake and extract phase (miscella) is the next step in the process.

The de-oiled cake is brought in contact with live steam in an agitated vessel and the desolventized cake is discharged and sent to the pelletizing plant. Fractional distillation under vacuum and at high temperature is used in the recovery of the solvent from the miscella. The reclaimed solvent is recycled to the extraction circuit and the oil is stored for subsequent refining.

Refining

Degumming, filtration, deodorization, and removal of the free fatty acid (FFA) form part of the physical refining process. The chemical refining step is aimed at controlling the triglyceride compositions, iodine value, slip melting point, fat and mineral contents e.g. iron, copper, and phosphorous. More details on the refining process are covered by Stage (1985), Morgan et al (1985) and Swoboda (1985). The resulting refined products and associated by-products are employed in various industrial applications. Having gained some background on the commercial process of vegetable oil extraction, it is necessary to look at the rural or indigenous extraction methods.

Rural Extraction Techniques

The extraction of oil from vegetable oil seeds (mostly palm, palm kernel, coconut and groundnuts) have been practiced since man was able to devise some basic domestic implements. The basic steps in the indigenous practices are:

- (a) Pre-extraction preparation of raw materials,
- (b) Extraction, and
- (c) Refining.

Some similarities exist between the basic steps in both rural and commercial practices but significant differences also characterize the scope and philosophy of these techniques. Extraction of palm oil is briefly reviewed.

Pre-Extraction Preparation of Materials

The ripe palm fruits are harvested and the bunches are chopped and covered with green leaves or mats to reduce evaporation and loss of moisture from the heap. This process will enhance fermentation and ease the release of the fruits from the bunches. The duration of coverage of the chopped bunches affect the quality of oil to be produced. For edible oil, one to three days will be the average time allowed but longer periods are necessary for non-edible oil. It should be noted that the FFA level increases with increased fermentation.

Edible and hard oil are the two main types of oil produced locally, the former through the sterile process and the latter through non-sterile methods. In the sterile process, the fruits are steamed for over six hours after which the fruits are beaten in large mortars. The mesocarp is separated from the nuts though mutually mixed. About two thirds of these nuts are removed from the crushed flesh (mesocarp) and the resulting meal is ready for oil extraction.

The production of hard oil is via the non-sterile method. Longer fermentation periods are required to ensure separation of the mesocarp from the nuts. The digested flesh and nuts are washed several times with copious amount of water and the fibre and nuts are removed by filtration. The liquid phase (emulsion) is left to stand for several days before the oil is separated.

It can be seen that the sterile process emphasizes quality of the oil produced while the non-sterile methods seek optimum extraction through washing with water as a solvent. Oil lost to the nuts and the flesh is minimum in the non-sterile process yet high total recovery is obtained with the sterile method. The extraction step is outlined below

Extraction

The flesh, partly devoid of nuts, is squeezed by hand if the quantity to be processed is very small. Hand press or mechanical press is used for larger production capacity. Pressure is applied on the flesh and the oil is squeezed out in proportion to the applied pressure. This means that the efficiency of the oil recovery decreases with the applied pressure, e.g. manual pressure hand press mechanical press. The pioneer oil mill is an adaptation of this concept with provision for increased pressure to improve extraction. At the instance when emulsion is observed from the press or at the limit of the applied pressure, the extraction is stopped. The de-oiled meal is used as fuel. The oil and nuts are stored for refining and further processing respectively.

In the non-sterile extraction process, the emulsified phase is stirred briskly in the presence of infiltrate air. Hydrophobic oil particles, produced by the stirring process, attach themselves on the air bubbles and float to the surface of the vessel. The float is skimmed off regularly and boiled to separate the oil from water and FFA sludge. Significant loss in recovery is experienced at this stage due to low efficiency of bubble-oil contact and the inability to completely decant the oil from the sludge.

