PROCESS DESIGN IN DEGUMMING AND BLEACHING OF PALM OIL

RESEARCH VOTE NO: 74198

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“I declare that thesis entitled PROCESS DESIGN OF DEGUMMING AND BLEACHING OF PALM OIL is the result of my own research except for some information which the source for each one has been stated.”

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Date : OCTOBER 2006

Dedicated to my beloved father, Haji Mohd Zin Haji Salleh for his constant encouragement and motivation. To my beloved mother, Hajah Gayah Jali for her inspiration and to my forever supportive family members.

Not to forget, this work is lovingly and respectfully in honour of my wonderful husband, Mohd Nizar Mohtar

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ABSTRACT

Palm oil (*Elaeis guineensis*) industry is one of the major contributors to the Malaysia economic growths. The palm oil supply from Malaysia has increased apparently over the last two decades due to vast increase in production. Crude palm oil (CPO) obtained from the mesocarp of palm oil fruit undergoes several stages of refining processes in order to produce refined, bleached palm oil (RBDPO). Currently, in Malaysia, the type of refining used is physical refining. This method involved the processing stages of degumming, bleaching and deodorization. From refiners point of view, degumming and bleaching processes are recognized as critical areas in the palm oil refinery and need to be carefully monitored, because any imperfection during these processes will tremendously affect the later stages of refining processes and finally affect the finished product. These stages are identified as the major contributors to the total operating cost of the plant due to the cost of chemicals (phosphoric acid and bleaching earth) that are being used in these processes. In this research, a process model for degumming and bleaching operation will be designed in order to help the refiners to predict the exact ratio of phosphoric acid and bleaching earth to the crude palm oil. By doing so, we hope to reduce the operating costs and time of the overall palm oil refining process. Therefore at the end of this research, with the ANN model designed, the bleaching and degumming efficiency can be improved thus can help to boost the Malaysian palm oil industry.

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ABSTRAK

Industri kelapa sawit (*Elaeis guineensis*) adalah merupakan salah satu penyumbang terbesar kepada pembangunan ekonomi Malaysia. Bekalan minyak kelapa sawit dari Malaysia telah meningkat dengan ketaranya sejak dua dekad yang lalu disebabkan oleh peningkatan yang besar di sektor penghasilan. Minyak kelapa sawit mentah yang disuri daripada bahagian mesorkapa buah kelapa sawit akan diproses melalui beberapa peringkat penapisan dan penulenan bagi menghasilkan minyak kelapa sawit tertapis, terluntur dan ternyahbau (RBDPO). Kini di Malaysia, kaedah yang digunakan adalah kaedah Proses Penulenan Fizikal. Proses penulenan secara ini melibatkan beberapa peringkat iaitu pennyah-gam degumming, pelunturan dan penyulingan. Pada pendapat mereka yang terlibat dalam industri penulenan kelapa sawit ini, mereka dapati bahawa peringkat degumming dan pelunturan adalah merupakan bahagian-bahagian yang kritikal bagi proses penulenan kelapa sawit, dimana pemantauan secara teliti adalah diperlukan kerana sebarang ketidaksempurnaan semasa peringkat-peringkat ini akan teruk menjejaskan peringkat seterusnya dan akhirnya memberi impak kepada hasil akhir. Peringkat-peringkat proses ini juga dikenalpasti sebagai penyumbang besar kepada keseluruhan kos operasi penulenan kelapa sawit disebabkan bahan-bahan kimia yang digunakan didalam peringkat-peringkat ini iaitu asid fosforik dan bleaching earth. Oleh itu, didalam projek penyelidikan ini, satu peoses model bagi operasi degumming dan pelunturan, akan direkabentuk bagi membantu refiners meramalkan nisbah yang tepat bagi asid fosforik dan bleaching earth kepada minyak kelapa sawit mentah. Dengan begiitu, kami berharap dapat mengurangkan kos operasi dan masa bagi keseluruhan proses penulenan kelapa sawit. Oleh itu, di akhir penyelidikan ini dengan ANN model yang direkabentuk maka efinsensi operasi degumming dan pelunturan dapat diperbaiki dan dapat membantu meningkatkan industri minyak kelapa sawit Malaysia.

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AAS	-	Atomic Absorbance Spectrophotometer
Abs	-	Absorbance value
ANN	-	Artificial Neural Network
AOCS	-	American Oil Chemists’ Society
AOTD	-	Analytical Olecochemical Technology Division
AV	-	Anisidine Value
BE	-	Bleaching Earth
CF	-	Calibration factor
CPO	-	Crude Palm Oil
Cu	-	Copper
DBPO	-	Degummed Bleached Palm Oil
DG	-	Diglycerides
DOBI	-	Deterioration of Bleachability Index
DOE	-	Design of Experiment
E	-	Error
EDTA	-	Ethylene Diamine Tetraacetic Acid
Fe	-	Iron
FFA	-	Free Fatty Acids
FFB	-	Fresh Fruit Bunches
g	-	gram
GDP	-	Gross Domestic Products
H_2O	-	Water
IV	-	Iodine Value
kg	-	kilogram
meq	-	milliequivalent

MG	-	Monoglycerides
MIBK	-	Methyl Isobutyl Ketone
M & I	-	Moisture and Iodine
ml	-	milliliter
ML	-	Moisture loss
mm	-	millimeter
mmHg	-	millimeter mercury
MPOB	-	Malaysia Palm Oil Board
MT	-	metric tonne
N	-	normality of solution
Na	-	sodium
NaOH	-	sodium hydroxide
NN	-	neural network
nm	-	nanometer
NPO	-	Neutralized Palm Oil
NHP	-	Non-Hydratable Phosphatides
P	-	Phosphorus
PA	-	Phosphatidyl acid
PC	-	Phosphatidyl choline
PE	-	Phosphatidylethanolamine
PFAD	-	Palm Fatty Acid Distillate
pH	-	potential hydrogen
PI	-	Phosphatidylinositol
PIPOC	-	Proceedings of International Palm Oil Congress
PORAM	-	Palm Oil Refiners Association of Malaysia
PORIM	-	Palm Oil Research Institute of Malaysia
POFP	-	Palm Oil Familiarization Programme
ppm	-	part per million
PV	-	Peroxide Value
RBDPO	-	Refined Bleached Deodorized Palm Oil
RF	-	Refining Factor
RM	-	Ringgit Malaysia, monetary unit of Malaysian
RMS	-	root mean square
T	-	Temperature

TG - Triglyceride

UV - Ultraviolet

V - volume of NaOH solution used in ml

Vs - volume in ml of sodium thiosulphate solution of normality N used for the determination

Vb - volume in ml of sodium thiosulphate solution of normality N used for the blank test.

W - weight of sample/test portion

α - Alfa

β - Beta

γ - Gamma

d - Delta

c - quantity of residual substance dissolved

K - constant unrelated to the amounts of solute and adsorbent

m - quantity of adsorbent

n - constant unrelated to the amounts of solute and adsorbent

x - quantity of substance adsorbed

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FFA - Free Fatty Acids
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g - gram
GDP - Gross Domestic Products
H_2O - Water
IV - Iodine Value
kg - kilogram
meq - milliequivalent
MIBK - Methyl Isobutyl Ketone

M & I - Moisture and Iodine

ml - milliliter

ML - Moisture loss

mm - millimeter

mmHg - millimeter mercury

MPOB - Malaysia Palm Oil Board

MT - metric tonne

N - normality of solution

Na - sodium

NaOH - sodium hydroxide

NN - neural network

nm - nanometer

NPO - Neutralized Palm Oil

NHP - Non-Hydratable Phosphatides

P - Phosphorus

PA - Phosphatidyl acid

PC - Phosphatidyl choline

PE - Phosphatidylethanolamine

PFAD - Palm Fatty Acid Distillate

pH - potential hydrogen

PI - Phosphatidylinositol

PIPOC - Proceedings of International Palm Oil Congress

PORAM - Palm Oil Refiners Association of Malaysia

PORIM - Palm Oil Research Institute of Malaysia

POFP - Palm Oil Familiarization Programme

ppm - part per million

PV - Peroxide Value

RBDPO - Refined Bleached Deodorized Palm Oil

RF - Refining Factor

RM - Ringgit Malaysia, monetary unit of Malaysian

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GLOSSARY

Acid Conditioning	Precipitation of hydratable and non-hydratable phosphatides and trace metals by mixing and reacting crude oil with acid. Caustic may also be added to neutralize the acid and create a small amount of soap.
Antioxidant	A substance that slows or interferes with the reaction of a fat or oil with oxygen. The addition of antioxidants to fats or foods containing them retards rancidity and increases stability and shelf life.
Anisidine Value	A measurement of a triglyceride's secondary oxidation determined by the amount of aldehydes formed as peroxides decompose.
Bleaching	Removal of color and oxidizing bodies, residual gums, soap and trace metals by mixing oil with special adsorbents (silica and/or bleaching earth). The adsorbents containing the mentioned impurities are then removed by filtration.
Carotenoids	A large group of isoprenoid structures with different numbers, positions and configurations of conjugated double bonds. β – carotene is a precursor of vitamin A in animals.
Chlorophyll	A natural, green coloring agent vital to a plant's photosynthesis process which is removed from the oil through the neutralizing and bleaching processes.
Degumming	A step in the refining of oils and fats. The addition of phosphoric acid to the crude oil results in the separation of all or some of phospholipids which present.

Deodorization	A process of removing the fatty acids, odor, flavor and destabilizing impurities, as well as some color bodies by subjecting the oil to high vacuum and temperature, augmented by direct steam agitation, under conditions so that the impurities are vaporized and removed while the oil remains liquid
Dewaxing	Removal of small amounts of high melting components (waxes) that cloud the oil at storage temperature. The term is typically associated with processing of sunflower and rice bran oils. The oil is chilled then mixed with filter aid. After holding the oil for a certain period, the waxes become solid (crystallize) and can be removed by filtration. In some cases centrifugal separation is used in combination with degumming, neutralizing or water washing instead of filtration.
Diglyceride	The ester resulting from the chemical combination of glycerol and two fatty acids.
Elaeis guineensis	The oil palm, which is the source of palm oil (from the mesocarp) and palm kernel oil (from the kernel). It is native to Africa. Mainly grown in South East Asia and Africa.
Experiment	A set of measurements carried out under specific and controlled conditions to discover, verify, or illustrate a theory, hypothesis, or relationship. Experiments are the cornerstone of statistical theory, and are the only method for suggesting causal relations between variables. Experimental hypotheses cannot be proved using statistics; however, they can be disproved. Elements of an experiment generally include a control group, randomization, and repeat observations.
Fractionation	Removal of higher melting fractions (stearin) that solidify at higher than desired temperatures. This is done by cooling the oil or fat so that the stearin forms crystals that can be separated, usually by filtration. The term is mainly associated with processing of palm and similar highly saturated oils.
Free fatty acid	Fatty acid in unbound (underivatised) form. Oils and fats are mainly triacylglycerols contain in their natural (crude) state small amounts of free acids which may removed by processing.
Hydration	Precipitation and agglomeration of phosphatides by mixing and reacting acid conditioned or crude oil with water.

Hydrolysis	The chemical reaction of a triglyceride with water forming glycerine and free fatty acids
Iodine value	Classical method to obtain information on degree of unsaturation in fats and oils. An iodine-bromide (Hanus reagent) or iodine monochloride (Wijs reagent) reagent is reacted with the double bond and excess reagent (as iodine) is titrated with sodium thiosulphate solution.
Lauric acid	Dodecanoic acid, 12:0. It is a major component of oils such as coconut and palm kernel. Oils rich in lauric acid are generally known as lauric acid
Lauric oils	Oils containing 40-50% lauric acids (C_{12}) in combination with other relatively low molecular weight fatty acids. Coconut and palm kernel oils are principal examples
Lecithin	A mixture of naturally occurring phosphatides which has emulsifying, wetting, and antioxidant properties, a principal source of which is crude soybean oil
Linoleic acid	An important C_{18} diene acid, 9C, 12C-18:2, present in most seed oils, often in high proportions. It is the first member of the (n-6) family of polyenoic acids and is an essential fatty acid.
Lipoprotein	Any of the class of proteins that contain a lipid combined with a simple protein.
Lovibond value	A system to express the colours of refined oils and fats. Red and yellow galsses are used, of which the red glasses are standardised
Monoglyceride	The ester resulting from the combination of glycerol and one fatty acid
Neutralization	A removal process of free fatty acids (FFA), hydratable and non-hydratable phosphatides, trace metals and color bodies (precipitated by mixing and reacting with caustic) by centrifugal separation. The process is most often preceded by acid conditioned.
Oleic acid	Trivial name for 9-cis-octadecanoic, 18:1 (19-C). It is the most widely distributed of all fatty acids and serves biosynthetically as the precursor to the (n-9) families of acids. Present in high

	amounts in olive oil and high-oleic sunflower and safflower oils.
Oxidation	The reaction of oxygen with an oil or fat causing rancidity
Peroxide value	Oxidised fats contain hydroperoxide which liberate iodine from acidified potassium iodide. This is basis of an analytical method for measuring oxidation and the peroxide value is usually expressed as milliequivalents of active oxygen/kg (or mmoles of active oxygen/2 kg)
Phospholipids	A general description of lipids containing phosphoric acid (or other phosphorus containing acids) in appropriate ester form such as glycerophospholipids..
Phosphatides	The chemical combination of an alcohol (typically glycerol) with phosphoric acid and a nitrogen compound; synonymous with phospholipids. Commonly referred to as gums.
ppm	This concentration parameter expresses the amount of e.g solid particles in a solvent. 100 parts (e.g grams) of iron, Fe in RBDPO means that in 1 million parts (e.g grams) of RBDPO, 100 parts (e.g grams) of Fe are dissolved.
Refining process	Industrial technology to obtain edible oils from crude oils through processing steps such as degumming, neutralisation, bleaching and deodorization.
Refining Factor	A parameter is used to asses the efficiency of various stages of a refining process. This factor is dependent upon the yield of the product and the quality of the input and it is calculated as: $$RF = \frac{oil\ \ loss\ \ \%}{FFA\ \ \%}$$
Soapstock	The by-product from the neutralizing step of chemical refining consisting of soap, hydrated gums, water, oil, and other impurities.
Tocopherol	A naturally occurring antioxidant found in many vegetable oils
Triglyceride	The chemical combination of glycerol and three fatty acids

CHAPTER 1

INTRODUCTION

1.1 Research Background

The oil palm, *Elaeis guineensis,* is native to Africa. The commercial values of this crop lies mainly in the oil that can be obtained from the mesocarp of the fruit - palm oil and the kernel of the nut - palm kernel oil. In fact, oil palm is the only fruit that can give these two types of oil. Both are edible oils but with very different chemical composition, physical properties and applications.

Palm oil is used mainly for cooking such as cooking oil, margarine and shortening but also has non-food applications such as soap, detergent, cosmetics. Figure 1.1 shows the cross-section of palm oil fruits indicating the mesocarp and kernel of the fruit.

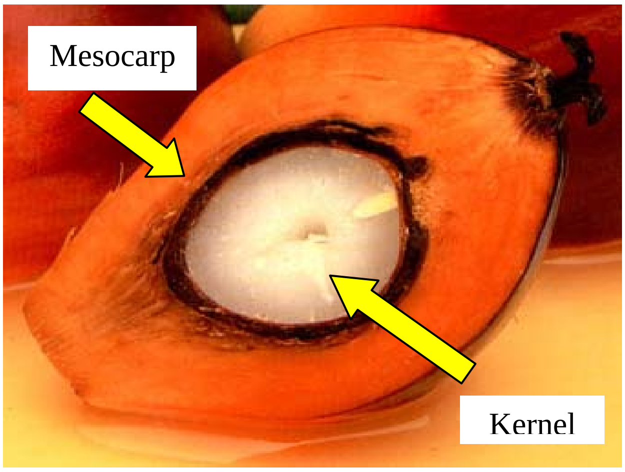

Figure 1.1: Cross-section of palm oil fruit

Although oil palm is native to Africa, Malaysia was the first country to embark on large-scale planting and processing. It had to develop its own technology and adopt innovative policies in order to boost production.

The first commercial oil palm estate in Malaysia was set up in 1917 at Tennamaran Estate, Selangor. However, it was only in the 1960s, oil palms were commercially cultivated in large scale in order to avoid over dependence on natural rubber which are major commodity during previous years. Since then, palm oil industry has expended rapidly and has emerged as the most remunerative agricultural commodity, overtaking the natural rubber (Arrifin and Fairus 2002).

The growth of the industry has been phenomenal and Malaysia is now the largest producer and exporter of palm oil in the world, accounting for 52 percent of world production and 64 percent of world exports in 1999. Table 1.1 and Table 1.2 below, shows world annual production and annual exports of palm oil according to the respective countries.

Table 1.1 : World Major Producers Of Palm Oil : 1994 - 2003 ('000 TONNES)

Country	1994	1995	1996	1997	1998	1999	2000	2001	2002	2003
Malaysia	7,403	7,221	8,386	9,069	8,319	10,554	10,842	11,804	11,909	13,354
Indonesia	3,421	4,008	4,540	5,380	5,361	6,250	7,050	8,030	9,200	9,750
Nigeria	645	640	670	680	690	720	740	770	775	785
Colombia	323	353	410	441	424	501	524	548	528	543
Cote d'Ivoire	310	300	280	259	269	264	278	220	240	251
Thailand	297	316	375	390	475	560	525	620	600	630
Papua New Guinea	223	225	272	275	210	264	336	329	316	325
Ecuador	162	178	188	203	200	263	222	201	217	247
Costa Rica	84	90	109	119	105	122	138	138	140	144
Honduras	80	76	76	77	92	90	97	108	110	112
Brazil	54	71	80	80	89	92	108	110	118	132
Venezuela	21	34	45	54	44	60	73	80	80	79
Guatemala	16	22	36	50	47	53	65	70	81	91
Others	1,265	1,676	815	869	844	832	879	919	922	940
TOTAL	**14,304**	**15,210**	**16,282**	**17,946**	**17,169**	**20,625**	**21,877**	**23,947**	**25,236**	**27,383**

Source : i) Oil World Annual (1999 - 2003) & Oil World Weekly (12 December, 2003)
ii) MPOB - For data on Malaysia.

Table 1.2 : World Major Exporters Of Palm Oil : 1994 - 2003 ('000 TONNES)

Country	1994	1995	1996	1997	1998	1999	2000	2001	2002	2003
Malaysia	6,750	6,513	7,212	7,490	7,465	8,911	9,081	10,618	10,886	12,248
Indonesia	2,173	1,856	1,851	2,982	2,260	3,319	4,140	4,940	6,379	6,830
Papua New Guinea	231	220	267	275	213	254	336	328	324	325
Cote d'Ivoire	148	120	99	73	102	101	72	75	65	63
Colombia	20	21	29	61	70	90	97	90	85	105
Singapore*	328	399	289	298	241	292	240	224	220	256
Hong Kong*	234	275	305	173	103	94	158	192	318	206
Others	876	791	711	860	680	787	884	1,107	956	1,083
TOTAL	**10,760**	**10,195**	**10,763**	**12,212**	**11,134**	**13,848**	**15,008**	**17,574**	**19,233**	**21,116**

Note : * - Includes Re-Exporting Countries
Source : i) Oil World Annual (1999 - 2003) & Oil World Weekly (12 December, 2003)
ii) MPOB - For data on Malaysia

Obviously now, palm oil has become an increasingly important vegetable oil in the world market and today is the largest traded vegetable oil in the world. It was recorded that in 2002, a trade surplus for palm oil industry is RM 54.1 billion compared to RM 50.9 billion in 2001. It shows that palm oil industry plays an important role in Malaysia's economy.

The palm oil refining industry is today among the most important manufacturing sectors in the country. Besides contributing to the Gross Domestic Product (GDP) and increasing employment opportunities in the country. The refining sector has contributed significantly to the growth of palm oil industry in Malaysia (Arrifin and Fairus 2002).

The palm oil industry can be divided into several main sectors according to their activities, namely:

(i) plantations - planting of the oil palm and harvesting of the fresh fruit bunches (FFB)
(ii) palm oil mills – processing of the FFB into crude palm oil (CPO) and palm kernels
(iii) palm kernel mills – extraction of the palm kernel oil
(iv) palm oil processing – refining and fractionation, production of downstream products

This research study will focus on the degumming and bleaching processes of the processing sector, which is in the refining process of palm oil.

Malaysia's refining industry first started in 1972, where there were only 4 refineries in operation during that time, but at the end of 2002, there are already 47 refineries in operation with total refining capacity of 16.14 million tonnes of CPO per annum. Most of these refineries are located close to the ports to facilitate exports for example in southern region of Malaysia most of the palm oil industry are located in Pasir Gudang , Johor where the port is situated.

When the palm oil refining industry first started in 1970's, it had employed mainly alkali or chemical refining process as alkali refining was the more establish process for edible oil at that time. This mode produced neutralized palm oil (NPO) and its products. In the late of 1970s however, physical refining began to be preferred method as it is more cost effective (Ahmad and Mohammad, 1993). Further elaboration on physical and chemical refining will be explained in Section 2.5 of Chapter 2: Literature Review, in this thesis.

Currently in Malaysia, all palm oil refining plants are using physical refining method as it is proven to be more cost effective compared to chemical refining method. In general, physical refining of palm oil consists of 2 stages namely pre-treatment stage and deodorization stage. Pre-treatment stage involves degumming and bleaching of palm oil where the aim of the processes is to remove the undesired impurities that affect the stability of final oil products. The aims of degumming and bleaching processes are achieved by means of chemicals used to react and adsorb the unwanted impurities. The chemicals used for these processes are phosphoric acid and bleaching earth. Detailed elaboration on degumming and bleaching methods will be further discussed in Chapter 3: Theory of Degumming and Bleaching.

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1.2 Research Objective and Scope

The objective of this research is to design a process model for physical refining of degumming and bleaching operation of palm oil refinery that is able to predict the suitable ratio of phosphoric acid and bleaching earth to the crude palm oil. This process model should be able to operate at various quality of crude palm oil fed and yet ensuring efficient separation. By doing so, we hope to reduce the operating costs and time of the overall palm oil refining process.

In achieving the objective of the research there are a few important tasks need to be carried out and three research scopes have been identified for accomplishing the objective. The scopes are:

1. Identification of the most optimal operating parameters and conditions for degumming and bleaching processes. This is done through surveys and plant visits to palm oil refineries throughout Malaysia. Initially questionnaires were distributed to all registered palm oil refineries in Malaysia. Then, based on responses obtained; the most optimal operating temperature, pressure, retention time, most common chemicals used, acceptable quality of crude palm oil (CPO) as feed and refined bleached deodorized palm oil (RBDPO) as the output are identified. At the same time, plant visits are also conducted in order to get overview from refiners and to familiarize with plant operation.

2. Design a lab scale experiment for degumming and bleaching in order to get the data for modelling. This lab scale experiment is designed as degumming and bleaching processes are carried out simultaneously and based on the most optimal conditions and parameters identified. During this stage, the number of experiments need to be run are determined through the design of experiment (DOE) method.

3. In experimental stage, some suitable quality checking experiments for the crude palm oil (CPO) and degummed bleached palm oil (DBPO) quality are also being identified during this stage. The parameters that are being measured in this experiment are; (i) FFA (free fatty acid) content, (ii) DOBI

(deterioration of bleachability index) value, (iii) peroxide value, (iv) phosphorus content, (v) iron content and (vi) moisture content. The deguming and bleaching process are carried out for 30 minutes, under 50 torr vacuum and temperature of 100^{o}C, using rotavapor unit. Based on the experimental results obtained, some deductions would be done on predicting the suitable ratio of phosphoric acid and bleaching earth to be added in the degumming and bleaching processes.

3. Develop feed forward back propagation neural networks modelas by using artificial neural networks (ANN) toolbox from Matlab V.7.0 to model and simulate processes and thus predicting the suitable ratio of phosphoric acid and bleaching earth to the crude palm oil. In this study, comparison of deductions between experimental findings and model developed would be done and a concrete conclusion will be made based on that.

1.3 Problem Statement

From refiners' point of view, degumming and bleaching process stage are recognized as critical areas in the palm oil refinery. Separation of minor components at these stages must be carefully monitored because any imperfection during these processes will tremendously affect the later stages of refining processes and finally affect the finished product. These stages are considered critical as it is identified as major contributors to the total operating cost of the plant due to the cost of chemicals (phosphoric acid and bleaching earth) that are being used in these processes.

