THE INFLUENCE OF PLATE AND PACKED COLUMN ON THE MASS TRANSFER OF FREE FATTY ACIDS FROM PALM OIL MIXTURES.

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1 SUMMARY.

Mass Transfer of free fatty acids in Palm Oil in Packed and Plate Column.

Stream stripping of free fatty acids from oil and splitting of oil into fatty acids are the key processes in the Malaysian Palm oil and Oleochemicals industry. Both consumes an enormous amount of high pressure steam to provide heat and vacuum. Typically, as much as 30 % of the operating costs is due to steam consumption. Recently there has been a new impetus to use medium pressure and temperature to strip and split fatty acids.

This study investigated the <u>mechanisms, mass transfer rates and efficiency of the steam stripping of free fatty acids and splitting of oil to free fatty acids as well the influence of plate and packed columns scale-up on the process.</u>

The scope of research encompassed the determination of vapor-liquid equilibria of oils with free fatty acids composition ranging from 2 to 7 %. Bench Still experiments are used to determined the mass transfer rates and efficiency of vacuum steam stripping-splitting of free fatty acids. Pilot Plant Plate and Packed columns experiments were used to ascertain scale-up effects. Finally a mathematical correlation, modelling and simulation of the experimental results were conducted and compared with computer predicted results[2].

Bench still tests indicated that at low to medium operating conditions, both stripping and splitting compete for dominance depending on the f.f.a. Driving force[a]. The stripping rate equation is a second order equation of f.f.a. stripping rate with respect to f.f.a. Driving force. This is due to splitting becoming dominant as f.f.a. Driving force being reduced.

On the other hand, the Pilot plant Plate and Packed column tests indicated that at identical conditions, stripping is entirely dominant in the Plate and Packed columns. Furthermore, stripping rate has increased measurably and exhibit a third order equation.

Comparison between Plate and Packed column performances at varying vacuum pressures indicated that the Packed column has a higher stripping rate, more steam efficient and achieved a higher level oil purity. However, the Plate column reaches equilibria faster and stabilises faster albeit over a shorter range of conditions.

2 INTRODUCTION.

Theory of Vacuum Steam Stripping in Palm Oil Physical Refining.

Free Fatty acids are natural acids formed by enzymes in the Oil Palm. It is catalysed by the presence of iron ions and moisture. In the mill, the Oil Palm is sterilised to destroy these enzymes but residue free fatty acids still remains in the Palm Oil. At the refinery, the residual f.f.a. are separated from the oil either by chemical refining or physical refining. Initially, the Crude Palm Oil contains other impurities e.g. Natural gums which are both undesirable and inhibits the separation of f.f.a. from the oil. These are eliminated by bleaching and degumming. In physical refining, the f.f.a. are stripped at vacuum by steam. The vacuum reduces the boiling point of the f.f.a. and prevents carbonisation of the oil. Steam is further used to strip the f.f.a. from the oil since the acids are hydrophilic compounds.

Stripping performances are influenced by 4 major factors such as the amount and influence of trace components on vapor-liquid equilibria, the hydrodynamics of the oil flow in the column, mass transfer efficiency of internals and the heat transfer from steam to oil. Normally, vacuum operations has a column efficiency of 40 % [3]but the multiple effects of these factors could reduced efficiency. However, the operation must also take into account other quality requirements such as deodorisation to eliminate odors and the thermal degradation of carotene to enhance color. Thus, we have a compromise between efficient mass transfer of f.f.a. and the attainment of non mass transfer related quality requirements.

3 EXPERIMENTAL PROCEDURES.

Sampling, analysis and operation of experiments.

The experiments can be classified into two categories; experimental and computational. This is further divided into 5 phases of operations; sampling, analysis, bench scale experiments, pilot scale experiments and analysis of results.

3.1 Sampling.

To ensure that only the stripping of fatty acids from oil was studied, other inhibiting components had to be eliminated. Samples of Palm Oil with control doses of fatty acids ranging from 2 to 7 % were prepared for the experiments. At the same time, the vapor-liquid equilibria was computed using a proprietary computer properties database. Operating conditions and product specifications were predicted for the mixtures using short-cut and

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3 Peters & Timmerhaus, "PLANT DESIGN AND ECONOMICS FOR CHEMICAL ENGINEERS", 3rd ed, 1980, McGrAw-Hill.

rigourous distillation methods[4]. Experimental and control samples were prepared for the 736mmHg, 600mmHg and 426mmHg vacuum experiments as well as for an optimisation experiment.