Refining

The word 'refining' as applied in the indigenous oil extraction process is limited to the removal of some FFA by fire treatment. Soft oil produced by the sterile process is heated to boiling and a small quantity of water is added. Boiling is intensified and the mixture is allowed to stand for one or two hours before cooling to room temperature. Purified oil is decanted and the FFA - water sediment is disposed of as sludge. The purity and freshness of the oil is judged by the amount of sediment left after the refining process. The smaller the quantity of sludge, the higher the purity and vice versa.

Some important considerations in the indigenous extraction processes includes:

- i) The use of steaming (sterilization) to enhance oil extraction and improve purity
- ii) The use of water as a solvent to maximize extraction of oil from the meal and seeds
- iii) The use of air gas as a third phase in breaking the oil-water emulsion, and
- iv) The application of heat to promote phase separation.

Through the above principles were employed without explicable theoretical reference, their collective effectiveness justifies their consideration in developing an appropriate technology for Nigeria.

ALTERNATIVE TECHNIQUES

Concern over the use of Hexane as a solvent in the extraction of edible oil has led many researchers to investigate alternative solvents for use in the vegetable oil extraction industries. Wolf outlined some of the fears/ disadvantages in the use of hexane

- It is an expensive petroleum product of limited availability
- It forms explosive mixture with air
- It is extremely flammable
- Its vapours are toxic
- Recovery of hexane from oil and meal is energy intensive
- High solvent loss due to incomplete recovery.

Several alternative solvent systems have been examined as possible replacements for hexane, including alcohols, halogenated hydrocarbons, water and supercritical carbon dioxide (Beckel et al., 1984; McKinney et al., 1959; Lawhon et al., 1981; Friedrich and List, 1982; Khor and Chan, 1985; Arnold and Choudhury, 1962). Most of the systems investigated could not be commercialized due to toxicity and other health related implications. Water was evaluated as a solvent for the recovery of oil from soybeans and the system was deficient on the following accounts (Lawhon et al. 1981)

Low yield of oil

The need to operate at stringent sanitary conditions than needed with hexane processing.

It should be recalled that water was used as a solvent in the rural techniques of extraction when maximum recovery was desired from the meal. The low yield reported above can be attributed to emulsion formation. Advances in treating petroleum oil-field emulsions suggest that with appropriate combination of techniques, the low yield problem could be overcome. Sterile treatment at various process steps could reduce the sanitary problems mentioned above. The author believes that the problems cited above, with respect to water as a solvent, can be overcome by proper selection of technology

THE MODIFIED RURAL TECHNOLOGY

It is necessary at this point to look at some of the advantages of water as an extraction solvent:

1. It is non-toxic and a universal solvent
2. It is generally available and hence devoid of problems associated with materials and chemical importation
3. It is relatively cheaper than hexane and other petroleum-based solvents.
4. It has useful physical and chemical properties which are amenable to efficient phase separation

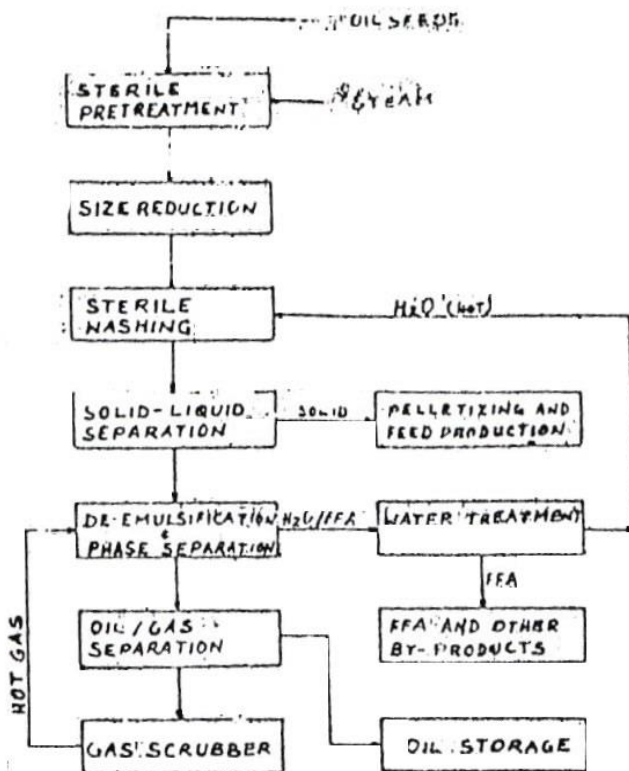


Fig 1. Flow diagram of the Proposed Oil extraction process from vegetable oil-seeds