In addition, Borner et al. (2003) also reported that bleaching is one of the most cost-intensive processes for refining vegetable oils caused mainly by the consumption of bleaching agents like bleaching earth and activated carbon, oil losses in the spent bleaching and in certain cases for disposal of the spent agents. Therefore, it's become an interest of all refineries to reduce the consumption of bleaching earth as much as possible. It is estimated about 20% of total operating cost are due to bleaching and degumming process and currently the price of bleaching earth is RM 700-800 per MT and RM 3000 per MT for phosphoric acid.

In current situation the amounts of chemicals added in are usually fixed within typical ranges of doses usually used throughout certain period. It means that, even though the incoming CPO has lower impurities or minor components content, the amount of chemicals added would not be changed. There is no system that can estimate the right amount of chemicals to be added in accordance with the quality of CPO used.

Thus, it important to have a process model which can suggest a suitable ratio of phosphoric acid and bleaching earth for the varying CPO quality.

1.4 Research Contributions

Even though application of Artificial Neural Networks (ANN) are now extensively employed in different branches of science and technology but in palm oil industry it is relatively a new scenario. Therefore, based on

(i) the present knowledge of palm oil processing method,
(ii) optimal operating parameters for the production of refined bleached degummed palm oil (RBDPO),
(iii) deduction from experimental findings and
(iv) the application of ANN technology in this research study,

it would introduce and enable transfer of this advanced technology to palm oil refiners. It is anticipated that once the ANN technology on palm oil processing have been developed, it will significantly reduced the operating cost of the plant since this technology will help the palm oil refiners easily predict the exact amount of phosphoric acid and bleaching earth to be used to refine the CPO fed in.

CHAPTER 2

LITERATURE REVIEW

2.1 Palm Oil (*Elaeis guineensis*)

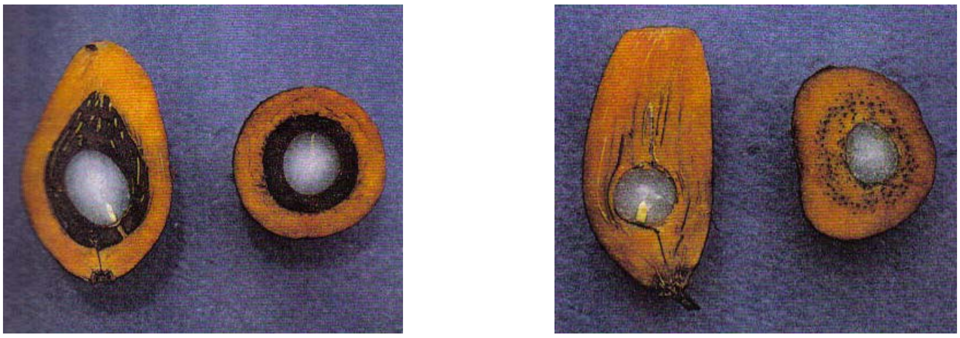
In 1434, a Portugese sailor, Gil Eannes first reported about oil palms (*Elaeis guineensis)* (Bockish, 1998). Today, they flourish mainly in the western part of Africa, Indonesia, Malaysia and most recently in Brazil and Colombia. Oil palm tree (Figure 2.1), grow up to 20 meter in height and grow best at temperature of 24-27^{o}C. Oil palm tree require a humid climate and the cultivated oil palm carry fruit from their fourth year onward and can be harvested for 40-50 years (Bockish, 1993).

Palm oil is derived from the fleshy part or the mesocarp of the fruit of the palm species *Elaeis guineensis* (Figure 2.2). However in Malaysia, *Tenera* (hybrid of *Dura* X *Psifera*) palm fruit is widely cultivated due to commercial and processing viability as harvesting becomes easier since the palm trees are relatively shorter, producing good fruit bunch and higher fruit oil content (Noor Azian, 1995).

Figure 2.1: Palm Oil Tree

Figure 2.1: *Elaeis guineensis*

Figure 2.3 shows three genotypes of palm oil where clear differences in their fruit morphology are revealed. The dura fruit form has a thick shell or endocarp surrounding the kernel whereas psifera form has no shell. The hybrid between the two, the tenera fruit form has a shell of intermediate thickness and a surrounding of fibre strands in the mesocarp (Noor Azian, 1995).



Dura (Thick-Shelled)

Pisifera (Shell-less)

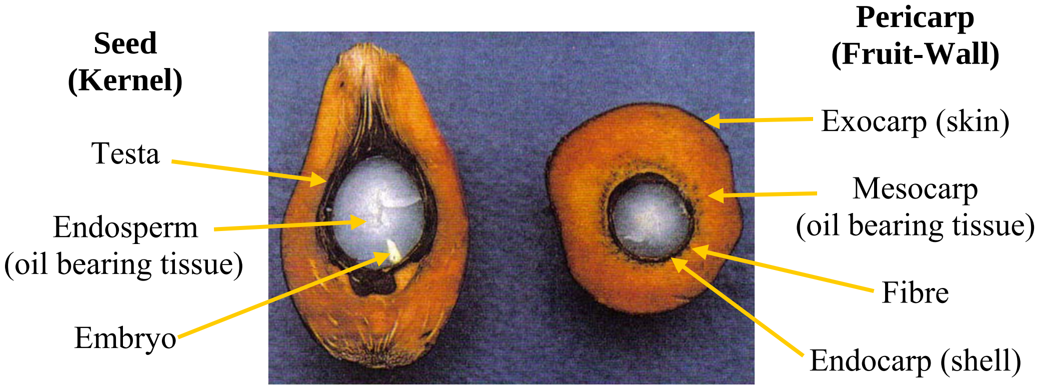


Figure 2.3: Tenera Hybrid obtained from Dura and Pisifera Parents (Noor Azian, 1995).

2.1.1 Palm Oil Fruit Composition

Mesorcarp accounts for about 60% of the total composition of palm oil fruit and crude palm oil is derived from this part. Figure 2.4 below shows overall composition of palm oil.

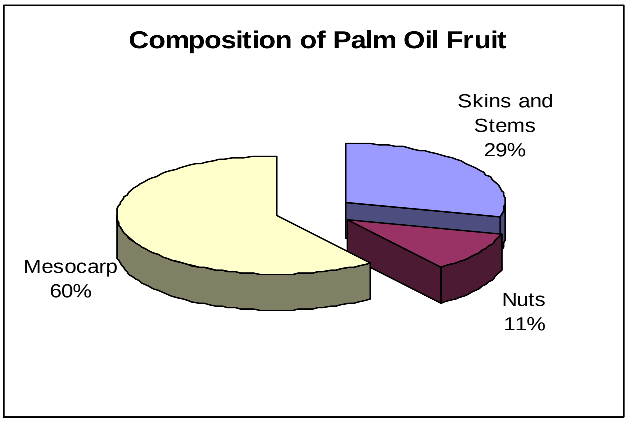


Figure 2.4: Composition of palm oil fruit (Bockish, 1993).

Figure 2.5 below shows the composition of mesocarp where the oil accounts for 39 % of the overall composition. Crude palm oil (CPO) is obtained from the mesocarp part of palm oil fruit after undergoing through several processes such as sterilization process, stripping, extraction and purification.

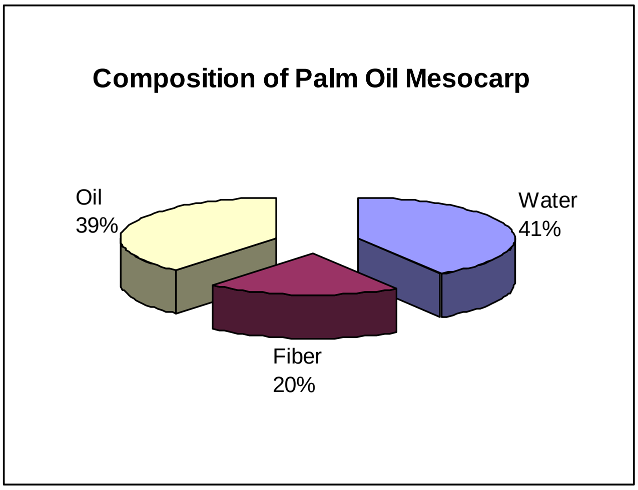


Figure 2.5: Composition of Palm Oil Mesocarp (Bockish, 1993).

2.1.2 Uses of Palm Oil

As mentioned, the oil palm produces two types of oils, palm oil from the fibrous mesocarp and palm kernel oil from the palm kernel. Palm oil and palm kernel oil have a wide range of applications; about 80% are used of food applications while the rest is feedstock for a number of non-food applications (Salmiah. 2000).

Among the food uses, refined, bleached and deodorised (RBD) olein is used mainly as cooking and frying oils, shortenings and margarine while RBD stearin is used for the production of shortenings and margarine. RBD palm oil, which is the unfractionated palm oil, is used for producing margarine, shortenings, vanaspati (vegetable ghee), frying fats and ice cream (Salmiah. 2000).

Figure 2.6 shows some examples of a number of palm-based food applications;

Figure 2.6: Variety of palm oil-based food products (Salmiah. 2000).

2.2 What is Crude Palm Oil ?

Crude palm oil (CPO) is the oil obtained from the mesocarp part of palm oil fruit. Figure 2.7 shows the processes undergone by fresh fruit bunches (FFB) to produce CPO. The crude palm oil (CPO) produced, is further processed to yield either red or bleached cooking oil or detergents.

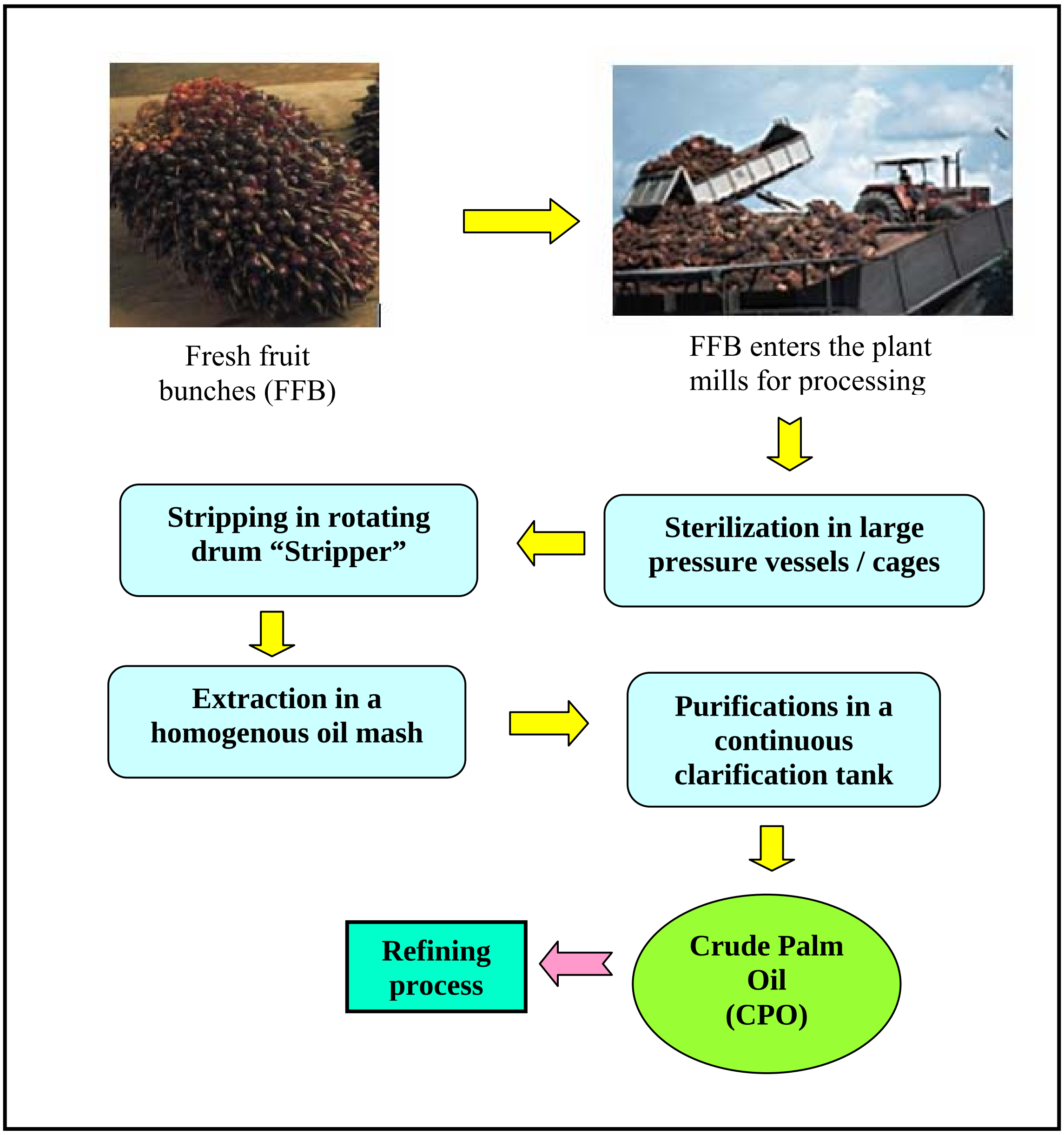


Figure 2.7: Flowchart of crude palm oil (CPO) production.

2.2.1 Composition and Constituents of Crude Palm Oil

Higuchi (1983), stated that crude vegetables oil commonly consists of desirable triglycerides, unsaponifiable matter together with small amount of impurities. Most of these impurities contribute undesirable effects to the oil, for instance colour, flavour, odour, instability and foaming. These impurities should be removed by a purification step in order to produce good quality of refined oil with minimal possible oil loss or damage to the oil and desirable materials such as tocopherols and carotenes.

The compositions of crude palm oil can be classified as a mixture of 5 main chemical groups (Abdul Azis, 2000) as per shown in Table 2.1 below.

Table 2.1: General Compositions Of Crude Palm Oil (Abdul Azis, 2000).

Group	Components in the group
Oil	- Triglyceride, Diglyceride , Monoglyceride - Phospholipids, Glycolipid and Lipoprotein - Free fatty acids
Oxidized Products	- Peroxides, Aldehydes, Ketones, Furfurals (from sugars)
Non-oil (but oil solubles)	- Carotene - Tocopherols - Squalene - Sterols
Impurities	- Metal particles - Metal ions - Metal complexes
Water Solubles	- Water (moisture) - Glycerol - Chlorophyll pigments - Phenols - Sugars (soluble carbohydrates)

Some of these chemical groups need to be removed partially or completely through the refining process in order to produce good edible oil that have better stability and keepability. Thus, in palm oil refineries the CPO produced undergoes degumming, bleaching and deodorization to order to obtain refined, bleached and deodorized oil (RBDPO).

Table 2.2 below, shows the typical composition of the main constituents of Malaysian crude palm oil. (Noor Azian, 1995)

Table 2.2: Typical composition of the main components
of Malaysian crude palm oil (CPO)

Constituent	**Crude Palm Oil**
Triglycerides, %	95
Free Fatty Acids, FFA, %	2 - 5
Red Colour (5 ¼ ” Lovibond Cell)	Orange red
Moisture & Impurities, %	0.15 – 3.0
Peroxide Value, PV (meq/kg)	1 -5.0
Anisidine Value, AV	2 – 6
β-carotene content, ppm	500-700
Phosphorus, P, ppm	10-20
Iron (Fe), ppm	4-10
Tocopherols, ppm	600-1000
Diglycerides, %	2-6

2.2.2 Quality Of Crude Palm Oil

In general, quality of crude palm oil is dependant on the contents of free fatty acids, moisture, heavy metals, DOBI, oxidized products and minor constituents such as phosphatides, carotenes and tocopherols.

(i) Free Fatty Acid (FFA)

The FFA content of crude palm oil is always used as an index of oil quality by the commercial oil refiners in Malaysia. FFA are formed when the bound fatty acids in triglyceride, diglyceride and monoglyceride molecules are split either by chemical or enzymatic hydrolysis.

According to Formo et al. (1979), the high moisture content of palm oil fruit is favourable to the enzyme action; hence this material should be processed promptly in order to yield oil with low free fatty acid (FFA) content. Palm oil fruits are susceptible to deterioration. Their lipolytic enzymes are so active that even under favourable conditions palm oil seldom produced with free fatty acid content less than 2 %.

Thus, crude palm oils that have low FFA are indicating that, the oil are being processed from fresh, unbruised fruits and carefully handled during production, storage and transportation. High FFA content must be avoided, as it will result in higher refining losses and possible bleachability problems during refining (Formo et al., 1979).

(ii) Moisture

The miscibility of oils and water, under certain conditions will hydrolyzed the triglycerides of oils to free fatty acids and glycerol (Formo et al.,1979)

$$\mathbf{C_3H_5(OOCR)_3 + 3HOH \longrightarrow C_3H_5(OH)_3 + 3\ HOOCR}$$

But under practical conditions, the rate of hydrolytic splitting of triglyceride molecule to produce FFA is negligible at 0.1 % moisture content. Therefore as quality control measure; crude palm oil should be stored with moisture content of below 0.1 % to prevent an increase in FFA and subsequent quality deterioration due to oxidation (Goh, 1991).

(iii) Heavy (trace) Metals

Heavy (trace) metals such as iron (Fe) and copper (Cu) are usually resulted from corrosion and mechanical wear at the mills and refineries. These metals are pro-oxidant and thus, detrimental to the oil quality.

Trace metals may be present as complexes surrounded by proteins, phospholipids and lipids or non-lipid carriers. In crude palm oil, trace metals can originate from contamination by soil and fertilizers. Trace metals can also be picked up from the palm oil mill, storage tanks, road tankers, pipelines and ships' tanks. The use of stainless steel for certain mill machineries which are subjected to constant wear and tear should help to reduce metal contamination.

Heavy (trace) metals catalyse the compositions of hydroperoxides to free radicals. Iron (Fe) increases the rate of peroxide formation while copper (Cu) accelerates the hydroperoxides destruction rate thereby increasing the production of secondary oxidation products (Sambanthamurthi, 2000).

Other metals reported in palm oil are manganese, cadmium and lead. These metals are found in very low concentrations and their effects on oxidation appeared to be negligible.

(iv) DOBI

DOBI (Deterioration of Bleachability Index) is simple parameter indicating the oxidative status of crude palm oil, its shelf life and refinability (Siew 1994). It is affected by quality of oil pal fruits, ripeness of fruits, post harvest storage period of fruits and storage of palm oil (Siew 1994).

DOBI is an indication of the bleachability of the crude palm oil based on the amount of carotenes still present in the crude oil and the amount of secondary oxidation products. A good easily bleached crude palm oil will have a DOBI of 4, while average quality crude will exhibit a DOBI of 2.5 to 3.

Table 2.3 below, shows the refinability of CPO according to DOBI values.

Table 2.3: Refinability of CPO according to DOBI values (Siew 1994).

DOBI	**Grade**
< 1.7	Poor
1.8-2.3	Fair
2.4-2.9	Good
3.0-3.2	Very good
> 3.3	Excellent

There are two factors affecting DOBI, namely (Siew 1994):

- **Quality of fresh fruit bunches (FFB)**
  DOBI values are significantly affected by freshness of FFB where in general, storage of oil palm bunches over four days normally shows significant reduction in DOBI values. Bruised fruits, under-ripe and over-ripe fruits are also affecting the DOBI values.

- **Storage of crude palm oil (CPO)**
  Generally, period of storage of CPO are also affecting the DOBI values as the period of storage are longer, DOBI values becomes poorer.

(v) Oxidized products

The formation of oxidized products, which are difficult to remove, will result in poor bleachability during refining and consequently will lead to refined oils will poor stability and keepability. Apart from that, off-flavours and odours in oils are normally caused by the reaction of oxygen with double bond of unsaturated fatty acids. Therefore, it is vital to prevent or minimize the deterioration in crude oils by avoiding aeration of the oil and by avoiding gross contamination with iron or copper. The oxidation conditions can also be hindered by avoiding the oil from exposure to light, elevated temperature and the presence of pro-oxidants.

(vi) Minor Constituents

Although present in small quantities in the palm oil, these minor constituents, to a certain extent, will affect the bleachability, stability and nutritional value of the palm oil. Phospholipids, which are complex esters that contain phosphorus, nitrogen bases, sugars and long-chain fatty acids, are the main culprit that needs to be removed during degumming by coagulating the phosphatides contents with phosphoric acid. The importance of removing this component will be discussed further in the next section on physical refining.

2.3 Finished Products (RBDPO) Quality

In Malaysia, the generally accepted trading specifications for crude palm oil are; 5 % maximum FFA; 2.5 % maximum moisture and impurities (Goh, 1991), while the Palm Oil Refiners Association of Malaysia (PORAM) standard specifications for refined palm oils are given in Table 2.3.

Table 2.3: PORAM Standard Specifications for refined palm oils

Parameter	**RBD Palm Oil**
Free Fatty Acids, FFA, (% as Palmitic)	0.1 max
Moisture and Impurities (%)	0.1 max
Iodine Value (Wijs)	50-55
Melting Point (^{o}C- AOCS Cc 3-25)	33-39
Colour (5.25" Lovibond Cell)	3 or 6 Red max.

2.4 Typical Analysis of CPO, DBPO and RBDPO

It is a goal for a refiner to maintain the quality of the palm oil products at the levels acceptable to the buyers or producing a better quality of finished product while minimizing the operating costs.

Therefore, to achieve this goal, quality control are applied at each important process stage in refining where suitable analysis are carried out in order to evaluate the efficiency of each process stage. Typical analysis for crude, bleached degummed oil and refined bleached deodorized oil are shown in Table 2.4 below (Er, 1985).

Table 2.4: Typical Analysis of Crude, Degummed, Bleached and RBD Palm Oil (Er, 1985).

Parameters	**Crude Palm Oil (CPO)**	**Degummed Bleached Palm Oil (DBPO)**	**RBD Palm Oil (RBDPO)**
FFA	2 - 5 %	3 – 5 %	~ 0.05
M & I	0.15 – 3.0 %	~ 0.2 %	~ 0.02
PV	1.5 -5.0	Nil	Nil
AV	2 – 6	2 – 6	~ 2.0
β-carotene content	500-600 ppm	-	-
DOBI	2 – 3.5	-	-
Phosphorus	10-18 ppm	~ 4 ppm	~ 3 ppm
Iron (Fe)	4-10 ppm	~ 0.15 ppm	~ 0.15 ppm
Copper (Cu)	~ 0.05 ppm	~ 0.05 ppm	~ 0.05 ppm
Colour (5.25” Lovibond Cell)	-	-	Red 2.0

In this study, the focus of the research is on the degumming and bleaching process. Therefore, it is important to monitor the quality of CPO input and the output of degumming and bleaching process, which is degummed and bleached palm oil (DBPO).

After degumming and bleaching processes, it is expected that there will be a small rise in FFA content due to the acidity of the phosphoric acid used. Moisture and impurities content in degummed and bleached oil (DBPO) should be reduced up to 0.02 % in order to avoid hydrolysis triglycerides, which will result in more FFA. Peroxide content in DBPO is totally being removed during the bleaching process.

The heavy metal impurities such as copper and iron are removed up to about 0.05 ppm and 0.15 ppm respectively in order to prevent excessive oxidation at olefin bonds of the oleic and linoleic acids resulting in rancidity.

Phosphorus content in CPO should be reduced up to about 4 ppm during bleaching in order to maintain the stability of oil produced. It is important to ensure that the DBPO qualities are within the desired quality range in order to monitor the efficiency of degumming and bleaching processes and hence will not affect the later stage of refining, deodorisation.

2.5 Refining Methods of Palm Oil

Refining process is a necessary step for the production of edible oils and fats products. The objective of this process is to remove the impurities and other components, which will affect the quality of finished product. The qualities of the finished product that need to be monitored are flavour, shelf-life stability and colour of the products (Leong, 1992).

In industry perspective, the main aim of refining is to convert the crude oil to a quality edible oil by removing objectionable impurities to the desired levels in the most efficient manner. This also means that, where possible, losses in the desirable components are kept minimal and cost effective.

