DEVELOPMENT OF OSCILLATORY FLOW REACTOR (OFR) FOR SYNTHESIS OF HYDRAULIC OIL

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by

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CERTIFICATION OF APPROVAL

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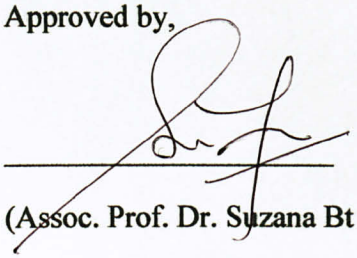

(Assoc. Prof. Dr. Suzana Bt Yusup)

UNIVERSITI TEKNOLOGI PETRONAS

TRONOH, PERAK

July 2009

CERTIFICATION OF ORIGINALTY

This is to certify that I am responsible for the work submitted in this project, that the original work is my own except as specified in the reference and acknowledgement, and that the original work contained herein have not been undertaken or done by unspecified sources or persons.

(Mohd Hasnul Adib Bin Mohamad Noh)

ABSTRACT

Long residence time reaction such as synthesis of hydraulic oil commonly performed in batch reactor. Even though continuous process is proved to be more efficient compared to batch process but if give certain disadvantages. Long residence time reaction in CSTR will give a wide range of residence time and these will result to the difficulties in control. Same goes for PFR as it requires a great length of narrow tubing for long residence time reaction that will lead to the control, pumping and operation difficulties. In addition, both problems cause the reactors to be expensive. The study on OFR has shown that these type of reactor has a potential to be more efficient compared to the other reactor for long residence time reaction. Therefore, the project is conducted to develop a conceptual design of OFR for synthesis of hydraulic oil thus produces a model of the reactor in a lab scale.

ACKNOWLEDGEMENT

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CHAPTER 1

INTRODUCTION

1.1 Background of Study.

Hydraulic oil refers to a fluid which is intended to transmit power or carry a load in various systems. Hydraulic oil is used in different stationary and mobile machines, such as cylinder performing a linear movement or rotating hydraulic motors. In addition to power transmission, the function of hydraulic oil is to lubricate mobile parts in the components of the system and to cool the system. Hydraulic oil has to fulfill the following requirements:

- Suitable viscosity at different temperatures.
- Sufficient pressure endurance.
- Non-foaming properties.
- Oxidation inhibition.
- Corrosion inhibition.
- Inert quality.

OFR is a relatively new type of mixing device for chemical/biochemical reactions under development in recent decades. It is generally understood to be a cylindrical tube or column containing periodically spaced orifice baffles in which a liquid or multiphase fluid is oscillated axially by means of diaphragms, bellows or pistons at one or both ends of the tube. The existences of the baffles allow a better mixing of the reactant as well as longer residence time compared to PFR. By means, it will produce much more product in a specific period of time.

1.2 Problem Statement.

Many long residence time processes such as hydraulic oil production are currently performed in batch, as conventional designs of PFR prove to be impractical due to their high length-to-diameter ratios, which lead to problems such as high capital cost, large footprint, high pumping costs and also difficult to control. Continuous processing tends to be more efficient than batch processing, and tends to be used at larger scales due to its economy of scale. However it is seldom used for long reactions as conventional continuous reactor designs have certain drawbacks which are:

- CSTRs: broad residence time distribution and tends to be expensive.
- PFRs: require great lengths of narrow tubing, causing control, operability, footprint and pumping problems.

1.3 Objectives and Scope of Study.

The objectives of the project are:

- To develop a conceptual design of OFR.
- To used the OFR for synthesis of hydraulic oil.
- To construct a model of the reactor for the process in a small scale.

The conceptual design may require a manual calculations as well as simulations such as polymath and etc. To fulfill the objectives above, the research and study on hydraulic oil production and reactor design need to be done.

CHAPTER 2

LITERATURE REVIEW

2.1 Hydraulic oil.

Hydraulic oil commonly made from synthetic esters synthesized from plant oils (vegetable oils). This is because the advantageous properties of vegetable oils include user friendliness and non-toxicity. In addition to this, they degrade in the environment, do not accumulate in the food chain of nature and are renewable raw materials.

Van der Waal and Kenbeek have presented a process for the preparation of synthetic esters from vegetable oils or animal fats (Proceedings of the Tribology 2000, 8th International Colloqium, Technische Akademie Esslingen, German, 14-16 Jun. 1992, Volume II). The process comprises first decomposing the glyceride esters of the starting material into fatty acid and glycerol and subsequently separating the fatty acid fraction into liquid and solid phases. The fatty acids of the liquid phase are separated by distillation into single fatty acids which can be further modified. Fractions containing a single fatty acid are esterified with no beta hydrogen polyols for preparing a synthetic ester.

The above process is known as esterification method. The fatty acids of the ester prepared according to the method usually contain less unsaturated double bonds which improves the oxidative stability. However, the cost of the process is extremely high, due to the multistage separation and purification reactions and the most severe condition (high pressure and temperature) required by the reaction. Moreover, it has been found that when fractions containing only a single fatty acid are reacted with polyol, plenty of mono and diglycerides are formed, so all the OH group of the polyol does not react. This

decreases the triglyceride yield and the raw material has to be recycled several times if the yield is to be improved.

The other method for preparing the synthetic esters from vegetable oil by avoiding the multistage reaction with several separations and recycling has been found. This process is known as transesterification (Merja Lamsa, 1999). Vegetable oils suitable as a starting material of the process are rapeseed oil, soybean oil, tall oil, high oleic sunflower oil, castor oil, olive oil, and others (Boris Krzan and Jose Vizintin, 2003).

In the process, a vegetable oil is first transesterified by reacting the vegetable oil with a lower alkanol to obtain a mixture of fatty acid lower alkyl esters. The lower alkanol used in the first transesterification reaction is preferably a C_1-C_4 alkanol, especially methanol or ethanol. The obtained mixture of lower alkyl esters of the vegetable oil is thus preferably a mixture of C_1-C_4 alkyl esters, especially a mixture of methyl and ethyl esters. The first transesterification reaction is illustrated by the following general reaction:

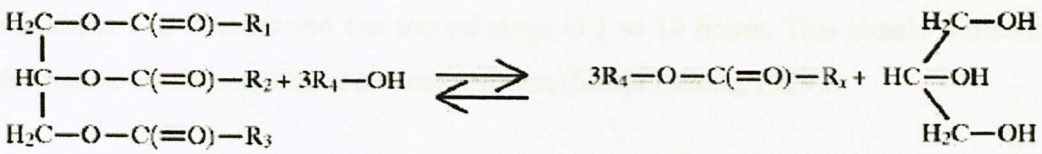

Figure 1: General reaction for first transesterification reaction.

Where R_1, R_2 and R_3 are fatty acids residues, R_4 is an alkyl residue, especially a C_1-C_4 alkyl residue, and R_x is R_1, R_2 or R_3. Glycerol is formed as byproduct.

In the second transesterification reaction, the mixture of fatty acids lower alkyl esters obtained from the first transesterification reaction is reacted with a no beta hydrogen polyol such as trimethylol ethane, trimethylol propane, trimethylol butane or pentaerythritol. The second transesterification reaction is shown in the general reaction in figure 2:

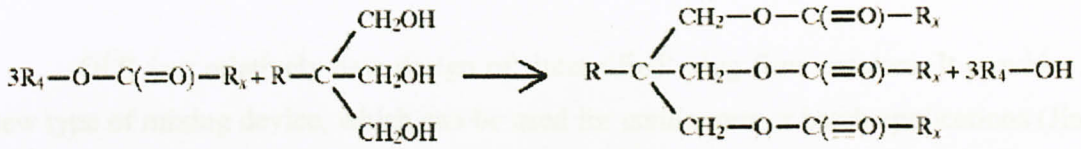

Figure 2: General reaction for second transesterification process.

Where R_4 and R_x have the same meanings as in the figure 1 and R is a C_1-C_6 alkyl group, especially a C_1-C_4 alkyl group, or a CH_2OH group.

The synthetic ester obtained from the second transesterification reaction is recovered and, if desired, purified by conventional methods, for example by neutralization and washing with an aqueous acid.

According to the invention the second transesterification reaction may preferably be carried out in two stages. The reaction temperature of the first stage being from 50°C to about 110°C while the second stage is from 110°C to about 160°C. Reaction time may vary for example from two to twelve hours. Preferably the reaction time for the first stage is about 1 to 7 hours and the second stage is 1 to 10 hours. This clearly indicates that the reaction is a long residence time reaction (Merja Lamsa, 1999).

Additives will be added into the synthetic ester to improve their poor stability properties such as poor thermal and oxidative stability due to unsaturated and polyunsaturated fatty acid (Merja Lamsa, 1999). The table 1 below shows the common additives that have been used in the process:

Additives	Examples
Oxidation inhibitors.	Amines and phenols.
Antiwear agents and corrosion inhibitors.	Phosphates or sulfonated.
Antifoam agents.	Metal sulfonates, metal phenates, polyesters or silicones.
Viscosity index improvers.	Polyisobutenes, styrene-butadiene and ethane-propene-copolymers.