3.2 Analysis.

Two types of analysis were conducted during the experiments; on-line and off-line analysis. The off-line analysis uses the acids titration method and gas chromotography to determine the acidity content and compositions. Whilst an on-line analysis technique was developed to extract vapor and liquid samples out of the rig at regular interval for immediate analysis. Three instruments were used and all readings were cross referenced and cross tabulated for accuracy.The on-line analytical methods are now being studied as a separate research program.

3.3 Bench-Scale Experiments.

Two bench scale experiment were conducted to determine the actual vapor-liquid equilibria and to evaluate stripping-splitting of f.f.a. for non-mass transfer enhanced conditions. Vapor and liquid samples were extracted periodically from the still and analysed. Off-line samples initially analysed and then stored under cool conditions for further analysis later.

3.4 Pilot Plant Scale Experiments.

After the bench scale experiments, the results were used as a basis to determine the optimum operating time, compositions and conditions for the pilot plant experiments. In this study, a 20 plate and a 1 metre packing columns were used. Vapor and liquid samples were obtained for this study.

3.5 Analysis.

Five techniques of acidity tests were done and cross checked in this study to evaluate the errors and limits of errors for each experiment. Results of the bench scale tests were compared w.r.t. varying vacuum pressures, acid composition and operating time.

The Pilot Plant test results were compared to the bench scale results and with each other to determine mass transfer rates and efficiencies.

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4 Mustafa Kamal (Dr) Abdul Aziz,"PROCESS MODELLING AND SIMULATION OF PALM OIL PROCESSES-poster paper." 5th Malaysian Institution of Chemical Engineers Symposium, June 26-28, 1989, Putra World Trade Centre, Kuala Lumpur.

Finally, the results were used to determined an optimum operating conditions w.r.t. vacuum pressures, steam flowrates and steam temperatures. The optimum values were compared to the computer predicted values.

4 RESULTS AND DISCUSSIONS.

Computational, Bench and Pilot Results.

As mentioned earlier, 4 types of results were sought in this project; vapor-liquid equilibria, mass transfer rates, column efficiency and scale-up effects of plate and packed columns.

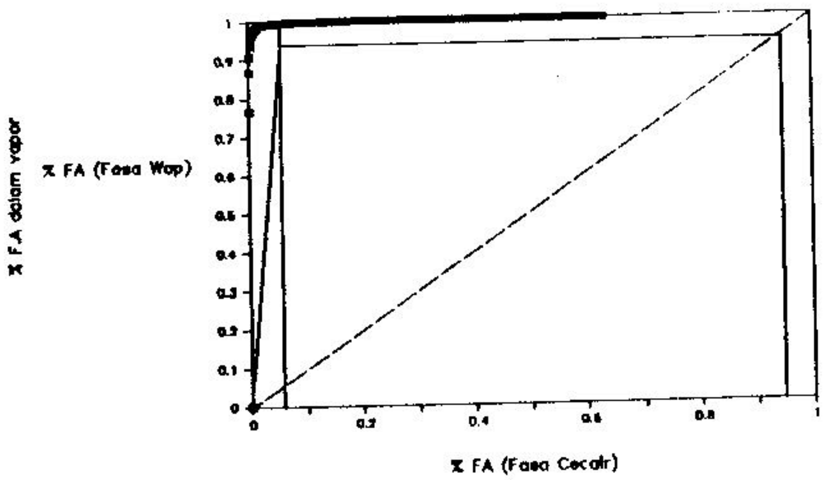
4.1 Vapor-Liquid Equilibria.

Computer generated vapor-liquid data indicated that the fatty acids-oil curve is highly convexed imposing extreme difficulties in short-cut design techniques i.e. McCabe-Thiele Pseudo-Binary method and large errors in calculations e.g. 70 % inaccuracy between rigourous and short-cut methods.

Furthermore, the presence of trace components and free liquid water in the fatty acids causes distortion of this vapor-liquid equilibria reducing theoretical column efficiency. The effects of these trace components will be the subject of the next study.

Mencari Bil.Plat

(Kaedah Mc -Cabe Thiele)