The objectionable substance or impurities in palm oil maybe biogenic i.e synthesised by plant themselves but they can be impurities taken up by the plants from their environment (Borner et al., 1999). The impurities maybe acquired during upstream of bleaching process which are extraction, storage or transportation of the crude palm oil from mill to the refinery.

It is important to have proper refining process in order to produce high quality of finished products with specified quality range and meet users' requirements. There are 2 basic types of refining technology available for palm oil:

(i) Chemical (alkaline) refining
(ii) Physical refining

The differences between these 2 types are basically based on the type of chemicals used and mode of removing the FFA.

In its early years of inception, the palm oil refining industry of Malaysia was mainly alkali-refining based. Alkali refining then was the more established process for edible oil. It was not until the late 1970s, that physical refining of palm oil in Malaysia started to emerge as a better alternative, in many ways, to alkali refining (Yusoff and Thiagarajan, 1993). Over the years, physical refining has proven to be very successful for palm oil and modern refineries in Malaysia are mainly using physical refining routes.

Physical refining appears to practically replace the use of chemical (alkali) refining in palm oil as the consequence of high acidity content (FFA) in chemically refined oil. The deacidification (deodorisation) process stage in the physical refining is able to overcome such situation. Apart from that, according to the literature, this method is preferred because it is acknowledged to be suitable for low-content phosphatides vegetable oil such as palm oil. Thus, physical refining is proven to have a higher efficiency, less losses (refining factor (RF) < 1.3), less operating cost, less capital input and less influent to handle (Yusoff and Thiagarajan 1993).

Refining factor (RF) is a parameter used to assess the efficiency of various stages of a refining process. This factor is dependent upon the yield of the product and the quality of the input and it is calculated as:

$$RF = \frac{oil\ \ loss\ \ \%}{FFA\ \ \%}$$

The RF is usually quantified for various stages of refining process individually and monitoring of the RF in the refinery is usually by means of weight calculated from volumetric measurements adjusted for temperature or by using accurate cross-checked flow meters (Leong, 1992).

In general, chemical refining requires more processing stage, more equipment and more chemicals compared to the physical refining. The processing routes for chemical and physical refining are as per illustrated in Figure 2.6.

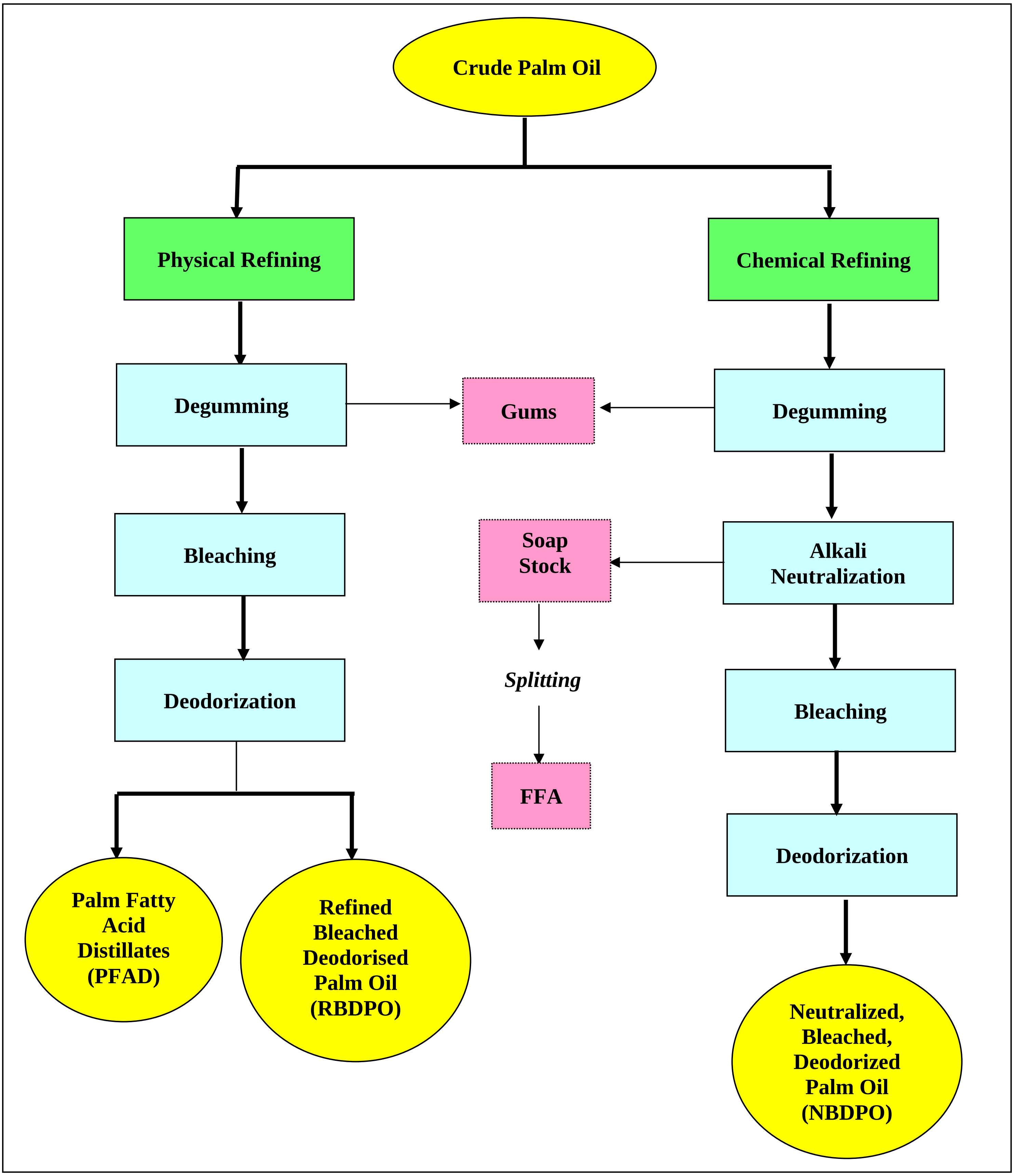


Figure 2.6: Chemical and physical refining routes.

2.5.1 Chemical (Alkaline) Refining

Chemical refining or also known as alkali refining is a conventional method used to refine the crude palm oil (CPO). Its normally involves 3 stages of refining processes namely;

2.5.1.1 Gum Conditioning and Neutralization

In this process stage, the phosphatide portion of oil is either removed or conditioned during this stage by addition of some additives/agents under specific condition. The most common additives for this purpose are phosphoric acid and citric acid.

A specified quantity of any one or mix of such additives/agents is mixed in the oil charge for a specific period and process parameters. This causes a separation of phosphatides from oil and they are removed after certain settling time. Sometimes these separated gums are not directly removed but with soap stock formed during neutralisation.

The oil mass is then neutralised with alkali for removal for free fatty acid in the form of soap stock. This soap stock is removed from oil mass by gravity separation method. For removal of alkali traces, oil is washed with hot water. The chemical reaction involved in this operation is as follows:

$$\mathbf{R\text{-}COOH + NaOH \longrightarrow RCOONa + H_2O}$$

2.5.2.1 Bleaching and Filtration

The neutralised washed oil is then taken to the second step in refining, which is bleaching. In this operation, the oil is taken into cylindrical vessel with agitator called `Bleacher' and kept under vacuum and heated up to 90^{o}C with steam.

The moisture from oil is thus evaporated and oil becomes dry. The dried oil is treated with bleaching earth (fuller's earth) and carbon. These bleaching agents will adsorb most of the residual colour of the remaining oil after neutralisation.

The mixture of oil and bleaching agent is filtered through a standard plate and frame press for separation. The clear oil obtained is bleached oil and is very much lighter in colour than the neutralised oil.

The oil charge is dehydrated under vacuum to avoid any further deterioration due to oxygen. In this operation, the coloured pigments in oil are adsorbed by certain bleaching agents under specific conditions.

2.5.2.3 Deodorisation

The oil after bleaching is practically pure, but contains minute quantities of original odoriferous matter and also the chemicals used during neutralisation process. This bleached oil are then sent to a cylindrical vessel called `Deodoriser'.

The Deodoriser is kept under very high vacuum and the bleached oil is then heated at high temperature 200^{o}C with high-pressure steam and open steam is passed through the oil. The volatile materials are evaporated off with some carrier (commonly direct steam).

This oil is then cooled and clarified through a Filter Press to get sparkling oil. The purpose of deodorisation is to make oil blend and tasteless. In this process, the peroxide value of oil is brought down as minimum as possible.

2.5.2 Physical Refining

Physical refining is a modern alternative for processing crude palm oil where the removal of free fatty acid is by distillation at higher temperature and low vacuum. This replaces chemical reaction mode using an alkali (caustic soda) in chemical refining.

Physical refining is also known as deacidification (deodorisation) by steam distillation in which free fatty acids and other volatile components are distilled off from the oil using effective stripping agent which is usually steam under suitable processing conditions. (Meirelles and Ceriani, 2005).

In physical refining FFA is removed during the final stage; deodorisation, in form of palm fatty acid distillate (PFAD) including the carotenoids pigments, volatiles impurities and oxidation products.

The advantages of this physical refining method over the chemical refining method of palm oil refinery are;

- Better yields
- High Quality of fatty acids as by-products
- Good oil stability
- Simultaneous distillation of fatty acids and deodorization
- Lower cost of equipment
- Simplicity of operation

Figure 2.7 shows the simplified flowsheet of physical refining which consists of pre-treatment stage (degumming and bleaching) and deodorization stage.

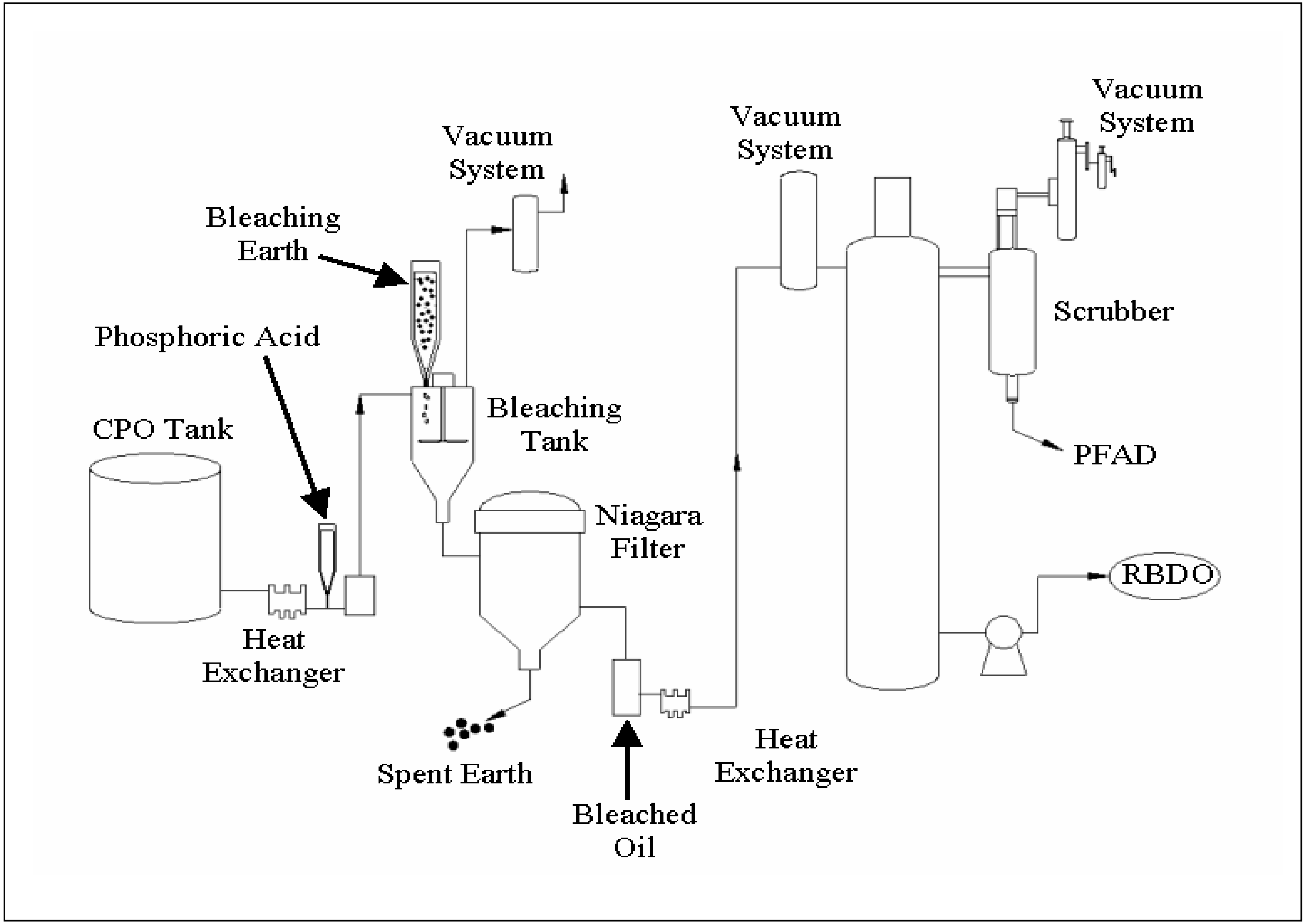


Figure 2.7: Simplified Flowsheet of Physical Refining

2.5.2.1 Pre-treament - Degumming and Bleaching Process

Degumming Process

The main objective of this degumming is to remove the unwanted gums, which will interfere the stability of the oil products in later stage. The objective is achieved by treating the crude palm oil (CPO) with the specified quantity of food grade acid normally phosphoric or citric acid of certain concentration.

The main component of contained in the gums, which has to be removed, is phosphatide. It is vital to remove the phosphatides content in the crude oil because the presence of this component will impart undesirable flavor and color, and shorten the shelf life of oil. The phosphatides emulsifying action is the main culprit that causes the oxidative instability of the crude palm oil (CPO).

In this unit operation, the incoming crude palm oil is first heated up to a temperature of about 90^{o}C - 110^{o}C before it is treated with phosphoric acid. The dosage of phosphoric acid normally used is within the range of 0.05 – 0.1 % of oil weight with acid concentration approximately about 80 - 85 %. It is meant to decompose the non-hydratable phosphatides as well as to coagulate the phosphatides making them insoluble and thus easily removed during bleaching.

Excessive amount of phosphoric acid are need to be avoided as it may cause rise in phosphorus acid and this excessiveness may be difficult to be removed and contribute to further refining problems.

Bleaching Process

During bleaching process in palm oil refinery, degummed oil is treated with bleaching earth and heated up to a temperature of about 100^{o}C before entering the vacuum bleacher. The dosage of acid activated clay used is typically within the range of 0.5 – 2.0 % by weight of oil and the contact time with continuous agitation is about 30 minutes.

During this stage, trace metal complexes such as iron and copper, pigments, phosphatides and oxidation products are removed by adsorptive effect of the bleaching earth. Any residual of phosphoric acid are removed during this stage too. The bleached oil is then filtered on industrial filters such as plate and frame filter press or vertical leaf pressure filter.

2.5.2.2 Deodorisation

The filtered oil (DBPO) is then channelled into the deodorizer for the deacidification and deodorisation treatment. This process utilises a combination of high temperature heating approximately about 240 - 260^{o}C, under vacuum (2 - 4 mmHg) and direct steam injection of about 2.5 - 4.0 % by weight of oil (Leong, 1992).

During this deodorization process, free fatty acid (FFA) in the form of palm fatty acid distillate (PFAD) is removed as refining waste, at the upper section of deodorizer. Apart from FFA, carotenoids pigments, primary and secondary oxidation products are also being removed as it may contribute to off-flavours. The deodorised oil is then cooled before it is filtered by means of a polishing filter and sent to storage tanks.

2.6 Physical Refining Versus Chemical Refining for Palm Oil

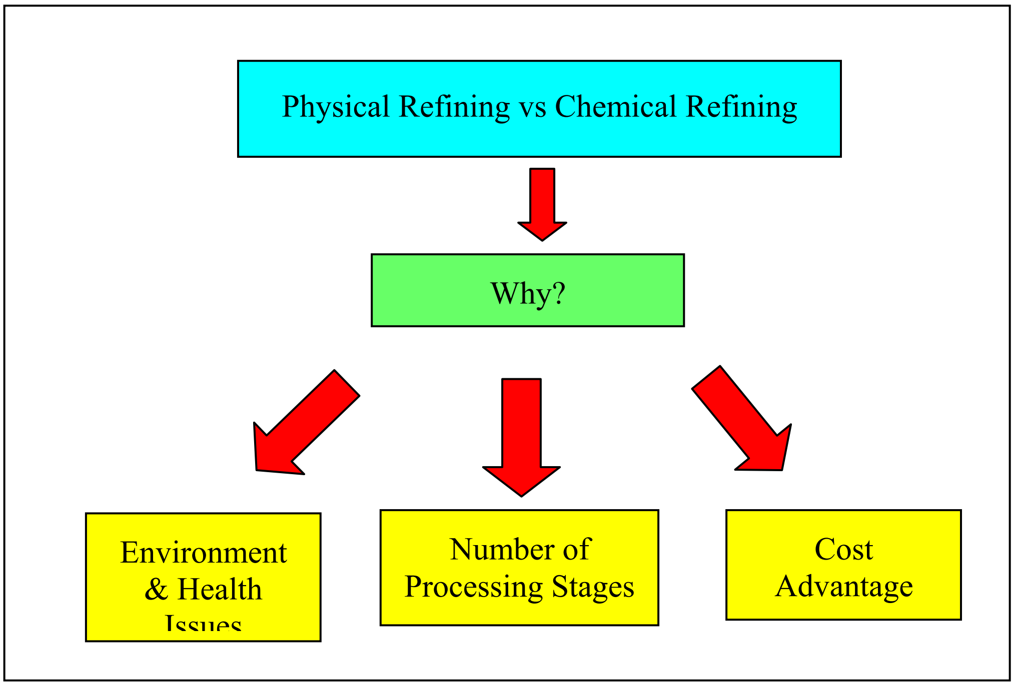


Figure 2.8: The flow chart for physical refining versus chemical refining for palm oil.

2.6.1 Environment & Health Issues

It is likely that when chemical refining were invented, environmental pollution and chemical usage in food processing industry were not become such main issues and thus, the chemical refining steps involved seem to have been fully acceptable. However, as consumers become more concern in health implications when consuming chemically refined oil and the legislation authorities becoming more stringent on environmental pollution, physical refining seems to be a better alternative for food processing (Noor Azian, 1995).

2.6.2 Number of Processing Stages

According to Noor Azian (1995), chemical refining requires more processing stages than physical refining and the processes involved in physical refining are simpler since it involves fewer step. From Figure 2.6, it can be clearly seen that, physical refining requires 4 processing steps to produce refined bleached deodorized palm oil (RBDPO) whereas chemical refining requires 5 processing steps to produce neutralized bleached deodorized palm oil (NBDPO).

2.6.3 Cost Advantage

Fewer processing steps, indicates that operating cost is considerably minimised. Thus, apparently physical refining requires less operating cost compared to chemical refining. Apart from that, chemical refining have to deal with the problem of disposing soapstock, which involves a high energy and high effluent treatment cost. Hence, its make chemical refining least favourable towards palm oil refiners (Noor Azian, 1995).

2.7 Factors Affecting Degumming and Bleaching Processes of Crude Palm Oil

There are several factors affecting the refining process of crude palm oil, the factors that are mentioned here are particularly focussed on the upstream operation of the refining process, which are degumming and bleaching. The factors are, quality of CPO, bleaching time, operating temperature, operating pressure, phosphoric acid dosage and bleaching earth dosage.

2.7.1 Quality of CPO

The quality of the crude palm oil is of utmost importance to the refiners as it relates directly to the processing cost and the product's shelf life (Wong, 1983). This is because refiners are requested to meet stringent specifications. Refiners want a crude palm oil (CPO), which can be easily processed to a bland and light coloured oil with good oxidative and colour stability. All requirements should preferably be obtained at the minimum refining cost, in other words, low oil losses with minimal use of bleaching aids. Therefore, an ideal CPO should posses the quality as shown in Table 2.5 below;

Table 2.5: Ideal quality targets of Crude Palm Oil (Ai, 1990)

Parameters	**Crude Palm Oil (maximum)**
FFA, %	3.5
M & I,%	0.25
Peroxide value, meq/kg	1.0
Anisidine value	5
β-carotene content,ppm	500-800
DOBI	2.5
Phosphorus,ppm	15
Iron (Fe),ppm	5
Copper (Cu),ppm	0.1
Colour (5.25" Lovibond Cell)	-

2.7.2 Bleaching Time

Bleaching time/period is one of the variables that can be controlled although it is often restricted by the production rate constraints. Based on Howes et al. (1991) works, from on Figure 2.9 below, it can be clearly seen that colour and FFA content continue to fall with increasing bleaching earth, but the peroxide value are minimizes after 30 minutes. The continuing fall of colour is attributed to the "heat bleaching effect".

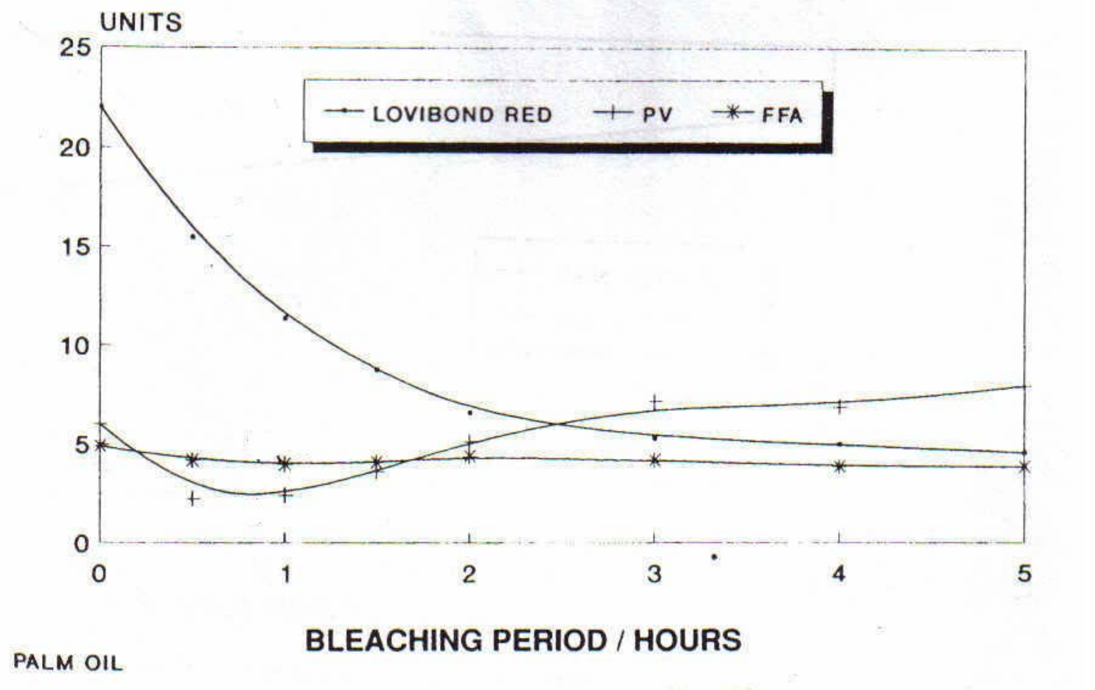


Figure 2.9: Effect of Bleaching Time on Oil Colour, Peroxide Value (PV) and Free Fatty Acid (FFA)

2.7.3 Bleaching Temperature

Bleaching temperature is one of the factors that affecting the performance of bleaching and degumming processes of crude palm oil. Bleaching temperature is discovered to be affecting the colour of oil, the FFA content of the oil and the PV and AV values of the oil.

