SUMMARY AND CONCLUSIONS

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The current Indian lube oil production does not meet its demand and import bill for petroleum products including lubricating oil has been ever increasing .

Different grades of lubricating oils are made from heavy petroleum distillates or lubricating oil fractions boiling in the range of 350-550 ^{o}C involving following separation steps (Nelson, 1958; Marple and Landry ,1965).

a) Solvent extraction of polycyclic aromatic hydrocarbons present in the vacuum distillate to improve the viscosity index.

b) Solvent dewaxing of the waxy raffinate by chilling and filtration for improvement of flow properties of the oil which is measured by pour point.

Solvent dewaxing operation in the lube oil manufacturing process is by far the most expensive and labour intensive to achieve the desired pour point . Moreover redesigning the entire process conditions becomes essential on variation of composition of the feed stock. Some desirable saturates get separated by chilling operation (Ramage 1986) . Consequently, Assam crude remained unsuitable as feed stock for lube production and indigenous production of lube depends on imported Gulf crude.

In order to reduce cost of operation and to increase yield development of differnt dewaxing methods are in progress.e.g; Catalytic dewaxing (Bill and Marmin, 1979), Urea adduction (Rogers,1957) , and molecular sieve adsorption (Symoniak, 1980) . However , there are some advantages and disadvantages associated

with each of these processes.

Dewaxing of lighter petroleum fraction i.e; Gas oil (b.p. 250-350 ^{o}C) by biological system has progressed much in the last two decades and the process was applied successfully for commercial production of Single Cell Protein (SCP) and dewaxed gas oil (Laine and du Chaffaut, 1975 ; Moo young , 1976 ; Litchfield, 1977). However choice of petroleum fraction as feed stock for SCP production was restricted to middle distillate (250-350 ^{o}C) and use of heavier petroleum fraction (350 - 500^{o}C) for this purpose has not yet been reported.

With a view to develope a cost-effective simple technology for production of lube oil and SCP , microbial dewaxing of lubricating oil fraction would be worth exploiting .

The present work was thus aimed at the following objectives :

i) Development of a dewaxing process on heavier petroleum fraction at ambient conditions applying microorganisms.

ii) Operational flexibility for the use of feed stocks (vacuum distillates) obtained from different crudes.

iii) SCP (biomass) obtained as by product of the dewaxing process can be substituted in the cattle feed as compared to slack wax obtained from the solvent dewaxing process which does not have market due to high oil content (25-35 %).

The following five chapters in the thesis described the different aspects of the work .

1. Introducing the present status of the dewaxing technology for lubricating oil manufacture , the limitations of the processes were highlighted in Chapter I .

2. Progress made on SCP production and dewaxing of gas oil fraction as feed stock was summarised from the available literature in Chapter II . Among the different hydrocarbon utilising microorganism , yeast strain , *Candida* was found sucessfully applied for the production of SCP and deweaxing of gas oil.

3. Chapter III covered the experimental set up , equipment and chemicals used for the study . Methods followed for the determination of different properties of oil and biomass specially , the determination of specific growth rate and power input to the culture fluid were also described in this chapter. Among the feed stocks used in the Study , one (sample 'A') was processed from Assam crude found unsuitable for lube production in Barauni Refinery due to its high paraffin content whereas the other sample B processed from Gulf crude is a regular feed stock for lube oil production.

4. In Chapter IV the experimental results obtained in the following aspects of microbial dewaxing of heavier petroleum fraction were discussed.

4.1. *Candida tropicalis* , a locally isolated Yeast strain was found higher adaptation capacity than that of *C lipolytica*

in the shake flask culture studies. Hence the strain *C.tropicalis* was selected for further studies.

Three different stages of interactions were observed between the cells and semisolid hydrocarbon droplets.

i) Attachment of yeast cells with large oil droplets .

ii) Emulsification of large oil droplets and formation of smaller drops of hydrocarbon during growth phase of the strain.

iii) Pseudo-solubilisation of oil/ water emulsion containing majority of yeast cells in free state at the completion of growth phase.