5. It has been effectively utilized in rural oil extraction processes to maximize oil recovery.

A modified rural technology for vegetable oil extraction is being proposed based on the following facts:

- (a) De-emulsification of a first order system can be achieved by application of sufficient kinetic energy to the molecules of the dispersed and continuous phases in the system.

Increase in temperature enhances phase separation of an oil-in water emulsion by modifying the densities, viscosities and interfacial tension (surface tension)

Centrifugation, filtration and chemical treatment have been used effectively to break emulsion (Anonymous 1984)

Oil is hydrophobic

Figure 1 is a schematic of a flow diagram depicting the process steps necessary for oil extraction using water as a solvent. Brief description of these steps follow.

Sterilization Pre-treatment

This is similar to the processes used in edible oil extraction described earlier. Steaming of fresh fruits and bunches to soften and enhance removal of the mesocarp from the nuts is accomplished by this process

Size Reduction

The sterilized oil seeds are crushed and ground to increase the available reaction surface for the extraction. It is essential to reduce the particles to a size that will yield a specific surface area greater than 600cm^2 . The size commonly encountered in conventional practices gives about 240cm^2 specific surface area. This is considerably larger than the liberation size.

Sterile Washing

Hot water or steam is used to wash the crushed seeds and the oil is extracted into the liquid phase. The emulsion or liquid phase has a good electric conductivity and does not wet hydrophobic surfaces. Counter-current decantation (C.C.D.) technique will be effective in washing the meal to minimize oil loss.

Solid-Liquid Separation

The de-oiled meal and other solid particles are removed from the emulsion by filtration. The resulting solid portion is compacted for use in animal feed production or as fuel. The filtrate is pumped into a storage tank for further processing.

De-Emulsification

This is the crucial step in the extraction process. The hydrophobicity of oil is exploited at this stage by adding appropriate surfactant to reduce the surface tension of water. Air or selected gas is pumped into a mixing chamber where the emulsion and water are mixed. The multiphase mixture is then pumped through an orifice or a gas pump (Figure 2). The output from the orifice is fed into a circulation tank as shown in Figure 3. Phase separation occurs in the tank as the air, oil, and water will settle out in response to their densities. The various output phases are tapped; oil is sent for refining while the air and water will be cleared and recycled.

THEORETICAL CONSIDERATIONS

The Centrifuge

Sedimentation centrifuge has found application in many industrial separation processes. All components of a system comprising one or more particles suspended in a continuous liquid phase that is enclosed in a rotating cylindrical container experience centrifugal force. This force is represented by the equation.

$$F_c = m\omega^2 r$$

where,

F_c = centrifugal force

m = mass of particle

ω = angular velocity

r = radius of path

From equation (1), the angular acceleration is given as

$$a_c = \omega^2 r \tag{2}$$

In the centrifuge, the particle is acted upon by gravitational force (w) and the centrifugal force (F_c). If the gravitational force is represented by the equation

$$w = mg \tag{3}$$

where g = acceleration due to gravity; the comparison of the two forces is a measure of the power of the centrifuge. The ratio of centrifugal force over the gravitational force is known as the Relative Centrifugal Force (RCF)

$$RCF = \omega^2 r / g \quad (4)$$

Substituting $\omega = 2\pi n$ and $r = D/2$ in the above equation and writing in familiar units.

$$RCF = 3.55 \times 10^{-5} n^2 D \quad (5)$$

where n = revolution per minute, (rpm)