Table 1: Additives for hydraulic oil.

2.2 Oscillatory Flow Reactor (OFR).

OFR is a relatively new design of 'intensified' plug flow reactors. It provides a new type of mixing device, which can be used for continuous or batch applications (Jian and Ni, 2003). In contrast to the conventional PFR, the OFR has equally spaced orifice plate baffles which divide the tubular reactor into single chambers. The orifice might be a single-hole or multiple-hole orifice baffle (Stonestreet and Harvey, 2002). When the fluid is oscillated, the baffles cause the formation of vortices, which leads to both radial and axial mixing. Thus, the chambers each act like a single stirred tank reactor, while the fluid is streaming through them. The chaotic flow patterns lead to instability and turbulence within the reactor and provide intensive, uniform mixing. Figure 3 shows the typical configuration of OFR with single-hole orifice baffle.

Figure 3: Typical configuration of OFR.

Oscillation can be carried out in two different ways: by oscillating the fluid itself or the baffled device. The OFR has a number of advantages over other reactors which might make it useful for special applications. One major advantage is the good, uniform mixing, which provides a good heat and mass transfer. The good mixing in OFR also results in shorter reaction times. Shorter reaction times lead to a reduced length-to-diameter ratio in comparison to conventional plug flow reactors and therefore a more

compact design. Thus the OFR is ideal for long reactions. The figure 4 below shows the flow pattern in OFR.

Figure 4: Flow pattern in baffled tube.

The key future of oscillatory flow mixing is the intensity of mixing can be controlled by altering the frequency and amplitude of oscillation. This will allow a wide range of mixing condition to be achieved, from "soft" mixing, where Oscillatory Flow Reynolds Number, Re_o is in the range of 50 - 500, to the most intense, corresponding to mixed flow (a single continuous stirred tank reactor, CSTR), where Re_o is over 5000. Figure 5 shows the nature of mixing patterns in various mixing condition.

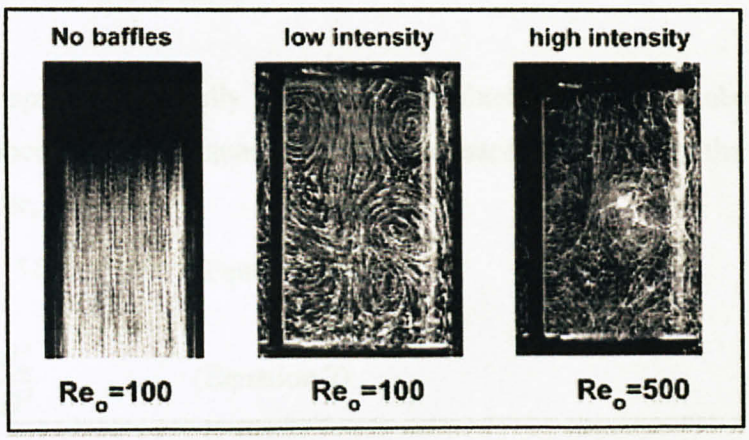

Figure 5: Mixing patterns in various mixing condition.

In the first image, there is no baffle inside the tube which resulting to no radial mixing. While in the second image, the existence of orifice baffle inside the tube cause a typical vortex formation, and the third image shows how the mixing intensity is increased by increasing the oscillation intensity; reflected by the higher value of Re_o (Stonestreet and Harvey, 2002).

The main advantage of OFR is that it offers a means to perform reactions which require long reaction times (of order hours) in a reactor of greatly reduced length-to-diameter ratio. This is achieved while maintaining plug-flow residence time distribution, RTD characteristics, effective mixing, and high heat and mass-transfer rates. Such requirements are difficult to achieve (for long reaction time processes) in tubular reactors relying on throughput alone to achieve mixing. These features make it possible to consider performing certain reactions continuously, which previously were only possible in batch.

2.2.1 Geometric and Dynamic Similarity.

The geometric similarity of the scaled up OFR is assured by keeping two geometric ratios constant which are:

- Baffle spacing, L.
- Baffle orifice open area, S.

The baffle spacing is usually expressed as a fixed ratio of the tube diameter and baffle orifice open area is the square of orifice diameter, d_o divided by the square of tube internal diameter, D.

$$L = 1.5D \qquad \text{(Equation 1)}$$

$$S = \frac{d_o^2}{D^2} \qquad \text{(Equation 2)}$$

In typical operation of an OFR, S is in the range of 0.2-0.4, usually 0.25, means the orifice diameter is half the tube diameter.

Key dimensionless numbers should be kept in a similar range on the full scale, as on the lab or pilot scale. For oscillatory flows, the key dimensionless numbers are net flow Reynolds number (Equation 3) and oscillatory Reynolds number (Equation 4):

$$\mathrm{Re}_n = \frac{\rho u D}{\mu} \qquad \text{(Equation 3)}$$

$$\mathrm{Re}_o = \frac{x_o \omega D \rho}{\mu} \qquad \text{(Equation 4)}$$

For a sinusoidal oscillation, x_o is the center-to-peak amplitude, ω, the frequency of oscillation, D, the tube diameter, ρ, the density, u, the superficial velocity and μ, the viscosity. Both dimensionless numbers can be related in defining the velocity ratio, ψ (Equation 5).

$$\psi = \frac{\mathrm{Re}_o}{\mathrm{Re}_n} = \frac{x_o \omega}{u} \qquad \text{(Equation 5)}$$

To produce fully reversing flows, Re_o must be larger than Re_n which mean the velocity ratio will always greater than 1. This is to ensure that the superimposed oscillation will dictate the mixing regime. But, it is important to note that the minimum values for Re_o and Re_n are 100 and 50 respectively.

2.2.2 Residence Time Distribution (RTD).

An investigation of the residence time distribution (RTD) behaviour of the laboratory scale oscillatory flow reactor has been performed by University of Cambridge to investigate and quantify the relative contributions of the oscillatory flow and bulk flow components in the baffled tube reactor. The effect of the bulk flow conditions on the RTD response is shown in Figure 6 below, which plots N against the velocity ratio. Each curve on the graph represents the complete set of data obtained by varying the amplitude and frequency for a particular value of net flow Reynolds number. It can be seen from the graph that for each flow rate considered there is a clear range for the velocity ratio for which N is largest, and thus where the system is operating closest to the ideal value of N (80). Also, the higher the net flow rate, the higher is the magnitude of N

(i.e. the closer to the ideal *N*) in the optimum region. This is probably due to the fact that for the higher flow rate, the residence time is correspondingly shorter, which means there is less chance for axial dispersion to occur, and this translates to a higher overall value for *N*. Based on the study, the velocity ratio is important in determining the RTD performance in an OFR (Stonestreet and Harvey, 2002).

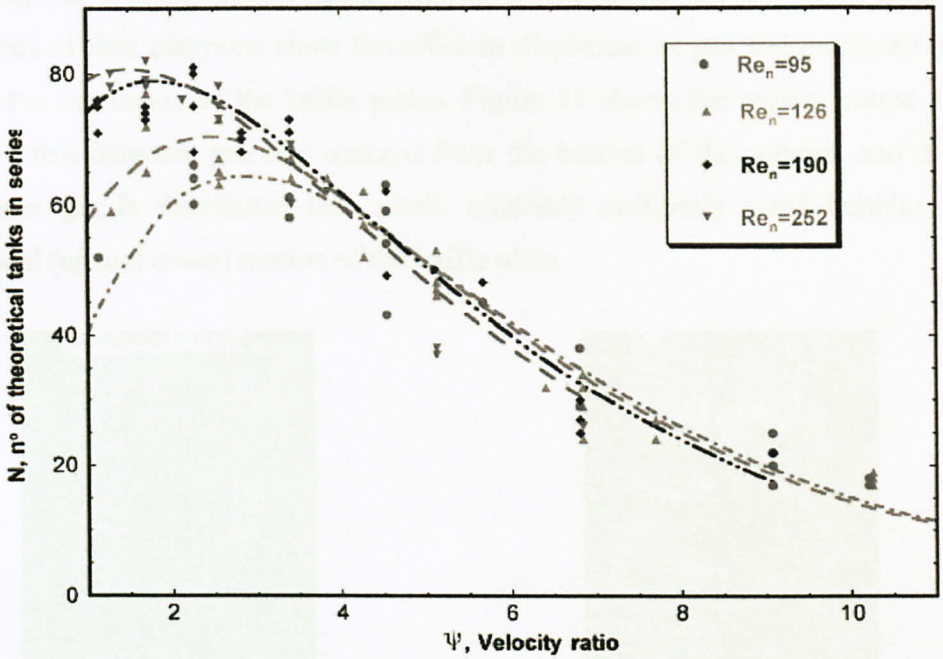

Figure 6: RTD behavior for OFR.

The data points show the experimentally measured RTD response according to the standard tank-in-series model. It can be seen that the number of tank in series is maximized around velocity ratio, ψ equals to 2.5 for each values of Re_n. The study recommended that the value of velocity ratio, ψ is in the range of 2 to 6 in order to ensure a close approach to plug flow behavior (Stonestreet and Harvey, 2002). For an OFR, the number of tanks in series can be considered as the number of baffle cavities in the reactor tube by assuming the RTD is that of perfect plug flow.

