STUDY OF FILTRATION DILUTED CRUDE PALM OIL SLURRY USING LABSCALE PRESSURE FILTER

By
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Abstract

A labscale pressure filter has been developed to study the filtration behaviour of diluted crude palm oil slurry. The apparatus mainly consists of two parts viz. a mixer and a filter which are placed in a oven for controlling operating temperature. The system is connected to a data aquisation system for recording data of applied pressure, medium pressure, filter temperature, weight of filtrate and mixer speed in a spread sheet. Filtration of DCPOS was carried out at a temperaure range 60°C - 90°C and applied pressure 1 bar - 4 bar using a polyester multifilament woven cloth as filter medium. The results have shown that the filter system was fully capable and suitable to be used to study filtration behaviour of DCPOS. The optimum process condition to give the highest filtration rate was at 2 bar and 90°C. The effect of pressure and temperature to the filtrate flowrate and cake compressibility were also discussed.

Introduction

Diluted crude palm oil slurry (DCPOS) is a slurry which is produced from the dilution of crude palm oil slurry (or undiluted crude palm slurry) in the conventional clarification process. Figure 1 shows the the flowchart of production of crude palm oil by conventional milling process.

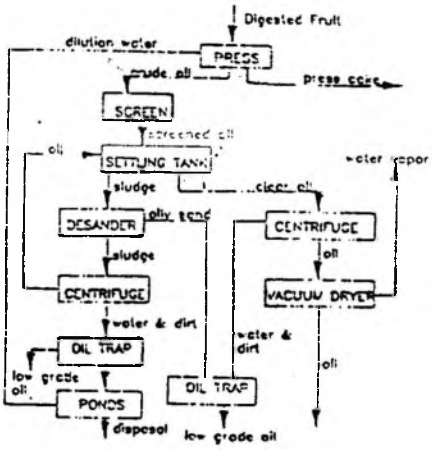


Figure 1: Flowchart of Conventional Milling Process

Dilution is initially carried out to reduce the slurry viscosity to ease separation of oil from its mixture in the clarifier. The main composition of slurry is water, oil and non-oily solids. Water soluble material were also found in the slurry which is consist of carbohydrate, protein, tocopherol, etc[1]. The composition of non-oily solid (DCPOS) is not constant due to the diffrent quality of the palm fruits, uneven flow of undiluted crude palm oil slurry (due batch oil extraction method) and open-loop control of hot water for dilution.. The typical composition of of diluted crude palm oil slurry is 21% - 25% oil, 66% - 72% water and 6 - 7% non-oily solids[2]. The typical density of the slurry is shown in Figure 2a where it is inversersely proportional to temperature. The DCPOS behaves as non-Newtonian fluid i.e. Bingham plastic type fluids and its viscosity deacreases with increasing temperature (Figure 2b). Non-oily solids consist of cell debris, fibres, calyx leaf and dirts as depicted in Photograph 1. The particle-size distribution of NOS particles is presented in Figure 3. The shape of non-oily solids were described quantitatively in term of factors of elongantion(L/B), area (A/(LxB)) and sphericity ($P^2/4A$) is given in Table 1. The true density of NOS particles in the range 0.92 - 1.34 g/cm^3. A typical particle-size distribution of the is presented in Figure 3.