D = inside diameter of tube or bowl, cm

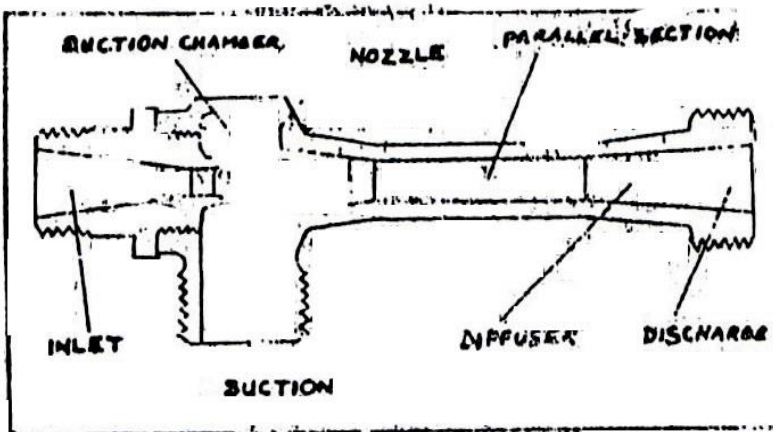


Fig. 2. Multiphase Mixer, and Nozzle

The range of values encountered in commercial application is 200 RCF: 125,000. (Anonymous, 1984)

In a two phase system, the separation characteristics can be analyzed using the difference between the dispersed or solid density (P_s) and the continuous phase or liquid density (P_L).

For

$P_s - P_L < 0$ solid is skimmed from surface of continuous phase,

$P_s - P_L = 0$ No phase separation,

$P_s - P_L > 0$ Solid migrate to the well of the centrifuge..

In treating vegetable oil emulsion, P_s represents the density of oil and P_L is the density of water. It can be seen that for any given temperature, $P_s - P_L$ will give negative values. This suggests that oil will be skimmed off the water at the completion of separation.

It is also possible to develop other simple devices capable of producing the range of relative kinetic force obtained in the centrifuge. Figure 2 depicts one of such devices which will be analyzed in the next paragraphs.

The nozzle

When a fluid is flowing through a variable diameter pipe (duct) the velocity of the fluid changes with the change in the diameter of the duct. For a constant mass flow condition, the velocity of the fluid in the pipe (duct) is inversely proportional to the pipe diameter. A particle in the fluid will acquire some kinetic energy due to the fluid motion. The force can generally be represented by F_N for one-dimensional steady flow condition.

$$F_N = P A V_x^2 \quad (6)$$

where

F_N = Force acting on the particle;

P = density of the particle;

A = cross-sectional area of the tube;

V_x = velocity of fluid along x-direction.

When the flow is through a circular pipe, the area is given by $A = \pi d_t^2/4$ and is substituted in equation (6)

$$F_N = \pi/4(\rho d_t^2 v^2)$$

where d_t = diameter of pipe or tube.

From equation (1)

$$F_c = PZ\omega^2 r$$

where Z = volume of particle.

For a spherical particle, the following equation is derived

$$F_c = \pi/6 \rho d_s^3 \omega^2 r$$

And d_s = diameter of particle.

The ratio F_N/F_c will be known as the relative nozzle advantage (RNA)

Hence

$$RNA = (1.5 d_t^2 v^2) / (d_s^3 \omega^2 r)$$

This nozzle force can be compared to the gravitational force to obtain the corresponding Relative Nozzle Force (RNF)

$$RNF = RNA \times RCF \quad (11)$$

Hence

$$RNF = 62.65 Q^2 / d_s^3 d_t^2 \quad (12)$$

where Q = volume flowrate (gpm).

It can be seen that the value of RNF is directly dependent on the square of the volume flowrate and inversely dependent on the cube of the particle size and the square of the pipe diameter. Equation (12) is a design equation which can be manipulated to obtain the required condition needed to effect phase separation for a given multiphase system.