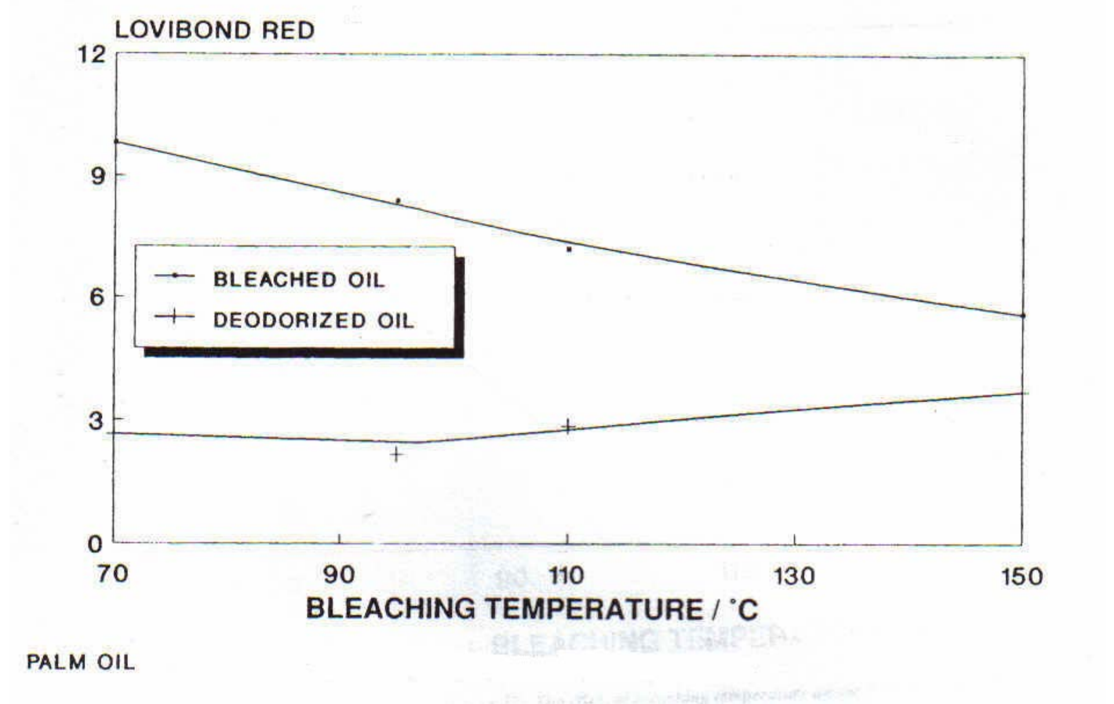


Figure 2. 10: Effect of Bleaching Temperature on Oil Colour

Based on the Figure 2.10, it can be noted that the bleached oil colour tends to continuously fall as the temperature is increased, but the deodorized oil colour reaches a minimum when the bleaching temperature is about 100°C. It is also deduced that, the fall in bleached colour with increasing temperature is due to the heat bleaching effect.

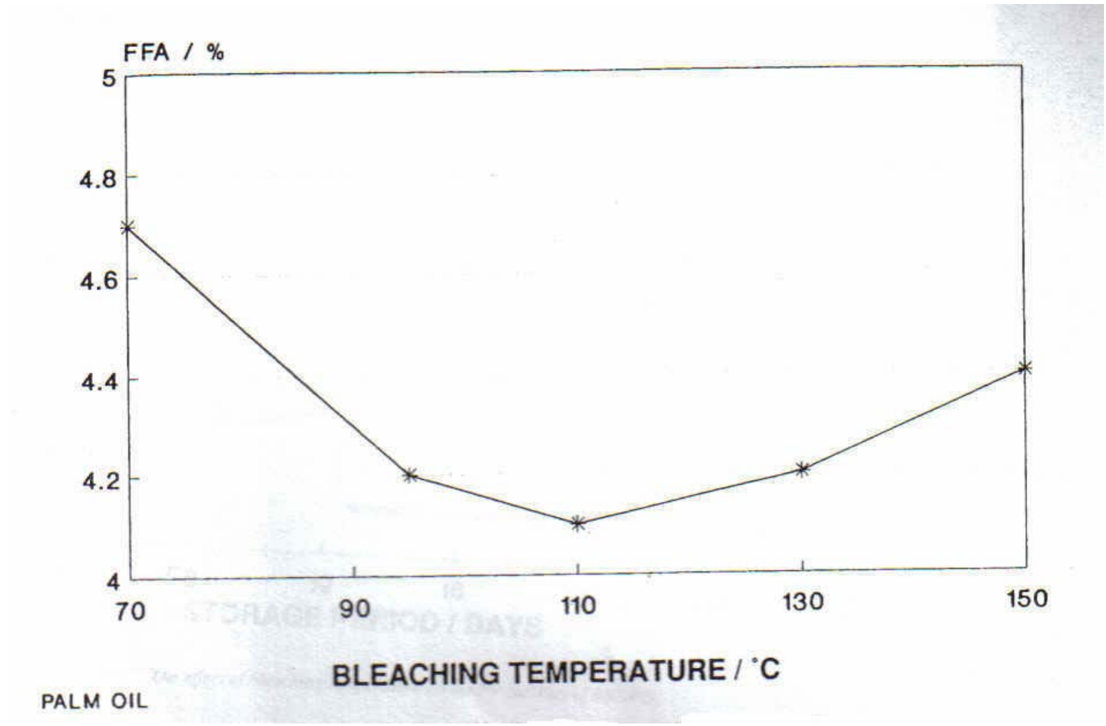


Figure 2. 11: Effect of Bleaching Temperature on Free Fatty Acid (FFA)

From Figure 2.11, it can be clearly seen that the FFA of bleached oil is minimised when the bleaching temperature is about 110°C.

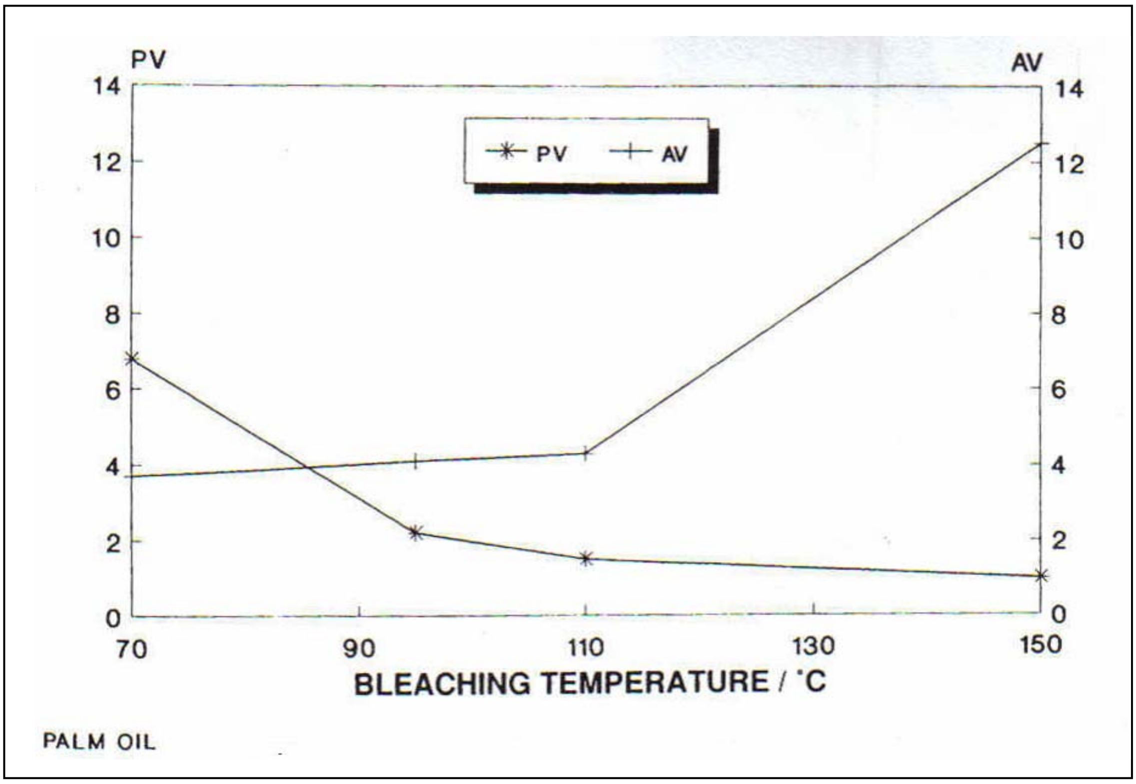


Figure 2. 12: Effect of Bleaching Temperature on PV and AV of Palm Oil

Figure 2.12 shows the effect of bleaching temperature on peroxide value (PV) and anisidine value (AV) of palm oil. From the graph, it can be seen that the peroxide value decreases with increasing bleaching temperature, however it effect is vice versa for anisidine value as the AV value rises rapidly as temperature increases particularly at temperature above 1110^{o}C. This indicates that the secondary oxidation products are not effectively being removed at higher temperature. The total oxidation of the oil is seems to be minimum at temperature about 100^{o}C (Howes et al., 1991).

2.7.4 Bleaching Earth Dosage

Bleaching earth dosage is also one of the factors that can influence the efficiencies of degumming and bleaching processes. This factor is one of the easiest variables to modify.

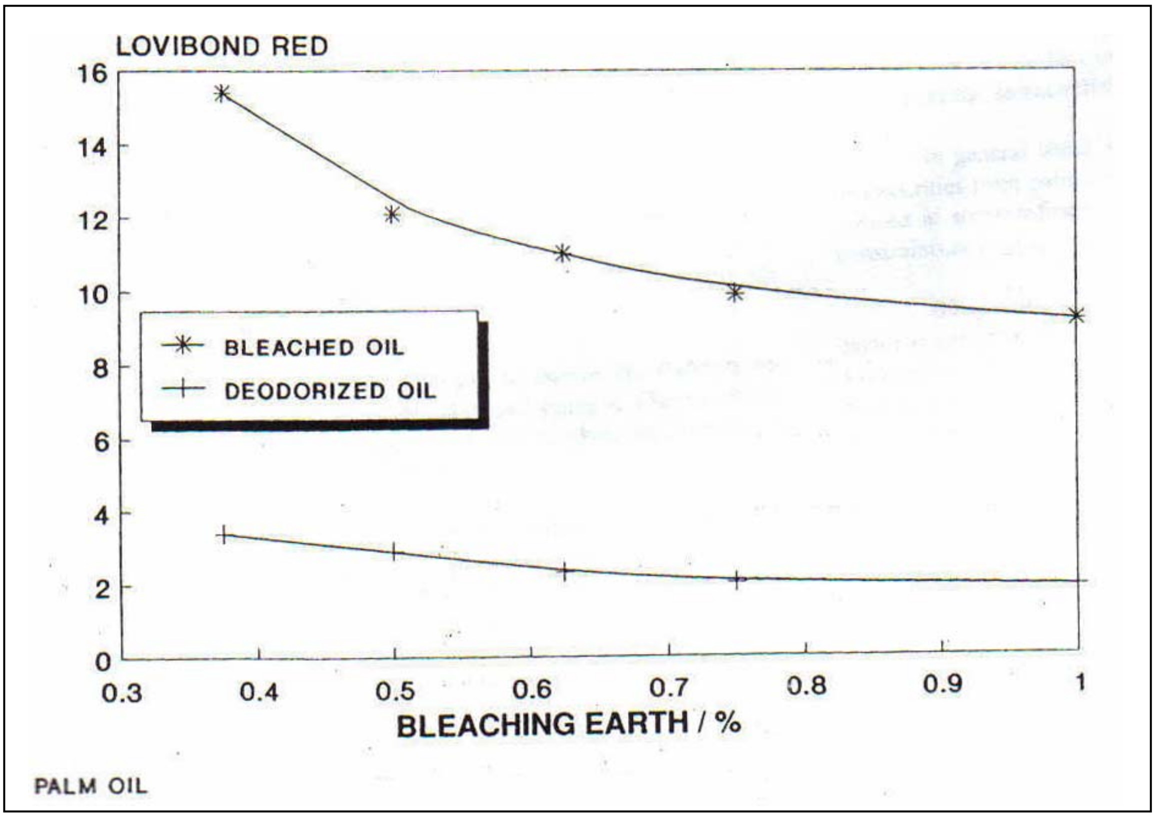


Figure 2. 13: Effect of Bleaching Dosage on Colour of Palm Oil

From Figure 2.13, it can be seen that the colour of bleached and deodorized oil continue to fall with increasing earth dosage. It is claimed that at high bleaching earth dosage, the bleached oil colour reduction is mainly due to the carotene adsorption which could be remove more cost-effectively during deodorization stage (Howes et al., 1991). The optimum earth dosage will depend on the quantity and the nature of impurities in the crude palm oil.

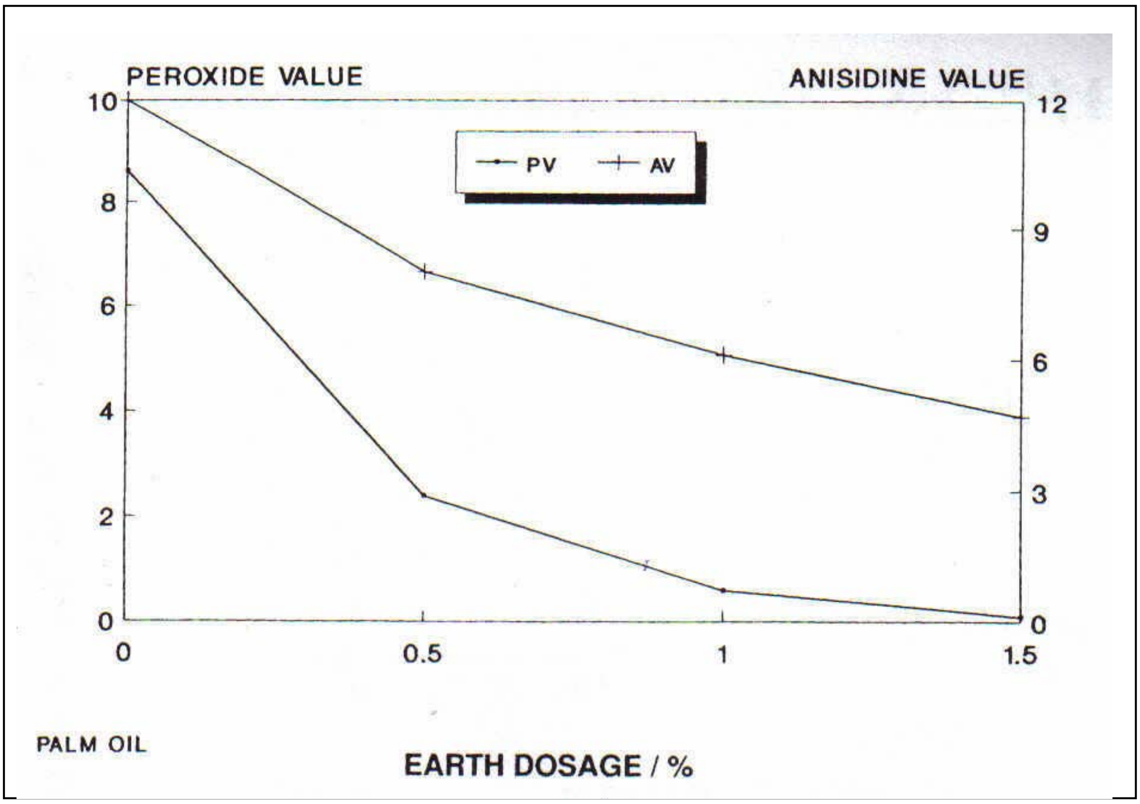


Figure 2. 14: Effect of Bleaching Dosage on PV and AV of Palm Oil

Figure 2.14 shows the effect of bleaching dosage on peroxide value (PV) and anisidine value (AV) of palm oil. Based on this graph, it can be deduced that PV and AV are reduced with increasing bleaching earth dosage. This condition is contrast with the reduction in PV alone when high bleaching temperature is utilized.

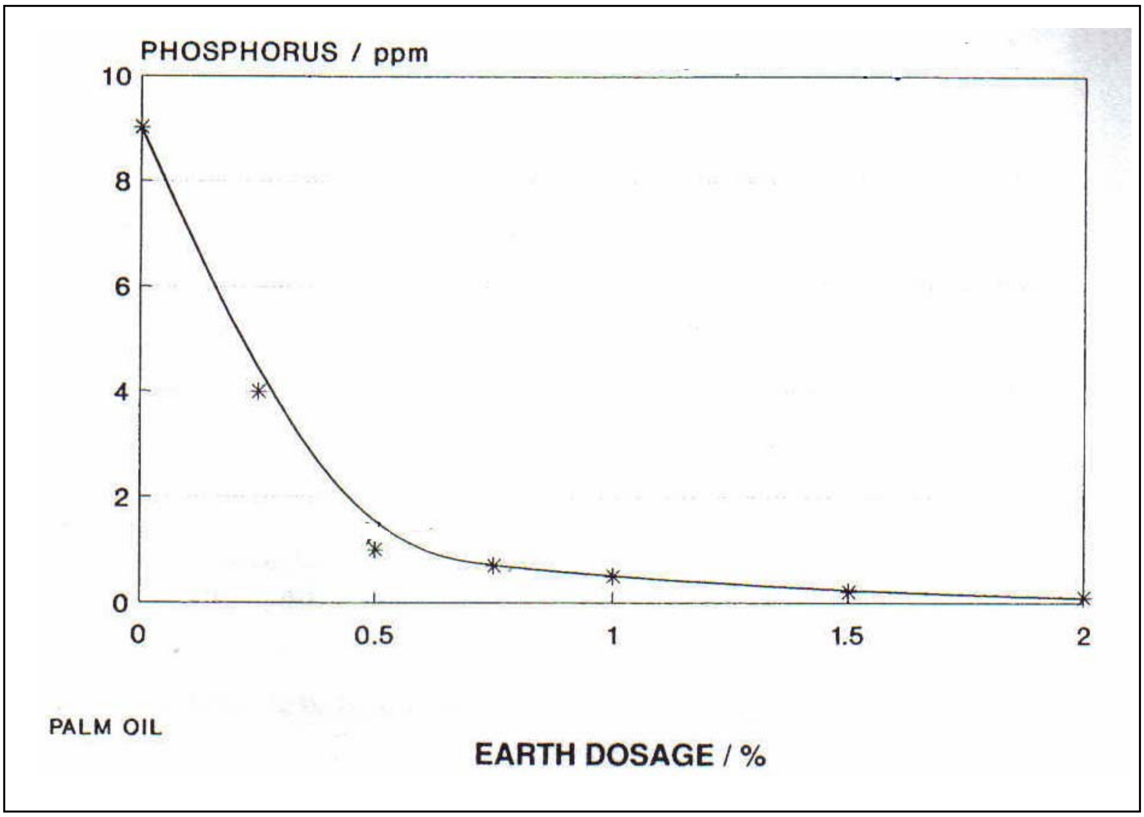


Figure 2. 15: Effect of Bleaching Dosage on Phosphorus Content of Palm Oil

Based on figure 2.15, it is found out that the phosphatides removal are improved by increasing the bleaching earth dosage.

2.7.5 Phosphoric Acid Dosage

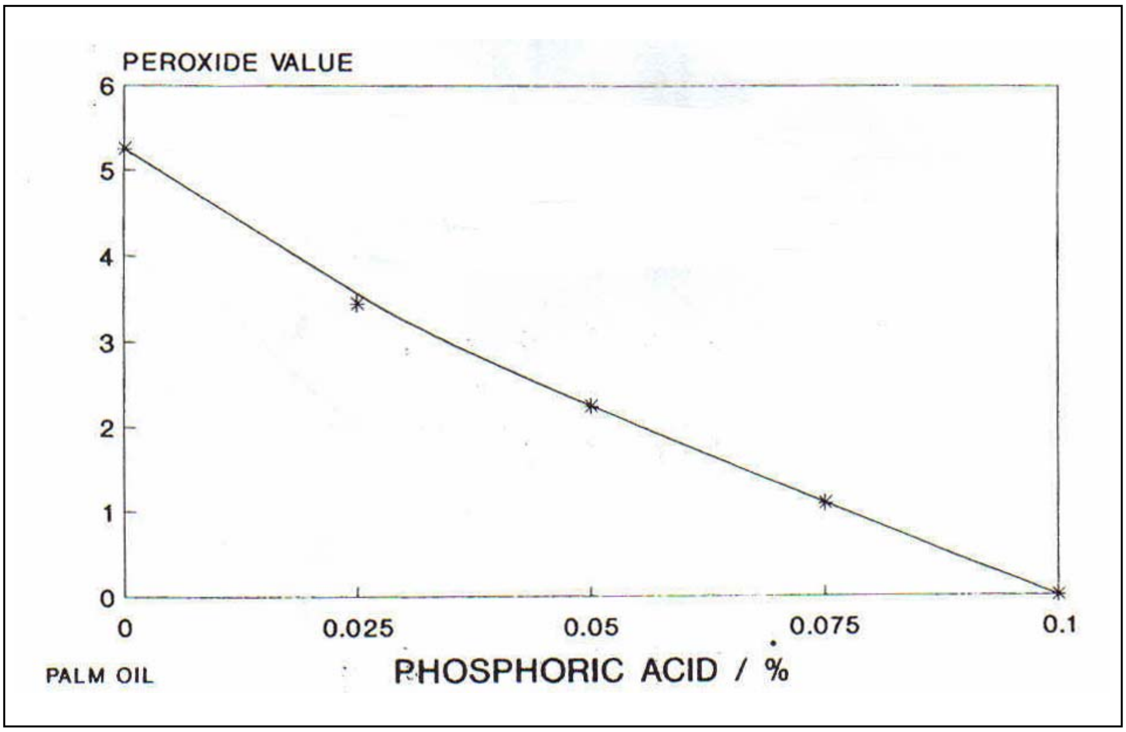


Figure 2. 16: Effect of Phosphoric Acid Dosage on PV of Palm Oil

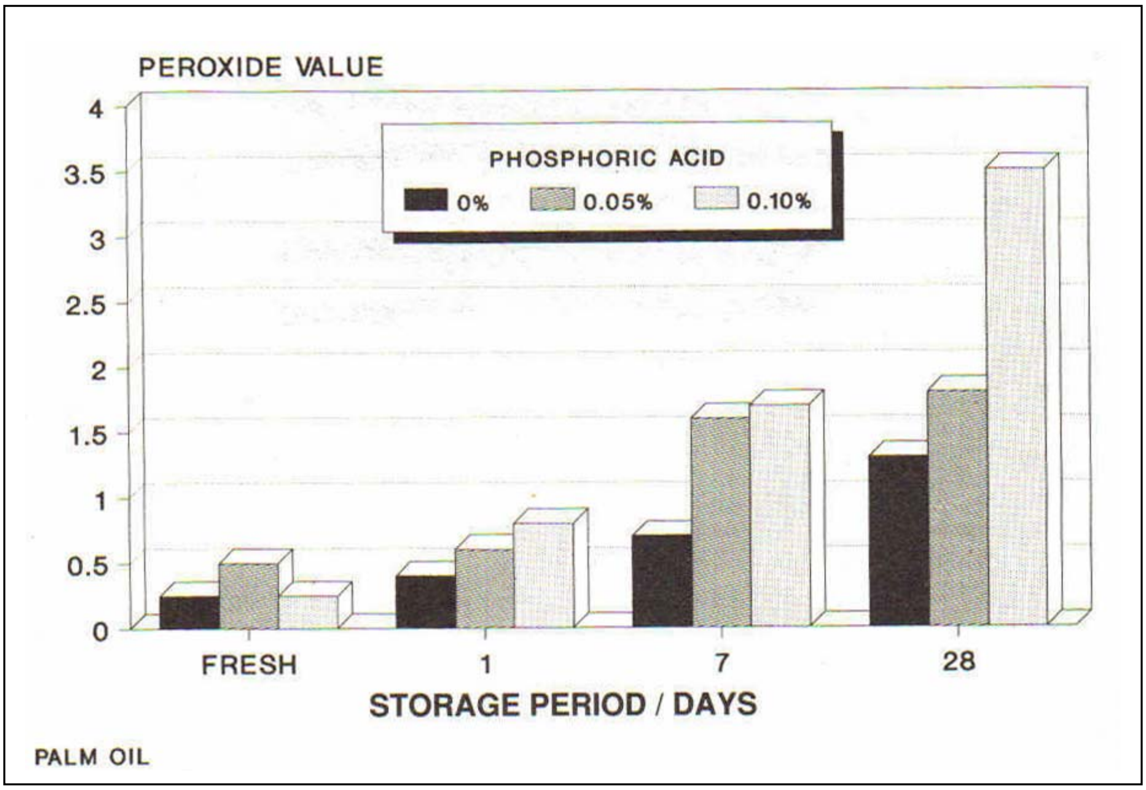


Figure 2. 17: Effect of Phosphoric Acid Dosage on PV of RBDPO

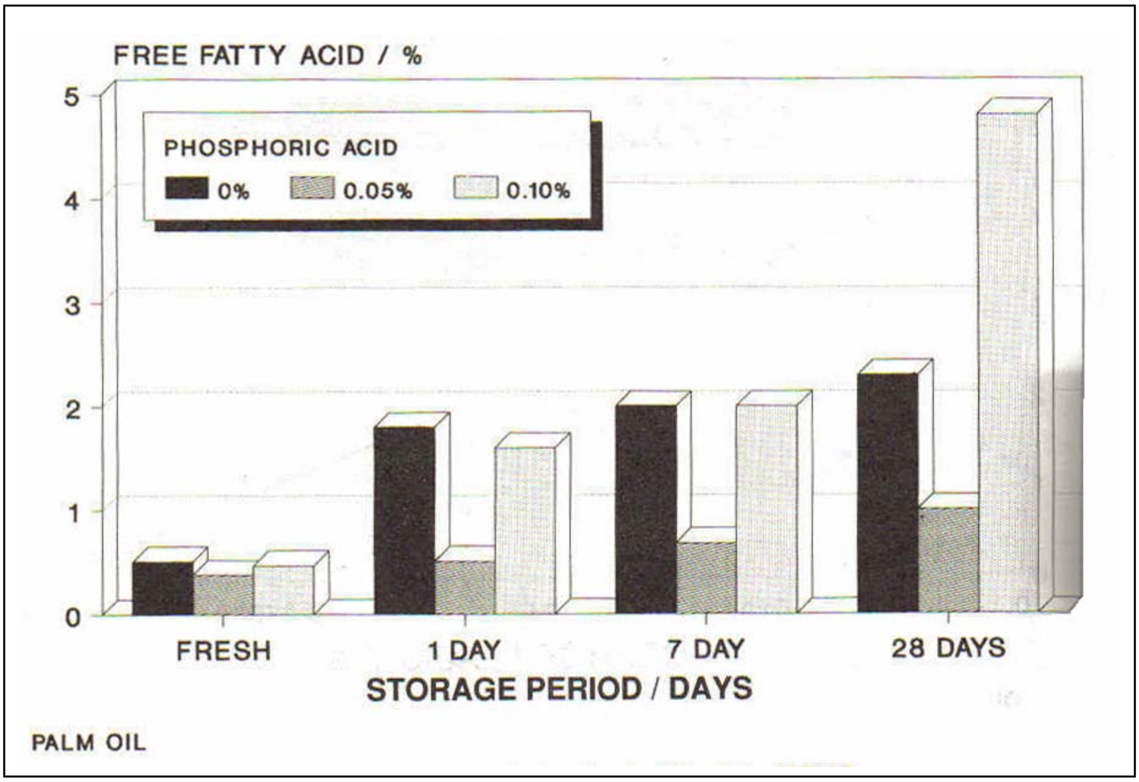


Figure 2. 18: Effect of Phosphoric Acid Dosage on FFA of RBDPO

CHAPTER 3

THEORY OF DEGUMMING AND BLEACHING

3.1 What is Degumming ?

Technically, degumming is referred as an operation of purification of seed oils, which normally contain impurities in the colloidal state or dissolved in them (Bernardini, 1985).