2.2.3 Enhancement of Mass Transfer.

The effect of flow oscillations on mass transfer enhancement has been well characterized in recent years, both for reciprocating plate columns (Baird et al, 1996) as well as for oscillatory flow in baffled tubes. The effect of oscillatory flow on gas dispersion in a reciprocating plate column is shown in the diagrams below. The sequences of four diagrams show the efficient dispersion of gas and increased hold-up due to the oscillation of the baffle plates. Figure 11 shows the region around a baffle plate. In this diagram, gas was sparged from the bottom of the column, and it can be seen how gas is distributed into small, relatively uniformly sized bubbles by the sinusoidal (up and down) motion of the baffle plate.

Figure 7: Gas sparged from base

Figure 8: Start of plate oscillation

Figure 9: After one complete oscillation

Figure 10: After many oscillations

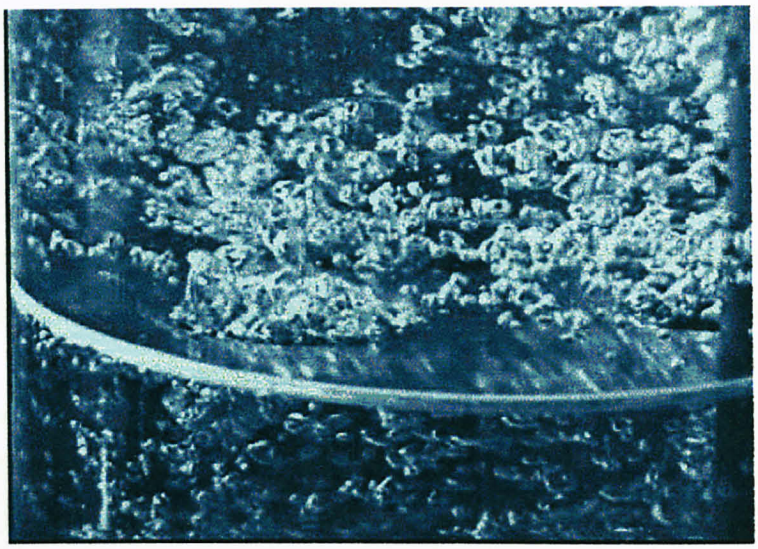

Figure 11: Flow visualization of bubble dispersion in an oscillatory flow.

In a recent study, the effect of oscillatory flow in baffled tubes for the oxygen/water system has been investigated. The apparatus consisted of a vertically mounted 26 mm inner diameter perspex tube of length 0.6m. Fluid oscillations were provided by a rubber diaphragm and electromagnetic oscillator. Air was sparged from the base of the tube, and the oxygen-water mass transfer coefficient, k_La, was determined for different oscillatory conditions and three different types of baffles: helical, central (disk-type), and wall mounted orifice-type. Photographic visualization of fluid mechanics and gas bubble behavior was also performed, which revealed that the oscillatory baffled flow was able to modify the bubble trajectories, which seen to move around the inside the volumes of the baffle cavities. At very intense levels of oscillation, the bubbles could be seen trapped within each cavity for at least several seconds (Hewgill, Mackley, M.R., Pandit, A.B. and Pannu, S.S. et al, 1993).

In a system without baffles, mass transfer with oscillatory flow was similar to a bubble column. The application of fluid oscillations in the absence of internal baffles had little effect on the measured k_La values. However, it was shown that if orifice-type baffles (spaced at 1.5 tube diameters) and fluid oscillations were present, up to six-fold increase in k_La relative to a bubble column was obtained. Experimental k_La values at

various superficial gas flowrates, coupled with power density calculations using a quasi-steady model were used to fit a correlation for k_La, which is given as follows:

$$k_La = 0.00495\ (e_v)^{0.3593}\ U_g^{0.4}$$

where e_v is power density (w.m^{-3}), and U_g is the superficial gas velocity (mm/s). This correlation can be compared to Lineks correlation for stirred tanks (Linek et. al., 1987).

If these two correlations are compared graphically, as depicted in the graph below, it can be seen that the oscillatory flow system is more energy efficient than gas sparged stirred tanks. For a given power density, the k_La value obtained for oscillatory flow is of order twice that of the stirred tank value, for all values of power density over 200 w.m^{-3}.

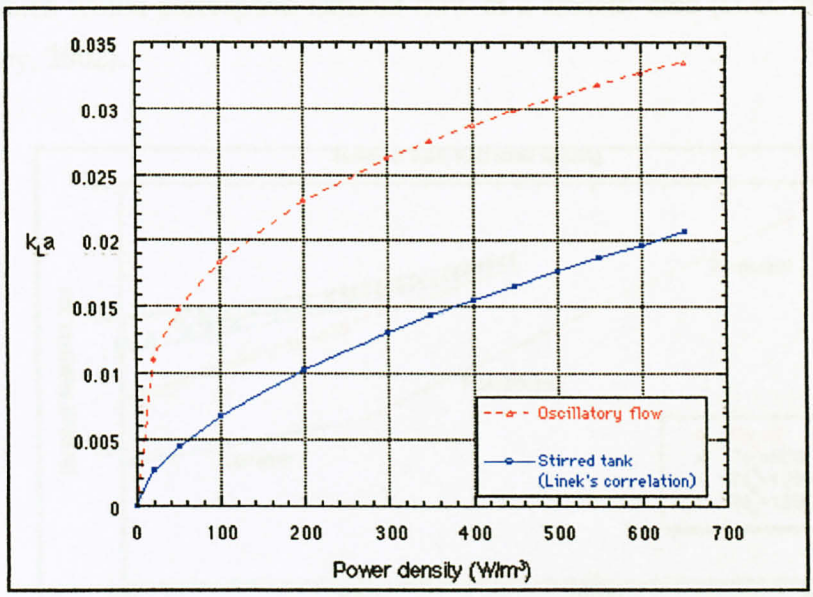

Figure 12: Graph of k_La versus power density for oscillatory flow and Lineks correlation.

The use of oscillatory flow for mass transfer enhancement, coupled with the fine control over mixing intensity suggests that an oscillatory flow mass transfer device would have potential for certain shear sensitive applications where a stirred tank would

be inappropriate, such as fermentation reactions. A preliminary study (Harrison and Mackley, 1991) has demonstrated the feasibility of an oscillatory flow bio-reactor. The capability of oscillatory flow to effectively mix two phases also suggests its use in gas-liquid reactions where diffusion is performance limiting, and scale-up difficulties of stagnant zones would be minimised, which can occur in stirred tank devices.

2.2.4 Enhancement of Heat Transfer Rates.

Heat transfer has been investigated in a two pass reactor configuration, using a light mineral oil pumped through the tube side and water in the shell as the cooling fluid at a high enough flowrate to ensure that the limit to heat transfer was on the tube side. The heat transfer coefficient (Nusselt number) has been investigated as a function of the oscillatory flow component superimposed on a bulk flow in a low Re region, i.e. Re<1000, which would correspond laminar flow in a smooth tube (G.G. Stephens and M.R. Mackley, 2002).

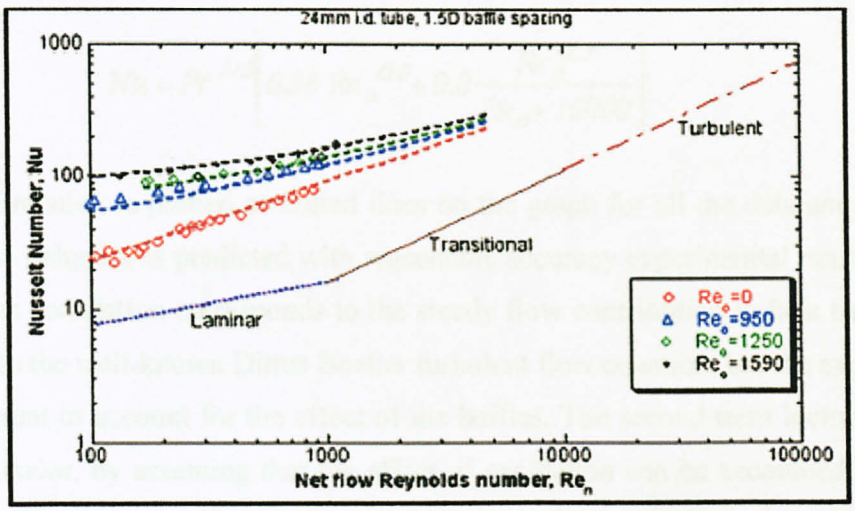

Figure 13: Heat transfer enhancement in OFR

The graph above shows the results, where the Nusselt Number (Nu) is plotted against the bulk flow Reynolds number. The oscillatory conditions 0, 6, 8, and 10Hz frequencies at a fixed amplitude of 5mm are shown, corresponding to an oscillatory Reynolds number = 0 (steady flow), 950, 1250, and 1590 respectively. Also shown are

standard heat transfer correlations for laminar and turbulent flow assuming smooth tube with steady flow. This gives an overview of the heat transfer regimes corresponding to different types of flow behavior.