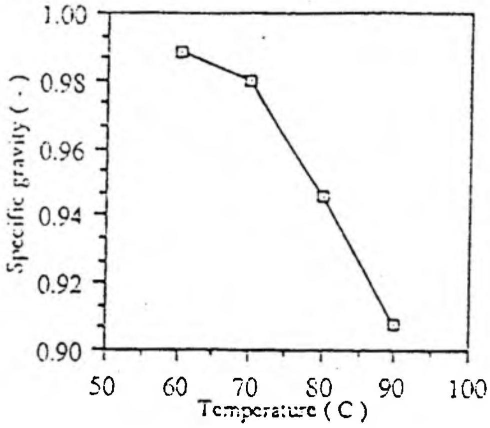


Figure 2a: Density of slurry Versus Temperature

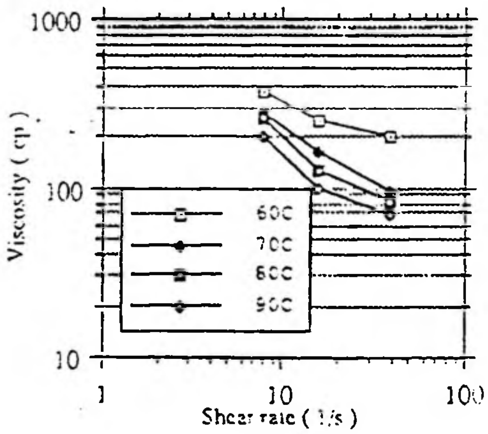


Figure 2b: Rheogram of Diluted Crude Palm Oil Slurry

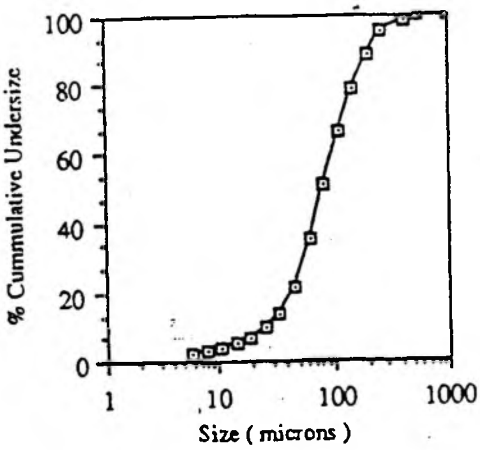
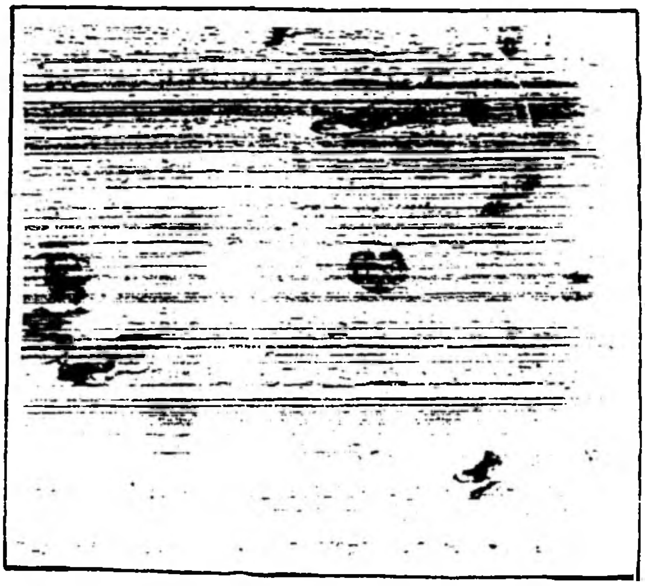


Figure 3: Partice-size distribution of Non-oily Solids



Photograph 1: Non-oily Solids Particles

Table1: Shape Factors of Non-Oily Solids

Type of Particle	Number	Elongation Factor	Area Factor
Cell Debris	17	1.0 - 2.5	0.5 - 1.0
Fiber	2	4.0 - 4.3	0.2
Calyx	1	1.4	0.7

The conventional milling process has been used for twenty years. It involves sterilisation, thresing, digestion, pressing, and clarification. The heart of clarification system is clarifier where the oil is continuously separated by sedementation. The perfomance of the clarifier depends on the slurry concentration, temperature, and cross-sectional area of clarifier. This method is inefficient when dealing with a larger volume of the slurry (usually occurs during peak harvesting season) and high concentration of slurry. The optimum efficiency obtained by diluting the undiluted crude palm oil slurry (UCPOS) to 30% dilution, maintaining settling time to for 4 - 5 hours duration and operating temperature above 90°C[3]. Clarification trials using decanter were reported by several reseachers [4,5,6]. The system would be profitable if oil loss is reduced through good mill operation and process condition[7].

Filtration of crude palm oil slurry has been considered long time ago[8]. But trials using rotary vacuum filter were not succeful due to the fast clogging of filter cloth. The cake resistance of filtration undiluted crude palm oil slurry (calculated by assuming that only oil in the filtrate) in between 0.4 - 21.6 x 10^{11} m/kg for the pressure range 2.9 - 3.5 bar and temperature range 60°C - 80°C[9]. The value indicates that the slurry can be filtered since these values are in the range moderate separation (Table 2).

The method has two advantages (i) reducing pollution problem by reuse of water since it contains very small quantity of suspended solids, (ii) increasing clarifier efficiency by intergrating the filter with clarifier where the function of filter is to reduce the solids content in the slurry. However the viability of the technique would depend on filtrate flowrate and oil loss.

In this study a labscale pressure filter has been developed to study the filtration behaviour of DCPOS at diffrent pressures and temperatures. Results of filtration of DCPOS with filtration of non-oily solid slurry (NOSS) are compared with filtration of non-oily solids slurry (NOSS), prepared crude palm oil slurry (PCPOS), and undiluted crude palm oil slurry (UDCPOS).

Table 2: Clasification of Separation by Filtration[10]

Ease of Separation	α (m/kg)
Very easy	10^{9}
Easy	10^{10}
Moderate	10^{11}
Difficult	10^{12}
Very Difficult	10^{13}

Cake and Medium Resistance Equations

Filtration of DCPOS involves two-phase flow of filtrate i.e water and oil. Calculation of cake and medium resistance by conventional filtration equation require measurement of density and viscosity of filtrate. In-situ measurement of viscosity and density of two-phase of liquids flowing through a compressible cake and filter medium is cumbersome. Furthermore no reliable method has been developed for this purpose. Therefore calculation of cake and medium resistance of filtration of DCPOS was carried based on the following assumption:

i. The oil phase is dispersed while water remains as continuos phase.
ii. The system temperature is constant.
iii. The pressure accross the cake is constant.
iv. The applied pressure is much larger than capillary pressure. Therefore it is assumed that no separation occurs due to capillary pressure.
v. The oil phase coaleescence occurs at the outflow of the filter medium.