Fats and oils contain complex organo-phosphorus compounds referred to as phospholipids (phosphatides) or more usually, as gums. Phospholipids should be removed because of their strong emulsifying action and if they are not removed, the oil will went through undue darkening during deodorization at high temperature (Kim et al.,2002). The phospholipids (phosphatides) are removed during processing by a variety of treatments collectively referred to as degumming. The treatment usually involves hydration with water,orthophosphoric acid, and polybasic organic acids either singly or in combination, followed by centrifuging the precipitated material or by its adsorption on bleaching earth or filter.

In more simple words, degumming is a process of removing the unwanted gums, which will interfere the stability of the oil products in later stage. The objective is achieved by treating the crude palm oil (CPO) with the specified quantity of food grade acid normally phosphoric or citric acid of certain concentration**.**

3.1.1 Types of Degumming

There are 6 types of degumming process in vegetable oil industry. The differences between all these types are based on methods of processing, chemicals used and the content of phosphatides in the crude vegetable oil. The types of degumming process are ;

3.1.1.1 Dry Degumming

Dry degumming process involved removal of gums through precipitation by acid conditioning and via filtration during the bleaching process, not via centrifugal separation. This process is used for low-phosphatides oil such as palm oil, lauric oils, edible tallow and suitable to be used for preparing oils for subsequent physical refining. This type of process eliminates bleaching, as separate processing step thus, it is cost-advantage and it is a well-proven process. Figure 3.1 below shows the flow diagram of water-degumming process.

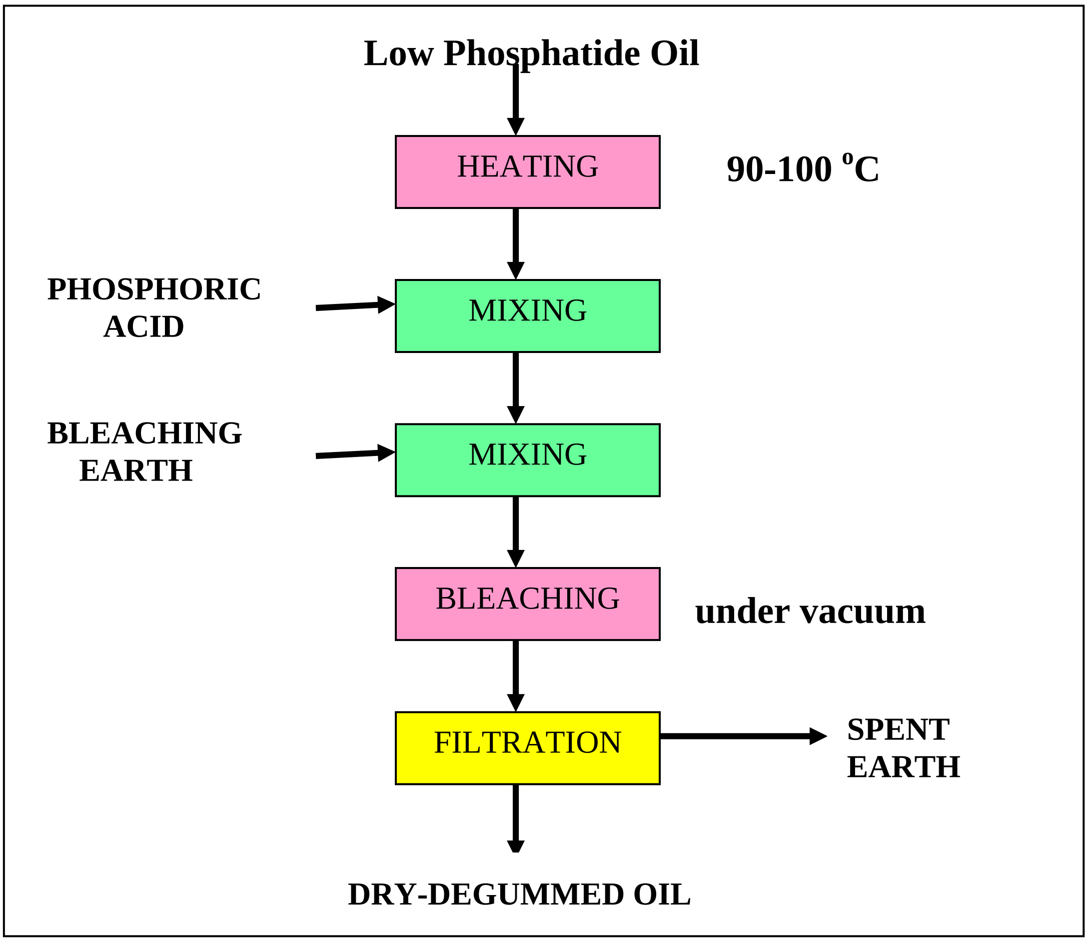


Figure 3.1: Flow diagram of dry degumming process

3.1.1.2 Water Degumming

Water degumming is a process of removing gums through precipitatation by pure water hydration of crude oil via centrifugal separation. This method is used when extracting gums for production of lecithin, soybean oil and for crude oil with 200ppm phosphorus content. In this process, water is the main agent used to remove the hydratable phosphatides from vegetable oils and it can be carried out in batch or continuous procedure depending on the type of the oil the be degummed and amount of oil to be processed.

This process is insufficient to be used for subsequent physical refining. Figure 3.2 below shows the flow diagram of water-degumming process

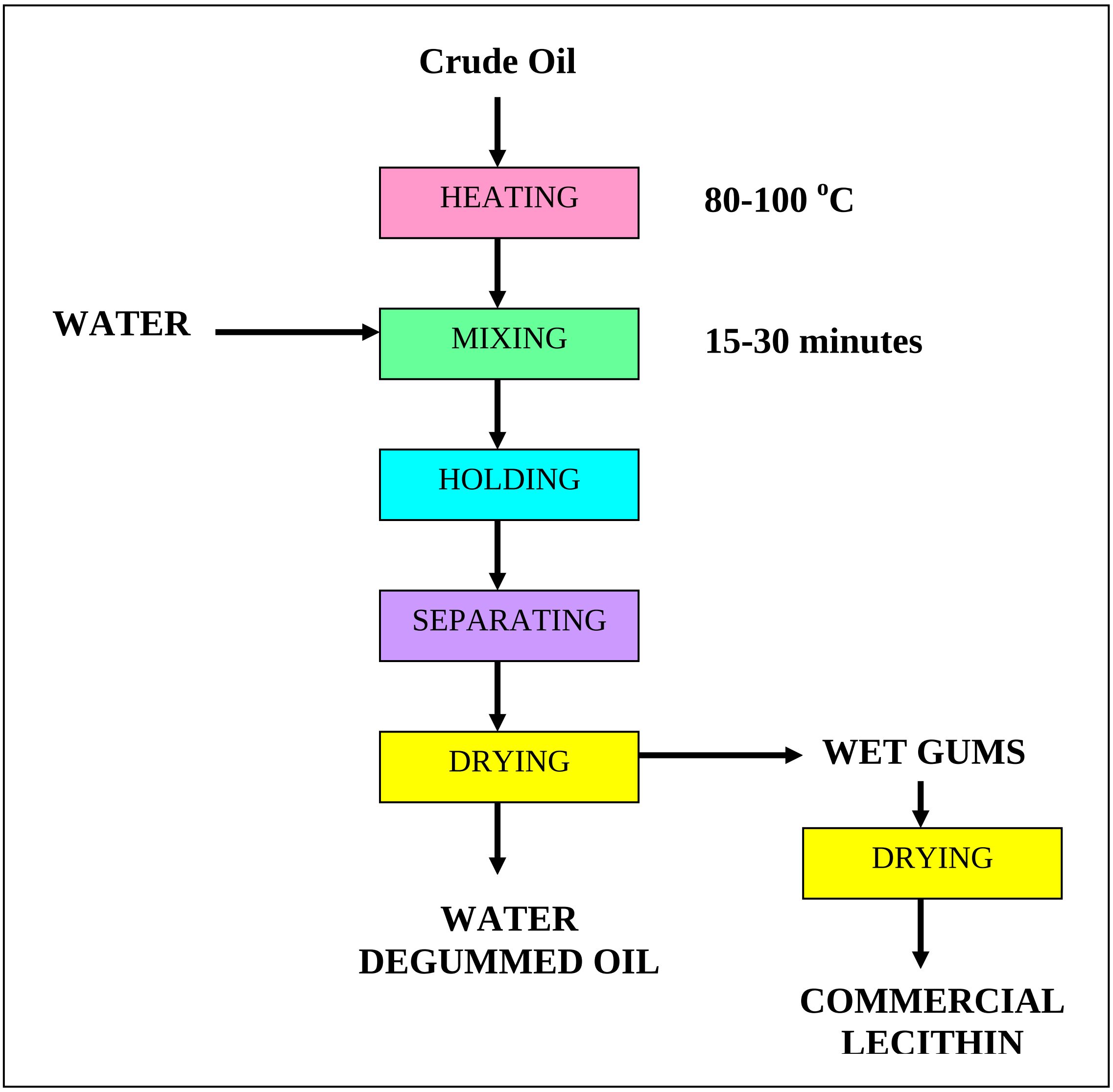


Figure 3.2: Flow diagram of water degumming process

3.1.1.3 Acid degumming

In this acid degumming process, gums are precipitated by some form of acid conditioning process and subsequently removed by centrifugal separation. In this process method, the gums can be hydrated at temperature higher than 40 °C and the process may lead to some dewaxing which usually associated with processing of sunflower and rice bran oils. In organic refining process, dilute organic (citric) acid is normally used and the removal of residual phosphatides is by bleaching using silica hydrogel.

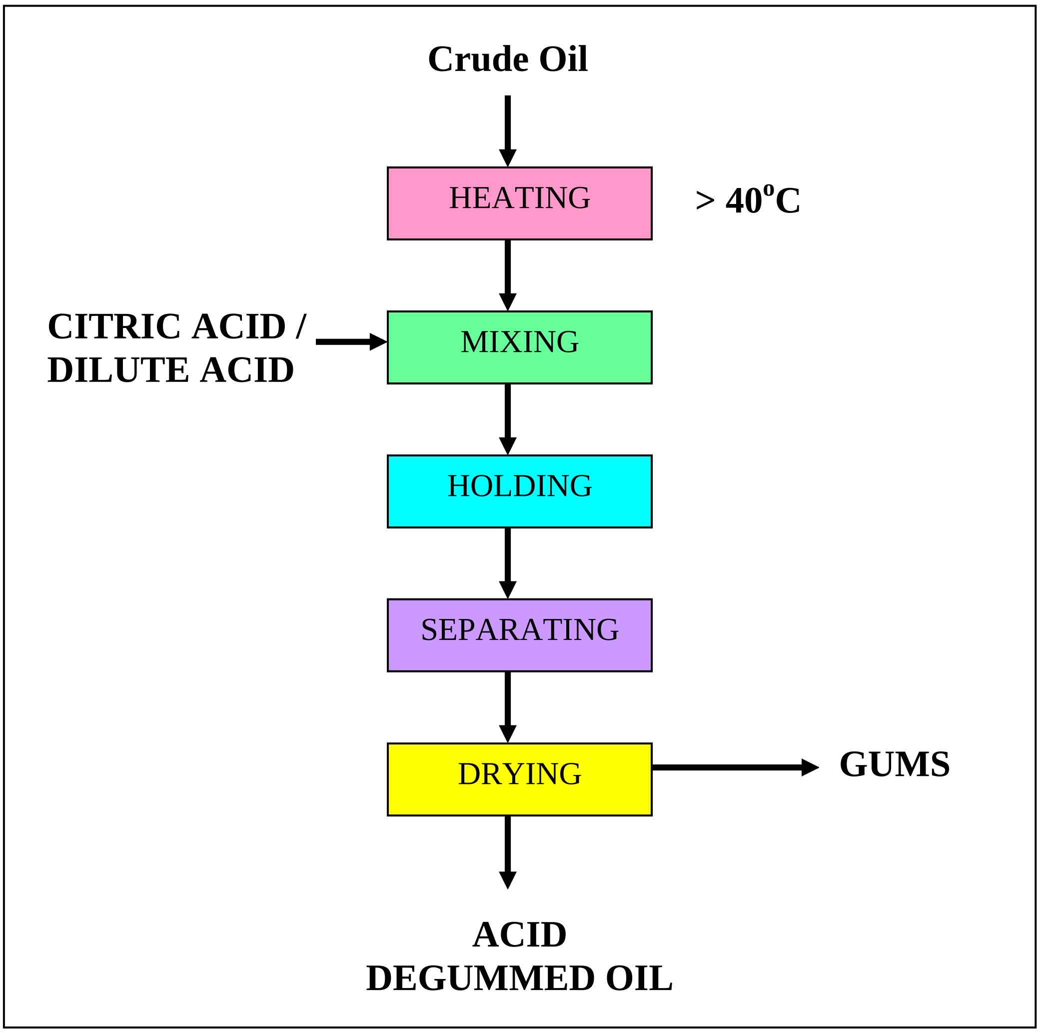


Figure 3.3: Flow diagram of acid degumming process

3.1.1.4 Enzymatic degumming

Enzymatic degumming is a special degumming that enhanced by using some food-grade enzymes. Types of oil that uses this process method are soybean oil and rapeseed oil. The advantage of enzymatic degumming is no soapstock is produced so no oil losses due to soapstock separation.

3.1.1.5 EDTA- degumming

EDTA degumming is a physico-chemical degumming process. It involves a complete elimination of phospholipids by a chelating agent, Ethylene Diamine Tetraacetic Acid (EDTA), in the presence of an emulsifying additive.

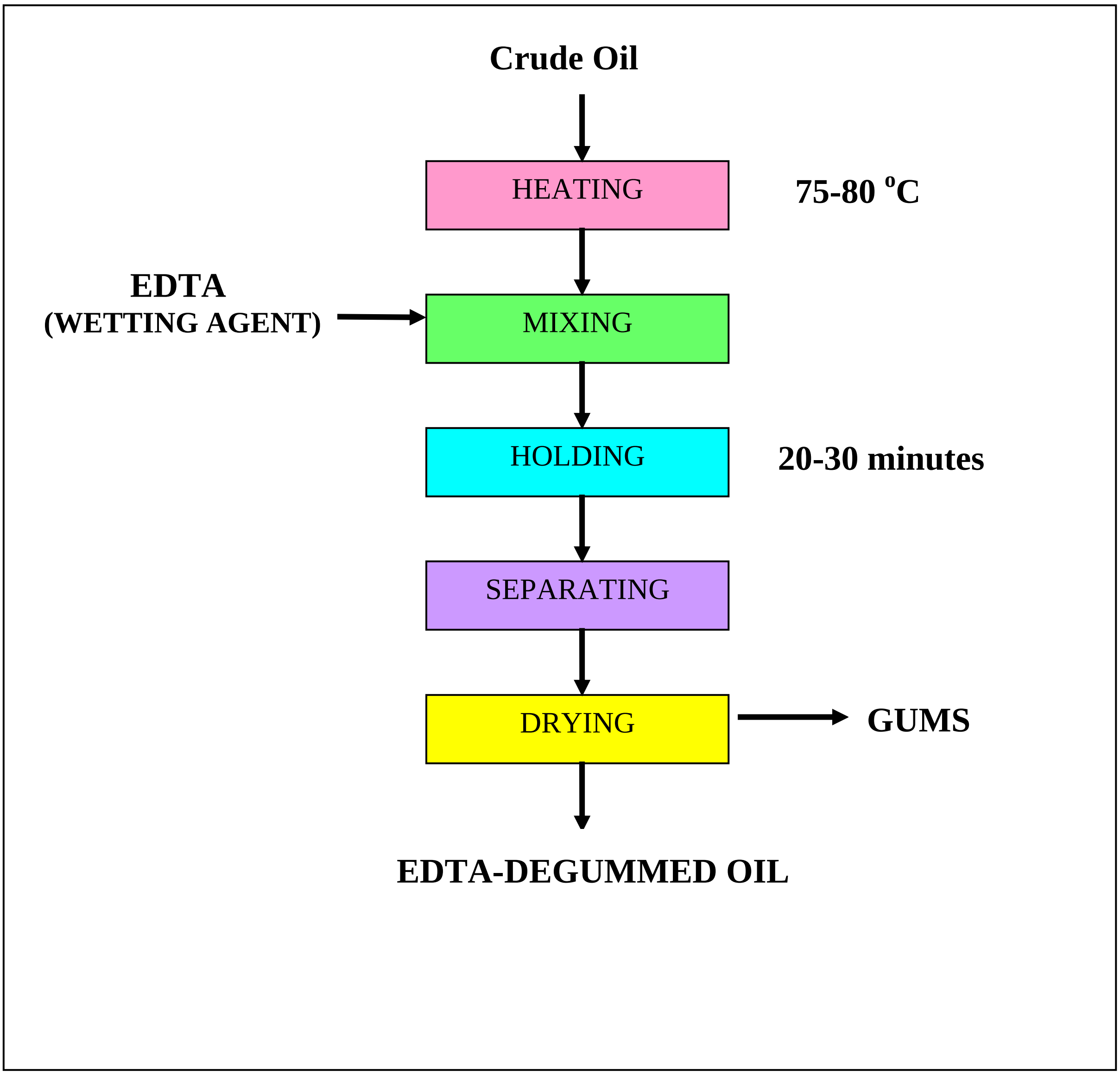


Figure 3.5: Flow diagram of EDTA-degumming process

3.1.1.6 Membrane degumming

Membrane degumming process is usually used in extraction plant. According to Lin et al. (1997), membrane separation is primarily a size-exclusion-based pressure-driven process. It separates different components according to the molecular weights or particles sizes and shapes of individual components and dependent on their interactions with membrane surfaces and other components of the mixture.

During oil processing, miscella, which contain 25-30% of crude oil and 70-75% hexane are obtained from extraction prior to solvent removal. Phospholipids can be separated from triglyceride in the miscella stage using appropriate membrane.

The membrane-based crude oil degumming produces permeate and retentate containing triglyceride and phospholipids, respectively. The majority of the coloring materials and some of the FFAs and other impurities are included in phospholipids micelles and removed as well (Lin et al., 1997).

This processing method is typically used for cottonseed oil. Figure 3.6 below shows the flow diagram of membrane degumming of crude vegetable oils.