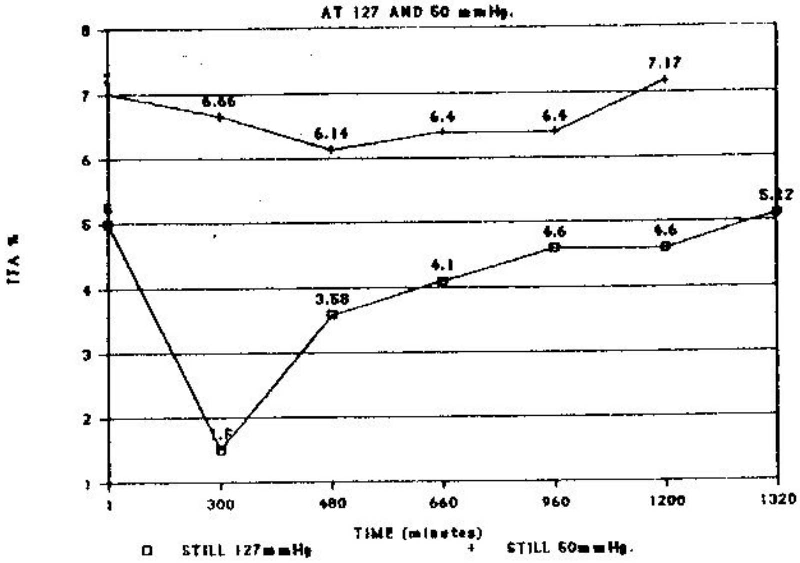


4.2 Bench Scale Experiments.

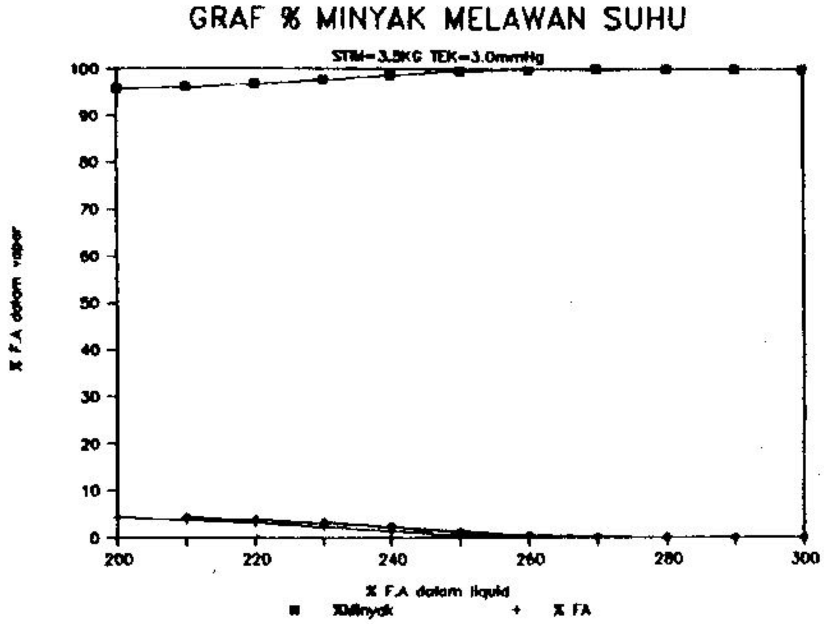
Stripping at low to medium conditions is related to the f.f.a. Driving force in the liquid and vapor phases. At 600mmHg, an 5 % acid-oil sample strips rapidly (less than 2 minutes) initially until an certain equilibrium f.f.a. Driving force is reached and then hydrolysis occurs slowly and increasing f.f.a. to certain driving force value. This is a cyclic phenomena at low conditions. In contrast, at 700mmHg, even a 7% sample cannot induced stripping but hydrolysed slowly.

In short, the stripping rate equation is a Quadratic equation with the stripping rate being faster but the hydrolysis rate being slower and the rate determining step in this reaction. This is explained by the fact unlike stripping, the triglycerides must decomposed to monoglycerides before hydrolysis occurs.The experiment further showed that the residence time required for stripping in a single plate is very short