Hence, the property of filtrate which consists of oil and water flowing through the cake and medium can be averaged as follow:

i) Density of filtrate

$$r_{ow} = x\, r_w + (1-x) r_o \qquad (1)$$

ii) Viscosity of filtrate

$$m_{ow} = x\, m_w + (1-x)\, \mu_o \qquad (2)$$

Modification of c^* (weight of the dry solids deposited on the cake when a unit volume of filtrate obtained) proposed by Gale [11] is,

$$c^*_{ow} = w_s\,(1 - w_s/w_c)/\mu_{ow} \qquad (3)$$

From here, the equation of cake and medium resistances of diluted crude palm oil can be developed by modification conventional filtration equation and result of plot time/volume (t/v) versus volume (v).

i) cake resistance,

$$\alpha_{ow} = \frac{\text{slopex } 2 \times A \times P)}{\mu_{ow} \times c^*_{ow}} \qquad (4)$$

ii) medium resistance

$$R_{ow} = (\text{intercept} \times A \times P)/\mu_{ow} \qquad (5)$$

alternatively R_{OW} also could be quatiti fied by measuring pressure at the top of filter medium P_m and calculated using the following equation:

$$R_{ow} = P_m / \mu_{ow} q \qquad (6)$$

The imperical equation of relationship of α_{OW} and P is given by,

$$\alpha_{ow} = \alpha_{o,ow} (P)^n \qquad (7)$$

The value of n can be obtained by plotting log α_{OW} versus log P and slope of the graph is equal to n.

Apparatus

Figure 4 shows the schematic diagram filtration rig to study the filtration of DCPOS at constant pressure and temperature. It mainly consists of a mixer and a filter which is placed in the oven.

Mixer has 15 cm ID, 20.5 cm height and made from stainless steel. It is accomodated with a stirrer, a sample inlet facility, a thermocouple, a sampling facility and an inlet of compressed nitrogen gas. A 1/2" globe valve is fitted between the mixer and the filter.

Filter has cylinderical shape 10 cm ID and 4 cm height and also made from stainless steel. Five 1/4" NPT holes at the top of filter are used to to equip five 1/8" pressure tubing to measure medium and cake hydraulic pressures. The head of the first tubing touches the surface of filter cloth, the second, the third, fourth and fifth heads tubing are positioned at 5 mm,

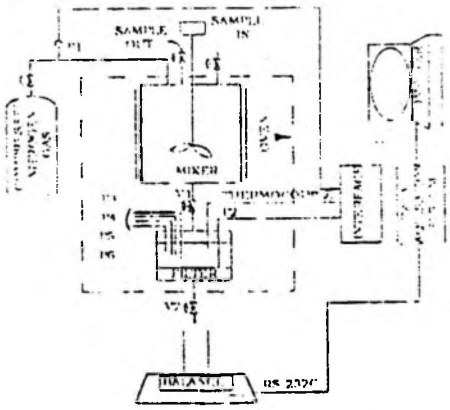


Figure 4: A Schematic Diagram of the Filtration Rig

10 mm, 15 mm and 20 mm above the first tubing head. The tubing are connected to 5 - 25 mA pressure tranducers.

The temperature of slurry in the mixer is increased to desired temperature using a belt heater fitted with a on-off temperature controller. The apparatus is installed in fabricated oven to control the temperature to desired test temperature.

The filtrate consist of oil and water is collected in 250 ml glass measuring cylinder which is placed on the top of digital balance. The balance can measure the weight from 0 -300 grams with 0.1 gram accuracy. It is interfaced to computer via RS-232C. The balance can be set to zero prior weighing and also can be set to send data in desired sampling interval.

Burr-brown data aquisation is used to acquire data of weight of filtrate, medium and hydraulic pressures, filter temperature and stirrer speed. The system compa-tible to MS-DOS operating system in which it is linked to a potable computer and to a printer for recording and displaying data. The system is configured with a software, an analog input board, a series termination panel and anumber of analog signal cables. Number of channel for this study are 10 i.e. & analog (6 pressure channels and 1 time channel), 1 parse and 1 time. The maximum period of data aquisation is 3600 seconds for 60 seconds sampling rate between the sampling data. The data is displayed in numeric form on computer monitor for easiness monitoring of filtration process.

Material and Method

i) Material

The slurry was filtered using polyester multifilament twill weaves with air permeability of 20 $cm^3/cm^2/sec$.

The samples of DCPOS were provided by a palm oil mill. The samples were taken from a sampling point which is located just below vibrating screen.

ii) Method

Initially the filtration rig in the oven was heated up to desired temperature for 4 hours. Then the temperature was kept constant by oven's temperature controller. The sample of diluted crude palm oil slurry was heated at 60^oC and then homogenised for 1 minute. by stirring. A 600 ml of homogenised slurry was placed into the mixer of filtration rig. The slurry was continuosly homogenised by stirring at 1200 rpm. If the slurry's temperature below the desired test temperature then it is heated up using belt heater which is fitted with temperature controller. At the begining, all the pressure reading (P1, P2, P3, P4, P5 and P6) were set to zero. Then the pressure valve was opened slowly until P1 (displayed on the monitor) had reached to the desired pressure. A 50 ml sample was taken through the sampling facility to check its composition.