It can be seen that for a given throughput (i.e. net flow Reynolds number), the heat transfer increases with increasing oscillatory Reynolds numbers. At high throughput, the oscillatory curves approach the steady flow curve, as the net flow component becomes larger relative to the oscillatory flow component. However, at low values of net flow, high rates of heat transfer are still obtainable provided the oscillatory flow component is present. For example, for an oscillatory Reynolds number = 1590, it can be seen that the Nusselt number is of order 120 where the net flow Reynolds number=200. This corresponds to heat transfer which may be obtained from turbulent flow in a smooth tube where Re=10 000.

In order to provide a design correlation for oscillatory flow heat transfer, we have modeled the heat transfer data phenomenological, as follows:

$$Nu = Pr^{1/3}\left[0.36\,Re_n^{0.6} + 0.8\frac{Re_o^{1.7}}{Re_n + 10000}\right]$$

The correlation is plotted as dotted lines on the graph for all the data and it can be seen that the behavior is predicted with reasonable accuracy experimental range. The first term of the correlation corresponds to the steady flow contribution to heat transfer, and is similar to the well-known Dittus Boelter turbulent flow equation, but the exponent of Re_n is different to account for the effect of the baffles. The second term includes the oscillatory behavior, by assuming that the effect of oscillation can be accounted for by adding the oscillatory term to the steady term. In the case for steady flow, the correlation reduces to the baffles only curve plotted on the graph. When the net flow Reynolds number is much greater than the oscillatory flow Reynolds number, the oscillatory term is small in relation to the steady term, and the curve collapses onto the steady flow behavior again. In contrast, when the throughput is zero, batch heat transfer will be

predicted by the non-zero second term. This correlation can be used to calculate Nu in the reactor tube a priori, given the net flow and oscillatory conditions.

It is clear from these results that Oscillatory Flow Reactor has a significant enhancement effect on tube-side heat transfer, particularly in the low Reynolds number throughput regime, which would correspond to long residence times in the reactor. Furthermore, the heat transfer enhancement can be controlled by the oscillatory mixing independently of any net flow, and in this respect, oscillatory flow reactor offers a strategic advantage over static mixer reactors which would not offer the same degree of thermal control.

CHAPTER 3

METHODOLOGY

3.1 Project Methodology.

Step 1: Research and problem identification.

- The research will be done on reactor design and hydraulic oil production by referring to the articles, journals, books, and etc.

Step 2: Selection on reactant and catalyst of hydraulic oil production.

- The selection is based on the research that has been done.

Step 3: Conceptual design of OFR for the process.

- This will include various parameters such as number of oscillation, length, tube diameter, choice of baffle design and distance between the baffles.
- This step may also involve simulation software such as polymath.

Step 4: Material selection and economic optimization.

- Before constructing the reactor model, the selection of material need to be done in order to minimize the project cost.

Step 5: Construction of OFR model.

- The reactor model will be constructed in a small scale.

Figure 14: Project methodology.

3.2 Hydraulic Oil Process Methodology.

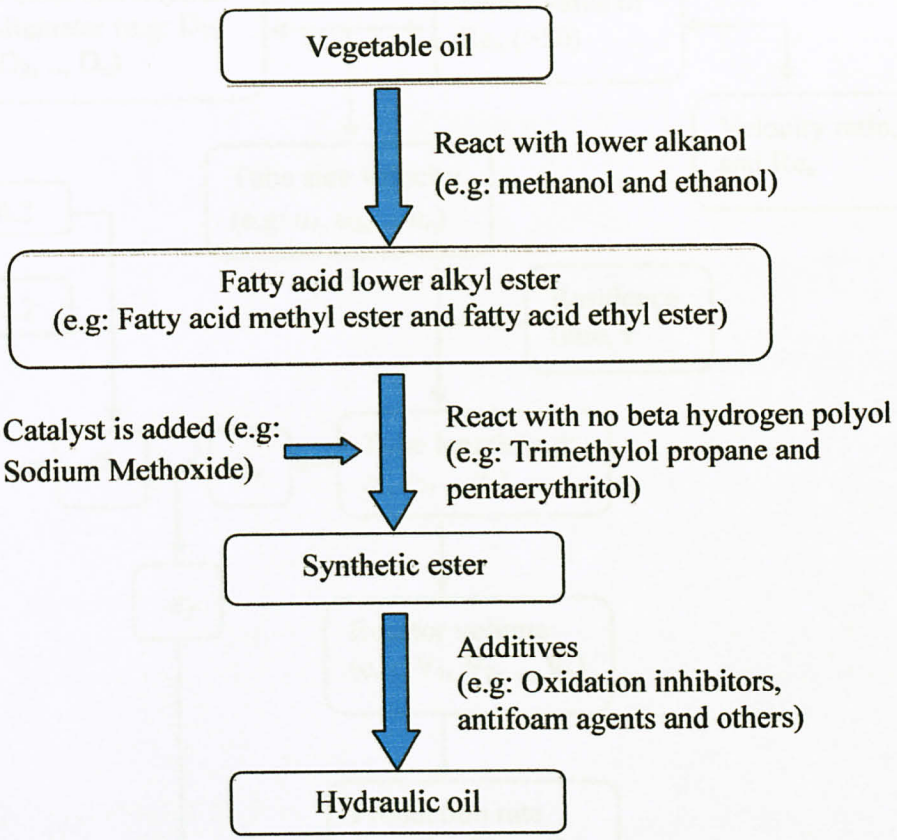

Figure 15: Hydraulic oil process.

3.3 OFR Design Methodology.

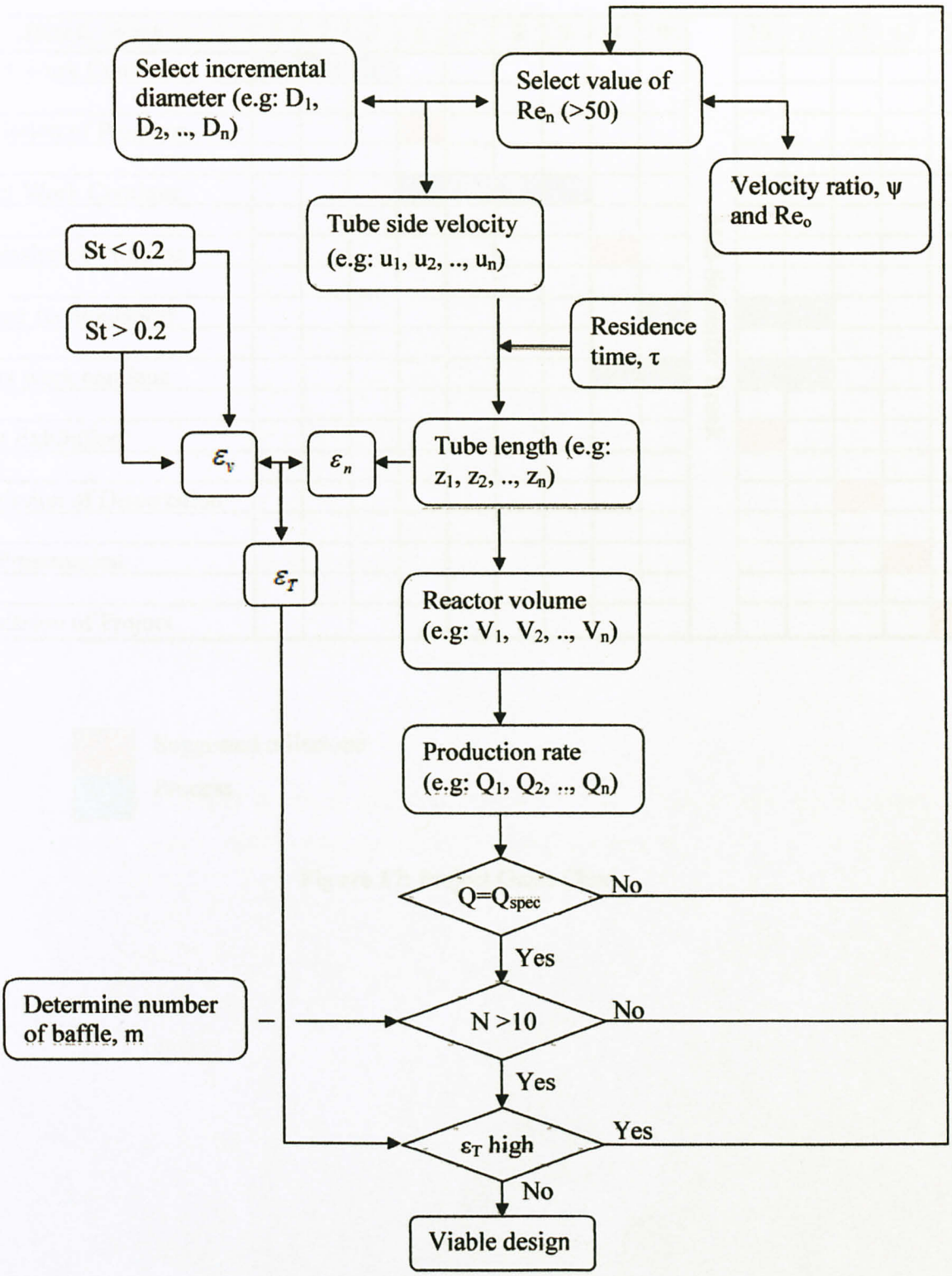

Figure 16: OFR design methodology.