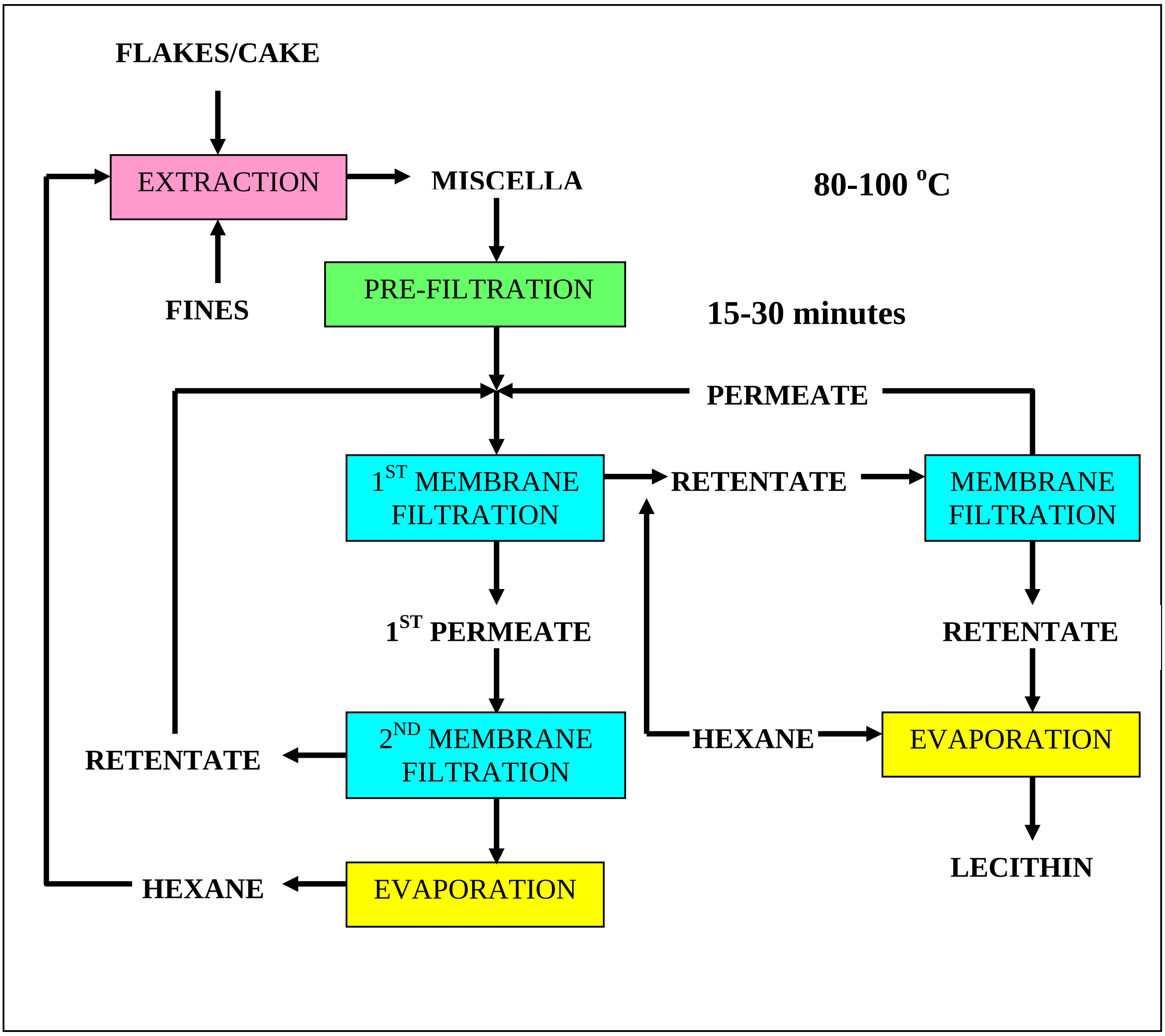


Figure 3.6: Flow diagram of membrane degumming process

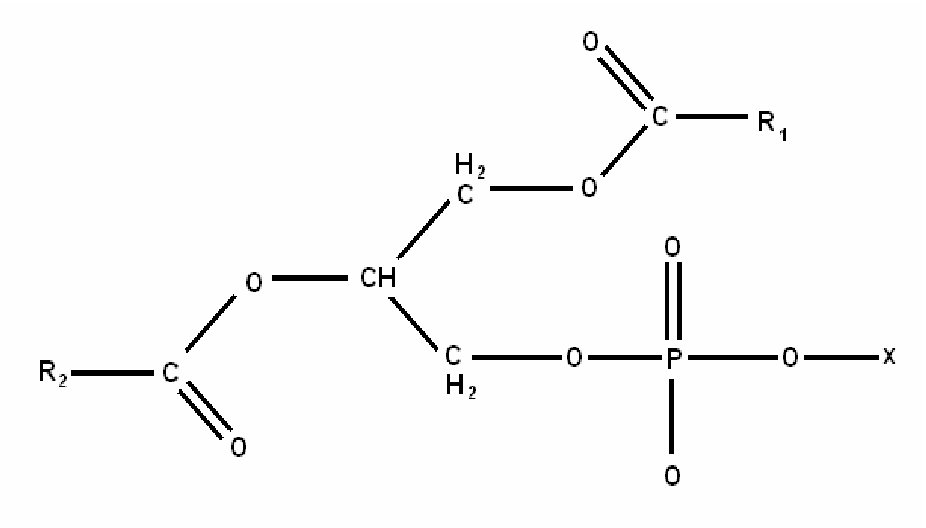
3.1.2 Process Theory of Degumming

Theoretically, phospholipids, proteins and carbohydrates, vegetable gums and colloidal components have negative influence towards the keepability of oil. They are considered as undesirable substances in refining because they increase the oil loss and hamper other operations. Therefore, oils that have certain amount of these substances should be degummed in order to remove all those substances.

There are 2 kinds of phospholipids exists, those that hydratable and those that cannot be hydrated (non-hydratable phosphatides -NHP). Hydratable phospholipids can be removed easily by the addition of water where the process can be conducted rapidly at elevated temperature or slowly at low temperature. However the temperature should stay below the temperature at which the phospholipids hydrate starts to become liquid crystals (usually ~ 40 ^{o}C). By taking up water, phospholipids lose their lipophilic character and become lipophobic and thus precipitate from oil (Bockish, 1998).

Whereas, for non-hydratable phospholipids, its have to be converted to hydratables ones. The conversion of non-hydratable phospholipids to hydratable is done usually through acidulation followed by neutralisation. Traditionally, acids that are being used are usually sufficiently strong to hydrate phospholipids without hydrolyzing the triglycerides. At present, citric or phosphoric acid is normally being used for any type of vegetable oil. However, phosphoric acid is more preferred by the palm oil refiners in Malaysia because of lower unit cost and easier handling (Thiagarajan and Tang., 1991).

The main component of phospholipids is phosphatides. Figure 3.7 shows the chemical structure of phosphatides.



X = choline (phosphatidyl choline or PC)
X = ethanolamine (phosphatidylethanolamine or PE)
X = inositol (phosphatidylinositol or PI)
X = hydrogen (phosphatidyl acid or PA)

Figure 3.7: Chemical structure of Phosphatides

Composition of phospholipids of palm oil is shown in table 3.1 below;

Table 3.1: Composition of Phospholipids of Palm Oil (mole %)

Phospholipid	Percentage
Phosphatidylcholine (PC)	36
Phosphatidylethanolamine (PE)	24
Phosphatidylinositol (PI)	22
Phosphatidylglycerol	9
Disphosphatidylglycerol	4
Phosphatidic Acid (PA)	3
Lysophosphatidylethanolamine	2
Phosphatidylserine	trace
Lysophosphatidylcholine	trace

Phospholipids are present in relatively small quantities of about 5 – 130 ppm in palm oil as compared with other vegetables oils. Sambanthamurthi et al., (2000) mentioned that the solvent extracted mesocarp oil usually contained 1000-200 ppm phospholipids , however it only present at level of 20-80 ppm in commercial crude palm oil.

Phospholipids have been reported to show antioxidant affects. Their antioxidant-synergistic effects can be attributed to the sequestering of soluble pro-oxidant metal ions to form inactive species. Hudson and Maghoub also showed a synergism between phospholipids and naturally occurring antioxidants such as α-tocopherol and quercetin. Hydratable insoluble metal ions could also dispersed by phospholipids through miscellar action. Since phospholipids and glycolipids cause reverse micelle, vesicle or emulsion droplet formation, phospholipids can remove metal ions and their hydrophilic salts from the lipid phase to reduce oxidation.

3.1.3 Degumming Agents

There are two types of degumming agents that are usually being used in palm oil refining industry, which are phosphoric acid and citric acid.

3.1.3.1 Phosphoric acid

Phosphoric acid (H_3PO_4), is a colourless and odourless liquid. A food grade phosphoric acid with concentration of 85% is normally used palm oil refining process. It a colourless and odourless liquid .

3.1.3.2 Citric acid

3.1.4 Phosphoric Acid Versus Citric Acid as Degumming Agent for Palm Oil Refining

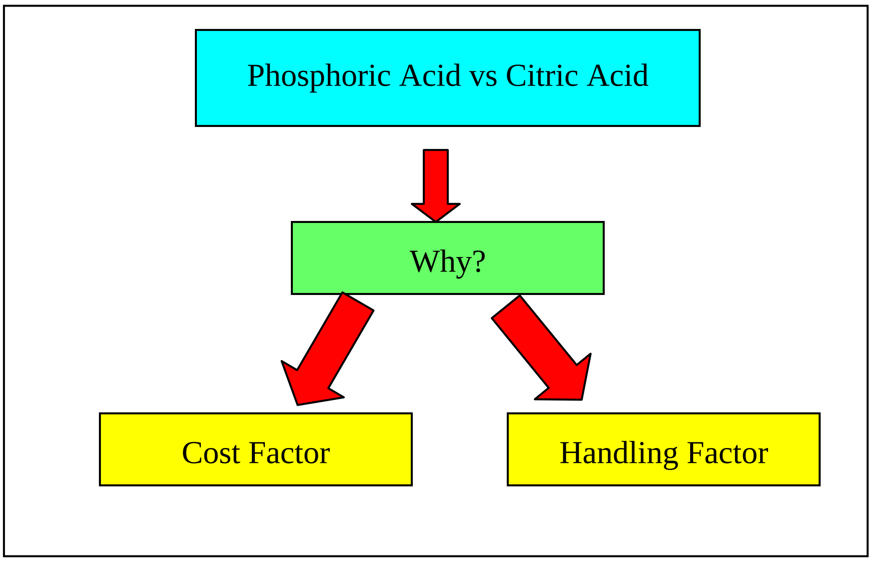


Figure 3.9: The flow chart for phosphoric acid versus citric acid as degumming agent for palm oil refining

3.1.4.1 Cost Factor

Cost factor is vital to be considered in running a refinery plant. Price of citric acid at the current market is much higher than the price of phosphoric acid. Nowadays, 1 MT of phosphoric acid cost abit RM 3000 whereas citric acid cost about RM 3400 per MT.

3.1.4.2 Handling Factor

3.2 What is Bleaching?

The term bleaching refers to the treatment that is given to remove colour-producing substances and to further purify the fat or oil. The usual method of bleaching is by adsorption of the colour producing substances on an adsorbent material. There a lot of adsorbent materials are being used in vegetable oil industry for examples; acid activated bleaching earth, natural bleaching earth, activated carbon and synthetic silicates.

Acid activated bleaching earth (fuller’s earth) or clay, sometimes called bentonite, is the adsorbent material that has been used most extensively. This substance consists primarily of hydrated aluminium silicate. Usually, bleaching earth does not remove all the colour producing materials, much of which are actually removed by thermal destruction during the deodorization process. Activated carbon is also used as a bleaching adsorbent to a limited extent.

3.2.1 Types Bleaching Methods

There are 3 types of bleaching methods can be used in edible oil industry (Gunstone and Norris, 1983) , namely :

3.2.1.1 Heat bleaching

Some pigments, such as the carotenes become colorless if heated sufficiently. However this will leaves the pigment molecules in the oil and may have adverse effect on oil quality. According to Gunstone and Norris, if this oil come into contact with air colored degradation products such as chroman-5,6-quinones from γ-tocopherol present, may be formed. These are very difficult to remove.

3.2.1.2 Chemical Oxidation

Some pigments for example carotenoids are made colorless or less colored by oxidation. But such oxidation invariably affects the glycerides and destroys natural antioxidants. Consequently, it is never used for edible oil but restricted to oils for technical purposes, such as soap-making

3.2.1.3 Adsorption

Adsorption is the common method usually used for bleaching the edible oil by using bleaching agents. Examples of bleaching agents are bleaching earths, activated carbon and silica gel. Bleaching agents normally posses a large surface that has a more or less specific affinity for pigment-type molecules, thus removing them from oil without damaging the oil itself.

3.2.2 Bleaching Process Theory

"Bleaching" process is always being misunderstood as just a mere process of decolourisation of oil and fats. In fact, "bleaching" is a process of selective removal of pigments and impurities by the physical and chemical (chemisorptions) interaction of an adsorbent with an oil or fat to improve its quality (Brooks, 1999). This process refers to the art of removing not only the colour pigments but also dirt, trace metals and various organic impurities that promote oxidation. Another term that can be used to describe bleaching process is purification process of vegetable oils.

Effective adsorption requires a large surface and practically high specific surface area (m^2/g) of a very porous adsorbent is should be used. The channels by which molecules reach this surface must be negotiable by the molecules concerned. The nature of the process must allow acceptably firm bonds, chemical or physical, between it and the adsorbate (Patterson, 1992).

Therefore, in order have maximum attainment of bleaching performance, an efficient bleaching earth is produced which having surfaces of the correct chemical composition and pore distribution selectively attractive to the detrimental components present in crude triglyceride oils.

Mathematically, bleaching or purification process follows the Freundlich adsorption isotherm. Adsorption isotherm is the equilibrium relationships between the concentration in the fluid phase and the concentration in the adsorbent particles at a given temperature. For the cases involving liquids, the concentration is often expressed in mass units such as part per million (ppm). The concentration of adsorbate on the solid is given as mass adsorbed per unit mass of original adsorbent.

Figure 3.11 below shows arithmetic graphs of some typical isotherm shapes (McCabe, 1993). Linear isotherm indicates the amount of adsorbed is proportional to the concentration in fluid. Favourable process is achieved when the isotherms are convex upward because a relatively high solid loading can be obtained at low concentration in the fluid. The isotherm for this favourable adsorption process is Langmuir Isotherm. The assumption made when deriving the Langmuir isotherm is that the process happens at uniform surface. However this relation only works well for gases that a weakly adsorbed (McCabe, 1993).

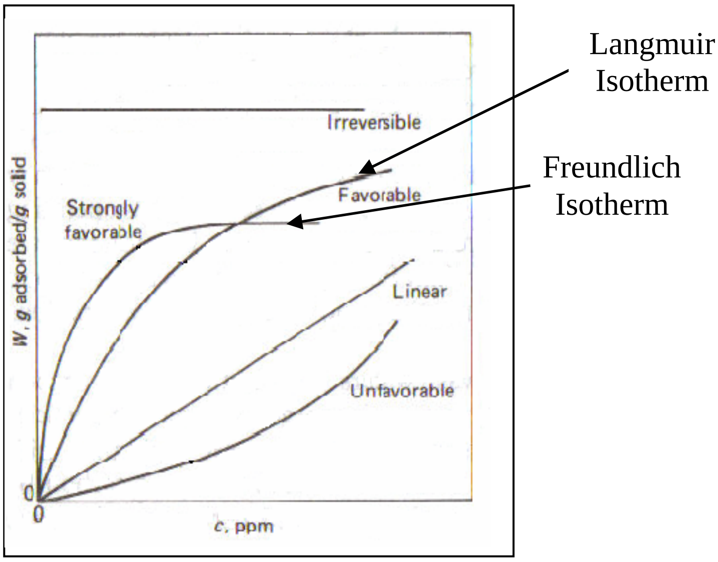


Figure 3.11: Adsorption Isotherms (McCabe, 1993).

For adsorption from liquids, Freundlich isotherm is applicable. This isotherm is of the strongly favourable type. Bleaching of palm oil falls under this type of isotherms as the bleaching process are involving liquids (oils). The equation for Freundlich isotherm for bleaching process can be illustrated as per below (Rossi et al., 2003);

$$\frac{x}{m} = Kc^{n} \qquad \text{.....3.1}$$

Where x = quantity of substance adsorbed

m = quantity of adsorbent

c = quantity of residual substance dissolved

K and n = constants unrelated to the amounts of solute and adsorbent

This Freundlich equation relates the specific adsorption (*x/m*), i.e the amount of substance adsorbed per unit of adsorbent weight, to residual solute concentration (*c*), at the equilibrium for a given temperature ((Rossi et al., 2003).

Equation 3.1 can be also expressed in logarithmic form;

$$\log\frac{x}{m} = \log K + n\log c \qquad \ldots..3.2$$

When graph of (*x/m*) vs. *c* is plotted, a straight-line graph is obtained with a slope equal to *n* and an intercept equal to *K*. The value of *K* determines the adsorption capacity of the adsorbent for specific solute whereas the value of *n* determines the ranges of bleaching in which adsorbent show its greatest effect. According to Rossi et al., if the *n* is high, the adsorbent will be effective for removing the first portions of colour but less efficient for reaching highest bleaching degree and if *n* is low it is vice versa. The value of *K* and *n* depend on the kinds of adsorbent and oil and also the operating conditions of bleaching process.

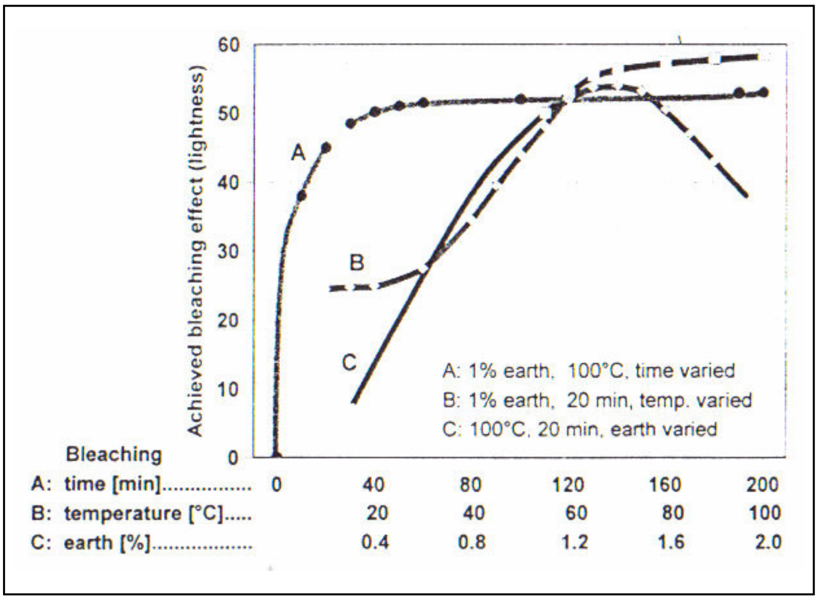


Figure 3.12: Bleaching effect of an oil dependent on different processing parameters (Patterson, 1992)

3.2.3 Mechanisms of Bleaching Process

Oils and fats are bleached in order to remove undesired colorants because these colorants can negatively affect the taste of the oil and in part because the colour would disturb the consumers, therefore on the whole, these colorants limit use and marketability. In addition to that, some particles or pigments that promote deterioration to oil quality is also being removed during bleaching process mainly due to their pro-oxidative properties that promotes oxidation (Bockish, 1998).

During bleaching or purification, the oil is brought into contact with a surface–active adsorbent, and then the undesired particles or other components are selectively retained on the pore surface and triglycerides escapes. Gradually, the concentration of undesired particles on the available surface-active of the adsorbent and the concentration remaining in the oil come into balance, so further exchange is negligible.

Best temperature for oil/adsorbent (clay) must be chosen, as well as duration of contact because an excess of either factor will impart undesirable side effects to the process. In order to have efficient use of adsorbent, any material like gum or soap should be removed at earlier stage (degumming) as the presence of these particles will compete for room on the adsorbent surface. That explains why an effective degumming process is required in refining process of vegetable oils.

Theoretically, coloured particles (or substances) that should be removed during bleaching are present in the oil either in dissolved or in a colloidal form. For both types, the process reaction happens at the surface of bleaching agent.

3.2.3 Bleaching Agents

There are few types of bleaching agents that are being used in vegetable oil industry such as acid activated bleaching earth, natural bleaching earth, activated carbon, synthetic silicates and synthetic resins.

3.2.4.1 Acid Activated Bleaching Earth

In general, bleaching earth is a decolourising agent, which will change the tint of any coloured oil to a lighter shade by changing the basic colour units in oil, without altering the chemical properties of the oil. Bleaching earths are normally used in the bleaching of vegetable oils and in the refining of mineral oils.

While some of these earths are naturally bleaching, some have to be treated with mineral acids. This activation results in the replacement of some of the aluminium ions by hydrogen ions from the acid. This further results in the creation of net negative charges on the clay lattice structure and creates the cation adsorption properties of the acid-activated bleaching earths clays. (Hymore, 1996)

According to Howes et al. (1991), acid activated bleaching earths (Figure 3.13) are in general produced from naturally occurring high-purity montmorillonite clays. The structural features of the clay are modified by treatment with acid. During this process the physical structure and chemical composition are altered in a controlled way to maximize specific properties.

Bleaching earth works based on its character of adsorption and ion exchange. The adsorption process is influenced by some factors as follow :

- particle size
- adsorbent polarity
- surface area
- pore volume
- pore size.

Figure 3.13: Acid Activated Bleaching Earths

3.2.4.2 Natural/Neutral Bleaching Earth

Natural bleaching clays are found in special strata and are naturally active. This material is also used for bleaching. According to Rossi et al. (2003), they are excellent metal adsorbents as they are able ;

(i). to decrease the levels of chlorophyll and colour bodies
(ii). to remove soaps and phospholipids
(iii). to minimizes free fatty acid increase during bleaching.

3.4.2.3 Activated carbon

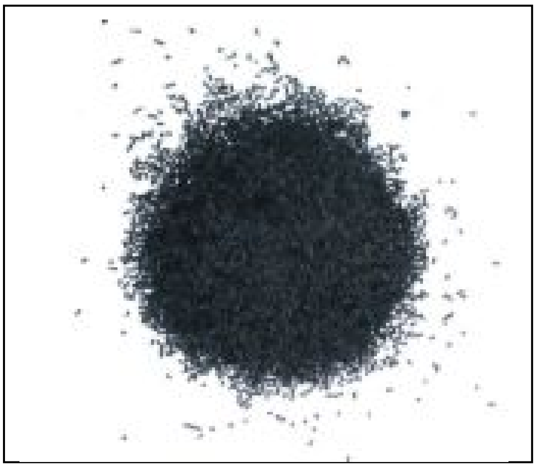


Figure 3.14: Activated Carbon

Activated carbon can be derived from a variety of coal s such as wood-based carbons and peat-based carbons. All the activated carbon (Figure 3.14) contain some micropore, mesopores and macropores characteristics but their proportions to one another vary substantantially depending upon the kind of starting material and the production procedures (Patterson, 1992). The bleaching action seems to be due to the large adsorbent surface of carbon. This large surface held in a small volume, would influence the surface tension of the compounds with which will comes into contact, thus causing adsorption. There are a large number of commercial grades of activated carbon that are used for adsorbing gasses and vapours, odours and colouring materials (Berdardini, 1985)

3.4.2.4 Synthetic silicate

Synthetic silicates are now commonly used in edible oil bleaching. Although synthetic silicates has a moderate capacity for pigment removal, small amounts of it are used in combination with bleaching clay and due to their synergic action the amount of bleaching clays needed to optimize the bleaching processes is reduced (Roosi et al., 2003). Silica had the capability of enhancing the earth’s ability to remove colour bodies, phosphorus and other minor components that affect the colour stability of the oil (Siew et al., 1994).

3.2.5 Acid Activated Earth Versus Natural/Neutral Earth for Palm Oil Refining

As mentioned previously, bleaching process is a crucial step in the refining of palm oil. This is because it is not only removes the colored compounds and impurities but also influence the stability of the final products. Refiners have a wide choice of adsorbents to be use and their selection of main criteria are cost and performance of the adsorbent materials. Numerous studies has been made for regarding the bleaching performance and the relationship between physicochemical properties of adsorbents and adsorption of trace constituents in palm oil (Cheah and Siew, 1999).

Studies on the performance of acid activated and natural/neutral-bleaching earth has been conducted by Howes et al., (1991) and Cheah and Siew (1999). In their studies, they found out that natural/neutral bleaching earths (non-activated) were less efficient in removing peroxides during bleaching. Based on Figure 3.15 (Howes et al., 1991) below, it can be clearly seen that for typical quality of palm oil, acid activated earths were more effective in removing peroxides.

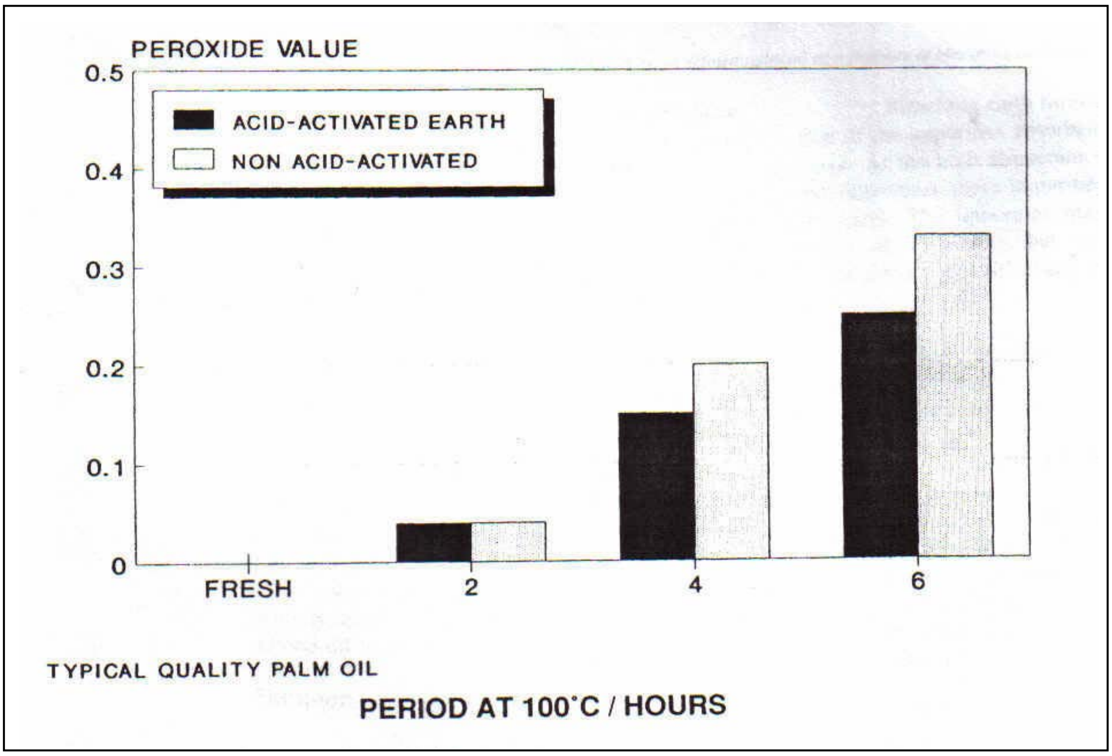


Figure 3.15: PV stability of Palm Oil as a Function of Bleaching Earth Type

Based on Cheah and Siew (1999) study, they concluded that in general, acid activated earths were more efficient in removing color components. This conclusion can be supported by Howes et al. (1991) study, where they plotted a graph of colour stability of palm oil as a function of bleaching earth type (Figure 3.16). From the graph, we can see that the colour pigments were removed better by using acid activated earth instead of natural/neutral (non-activated) bleaching earths.