The valve V1 (refer to Figure 4) and the data aquisation system was slowly opened to start filtration and the data aquisation was activated simultenously. Filtration tests were carried out at temperatures of 60^oC, 70^oC, 80^oC and 90^oC and pressures of 1 bar, 2 bar, 3 bar and 4 bar. The volumetric ratio of oil and water of filtrate was recorded manually every 5 minutes for the first 1 hour, 15 minutes after 1 hour to 3 hours, 20 minutes after 3 hour to 4 hours and 30 minutes after 4 hours of filtration. The wet cake and dry cakes were weighted and the oil content in the cake was determined using soxhlet apparatus.

Result and Discussion

i) Slurry Preparation

The most important in laboratory experiment is to obtain a reproducible slurry preparation method[12]. It is found that the DCPOS tend to separate into oil and sludge at temperature to homogenize the slurry i.e above 50^oC .

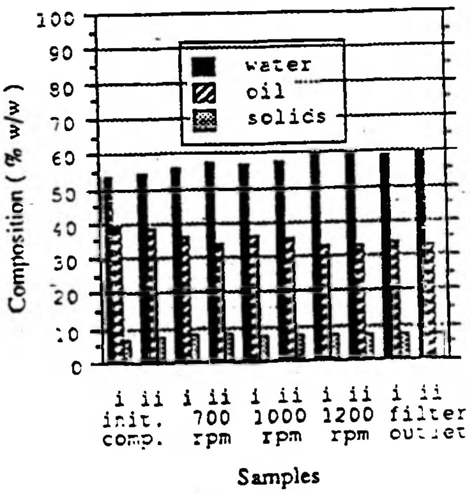


Figure 5: Result of Homogenation Test

A homogenationtest was carried out at 60°C to find appropriate speed of filter. stirrer to obtain a reproducible composition of the slurry. The test was divided into two stages, where the composition of slurry was determined immediately after homogenation, and in the second stage the slurry composition (in duplicate) was determined after it was homogenised for period 30 minutes in the filter mixer at rotation speed 700 rpm, 1000 rpm and 1200 rpm. Typical result of the homogenation test is presented in Figure 5. It shows that stirring at rotation speed of 1200 rpm gives a consistent slurry composition. Thus, for all sample preparation, the slurry was continuosly homogenation in the filter mixer by stirring the slurry at 1200 rpm.

ii) Effect of Pressure to Filtration Rate

Typical effect of pressure to the filtration rate of filtration DCPOS at operating temperature 60°C, 70°C, 80°C and 90°C is shown in Figure 6.

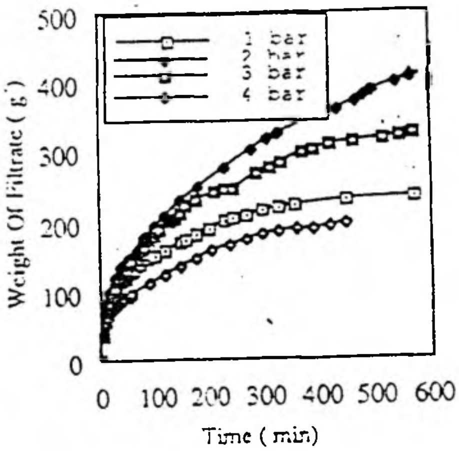


Figure 6: Weight of Filtrate(Filtration at 60 °C)

For each operating temperature the highest flowrate is obtained at applied pressure 2 bar followed by pressures 3 bar, 1 bar and 4 bar. The results reflected that the cake is inelastic where the filtrate flowrate was increased to maximum at 2 bar and then deacresed as pressure was increased. The reduction of the filtrate at temperature above 2 bar was probably due to the change of internal cake structure .

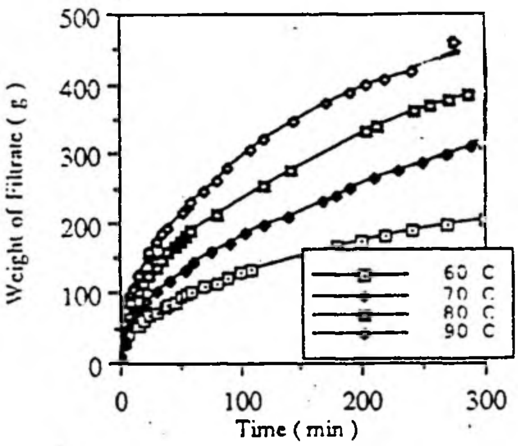


Figure 7: A Typical Example of Effect of Temperature on Filtration Rate

iii) Effect of Temperature to Filtration Rate

Figure 7 shows a typical effect of temperature on the filtration rate. Increasing temperature has increased the filtration rate significantly by reducing the viscosity (liquid resistance).

iv) Quantity Water and Oil in Filtrate

Figure 8 presents the typical quantity of water and oil in filtrate. The volume of water phase reaches maximum at early filtration (up to 40 minutes) and then reduces steadily until the end of filtration. The increment of volume of oil is quite steadily from beginning to the end of filtration.