Considering the differences in density between the oil, water and air, the linear displacement experienced by each particle of equal diameter due to an applied force will vary. Such variation can result in the stratification of the particles. By providing a quiescent path for the phases to settle out, the oil can be skimmed off while the air (gas) and water are cleaned and recycled in the circuit for optimum efficiency

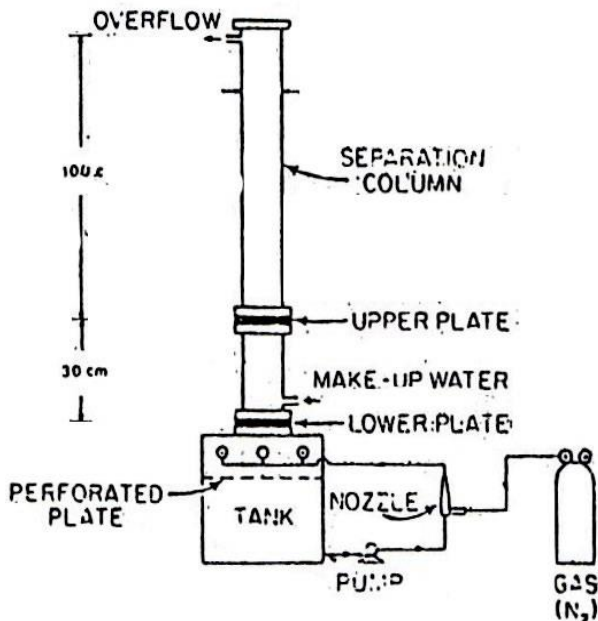


Fig. 3. Experimental Bubble Column Floatation Unit.

The Characteristics of a multiphase system was independently investigated using water and air mixture (Ette and Poirier, 1985). Data from this investigation will be used in analyzing the design equation. Also, data was collected from the extraction of an oil-in-water emulsion (coconut-oil milk) in a centrifuge.

RESULTS AND DISCUSSION

Oil extraction by the proposed technique involves a multiphase separation. An earlier work carried out by the author to investigate the dynamics of two-phase flow in a bubble column flotation unit will form the source for some of the data used in analyzing the model. Figure 3 depicts the unit from which data for volume flowrate (Q), particle diameter (d_p), and the orifice diameter (d) were obtained. The difference between this system and the proposed technique is that pure water was used instead of an emulsion, hence making the new process a three-phase system (oil-water-gas).

Production of spherical bubbles of fine particle size was accomplished by addition of 8ppm of surfactant to the water. The average measurable bubble diameter (d_b) was 0.098 mm. Much finer sizes were produced as 'cloud' at the instant of exit of the orifice which had a diameter (d_i) equal to 12.5 mm. Particle size was influenced by the flow-rate of the water (Q) which was varied from 5.8 to 7.0 gpm. The higher the flowrate, the finer the particles. For the analyses, $Q = 7.0$ gpm, $d_i = 12.5$ cm, and $d_b = 0.125$ cm.

Introducing the above values into equation (12) gives $RNF = 2.087 \times 10^9$. Compared with the RCF values for commercial centrifuge, RNF obtained from this system is 3.57×10^4 times the maximum obtainable value in the centrifuge. In addition is the ability to obtain such high values of g in the system. Its versatility and flexibility ensure adaptation of the unit to produce my desired condition for effective separation.

It was necessary to test the practical feasibility of the centrifuge separation process. Hence an oil-in-water emulsion (coconut-oil milk) was prepared for separation. The diameter of the centrifuge tube was 1.0 cm and the revolution was set at 1200rpm. It was possible to separate the oil (precipitate) from the water and the solid particles also settled down at the bottom at the centrifuge showing stratification due to density variation. Introducing these data into equation (5), the corresponding RCF value was equal to 51.12. From equation (12), $RNF = 62.65$ if the values of Q , d_p and d_i were set at unity respectively. This indicated that a minimum of 1.44×10^3 gallons of emulsion can be treated per day utilizing 22.6% separation advantage over centrifugal processing. The absence of mechanical moving parts in the design minimizes maintenance cost and down-time due to equipment failure. The system is portable and can be used for small-scale and large commercial production with minimum modification of the design variables.