3.4 Project Process Flow (Gantt Chart)

Detail/ Week	1	2	3	4	5	6	7	8	9		10	11	12	13	14
Project Work Continue															
Submission of Progress															
Project Work Continue															
Submission of Progress															
Seminar (compulsory)															
Project work continue															
Poster Exhibition															
Submission of Dissertation															
Oral Presentation															
Submission of Project															

Mid-Semester Break

Suggested milestone

Process

Figure 17: Project Gantt Chart.

CHAPTER 4

RESULT AND DISCUSSION

4.1 Design Calculation.

The design calculation has been performed using spreadsheet. A few set of calculation has been done and below is the selected set of calculation that gives the minimum percentage error of amount of product produced. For the design methodology, the initial reactor conditions need to be determined. Since the project is a lab scale reactor, the target production rate, Q_{Target} is 2 liters per hour. The residence time, τ for the reaction is 1 hour. The fluid viscosity, μ and density, ρ inside the reactor are 0.00396 kg/m.s and 881 kg/m^3 respectively. To ensure a close approach to plug flow, the velocity ratio, ψ is maintained at the value of 4. The net flow Reynold Number, Re_n is selected as 3. This should give a value of oscillatory flow Reynold Number, Re_o of 12 since $Re_o = Re_n \times \psi$.

The initial tube diameter, D is selected as 48 mm. To determine the required length of the reactor, the value of superficial velocity, u is needed.

$$u = \frac{\mu.\mathrm{Re}_n}{D.\rho}$$

$$= \frac{(0.00396kg/m.s)(3)}{(0.048m)(881kg/m^3)}$$

$$= 0.0003m/s$$

Therefore, the required reactor length, z

$$z = u.\tau$$

$$= (0.0003m/s)(3600s)$$

$$= 1m$$

Then, the length to diameter ratio will be 21.1. Based on the reactor length calculated and the tube diameter, the volume of the reactor, V can be determined.

$$V = z.\frac{\pi D^2}{4}$$

$$= (1m)\frac{\pi(0.048m)^2}{4}$$

$$= 0.0018m^3$$

Next, calculate the actual production rate, Q_{Cal} by the reactor and compare with the target production rate, Q_{Target}.

$$Q_{Cal} = \frac{V}{\tau}$$

$$= \frac{0.0018m^3}{1hr}$$

$$= 0.0018m^3/hr$$

Percentage error,

$$Error = \left|\frac{Q_{Target} - Q_{Cal}}{Q_{Target}}\right| \times 100$$

$$= \left|\frac{0.002m^3/hr - 0.0018m^3/hr}{0.002m^3hr}\right| \times 100$$

$$= 8.5\%$$

After completing the basic design calculation, the amount of power required to oscillate the baffles inside the reactor need to be calculated. This is important to ensure the required power can be maintained as low as possible to reduce the operating cost of the reactor. The power required to oscillate the baffles is the sum of power density, ε_n as well as the average power per unit volume, ε_v. In order to calculate the power density, the total number of baffles, m_t, baffle spacing, L, baffle orifice diameter, d_o, baffle

orifice open area, S, pressure drop in the tube, ΔP, and pressure enhancement ratio, ζ need to be identified.

Baffle spacing, L,

$$L = 1.5D$$
$$= 1.5(48mm)$$
$$= 72mm$$

Total number of baffles, m_t,

$$m_t = \frac{z}{L}$$
$$= \frac{1000mm}{72mm}$$
$$= 14baffles$$

Orifice diameter, d_o,

$$d_o = \frac{D}{2}$$
$$= \frac{48mm}{2}$$
$$= 24mm$$

Baffle orifice open area, S,

$$S = \frac{d_o^2}{D^2}$$
$$= \frac{(24mm)^2}{(48mm)^2}$$
$$= 0.25$$

Pressure drop in the tube, ΔP,

$$\Delta P = m_t \frac{\rho u^2}{2C_o}\left(\frac{1}{S^2} - 1\right)$$

C_o is a Standard Orifice Coefficient which is usually taken as 0.6.

$$= (14)\frac{(881kg/m^3)(0.0003m/s)^2}{2(0.6)}\left(\frac{1}{S^2}-1\right)$$

$$= 0.01kg/m.s^2$$

Pressure enhancement ratio, ζ,

$$\zeta = \left[1+\left(\frac{4\psi}{\pi}\right)^3\right]^{1/3}$$

$$= \left[1+\left(\frac{4(4)}{\pi}\right)^3\right]^{1/3}$$

$$= 5.11$$

Power density, ε_n,

$$\varepsilon_n = \Delta P\left(\frac{u}{z}\right)(\zeta)$$
$$= (0.01kg/m.s^2)\left(\frac{0.0003m/s}{1m}\right)(5.11)$$

$$= 0.000017\ W/m^3$$

To calculate the average power per unit volume, ε_v, the amplitude of oscillation, x_o, and frequency of oscillation, f need to be selected which is 0.01m and 1 Hz respectively. The empirical derived eddy mixing length, l, is usually taken as 0.009. Large eddies diminishing while smaller eddies developing near baffle and wall area with increasing gap sizes.

Average power per unit volume, ε_v,

$$\varepsilon_v = \frac{3m_t\rho(w)^3 x_o^2 l}{Sz}$$

$$= \frac{2(14)(881kg/m^3)[(2)(\pi)(1Hz)]^3(0.01m)^2(0.009)}{(0.25)(1m)}$$

$$= 32.79 W / m^3$$

Therefore, the power required for the oscillation, ε_T,

$$\varepsilon_T = \varepsilon_n + \varepsilon_v$$
$$= 0.000017 W / m^3 + 32.79 W / m^3$$
$$= 32.790017 W / m^3$$

For this set of calculation, the calculated power required is not too high so that the design is viable. The final configurations of the reactor are summarized in the table 2.

Tube diameter (mm)	48
Total tube length (m)	1
Number of tubes	1
Total volume (m^3)	0.0018
Volumetric throughput (m^3/hr)	0.0018
Total number of baffles	14
Baffles spacing (mm)	72
Space between baffles and column wall (mm)	0.5
Baffles diameter (mm)	47
Orifice diameter (mm)	24
Baffles orifice open area	0.25
Net flow Reynolds number	3
Oscillatory flow Reynolds number	12
Frequency (Hz)	1
Amplitude (mm)	10
Power required (W/m^3)	32.79

Table 2: Final configurations of OFR.

4.2 Heat Transfer Design.

In order to maintain the reaction temperature at an optimum temperature, the vertical section of the reactor is jacketed by hot oil. The hot oil system consists of heater, hot oil reservoir (storage vessel), and pumps. The hot oil from the reservoir is heated to a

certain temperature and pumped into the jacket. There will be a control valves at the inlet of the jacket connected to temperature indicator at both inlet and outlet of the OFR. The flow rate of hot oil used will be depending to the temperature at both inlet and outlet of the OFR. The hot oil that passes the jacket will be collected back in the hot oil reservoir before being circulated again in the system.

4.3 Reactor Tube and Jacket Pressure Design.

The design pressure for both reactor tube and jacket (P_D) are based on the maximum working pressure (MWP) with a 5% of safety factor. Since the reaction is totally in a liquid phase, the hydrostatic pressure exerted by the liquid for a certain liquid level in the reactor tube will determine the design pressure. Therefore, the design pressure calculation used for the project will consider the hydrostatic pressure in both reactor tube and jacket.

4.3.1 Maximum Working Pressure (MWP) in Reactor Tube.

Since the OFR should be fully filled, we can use 100% liquid level in the tube for the calculation.