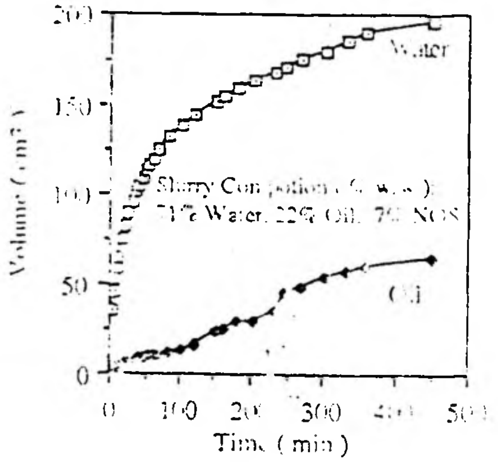


Figure 8: Volume of Water nad Oil Versus Time
(Filtration at 1 bar 60°C)

The changes of composition of filtrate (% per total volume) of water and oil with time is given in Figure 9. The water content is at maximum at early filtration and reduces linearly until the end of filtration whilst the changes oilcontent is reversed i.e. minimum at beginning of filtration and increased until the end of filtration.

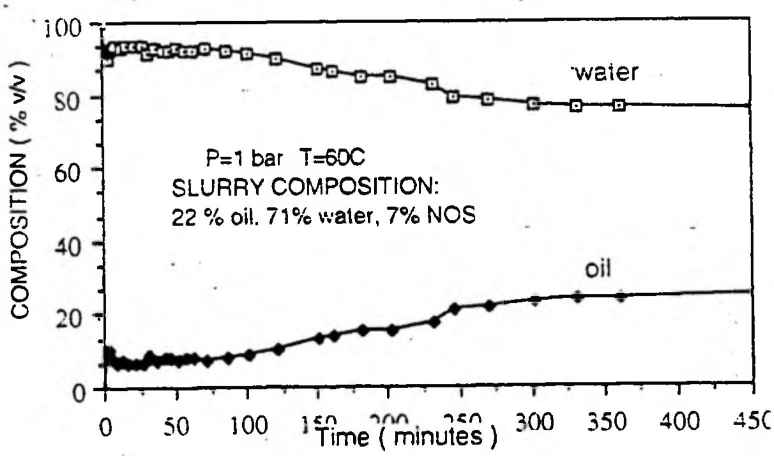


Figure 9 :Composition of filtrate Versus Time
(Filtration at 1 bar 60°C)

It means that no separation occurs from beginning to the end of filtration. The curves of water and oil in the graph illustrate the flowrate of continuos and dispersed phases through the cake and medium. The flowrate of oil (dispersed phase) slower than water (continuos phase) because the dispersed phase needs to coalease to a certain size before it can flow out[13].

v) Cake Hydraulic Pressures

An indirect method to measure porosity accross the cake is by measuring liquid pressure as funtion of distance through the cake[14]. Figure 10 shows a typical hydraulic pressures (PI, P2, P3), applied pressure (Pa) and medium Pressure (Pm) in the cake through filtration of diluted crude palm oil slurry. The hydraulic pressure ditributions almost independent of time which is opposed to filtration of silica slurry (Figure 11). The both filtrations were carried out at the same pressure but pressure drop accros the the cake of DCPOS only 0.4 psi which is much smaller than the pressure drop through the silica cake i.e. 3.2 psi. A slow filtration rate of filtration DCPOS

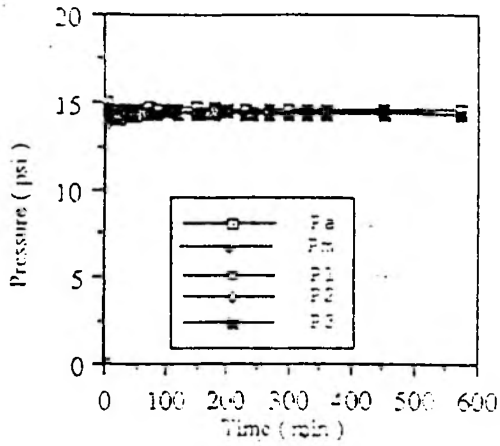


Figure 10: Plots of Pressures versus Time
(Filtration of DCPOS at 1 bar 60°C)

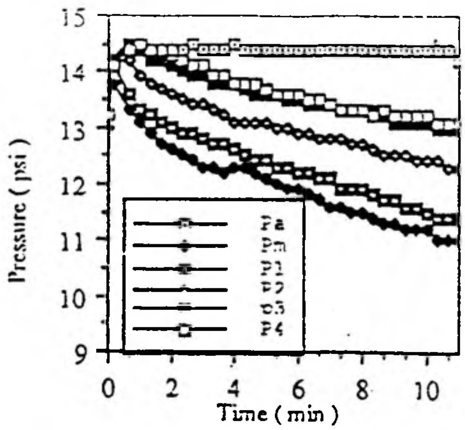


Figure 11: Plots Pressures Versus Time
(Filtration of Silica Slurry at 1 bar 25°C)

was related to the a slight pressure drop accros the cake and horizontal hydraulic pressure distributions. Under the same applied pressure the diffrence of filtration was 108 minutes. Other factors could affect the filtration rate of DCPOS are particle-size distribution and oil globules in the slurry. The NOS particles has wide range particle-size disribution (as shown in Figure 3). The wide particle-size distribution to cause media blinding and migration of fine particles in the pores. The oil globules were attributed a low fluid loss property and recognised as effective sealing property in the wall of bores holes [15]. The small particle size might reduce the porosity of the cake by migrating into the interstices[16]. Combination of these two factors of course would double the deficulty of filtration diluted crude palm oil slurry.