4.2. Optimisation of growth parameters have been recorded in Chapter 4.2. The optimum temperature range of 36-37^{o}C was observed for the maximum sp. growth rate of 0.27 h^{-1} for sample 'A' (C_{22}-C_{40}) and 0.38 h^{-1} for n.paraffin (C_{14}-C_{18}). Higher energy of activation (E $\sim$ 67.7 K J / moles) for growth of the strain on vacuum distillate 'A' was observed as compared to that of n. paraffins (E $\sim$ 58.5 K J / moles) perhaps due to emulsification constraint faced by the strain during growth on sample 'A'. Maximum decrease of pour point of dewaxed oil was observed at 37^{o}C which would be favourable because low cost of heat removal can be achieved by circulating tap water (25-30^{o}C).

The optimum pH range of 4 to 4.5 was observed for maximum sp. growth rate of the strain which would be favourable for avoiding contamination problem in large scale operation. Decrease of pH was observed during growth of the strain due to assimilation of ammonia as nitrogen source by

the strain . However increase in pH during stationary phase may be due to endogeneous metabolism of the strain.

Substrate concentration up to 40 g/l did not have any inhibitory effect on the growth and dewaxing of oil . However poor dewaxing and decrease of yield of biomass were observed at higher oil concentration (100 g/l) perhaps due to dispersion limitation of the hydrocarbon flocks in the fermentor used in the study .

4.3. Batch growth studies indicated that long lag phase of the distillate sample 'A' was due to emulsification of the semisolid hydrocarbon as compared to liquid mass of sample 'B '. Logarithamic growth phase indicated that dewaxing of vacuum distillates was a growth associated phenomena . Intial higher sp. growth rate and lower dewaxing rate were observed due to preferrential assimilation of short chain alkanes over long chain alkans which were assimilated in the latter phase of growth. Higher dewaxing rate of sample 'B ' was observed in comparison to sample 'A ' may be due to variation of composition of the distillate samples. However an overall pour point depression of 30^{o}C was observed for both the distillate samples .

A low yield of biomass was observed due to assimilation of long chain alkanes in comparison to n.paraffins of shorter chain length as carbon substrate . Yield and and productivity of dewaxed oil , however can be increased in semi-continuous operation as compared to batch operation perhaps due to higher concentration of bioemulsifier being present in the culture

fluid during semi-continuous operation which enhanced the assimilation of alkanes in a shorter period than the corresponding batch operation .

4.4. The rheological properties of the culture fluid indicated that higher viscosity and non - Newtonian charecteristics prevailed during initial growth phase due to emulsification of viscous oil samples . Hence the scale up studies were conducted based on the data of different parameters obtained at initial growth phase to avoide the failure of the system. The hydrodynamic behavior of the oil/water/cells flocks showed that the system is a dispersion limited process rather than limitation of mass transfer of oxygen in the system . It was confirmed from the scale up data that suitable criteria for scaling up of the system would be equal power per unit volume (P / V) . However one has to take care of the high motor power requirement in large stirred tank fermentor

Hence suitable choice of fermentor is required to make the system energetically efficient as compared to solvent dewaxing process. Recent development in the design of bioreactors indicates that JET FERMENTOR would be effective in processing dispersion limited system with lower input of power as compared to stirred tank fermentor. Increase of productivity of dewaxed oil may be expected in the jet fermentor due to its continuous operation as compared to batch or semicontinuous operation .However these aspects could not be studied further due to limitation of time.

4.5. The distillate oil-water-cell emulsion obtained from the fermentor was characterestically different from gas oil-water-cell emulsion . Hence application of leaching would be ideal for the recovery of dewaxed oil . Further studies are needed for the optimisation of the entire operation .

4.6. The analysis of dewaxed oil indicated that yeast strain *C.Tropicalis* selectively assimilated the long chain paraffinic hydrocarbons present in the vacuum distillate leaving the aromatics rings , naphthenic and branched paraffin hydrocarbons . Hence microbial dewaxing process in combination with dearomatisation process e.g; solvent extraction has the potential to become an cost-effective alternative technology for the production of lubricating oil .

The composition of biomass indicated that it is comparable with n.paraffin grown SCP . Hence there is a scope for utilisation of the biomass as SCP in the cattle feed as the demand for cattle feed is increasing .

There is ample scope for further studies on microbial dewaxing of heavier petroleum fraction on the above mentioned aspects and based on the data generated from those studies a true economic comparison can be drawn with the existing lube oil production procrsses.