$$h = 1 \times 1m = 1m$$

$$\rho_{Liquid} = 881kg/m^3 \ , \ g = 9.81m/s^2$$

Hydrostatic pressure exerted by the liquid,

$$P_{Hydrostatic} = \rho gh$$

$$= (881kg/m^3) \times (9.81m/s^2) \times (1m)$$

$$= 8.64kPa$$

Consider 5% noise in hydrostatic fluid due to agitation in the liquid,

$$P_{Hydrostatic'} = 8.64kPa + \left(\frac{5}{100} \times 8.64kPa\right)$$

$$= 9.07kPa$$

Tube operating pressure, $P_{Tube} = 20mbar = 2.03kPa$

Consider 5% noise in supernatant

$$P_{Tube'} = 2.03kPa + \left(\frac{5}{100} \times 2.03kPa\right)$$

$$= 2.13kPa$$

Consider 5% overpressure for safety relief valve,

$$P_{Tube''} = 2.13kPa + \left(\frac{5}{100} \times 2.13kPa\right)$$

$$= 2.24kPa$$

$$MWP_{Tube} = P_{Tube''} + P_{Hydrostatic'} = 11.31kPa$$

4.3.2 Maximum Working Pressure (MWP) in Hot Oil Jacket.

Jacket operating pressure, $P_{Jacket} = 101.33kPa$

Consider 5% noise in supernatant fluid,

$$P_{Jacket'} = 101.33kPa + \left(\frac{5}{100} \times 101.33kPa\right)$$

$$= 106.40kPa$$

Assume the hot oil jacket is fully filled,

$$h = 0.84m$$

Assume $\rho_{Oil} = 800kg/m^3$

Hydrostatic pressure exerted by hot oil,

$$P_{Hydrostatic} = \rho gh$$

$$= \left(800kg/m^3\right) \times \left(9.81m/s^2\right) \times \left(0.84m\right)$$

$$= 6.59kPa$$

$$MWP_{Jacket} = P_{Jacket'} + P_{Hydrostatic}$$

$$= 112.99kPa$$

4.3.3 Pressure Design for Reactor Tube.

$$P_{Design(tube)} = 1.05(MWP_{Jacket} - MWP_{Tube})$$

$$= 1.05(112.99kPa - 11.31kPa)$$

$$= 106.76kPa$$

4.3.4 Pressure Design for Hot Oil Jacket.

$$P_{Design(Jacket)} = 1.05(MWP_{Jacket} - P_{atm})$$

$$= 1.05(112.99kPa - 101.33kPa)$$

$$= 12.24kPa$$

4.4 Reactor Tube and Jacket Temperature Design.

The maximum operating temperature for the process is based on 20% safety factor. To determine the design temperature of the reactor tube and hot oil jacket, 10°C of additional temperature is added up to the maximum operating temperature for the process. This is important in order to ensure the safety of the equipment.

$$T_{Design} = T_{\max} + 10^{o}C$$

Table below summarized the design temperature for both reactor tube and hot oil jacket:

Reactor Tube	Hot Oil Jacket
$T_{Operating} = 130^{o}C$	$T_{Operating} = 150^{o}C$
$T_{Max} = 156^{o}C$	$T_{Max} = 180^{o}C$
$T_{Design} = 166^{o}C$	$T_{Design} = 190^{o}C$

Table 3: Reactor tube and hot oil jacket design temperature.

4.5 Material Selection.

There are many factors that need to be considered in selecting the material for OFR construction. For the project, the material must have the ability to resist a high temperature and corrosion.

Due to the presence of liquid phase in the reaction, the selection of stainless steel is highly favored over carbon steel. One of the major reasons is that carbon steel is not suitable for contacting liquid with the surface inside the reactor, due to its corrosiveness, and deemed undesirable for long term usage.

From the study, there are several grades of stainless steel that have the potential to be selected which are SS304, SS321, SS316, SS316L, and SS310. The table below summarized the description of each grade of stainless steel.

Grade	Description
SS304	• Good corrosion resistance in a wide range of atmospheric environments and many corrosive media. • Good oxidation resistance in intermittent service to 870°C and in continuous service to 925°C.
SS321	• Sustain a very high temperature up to 900°C. • Corrosion resistance is equivalent to Grade 304 but superior if the application involves service in the 425-900°C range.
SS316	• Excellent corrosion resistance in a wide range of atmospheric environments and many corrosive media (generally better than SS304). • Good oxidation resistance in intermittent service to 870°C and in continuous service to 925°C.

SS316L	• Immune from sensitization compared to SS316L (grain boundary carbide precipitation).
SS310	• Sustain a very high temperature up to 1150°C.

Table 4: Description of possible construction materials.

Hence, stainless steel of grade 316 is selected as the material for OFR construction.

4.6 Process Flow and OFR Drawing.

4.6.1 Process Flow.

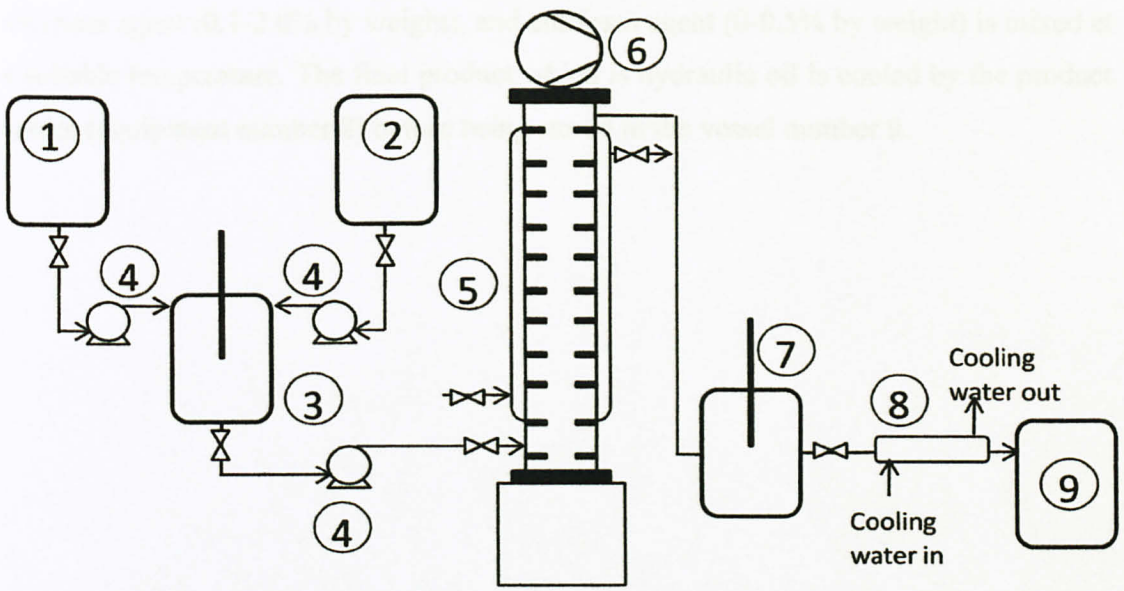

Figure 18: Process flow of synthesis the hydraulic oil using OFR.

A rapseed oil methyl ester (RME) of 0.65mol % is stored in vessel number 1 at a temperature of 60°C. Trimethylol Propane (TMP) of 0.19 mol % is stored in vessel number 2 also at a temperature of 60°C. Both reactants will pumped into vessel number 3 which is a pre-mix vessel to ensure a proper mixing before entering the OFR. The flow of both reactants is controlled by a ratio control since the mixture needs to be maintained

at a ratio of 3.8 : 1(RME : TMP). As the reactants flow into the OFR, a catalyst (sodium methoxide of 0.9 w/w % of the reactants) is added into the reactor. Then, the pressure inside the OFR is reduced to 20 mbar since this is the optimum operating pressure for the reaction. This job is done by using a vacuum pressure labeled by the number 6. The reactants will pumped into OFR by pump number 4.

Inside the OFR, the reaction temperature is raised and maintained at 130°C by the hot oil circulation equipped in the OFR system. The reason of maintaining this operating temperature is because the conversion of triesterss (TE) increase slightly until the temperature of 130°C, but at a temperature higher than 130°C, RME tends to vaporized and easier to be pulled away via vacuum pressure. After the reaction is completed, the product will flow into vessel number 7 where the additives such as oxidation inhibitor (0.1-2.5% by weight), pour point depresser (0-5.0% by weight), antiwear agent (0.1-2.0% by weight), and antifoam agent (0-0.5% by weight) is mixed at a suitable temperature. The final product which is hydraulic oil is cooled by the product cooler (equipment number 8) before being stored in the vessel number 9.

4.6.2 Basic Drawing of OFR.

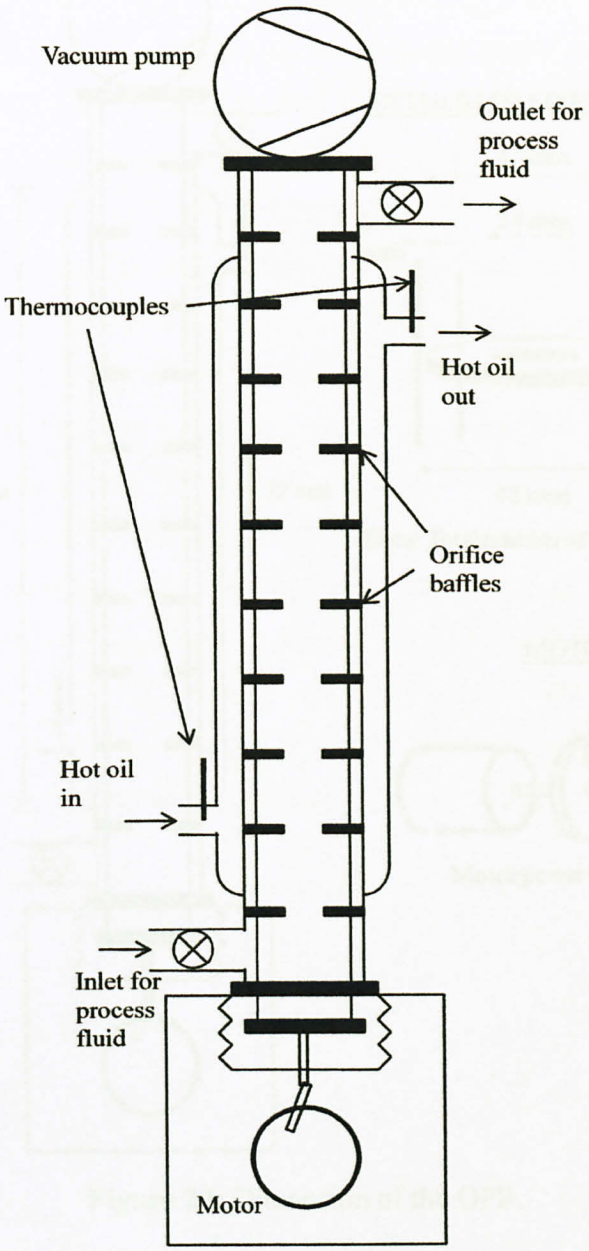

Figure 19: Basic drawing of OFR.