PLOT OF BATCH STILL EXPERIMENTS

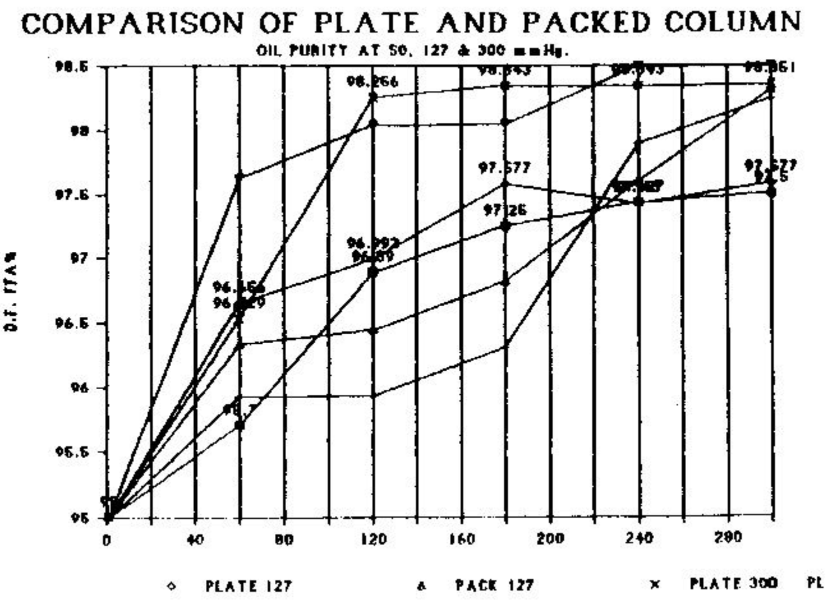


The next important point is that vapor-liquid equilibria of fatty acids-oil mixtures are highly convexed and furthermore distorted by the presence of trace components which will hinder stripping. By measuring this distortion, one is able to ascertain the maximum theoretical efficiency of the process for a particular Palm Oil composition profile.



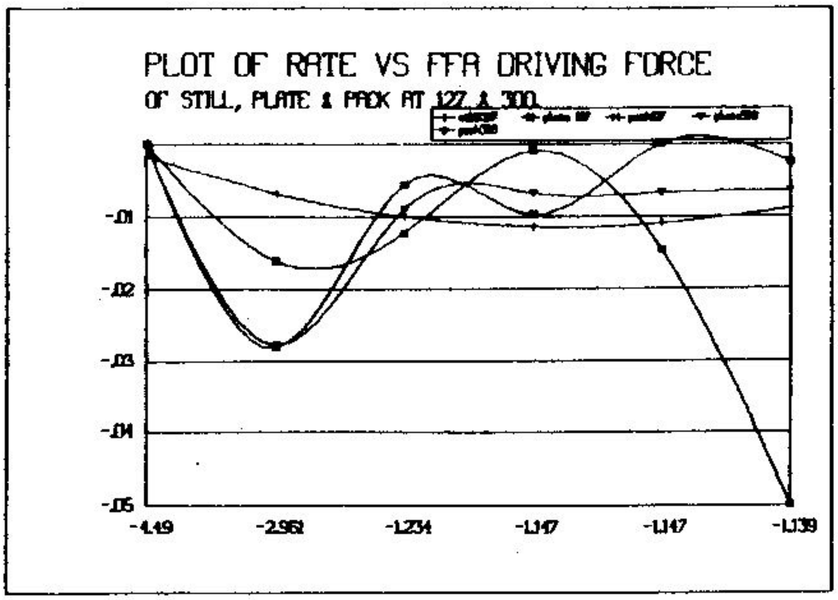
4.3 Pilot Plant Scale Experiments.

Comparison at 700mmHg and 650mmHg vacuum pressures showed that the packed and plate column enhanced stripping and suppressed hydrolysis. The stripping rate operates over a wider f.f.a. Driving force range at a higher stripping rate.

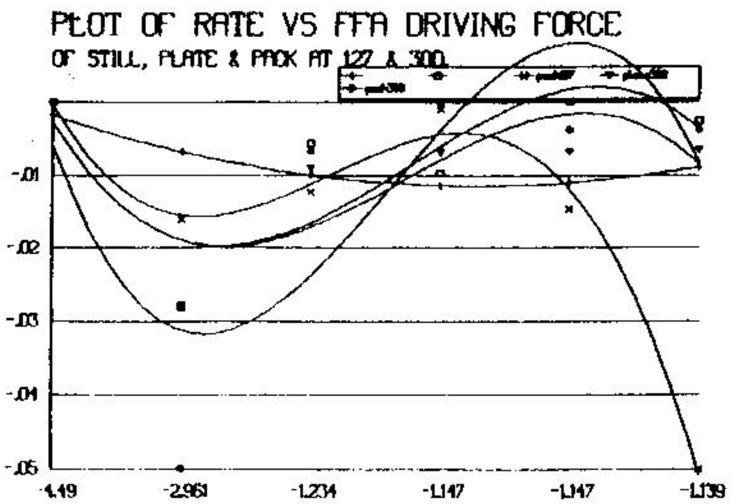


The plate column still exhibits a Quadratic order of equation but a higher stripping rate. Simply said, that the plate column reaches equilibrium faster quicker and inherently more stable but at the expenses of a shorter operating conditions and slightly lower stripping rate.

This inherent stability enables the plate column to compensate minor deviations and needs lesser process control.



Conversely, the packed column exhibit a distinct Cubic order of equation with a higher stripping rate and over a wider operating condition but has instability in moving from one regime to another. Responses to disturbances are faster and more sensitive unless well controlled. Invariably this means better process control is needed especially on the distribution of liquid and vapor.