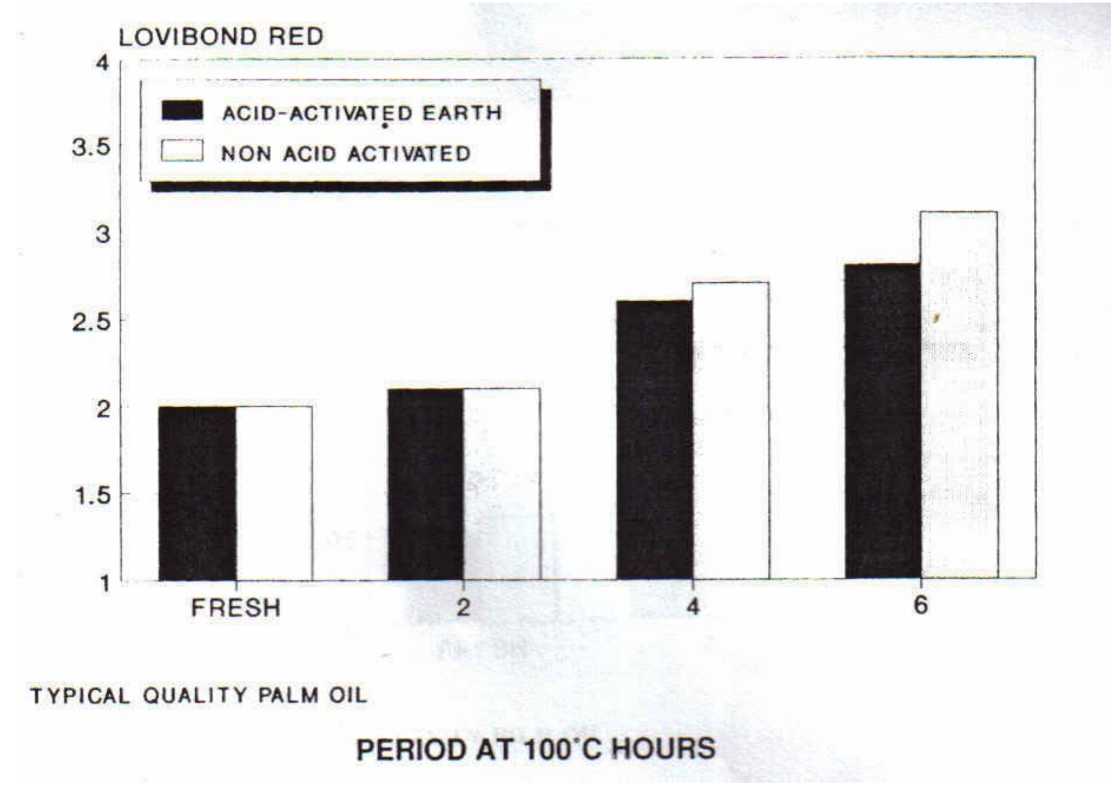
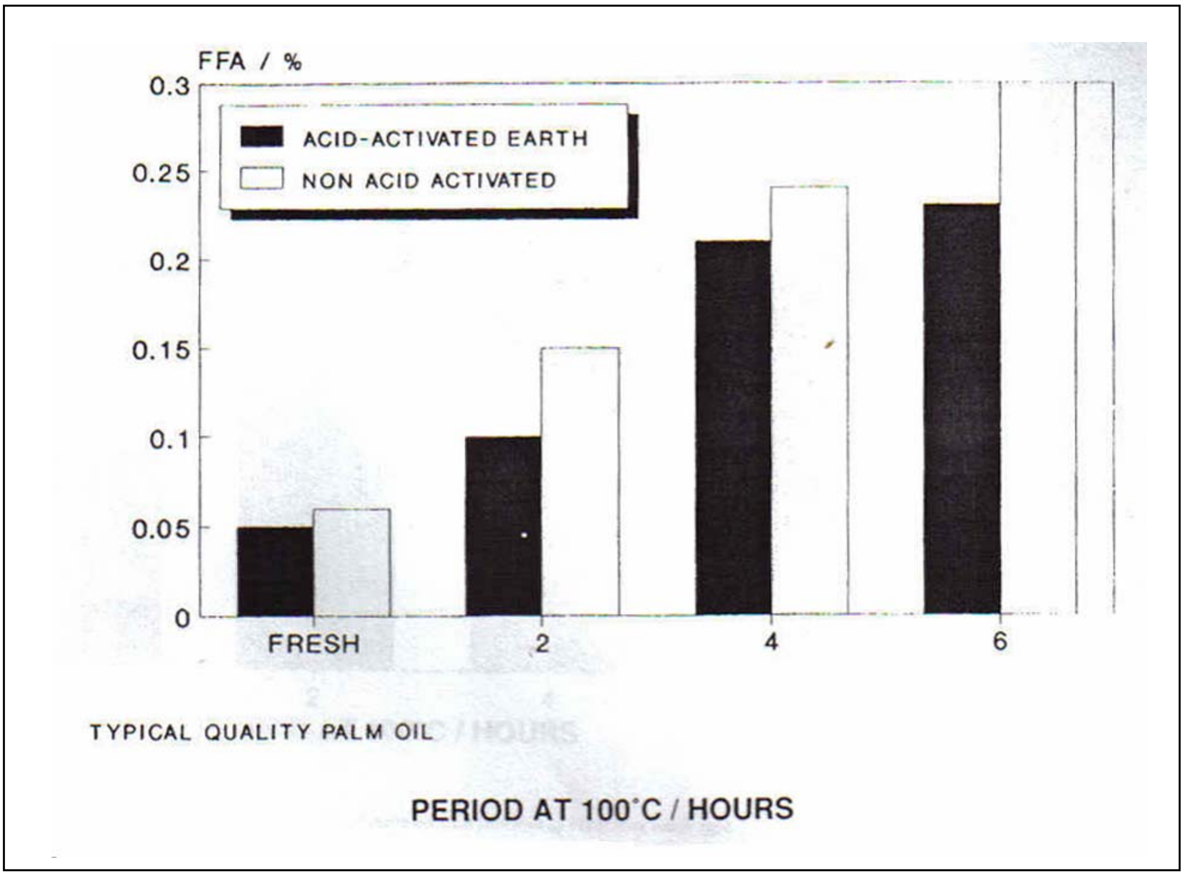


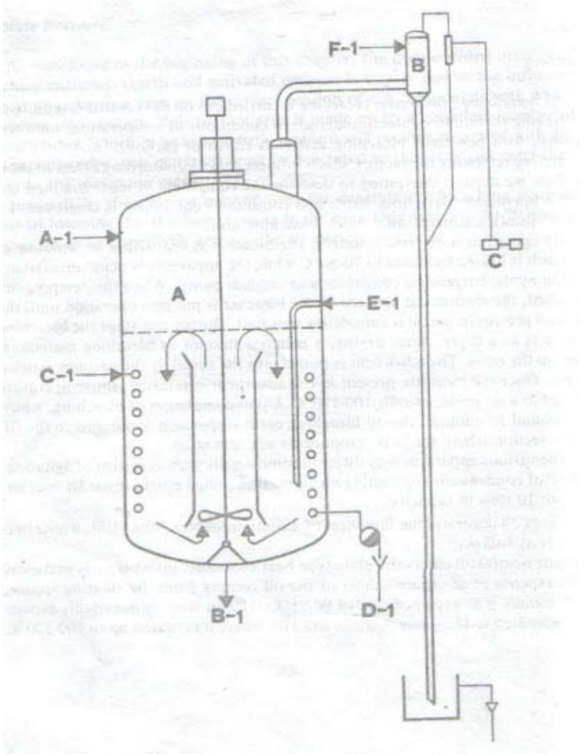
Figure 3.15: Color stability of Palm Oil as a Function of Bleaching Earth Type


FFA / %
0.3
0.25
0.2
0.15
0.1
0.05
0
ACID-ACTIVATED EARTH
NON ACID ACTIVATED
FRESH
2
4
6
TYPICAL QUALITY PALM OIL
PERIOD AT 100˚C / HOURS

Conclusion

3.2.6 Mode of Bleaching / Equipments for Bleaching

3.2.6.1 Batch Bleaching



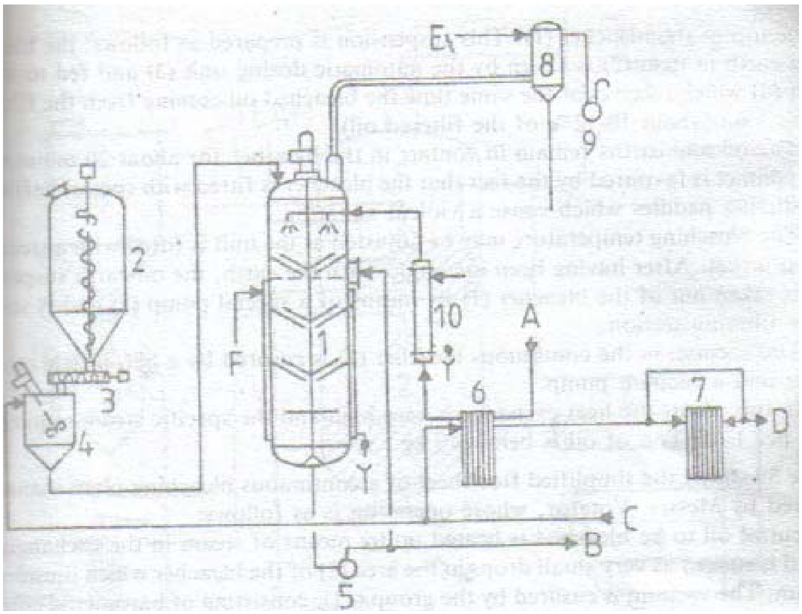
A : Bleacher
B : Barometric condenser
C : Vacuum pump

A1 : Oil inlet
B1 : Oil adsorbent outlet
C1 : Steam inlet
D1: Condensate outlet
E1 : Bleaching Material inlet
F1: Water Inlet

Figure 3.14: Batch Bleaching Unit

Figure 3.4 shows a conventional batch bleaching unit where its operation is rather simple. The bleacher is fed by a set amount of oil and heated by steam while the apparatus is maintained under vacuum by the barometric condenser and vacuum pump. When the desired temperature is reached, the mechanical agitator of the bleacher is put into action until moisture present in the oil is completely removed. During this stage the bleaching kettle will act as dryer. After drying, a metered amount of bleaching is added to the mass and the adsorbent is pulled into the vessel by the vacuum existing therein. Upon completion of bleaching, the oil-bleaching earth suspension is pumped to filtration section where the two components are separated (Bernardini, 1985)

3.2.6.2 Continuous Bleaching

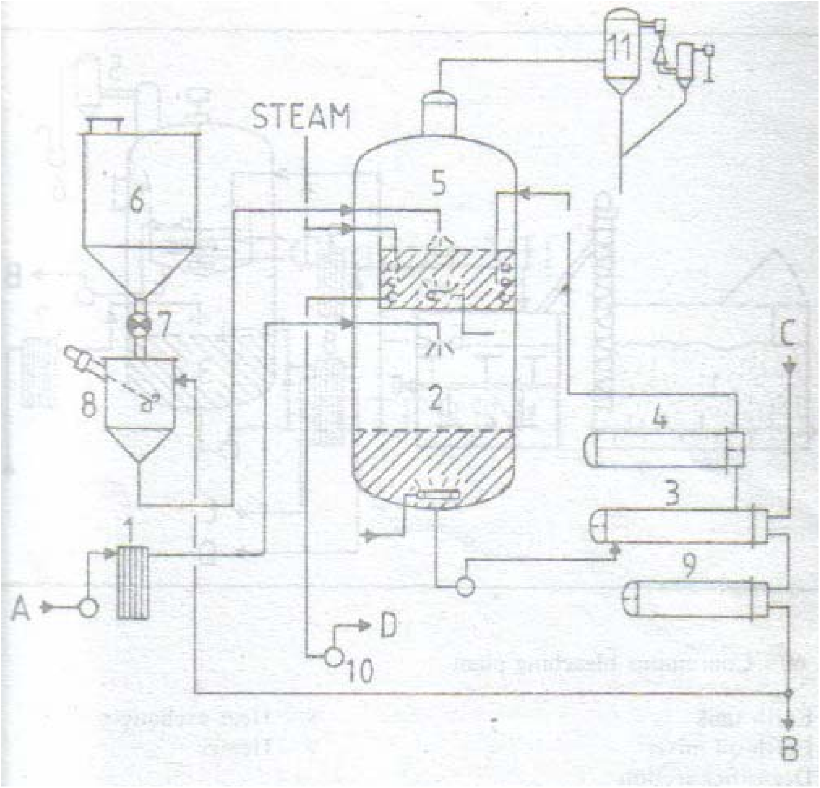


A: Crude Oil Inlet
B: Oil-Earth suspension to be filtered
C: Bleached oil inlet
D: Bleached oil outlet
E: Water Inlet
F: Steam Inlet

1: Continous bleacher
2: Earth tank
3: Earth dosing unit
4: Mixer
5: Earth-oil pump
6: Heat Exchanger
7: Cooling Unit
8: Barometric condenser
9: Vacuum Pump
10: Heat Exchanger

Figure 3.15: Continuous Bleaching Plant (Type 1)

There are a few types of continuous bleaching plant as per illustrated in Figure (3.5 - 3.7).



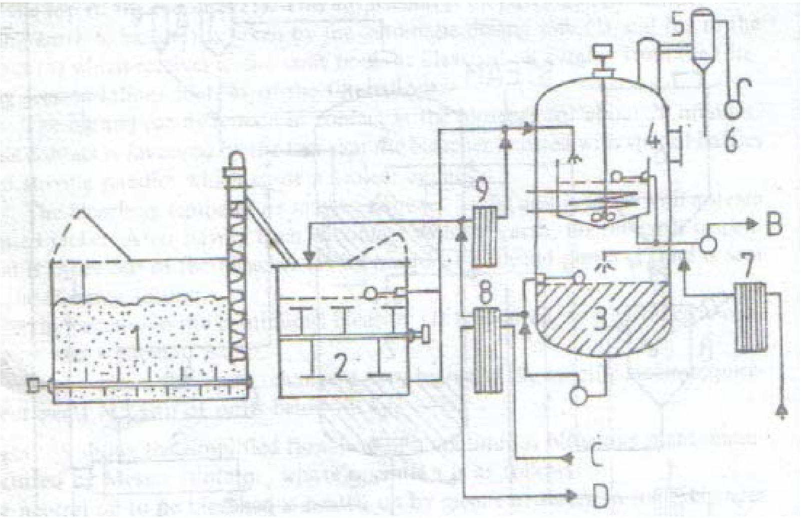
1: Heater
2: Degasifier
3: Heat Exchanger
4: Heater
5: Bleaching Unit
6: Earth Tank
7: Earth dosing unit
8: Mixer

9: Cooling Unit
10: Oil-Earth Pump
11: Vacuum System

A: Raw Oil inlet
B: Bleached Oil inlet
C: Bleached Oil Outlet
D: Oil-earth suspension to filtering

Figure 3.16: Continuous Bleaching Plant (Type 2)

Figure 3.6 shows the simplified process flowsheet of a continuous bleaching plant. In this plant, the crude oil to be bleached is heated by means of steam in the exchanger and is sucked as very small drops in the lower part area (2) of vacuumed bleacher which operation is ensured by the presence of barometric condenser and ejectors. Dry steam is injected to the oil mass in order to facilitate the operation and also to keep the mass in agitation. The oil is then taken by a pump and is sent to a series of heat exchanger to raise its temperature to a desired level and then sent to upper part area (5) of bleacher. The oil remains in contacts with the earth for a specified period of time and then the suspension is sent to filtering (Bernardini, 1985).



1 : Earth Tank
2 : Earth oil Mixer
3 : Degasifier section
4 : Bleaching Section
5 : Barometric Condenser
6 : Vacuum pump
7 : Heater

8 : Heat Exchanger
9 : Heater

A: Crude Oil Inlet
B: Oil-earth Outlet
C: Bleached oil inlet
D: Bleached oil outlet

Figure 3.17: Continuous Bleaching Plant (Type 3)

Figure 3.7 shows another type of continues bleaching plant. The main advantage of this type of plant is that the bleaching earth is loaded on the ground floor. The operation of this type of plant is nearly the same as the previous one.

3.2.7 Degumming and Bleaching Approaches in Malaysia Refineries

As mentioned earlier in Chapter 1, the scope of this research study is narrowed down to the first 2 stages, which are degumming and bleaching process. Currently, based on the survey conducted, it is found that nowadays in Malaysia the processes of degumming and bleaching are being carried out together but in two different approaches.

(a) For the first approach (refer to Figure 3.18 below), the phosphoric acid is mixed with small amount of crude palm oil in the mixer before it is sent to degumming vessel for more efficient mixing. This mixture will be further sent to bleaching vessel where the bleaching earth is added. The process will continue with filtration where the gums and spent bleached earth are separated from the degummed and bleached oil.

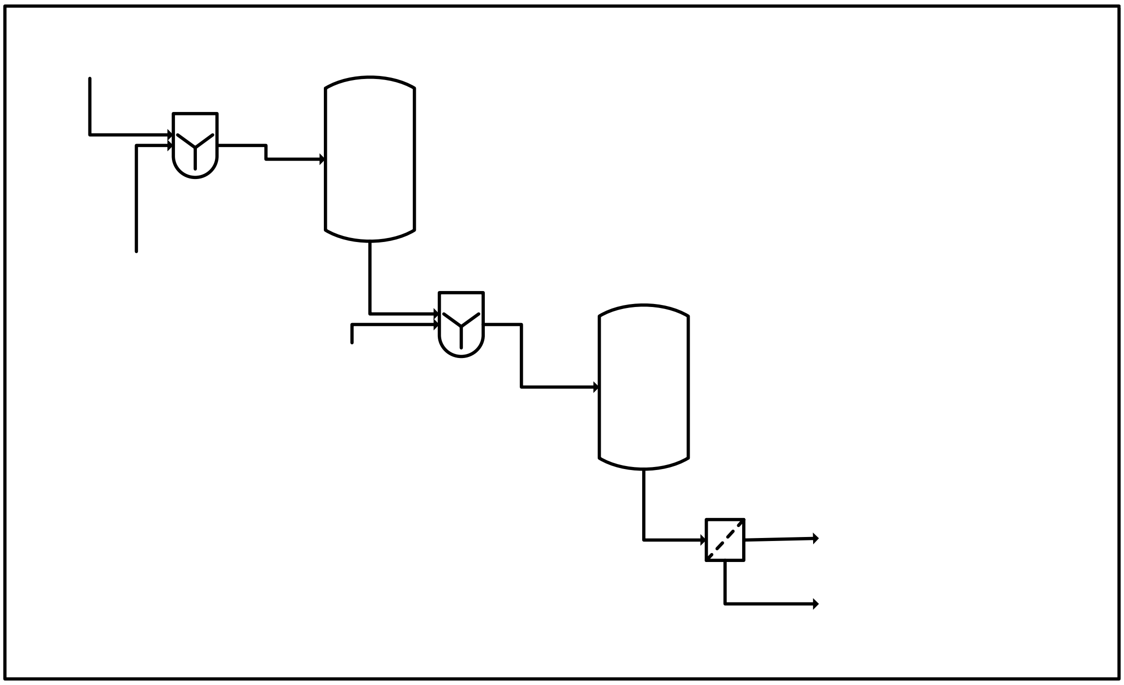


Figure 3.18: Process flow diagram of bleaching and degumming carried out in different vessels

The second approach that is found from the survey is described as below;

(b) For this second approach (refer to ***METHOD B*** below), small quantities crude palm oil (CPO) is mixed with phosphoric acid and bleaching earth in 2 separate mixers before entering the bleaching vessel. No degumming vessel is required. Degumming and bleaching took place simultaneously in bleaching tank. Filtration similar to the first approach is carried out after that.

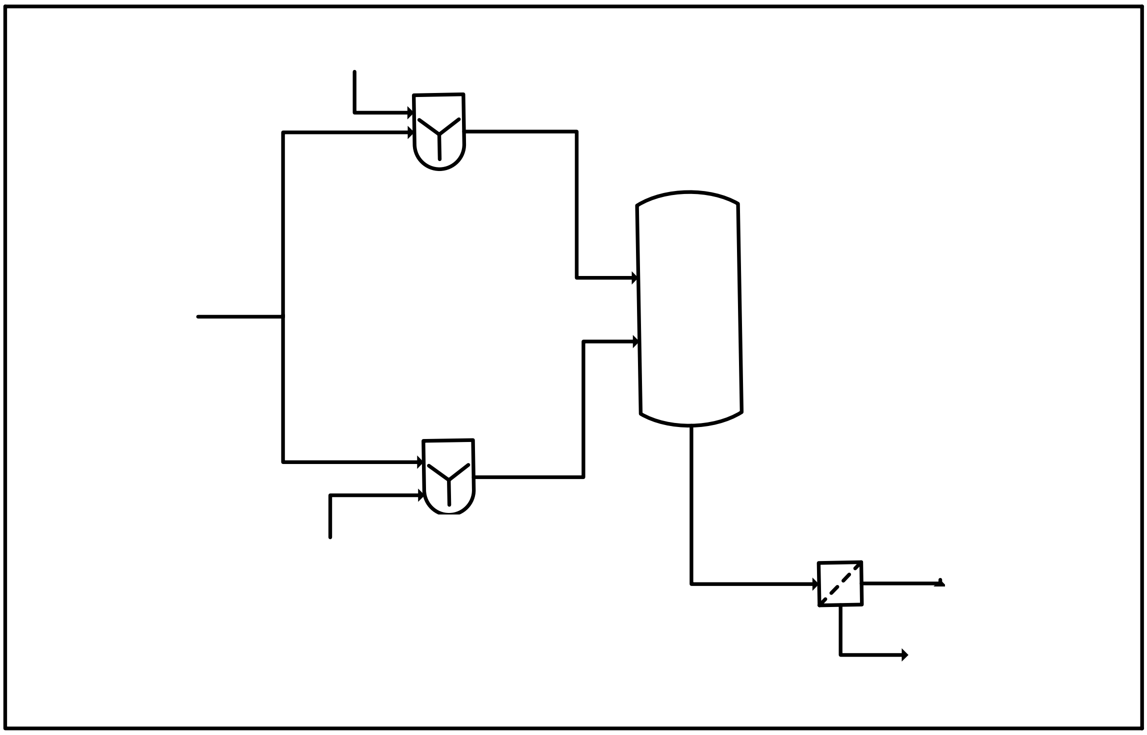


Figure 3.19: Process flow diagram of bleaching and degumming carried out in the same vessel

The second approach of processing (***METHOD B***) will be modelled in this research because of its simplicity. The model will be used to verify the performance of both methods of processing (***METHOD A and METHOD B***). If the performance of both approaches of processing can be predicted by this model, this proved that the second approach of processing is superior.

CHAPTER 4

DESIGN OF EXPERIMENT (DOE) & ARTIFICIAL NEURAL NETWORK (ANN)

4.1 Design Of Experiments

Design of Experiment (DOE) is a technique to lay out experimental research studies plan in most logical, economical and statistical way. Through this technique researchers can determine the most desirable design of product, best parameters combination for the required process, most robust recipe for formulation, most critical validation /durability test conditions and most effective data collection plan.