CONCLUSION

1. Nigeria has abundant vegetable oil-seeds from which oil could be profitably extracted on a commercial scale.
2. Cost of extraction chemicals and the potential health hazards inherent in these chemicals necessitate the need for alternative extraction methods.
3. Using water as an extraction solvent generally produces emulsion which decreases recovery of the oil.
4. De-emulsification can be accomplished by physical and chemical techniques including centrifugation, reduction in surface emission, etc.
5. A method of oil extraction/emulsion separation based on indigenous practices and particulate rheology and dynamics is proposed
6. The technique will provide sterile extraction with high production capacity and adaptation to small scale or large scale operation
7. It is capable of producing high g values which with appropriate surface treatment, particulate stratification will occur based on density differences.
8. The technique has the following potentials over the sedimentation centrifuge separation:
 - (a) High production capacity,
 - (b) Simplicity in design and the absence of mechanical moving parts, and
 - (c) Overall low maintenance and operation cost.

RECOMMENDATION

1. More studies should be carried out to establish the performance of the technique using some locally available oil-seeds.
2. The effect of orifice diameter, volume flowrate and particulate size on the separation process should also be investigated.

3. Screening of acceptable surfactants for surface tension modification should be carried out
4. Optimization of the process and provision of adequate parameters for plant design should be explored
5. Economic feasibility studies should also be carried out

REFERENCES

- Anonymous. 1984. *Perry's Chemical Engineer's Handbook*, 6th Edition, McGraw-Hill Inc., NY
- Arnold, L.K., R.B.R. Choudhury, 1962. Extraction of Soybeans with Four Hydrocarbon Solvents. *JAOCS* 37: 378
- Arnold, L.K., R.B.R. Choudhury, 1962. Ethanol Extraction of Soybean Oil. *JAOCS* 37: 379-380
- Beckel, A.C., P.A. Belier, and A.K. Smith, 1984. The Non-distillation Alcohol Extraction Process for Soybean Oil, *JAOCS*, Vol.25: 10-11.
- Etté, A.O., and D.R. Poirier. 1985. Dynamics of Two-Phase Flow in Bubble Column Flotation Units. A Report of Investigation Submitted, USBur Mines.
- Friedrich, J.P., and G.R. List. 1982. Characterization of Soybean Extracted by Supercritical Carbon Dioxide and Hexane. *Journal of Agricultural and Food Chemistry* 30: 192 - 193.
- Khor, H.T., and S.L. Chan. 1985. Comparative Studies of Three Solvent Mixtures for the Extraction of Soybean Lipids. *JAOCS* 62(2): 98 - 99.
- Lawhon, J.T., L.J. Manak, K.C. Rhee, K.S. Khoo, and E.W. Lusas. 1981. Combining Aqueous Extraction and Membrane Isolation Techniques to Recover Protein and Oil from Soybeans. *JAOCS* 46: 912 - 916, 912.
- McKinney, L.L., J.C. Picken Jr., F.B. Weakley, A.C. Eldridge, R.E. Campbell, J.C. Cowen, and H.E. Biester, 1959. Possible Toxic Factor of Trichloroethylene Extracted Soybean Oil Meal. *JAOCS* 81: 909 - 915.
- Mielke, S. 1985. Present and Future Position of Palm and Palm Kernel Oils in World Supply and Trade. *Journal of the American Oil Chemists' Society* 62(2): 193 - 197
- Morgan, D.A., D.B. Shaw, M.J. Sidebottom, T.C. Soan and R.S. Taylor. (1985) The Function of Bleaching Earths in the Processing of Palm, Palm Kernel and Coconut Oils. *JAOCS*, 62(2): 297 - 299
- Southworth, A. 1985. Palm Oil and Palm Kernels. *JAOCS*, 62(2): 250 - 254
- Stage, H. 1985. The Physical Refining Process. *JAOCS*, 62(2): 279 - 308.
- Swoboda, P.A.T. (1985). Chemistry of Refining *JAOCS*, 62(2): 287 - 292
- Wolf, Walter J. Research Needs for A New Solvent to Replace n-Hexane in Soybean Oil Extraction. From *Soybean As Food Source*.