vi) Calculation of Cake Resistance

A set of filtrations which consist of filtration of non-oily solids slurry (NOSS), undiluted crude palm oil slurry (UDPOS), prepared crude palm oil slurry (PCRPOS) and diluted crude palm oil slurry (DCPOS) were carried out to determine the suitablity of the equations (1) and (2). to be used for quantifying the cake and medium resistances of DCPOS. A graph of t/v versus v of the filtration of that slurries is presented in Figure 12. The slopes of the graph and the equations (1) to (2) were used to calculate the cake and medium resistances. The values of the resistances is presented in Table 1. The highest value of the cake resistance is found for filtration of DCPOS, followed by NOSS, PCPOS and UDCPOS. The data indicative that the calculation of viscosity and density based on weigted average and equationa (1) and (2) can be used sufficiently to calculate medium and cake resistances. Modification of conventional filtration equation can be used to quantify the cake resistance.

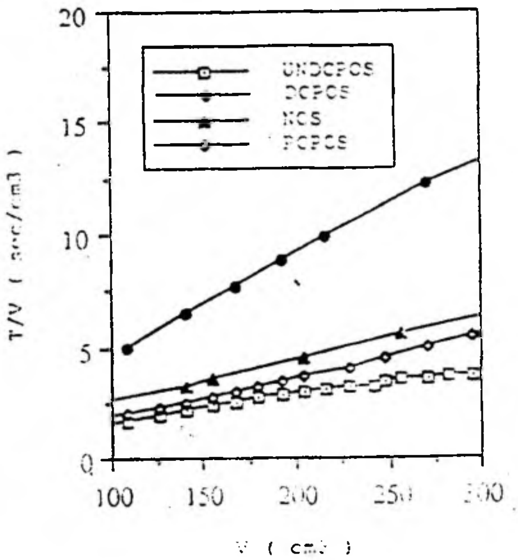


Figure 12: Plot T/V Versus V For Diffrent Crude Palm Oil Slurries

The slopes of the graph and the equations (1) to (2) were used to calculate the cake and medium resistances. The value of the resistances is presented in Table 2. The highest value of the cake

Table 3: Cake and Medium Resistances

SLURRY	T	μ	Ws	α x E-11	Rm x E-13
	(C)	(kg/ms)	(-)	(m/kg)	(1/m)
DCPOS	90	2596	0.06	101.0	126.0
NOS	23	946	0.07	67.4	14.7
PCPOS	90	8734	0.07	4.2	10.4
UDCPOS	90	6958	0.07	2.5	7.7

resistance is found for filtration of DCPOS, followed by NOSS, PCPOS and UDCPOS. The data shows that the calculation of viscosity and density based on weigted average and equationa (1) and (2) to calculate medium and cake resistances and modification of conventional filtration equation can be used sufficiently to quantify the cake resistance.

vii) Medium Resistance

The medium resistance of filtration DCPOS was calculated using equation 6. A typical profile of increasingly medium resistance (Rm) of DCPOS versus time is depicted in Figure 13. The figure shows that medium resistance is not constant as assumed in conventional filtration equation but increased steadily as filtration continues. The increasing of medium resistance of filtration DCPOS significantly diffrent with increasing of medium resistance of silica slurry (Figure 14).

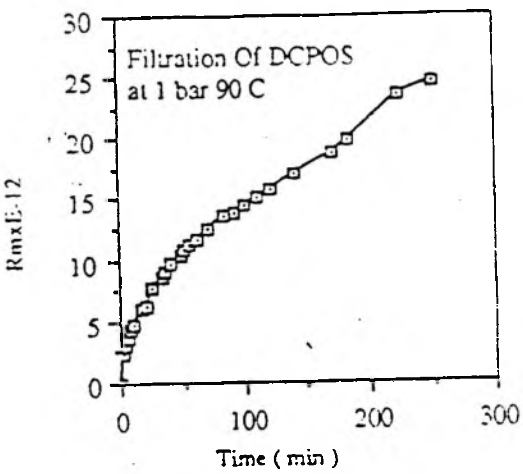


Figure 13: A Typical Example Medium Resistance Versus Time (DCPOS)

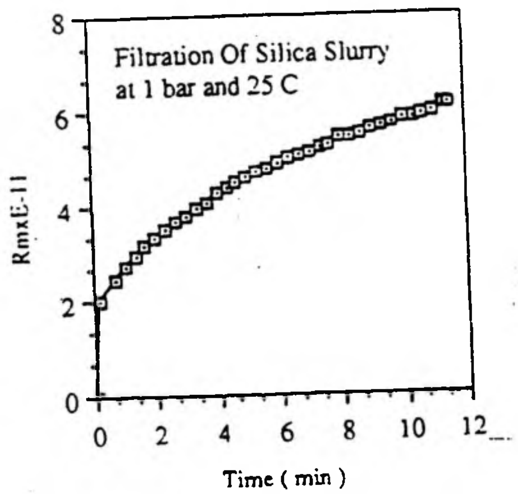


Figure 14: A Typical Example Medium Resistance Versus Time (Silica)

The size-distribution of silica powder is presented in Figure 15 where its mean size is 24.4 microns. The mean size of silica twice smaller than than the mean size of NOS particles where the latter has the mean size 41.7 microns (Figure 3). Since the mean size of silica smaller than the NOS particles the former particles have capability to penetrate into multifilament threads more easier than the latter and cause a subtantial changes of Rm.