4.6.3 Dimension of OFR.

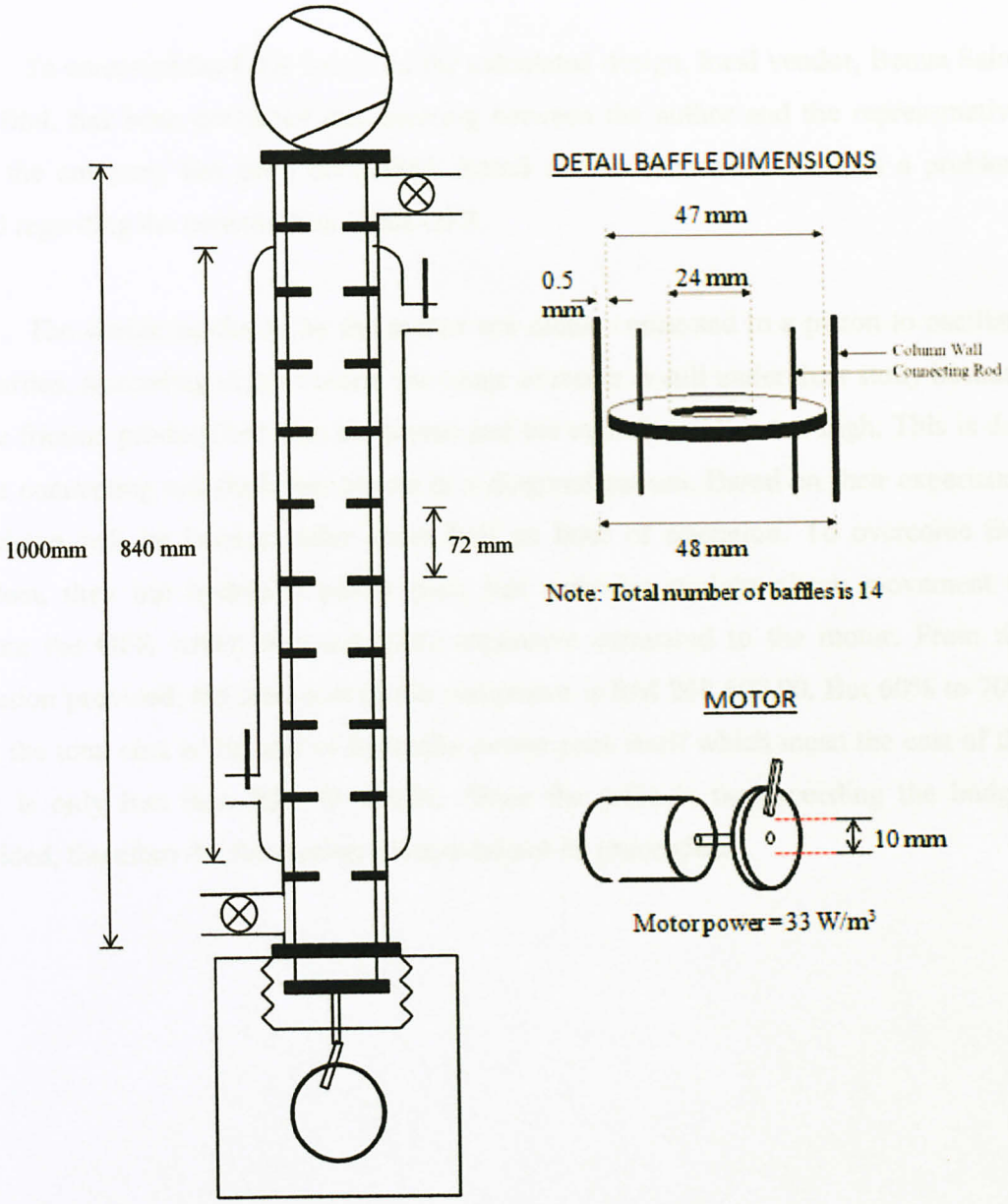

Figure 20: Dimension of the OFR.

4.7 Fabrication of OFR.

To construct the OFR based on the calculated design, local vendor, Benua Sains Sdn. Bhd. has been contacted and meeting between the author and the representative from the company has been conducted. Based on the discussion, there is a problem raised regarding the construction of the OFR.

The design produced by the author use motor connected to a piston to oscillate the baffles. According to the vendor, the usage of motor is still under their study because of the friction produce between the piston and the cylinder wall is too high. This is due to the connecting rod shaft that moves in a diagonal pattern. Based on their experience the piston will be jammed after about half an hour of operation. To overcome this problem, they use hydraulic power pack that provides straight piston movement to operate the OFR which is much more expensive compared to the motor. From the quotation provided, the total cost of the equipment is RM 268 600.00. But 60% to 70% from the total cost is the cost of hydraulic power pack itself which mean the cost of the OFR is only less than RM 90 000.00. Since the price is far exceeding the budget provided, therefore the fabrication process cannot be proceeded.

CHAPTER 5

CONCLUSION

The OFR can be designed based on the method that has been developed. Study has shown that OFR is suitable for a long residence time reaction such as the synthesis of hydraulic oil. This is due to the uniform and efficient vortex mixing as a result of oscillation of the orifice plate baffles inside the tube. Even though the OFR is not fabricated, but it proves that the cost of the OFR is not as high as other reactor used for long residence time reaction.

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PROPOSAL FOR FABRICATION & PROCESS CONTROL OF ONE (1) UNIT OSCILLATORY FLOW REACTOR AT UNIVERSITI TEKNOLOGI PETRONAS (UTP)

By

BENUA SAINS SDN. BHD

August 2009

1. OBJECTIVES AND SCOPE

1.1 OBJECTIVES

The main objective of this project is to construct a oscillatory flow reactor system which serves the following primary purposes:

- Process: Reactions assisted by oscillatory flow mixing

In this proposal, we will carry out the following service, henceforth called "Engineering and Implementation", to be materialised in engineering, procurement and fabrication and system start-up, henceforth called the "Deliverable", for the following systems of the UTP, pulsed baffled reactor project:

Service Provided:

- Design & Engineering
- Fabrication
- Process control integration
- System Start-up

System Related:

- Oscillatory Flow Reactor

Project Service							
Design & Procurement		Shop Execution				Validation	
Design &Engineering	Procurement	Fabrication	System integration			Commissioning	Start-up

Scope of the present proposal

2.2 ENGINEERING SERVICE

The aim of the proposal is to present our overall approach for the conduct of such a project and for the detailed engineering of your oscillatory flow reactor system.

We plans to achieve this goal for the scope of services listed previously, an overall approach and a step by step realization in different phases.

- Engineering - design of the oscillatory flow reactor to user requirement specification
- Procurement - specification of oscillatory flow reactor design & components
- Fabrication of the oscillatory flow reactor
- System integration with controls & instrumentation

For engineering, We will produce a Deliverable that will contain the amount of detail necessary to enable BENUA SAINS SDN BHD to undertake a ***lump sum price basis*** for the execution of the related works. The deliverable will be limited to the set of documents specified in article 3.1.

The whole engineering service will be solely based on the items listed in article 3.1, which are delivered by us. Alternatives to the requirements laid down in the documents and drawings as specified under article 3.1 are consider as void order and shall be charge according to work being carried out.

We will assume that the said process descriptions adequately reflect UTP wishes.

2.3 PROJECT SCHEDULE

16-22 weeks after received of official PO.

3. TECHNICAL TERMS AND SPECIFICATIONS

3.1 PROCESS SPECIFICATIONS

The process specifications to be taken into account when designing the installations specified under article 2.1 are based on discussion between Mr.Mahendran & Mr Adip of UTP. It is understood that we considers the information obtained as the necessary basis to elaborate the Detailed Design for the installation as UTP requires.

4. COMMERCIAL AND FINANCIAL CONDITIONS

4.1 PRICE

Description	Subtotal	Currency
Engineering, procurement, installation, control integration, test & commissioning for Oscillatory Flow Reactor at UTP Warranty: 18 months		
TOTAL: RINGGIT MALAYSIA TWO HUNDRED SIXTY EIGHT THOUSAND SIX HUNDRED ONLY	268,600.00	RM

Delivery lead time: 16-22 weeks upon receipt of official PO and execution of commercial terms.