4.4 Optimisation Experiments.

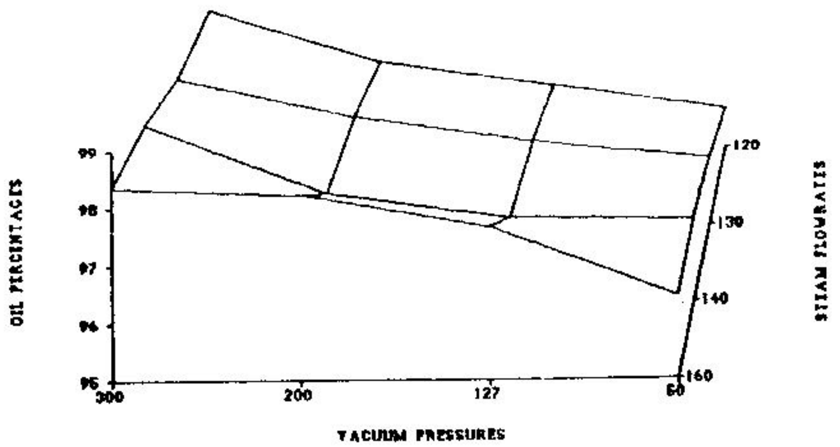
In optimisation, the Response Surfaces Methodology[5] was used to determine optimum conditions for a plate column. This was cross checked with computer predicted results

% OIL	PRESS	TEMP	STEAM
99.9054	3	265	3.50
99.9056	3	260	5.20
99.9070	3	240	17.0
99.9090	2	240	7.50

The optimisation experiment investigates the variation of oil purity attained w.r.t. steam flowrate, vacuum pressures and steam temperature. The Response Surface Curve was calculated for this experiment to determine the optimum point of oil purity attained w.r.t. steam flowrate and vacuum pressures.

PLOT OF VARYING VACUUM & STEAM FLOWRATE

FOR STRIPPING OF FATTY ACIDS



Knowing that the plate responses to these variations is a Quadratic order, a projection based on a Quadratic equation was calculated over a range of 120 to 165 litres per hour steam rate and 50 to 725mmHg pressure reduction.

This technique offers opportunity to optimised several contradicting variables to attain a multiple criteria objectives of Palm Oil purity, vacuum, temperatures, flowrates and time.

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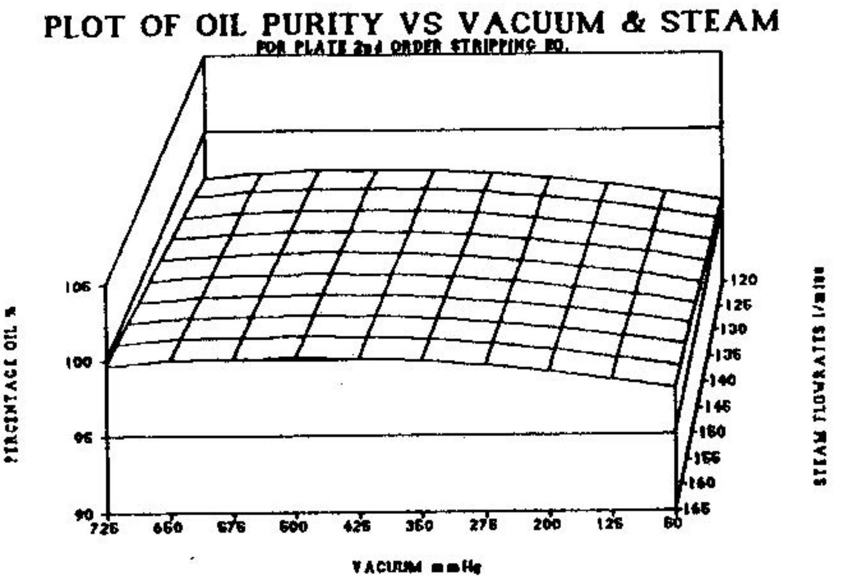
5 Khuri & Cornell, "RESPONSE SURFACES:Design and Analyses.",vol.81, 1987, Marcell Dekker Inc



5 CONCLUSION.

The study has given us a better insight of the mechanisms, mass transfer and efficiency involved in the stripping of free fatty acids from Palm Oil. Furthermore, we were able to determine the impact of plate and packed column on the same factors and its implications on process control and process design. Last but not least, these information could be used as a basis for an multiple criteria optimisation study based on fundamental chemical engineering principles. Future work will focus on trace component deviation on VLE data, determination of plate and packing efficiencies and multiple criteria optimisation.