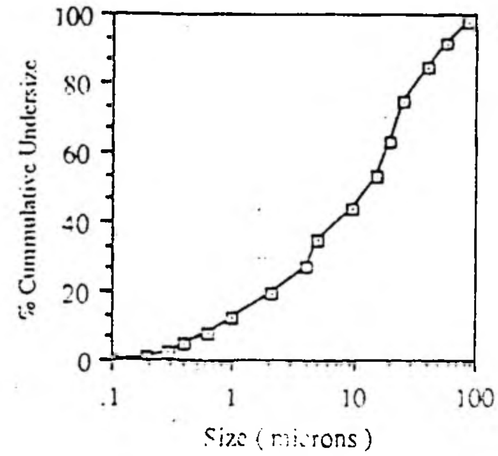


Figure 15: Particle-Size Distribution of Silica

viii) Effect of Temperature to The Cake Resistance

The cake resistance of filtration of DCPOS was calculated using of equation 4. Figure 16 shows the effect of temperature to the cake resistance. It deacreases linearly as temperature increases except for filtration at 2 bar. The deacreasing of the cake resistance could be due to the deacreasing of viscosity (low viscosity high filtration rate) of liquid and increasing the size of pores in the cake..There are two mechanisms may be involved in increasing the pore size of the cake.. The first mechanism is due to the flowing out of some water in the pores when its viscosity deacreses or when evaporation occured. The second mechanism is due to the. escaping of oil globules from the pores when their surface tension is reduced at high temperature. Reducing the oil globules surface tension would increase the coalesing between the globules and assit the movement of oil out of the pores. .Besides of these two factors, the porosity of the cake is also influenced by the size-distribution and dispersion of NOS particles.

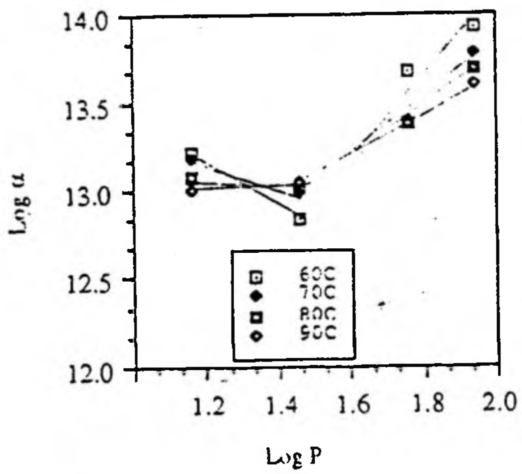


Figure 16: Plot Log α Versus Log P

The increasing of cake resistance for the filtrations at 2 bar probably due to the migration of the fine particles into the pores or increasing dispersion of solid particles during the slurry prepartion.

ix) Cake Compressibility

A plot of log a and log P is presented in Figure 17. It shows that the cake is compressible. The log α deacreses from log P = 1.17 (or 1 bar) to log P = 1.46 (or 2 bar) and then increases till log P = 1.94 (or 4 bar). The value of n (i.e cake compressibility) at diffrent temperature is calculated using equation 7. The values of n at diffrent temperatures are given in Table 4.The cake is very compresible

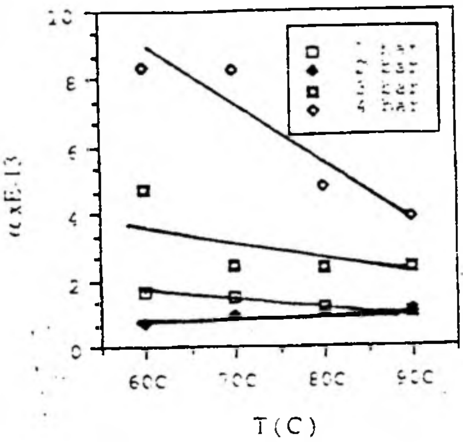


Figure 17: Plot α Versus Temperature

T (C)	n
60	1.08
70	0.93
80	0.79
90	0.77

Table 4: Cake Compressibility

at 60°C (n > 1) and becoming less compressible as temperature increases. Deacreasing of n possibly due to deacreasing of the amount of soluble material in water, oil globules and water in the pores as temperature is increased.

x) Cake Composition

A typical cake and slurry compositions from filtration of DCPOS at temperature range 80°C to 90°C and a pressure range 1 bar - 4 bar using labscale filter press is given in Table 5a. The compositions of cake and slurry of filtration using pilot scale filter press is presented in Table 5b. The compositions of cake and slurry of pilot and labs scales are not much diffrent. Hence the the labscale filter press can be used for filtration study of DCPOS. Result of analysis of variance to determine the effect of operating condition (pressure and temperature) to the cake composition of labscale filtration showed that the two parameters were not significantly effect the composition of the cakes. In another study, the cake composition obtained from filtration of diluted crude palm oil slurry (DCPOS) are compared with the composition of cakes from filtrations of prepared crude palm oil slurry (PCPOS), undiluted crude palm oil slurry (UDCPOS), non-oily solid slurry (NOSS), silica, cupric and talc slurries. The compositions of the slurries and cakes cakes are presented in Figure 18 and Figure 19 respectively . Figure 19 shows that the cakes of crude palm slurry have higher water content than inorganic cakes. The high water content in the former cakes could be due to the wide range particle-size distribution.