The above price is exclusive of :

- **Vacuum pumps, glassware for distillations etc**
- **Deviation Design. Procurement & Installation Fee will be invoiced to the buyer for any deviation designs.**
- **Main electrical supply to the control panel**
- **Product Runs**
- **Any foundation work, building jobs, plasterwork, mortaring and woodwork.**
- **Acid protection of foundations.**
- **Any sandblasting, primer or top coats of paint on site upon completion of assembly.**
- **Sound-proofing measures.**
- **All chemicals and consumables required for commissioning and trial run.**
- **Spare Parts**

- **Continuous Oscillatory Flow Reactor**

A) Continuous oscillatory flow reactor system

a) Reactor Vessel Body

- SS316 top plate
- Stainless steel body, Mo: 2-3% 300 series
- SS316 baffle plates
- Capacity: 2 L
- 1 nos of sampling point at lower reactor body
- Sampling ball valve, SS316 forged body
- Quick clamps for easy maintenance
- TIG welded with 99.999% purge
- Continuous operation temperature rating: 150 deg C max
- Band heater on reactor body for temperature control, maximum temperature 150 deg C.
- Necessary piping and valves
- 2 nos starting material (e.g palm oil) inlet metering piston pump, operated via timer control
- Stainless steel SS304 clamps
- All weld seams pickled and passivated
- Inner reactor baffle plates precision laser cut
- Baffle material: SS316L, 2B finishing

Qty: 1 unit

b) Reactor mounting

- Mounted on a skid, mild steel
- Provision for space for sample withdrawal

Qty: 1 unit

c) Pulsation unit

- Servo hydraulic driven piston pulsing, directly coupled to the reactor body bottom
- Variable frequency: 1-8Hz, maximum frequency is stroke length dependant, the higher the frequency, the lower is the maximum peak height
- Variable pulse height: 40mm peak to peak height (frequency dependant, at high peak height, the maximum allowable frequency is lower)
- Straight piston movement for pulse generation
- c/w stroke length controlled by servo-hydraulic
- Hydraulic pulsing is controlled via a PC with RS232 interface
- Total isolation of wetted area from piston housing
- Pulsation piston sealed with positive pressure activated seals
- Self lubricated piston seals
- Necessary sensors for closed loop movement control
- Piston made of SS316
- Piston block made of SS316, precision machined to +/- 10 micron accuracy

- Pulsation controller and interfacing card
- Industrial 24 V Programmable logic control
- Industrial control and manufacturing test applications
- Watchdogs, programmable power-up states, change detection, input filters, high current drive

Qty: 1 unit

d) Temperature sensor
- Temperature range: ambient +10 deg C to 150 deg C max
- 300 series, Mo:2-3% thermowell

e) Valves and piping
- Valve seat: PTFE or ME compatible
- SS316 body
- Manual operated
- Connection: BSP or NPT
- Tubing connection: SS316 twin ferrule fittings
    - Interconnecting piping: PTFE
        - Fittings: 1 lot of compression fittings
            - Metallurgy: Stainless steel SS316
            - Gaugeable
            - Pressure rating: 7800 psig to ASME B31.3
            - Ultimate tensile strength: 75 000 psig to ASME B31.3
            - NPT connection to ASME B1.20.1

f) Electrical control panel consisting of:
- Switch cabinet
- Material casing sheet steel, powder coated
- Type of enclosure IP 54
- Base plate for mounting the equipment, incl. wiring in plastic ducts
- Labelling of equipment – single and permanent
- Panel light for each control panel
- Socket outlet
- Floating contact for general alarm
- General alarm as signal lamp and electronic buzzer with acknowledgement
- Lockable mains switch with emergency off
- Control voltage supply :24 V DC / 230V AC
  Analog signals: 4 - 20mA
- Control/automation system with PC process control
- Input/output modules Analog and digital
- Electrical cabling within the plant frame work of motors, sensors and actuators in plastic ducts

g) Integration of pumps for raw materials
Material compatibility: methyl ester

i) Piping installation
   a. Either Teflon or fully annealed SS316L ASTM A269 tubing

b. Outer surface: 0.8 micron Ra max

h) Process to be mounted onto a coated mild steel skid

i) Feed tank

- 2 nos PP container, 10 liter
  - Non presurised
  - Capacity: 10 liters
  - Necessary connections
  - Non-stirred tank
  - Interconnecting piping: PTFE
    - Fittings: 1 lot of compression fittings
      - Metallurgy: Stainless steel SS316
      - Gaugeable
      - Ultimate tensile strength: 75 000 psig to ASME B31.3
      - NPT connection to ASME B1.20.1

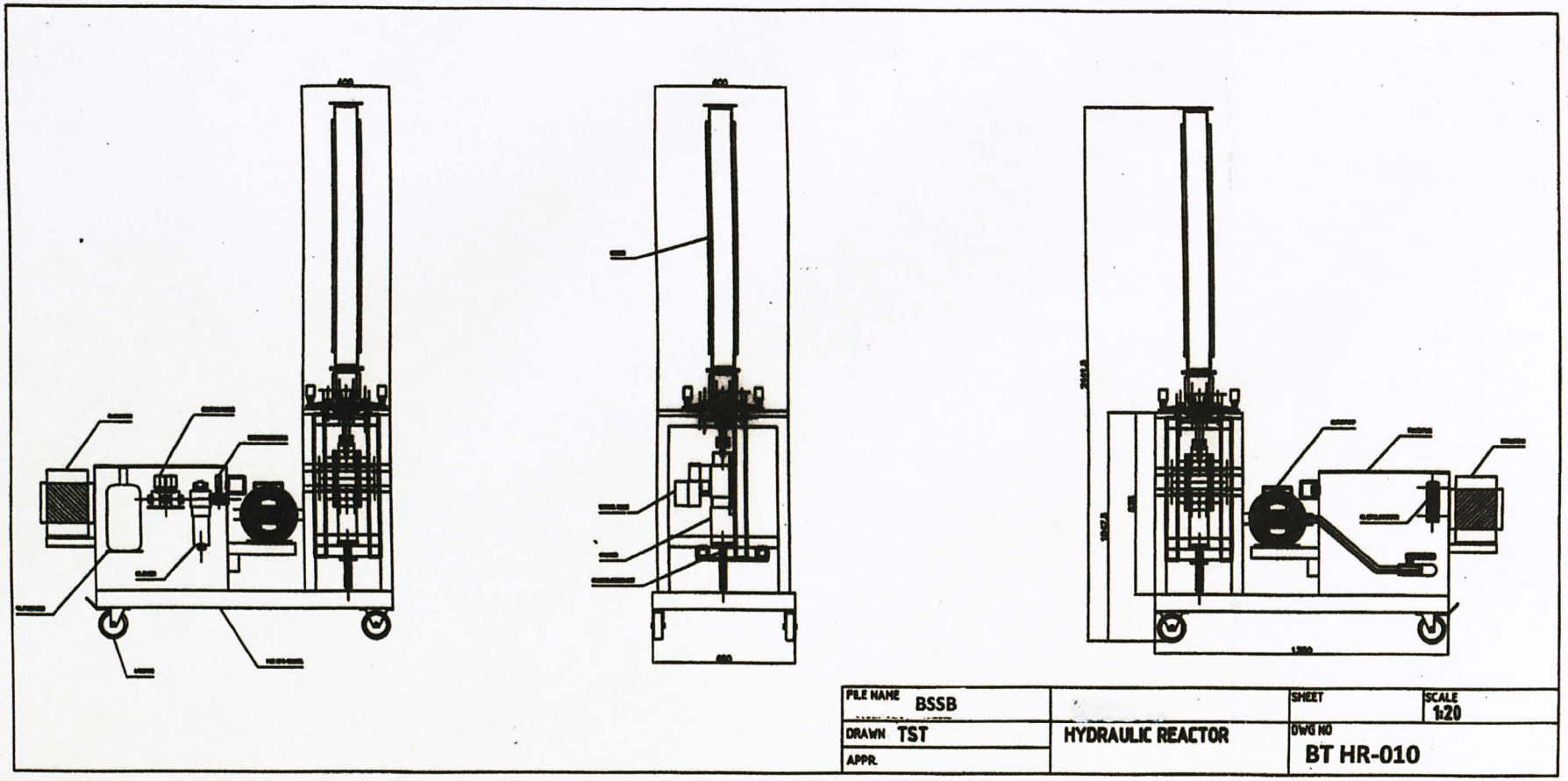
- 1 nos of 10L PP tank for product outlet
  - Capacity: 10 liters
  - Necessary piping connections
  - Non-stirred tank
  - Interconnecting piping: PTFE

l) Execution

- Reactor to mounted in a mild steel skid
- Piping (Teflon, stainless steel, PP) to be routed within the skid
- Base plate for mounting the equipment, ncl. wiring in plastic ducts
- Labelling of equipment - single and permanent
- Panel light for each control panel
- Socket outlet
- Floating contact for general alarm
- General alarm as signal lamp and electronic buzzer with acknowledgement

m) Field installation

- Deliver to Faculty of Engineering, UTP
- Start-up and commissioning



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APPR.			

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APPR.			