Table 5a: Composition of DCPOS and Cake at 80C and 90C (labscale tests)

T (C)	P (bar)	Cake Composition Water	Oil	Solids	Slurry Composition Water	Oil	Solids
80	1	69.9	19.2	13.0	68.5	25.8	5.7
80	2	69.4	21.4	9.2	70.7	23.3	6.0
80	3	67.6	22.4	10.0	67.2	25.9	6.9
80	4	67.2	23.1	9.1	65.5	28.3	6.2
90	1	70.6	19.0	10.4	71.7	22.6	5.7
90	2	70.1	17.3	12.6	72.1	21.6	6.3
90	3	65.6	21.3	13.1	71.3	21.6	7.1
90	4	71.2	17.2	11.6	69.7	23.5	6.9

Table 5b: Composition of DCPOS and Cake (pilot scale trials)

T (C)	P (bar)	Cake Composition Water	Oil	Solids	Slurry Composition Water	Oil	Solids
80	1.7	66.1	24.8	9.1	68.4	26.6	5.0
90	3.4	73.0	17.8	9.2	64.8	30.0	5.2
80	5.2	70.1	20.6	9.3	69.8	25.3	4.9

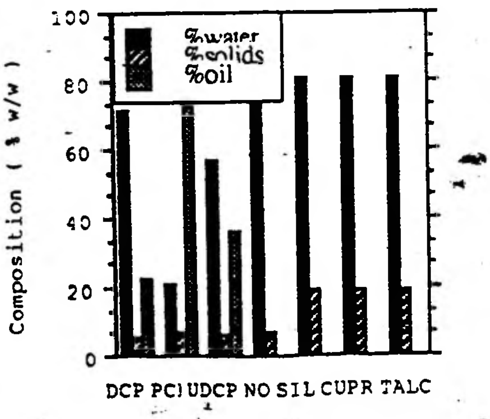


Figure 18: Composition of Diffrent Slurries

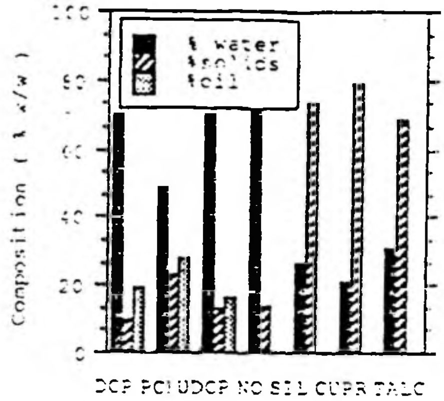


Figure 19 : Composition of Cakes of Diffrent Slurries

Conclusion

At first it is essential to determine that filtration rig and method which capable and suitable to be used for filtration of DCPOS. From a series of filtration test, it was found that the rig and method employed can produce a reproducible slurry composition. The rig capable to filter the DCPOS at temperature range $60^{o}C$ - $90^{o}C$ and pressure range 1 bar - 4 bar (14.5 psi - 87 psi.). The optimum filtration rate of DCPOS was found at 2 bar and $90^{o}C$. The flowrate of water phase faster than the flowrate of oil phase because the composition of water in the DCPOS slurry more than oil. The DCPOS is the most difficult to filter slurry followed by NOSS, PCPOS and UDCPOS. The pressure drop through the cake of DCPOS is too small and distribution of hydraulic pressures in the cake is almost independent of time. The modification of conventional filtration equation based on weighted average can be used for calculation of the cake resistance and might be extent to quantify the medium resistance. The cake resistance deacreses as temperature increases. The cake is highly compressible at $60^{o}C$ (n>1) and becoming less compressible as temperature increase. The medium resistance of filtration DCPOS is not constant (i.e. oppose to conventional filtration equation) but increases in form of parabolic curve.The cake composition which obtained by filtration rig (labscale) is similar to the cake produced from pilot scale filter press. Pressure and temperature were unsignificantly effected to the cake composition. The cake of palm slurries are hydrofobic than the cakes of silica, cupric oxide and talc.. This is correspond to the property of NOS particle i.e. has a wide particle-size distribution and diffrent shapes.

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Nomenclature

A = filtration area (m^2)
c^{*}_{ow} = dry suspended solids/ unit volume of filtrate(kg/m^3)
n = compressibility (-)
P = applied pressure (Pa)
P_2 = medium pressure (Pa)
q = superficial velocity (m/s)
Rm = medium resistance (1/m)
x = fraction of water in filtrate
ρ_o = densisty of oil (kg/m3)
ρ_w = density of water (kg/m3)
μ_o = viscosity of oil (kg/ms)
μ_w = viscosity of water (kg/ms)
μ_{ow} = weighted average of viscosity (kg/ms)
α_{ow} = cake resistance of filtration of DCPOS (m/kg)

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