DOE consists of a set of Experimental runs, which each run defined by the combination of each factor level (variables) and the analysis of the experiments. DOE helps to make product and processes more robust. DOE is a proven technique that continues to show increasing usage in chemical process industries especially for fast, cost saving and accurate result.

Historically, DOE technique was first developed by R. A Fisher in the 1920s to study the effect of multiple variables simultaneously (Dowey and Matthews, 1998). In his early applications, Fischer wanted to find out how much rain, water, fertilizer, sunshine are needed to produce the best crop and since that a lot of DOE technique developments has been done for industrial applications.

DOE technique now has become a very useful statistical tool to help us understand process characteristics and to investigate how inputs affect responses based on statistical backgrounds. In addition, it has been used to systematically determine the optimal process parameters with fewer testing trials (Park and Ahn, 2004). Thomas (1997) pinpointed that, the advantages of statistical design are ;

(i) to provide more information per experiment than unplanned experiments
(ii) to organize data collections and analysis information
(iii) to assess the information reliability in the light of experimental and analytical variation
(iv) to view the interaction of experimental variables, leading to more reliable predictions of the response data in areas not more directly covered by experimentation

4.1.1 Response Surface Methodology

There a several experimental design techniques that can be used in DOE such as Factorial design, Response Surface Method (RSM) design, Mixture design and Taguchi design. In order to select the best design method to be used, one must be carefully consider the objectives /goals for an experiment.

The types of experimental designs classified according to the experimental objective they meet; either it is for comparative objective or screening objective or response surface objective.

In response surface objective, the experiment that are designed are used;

- to hit a target
- to maximize or minimize a response
- to reduce variation by locating a region where the process is easier to manage
- to make a process robust

In this study, the objectives are; to hit a target (to find the exact dosage of phosphoric acid and bleaching earth and phosphoric acid to be added to CPO fed) and to maximize a response (to optimize degumming and bleaching process).

Therefore, Response Surface Method (RSM) technique is discovered to be is the most suitable design technique to be applied in this research study. RSM is also used as its analyze problems which response is influence by several variables and its measure the quality characteristics of a system. This method is based on polynomial surface analysis and it is a collection of mathematical and statistical techniques that are useful for the mode ling and analysis of problems in which a response of interest is influenced by several variables.

According to Dey et al., (2001) and Prado et al., (2004), the chosen method for optimisation of the response factor was efficient, relatively simple with time and material saving by using response surface methodology. It is found out that, the RSM technique has been successfully applied in the field of quality experimental work (Muralidhar *et al*., 2001; Amin and Anggoro, 2003; Varnalis *et al*., 2004; Jose *et al., 2004*).

Response surface method is created from factorial design and there are two categories of quadratic factorial designs, namely; central composite design (CCD) and Box-Behnken design. CCD contains an imbedded factorial or fractional factorial design with center points that is augmented with a group of `star points' that allow estimation of curvature.

Whereas, the Box-Behnken design is an independent quadratic design in that it does not contain an embedded factorial or fractional factorial design. In this design the treatment combinations are at the midpoints of edges of the process space and at the center. These designs are rotatable (or near rotatable) and require 3 levels of each factor. The designs have limited capability for orthogonal blocking compared to the central composite designs.

Thus, due to the limited capability of orthogonal blocking of Box-Behnken design, Central Composite Design is employed in this study,

4.1.2 Central Composite Design

A designed training data sets are more desirable than random experimental sets, due to their higher orthogonality (Lanouette *et al.*, 1998 and Kapur *et al.,* 2004). Central Composite Design (CCD) is well-liked among researchers to design the training data set and to analyze the influence of variables due to its ability to show which variables significantly affect each response. The central composite designs satisfy the general requirements of response surface designs, which are (Mead, 1992);

1. that the parameters of the model to be fitted can be estimated
2. that the number of treatment combinations is not allowed to become too large

the observation are spread fairly evenly over the region within which information about the surface is required.

Peng et al.(2002) point out that, to have a robust model for optimization, the CCD, generally is the best design for RSM optimisation. CCD allows us to show which variables significantly affect each response and performs optimisation on the value of variables that are found significant.

For example, CCD with three experimental factors employed 16 experiments. The experiments contain eight runs at two level (-1, +1), six star point (-∗, +∗) and two replicates at the centre point (0) to allow estimation of the error and provide a check on linearity.

4.2 Artificial Neural Network (ANN)

ANN modelling is a relatively new non-linear statistical technique where it can be used to solve problems that are not suitable using conventional statistical methods. Apart from that, application of ANN in modelling is about 20 times faster than numerical integration of a differential equations system (Parisi and Laborde, 2001). Therefore the application of ANN in various branches of science and process technology has become an attention by the researchers.

4.2.1 ANN Definition

Artificial Neural Network (ANN) is a data-processing technique simulating human brain analytical function, which has ability to learn by experience and is used when no exact mathematical relationship is available and when linear and nonlinear prediction problems must be solved. It is a computing tool made up of a number of simple but highly connected nodes (Baughman and Liu, 1995).

Neural networks posses the ability to learn what happens in the process without actually modelling the physical and chemical laws that govern the system. The success in obtaining a reliable and robust network depends strongly on the choice of the process variables involved as well as the available set of data and the domain used for training purposes (Nascimento, 1997).

One of the advantages of neural networks is less time demanding to be developed than the traditional mathematical models because ANN can also obtain a highly accurate mathematical model of the system without detailed knowledge of the system (Shene *et al*., 1999).

The goal of a neural network is to map a set of input patterns onto a corresponding set of output patterns. The network accomplishes this by first learning from a series of examples that contain the behaviour of an unknown function. The network then applies what it has learnt to a new input pattern. The network model should have the same behaviour as the desired function, predicting the appropriate output.

Artificial neural networks consist of a number of simple interconnected processing units, also called neurons, which are analogous to the biological neurons. Two important features of neural networks (NN) are;

1. the ability of supplying fast answers to a problem and
2. the capability of generalising their answers, providing acceptable results for unknown samples.

4.2.2 Network Topology/Architecture

An ANN consists of several different layers. A general topology/architecture of an ANN is shown in Figure 4.1 . The network consists of three layers, an input layer, a hidden layer and an output layer. .

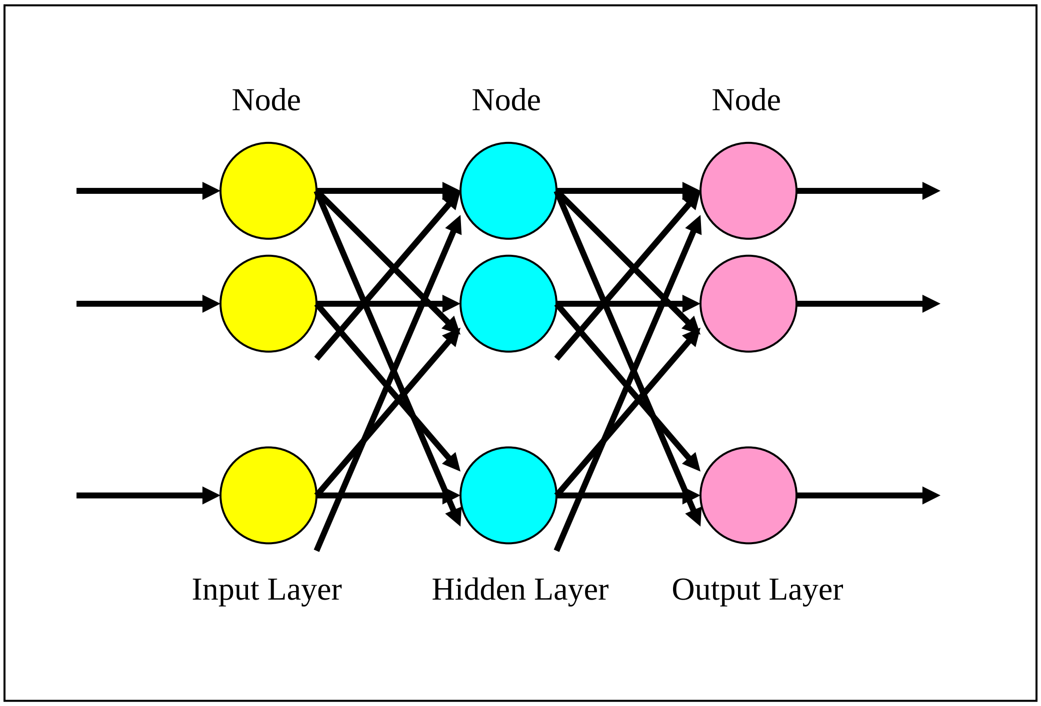


Figure 4.1: General Topology of three-layer perceptron ANN

There are a few functions that can be used in the neural e.g. Radial Basis Function, feed forward neural network (also know as preceptron) and Fuzzy Logic. A feed forward neural network is most commonly used because it is most applicable to science and engineering, least complicated and most straightforward to implement (Braughman and Liu, 1995).

Baughman and Liu (1995), stated that the feedforward connection is used where the outputs from a node feed into nodes in the subsequent layer .The first layer is the input layer which it receives information from an external source and feeds it into the network for processing. The second layer is called the hidden layer. It receives information from the input layer and processes it. The third layer is the output layer which receives the processed information from the hidden layer and sends the results to an external receptor.

4.2.3 Components of A Node

As mentioned previously, the node (neuron) as an input layer is the elementary component of an ANN. The function of a node is to process information from external inputs by its dynamic state response. This is why sometimes the node is also called as processing element.

The output from the node is then transferred to the next processing nodes called the hidden layer. A simple model of a node and its components is shown in Figure 4.2 below;

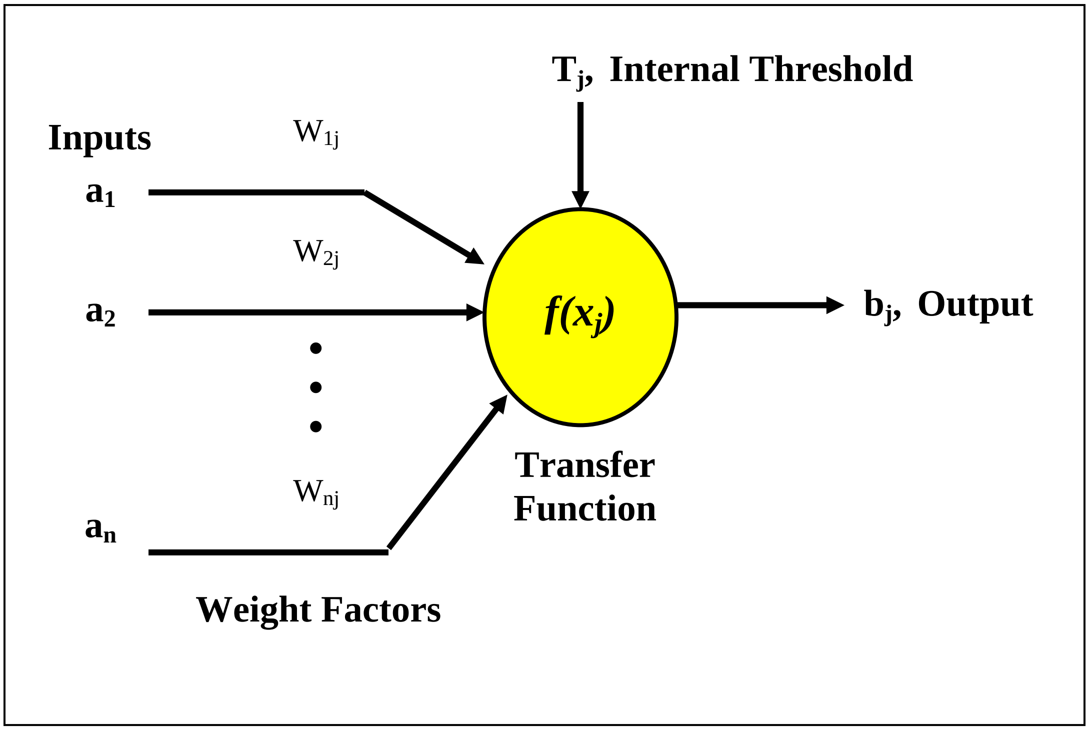


Figure 4.2: Simple model of a Node (Baughman and Liu, 1995).

4.2.2.1 Inputs and Outputs

The input to the node are represented by an input vector, a_n. The node manipulates these inputs to give the output, b_j. This output then forms part of the input to another node

4.2.2.2 Weight Factors

Each node usually will receive several inputs at the same time. There is also a weight factor, w_{ij} that affects the output from the node besides the component value of the input vector. According to Baughman and Liu (1995), every input vector is multiplied by its weight factor and the node uses the weighted input to perform further calculations.

The weight factors will determine how much each input will affect the output from the node. If the weight factor is large, it will excite the node. If the weight factor is small, it will inhibit the node and that input signal has little effect on the output

4.2.2.3 Internal Thresholds

The function of the internal threshold, T_j is to control the activation of a particular node. The total activation can be calculated by subtracting the internal threshold value from the sum of all weighted inputs. If T_j is large, node firing will be inhibited and vice versa.

4.2.2.4 Transfer Functions

The transfer function is the final factor governing the output from a node. The node calculates the dot product of the weight factor, w_{ij} and input vector, a_i. Afterwards, it subtracts the threshold T_j from it. This result will then be passed on to a transfer function, *f*(). The transfer function changes this result to an output. The function is generally chosen to be continuous and non-linear. Some of useful functions are the sigmoid, hyperbolic tangent and radial basis transfer function

4.2.4 Back-propagation Neural Network

The most common form of learning utilized in neural networks nowadays is error-correction learning. Previously, this technique is being neglected by mathematicians due to its incapability to work with neural networks with hidden layers. However, now through a technique called back-propagation, we can apply the error-correction learning to neural networks with hidden layers. According to Baughmann and Liu (1995), back-propagation requires a perceptron neural network defined as a network with only feed-forward interlayer connections (no intralayer or recurrent connection). Each layer must feed sequentially into the next layer.

The goal of back propagation training is to change iteratively the weights between the neurons in a direction that minimizes the error E, defined as the squared difference between the desired outputs of the actual outputs of the outputs output nodes, summed over the training patterns (training data set) and the output neurons according to the steepest descent method (Marini et al, 2003).

In back-propagation ANN, the structure of neural network consists of three fundamental layers;

(1) an input layer, where each neuron is associated to an experimental factor and receive information/signal from outside world, usually in the form of a data file

(2) layers of processing neurons, called hidden layers or intermediate neurons contained in one or more hidden layers allow nonlinearity in the data processing.

(3) an output layer, where each neuron is associated to the response and provide an answer for a given set of input values.

The signal moves from the input layer towards the output layer as per shown in Figure 4.3, and in this process each neuron uploads all the neurons of the successive layers, transferring a portion of the signal that has been accumulated. The portion of signal transferred is regulated by a transfer function (Marengo et al., 2004).

A feed-forward back-propagating ANN structure as illustrated in Figure 4.3 was used to develop yield prediction models. Data move through the layers in one direction, from the input through the hidden to the output layers, without loops as opposed to feedback networks.

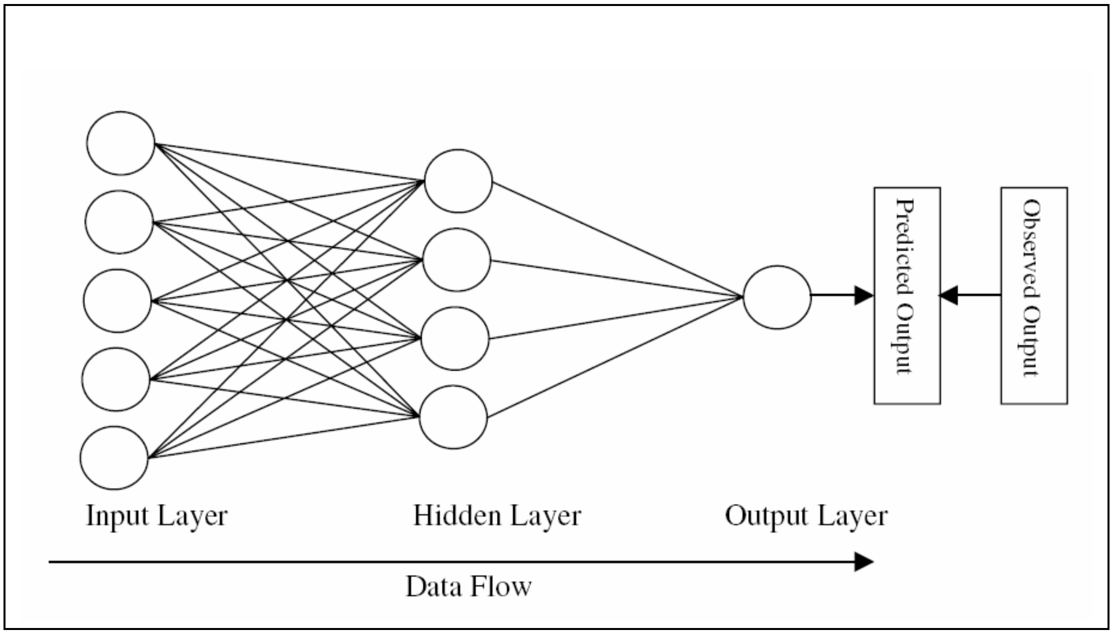


Figure 4.3: Layers and connections of a feed-forward back propagating artificial neural network.

In fully connected back-propagation ANN, each neuron in a given layer is connected to each neuron in the following layer by an associated numerical weight (Marini et al., 2003). The weight that connecting two neurons serves to regulate the magnitude of a signal that passes between them. Each neuron then possesses numerical bias term corresponding to an input of –1, whose associated weight has the meaning of threshold value.

Generally, feed-forward networks may be based on linear or non-linear transfer functions that affect the output from the input and hidden layers. Non-linear networks may be trained using supervised learning, learning by example with outputs, or unsupervised learning, self-organizing without outputs. Supervised learning uses known outputs to train the ANN and is more commonly used than unsupervised learning

Back propagation is a form of supervised learning where the error rate is sent back through the network to alter the weights to improve prediction and decrease error. The general process to build a neural network model included creating data sets for training and testing, training multiple networks with varied parameters, analyzing network results, and testing the models

4.2.5 Neural Network Development

The process of neural network development can be divided in three main phases (Baughman and Liu 1995);

(1) Design and training/learning of neural network
(2) Recall phase
(3) Generalisation phase

Prior to these three phases of neural network development, a few steps need to be taken first, which are database collection, normally through experimental works or obtain from literature review and also analysis and pre-processing of the data gained.

The design and training of neural network is the first phase of neural network development process. At this phase, the network is fed with a set of known input-output patterns. The weight factors of the nodes are adjusted until each input yields the desired output.

In learning phase, it is where the actual process of adjusting the weights factors until the desired outputs are obtained. In this way, the neural network (NN) needs to learn about the problem under study. This process usually starts with random values for the weights of the NN. As mentioned earlier, there are two types of learning method; supervised learning, learning by example with outputs, or unsupervised learning, self-organizing without outputs and back-propagation is a form of supervised learning.

In recall process, the performance of the network is checked and evaluated once the training phase is completed. The network is given an array of input patterns that was previously seen during the training phase. The output error from the network is then assessed. A well-trained network should produce outputs that deviate very little from the desired value.

The generalisation phase is where a new set of input patterns, which the network has not seen before, is introduced to it. The network output is then compared to the desired output. Therefore, the performance of the neural network in predicting new input patterns can be evaluated

4.2.6 Strengths of ANN

Even though application of ANN in modeling can be used to solve problems that are not suitable using conventional statistical methods but ANN is not a solid solution for all modeling problems and therefore it is important to understand the strengths and limitations of ANN with compared to first principle models or other empirical models. Baughman and Liu (1995) has stated that the strengths of ANN are :

1. *Information is distributed over a field of nodes*.
   In symbolic processing, the information is held at one fixed location but in ANN the distribution of information are wide and thus provides greater flexibility.

2. *ANN has the ability to learn.*
   There is an error-correction training technique in ANN, in order to encounter with any error occurred by adjusting the related signals and effectively the system will "learn" and in future will be working properly and better.

3. *Neural network allow extensive knowledge indexing*.
   Knowledge indexing is the capability to store large amount of data and can be easily being accessed. The network stores the informations in two forms which is the connection between nodes and the weights of every connection.

4. *Neural networks are better suited for processing noisy, incomplete or inconsistent data*.

In ANN, each node encodes a *microfeature* of input-output pattern, where it implies that each node will only affect the input-output pattern slightly and therefore minimizing the effects of noisy or incomplete data in any given node.

5. *Automated abstraction*

   ANN can determine the essentials of input-output relationships automatically as it will not need any *domain expert* (an expert in a problem-solving domain) to develop the knowledge base.

6. *Potential for on-line use*.

   The potential of the trained ANN for producing the result instantaneously has make it a desired feature for the on-line use.

4.2.7 Limitations of ANN

Baughman and Liu (1995) have described the limitations of ANN as follows:

1. *Long training time*

   Training time for ANN can consumed a lot of time especially for large networks and complex problems

2. *Large amount of training data.*

   ANN requires large amount of historical input-output data for a better model generalisation. If there is little amount of input-output data exists, ANN may not be suitable for modelling the system.

3. *No guarantee to optimal results.*

There is no guarantee that the resulting model is perfect for the system, even though the network contains parameters that can be tuned by the training algorithm. It can be that tuned model may be accurate in one region but inaccurate in another.

4. *No guarantee of 100 % reliability*

   The ANN model can be unreliable particularly when there is a limited training data.

5. *Good sets of input variables.*

   Selecting the input variables to give proper input-output mapping is difficult because it is not apparent that which input variable will give best results. Usually some trial and error method will be required in doing the selection. .

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CHAPTER 5

METHODOLOGY

5.1 Overall Research

The whole research study can be divided into 5 main parts:

(1) Identification of optimal operating parameters for bleaching and degumming processes from the surveys and plant visits conducted to refineries throughout Malaysia.

(2) Determination of optimum number of testing trials to obtain experimental data through Design of Experiment (DOE) method which using statistical tool, MINITAB 14.

(3) Determination of data of DOBI, FFA, peroxide value (PV), phosphorus content, moisture content and iron content of CPO and DBPO samples through experimental works.

(4) Models development of degumming and bleaching process using artificial neural network toolbox in MATLAB 7.0.

(5) Comparisons deductions between experimental findings and ANN model.

The main objective of this research study is to develop a model that can suggest a suitable ratio of phosphoric acid and bleaching earth dosage for degumming and bleaching processes. The research started with identification of optimal operating parameters for bleaching and degumming processes based on the surveys distributed to refineries throughout Malaysia and also based on plant visits conducted.

Surveys are done by distributing questionnaires to all the registered palm oil refineries in Malaysia. There are 2 set of questionnaires (refer to Appendix A and Appendix B) that has been distributed, with the objectives;

- to get on overall overview of the actual refining processing of palm oil available in Malaysia.
- to get preliminary related data on the refining operating process and
- to obtain further information on the operation of degumming and bleaching

Questions constructed in this survey were emphasizing on the degumming and bleaching section where some useful information are obtained such as:

Few plants visits are conducted in order to get familiarize with actual plant operation and to have a closer look at degumming and bleaching operation. The visits were accompanied by the supervisors, lecturers and research officers. During the visits, discussions are made with the industrial expertise in order to get their opinions, suggestions and feedback from their point of view regarding the proposed research.

Site visiting accompanied by the engineers are also conducted in which we are able to see the actual processing of palm oil, equipments used and controlling system of the refinery plants. Below are the details on the plant visits conducted:

Table 5.2: Details on the plant visit activities conducted.

Plant Visited	Date and time
1. **Felda Oil Products Sdn Bhd,** Lot 82, Jalan Besi Dua, 81700 Pasir Gudang, Johor	Date : 25th March 2004 Time : 10.30 a.m –1.00 pm
2. **Soctek Edible Oil Sdn Berhad,** Plo 8 & 9 Jalan Timah, Pasir Gudang, Industrial Estate, 81700 Pasir Gudang Johor.	Date : 25th March 2004 Time : 2.00 p.m – 4.30 pm
3. **Delima Oil Products,** Pandamaran Industrial Estate, P.O Box 204, Pandamaran, 42009 Port Klang, Selangor.	Date : 22nd May 2004 Time : 2.30 p.m – 4.30 pm
4. **Golden Jomalina Food Industries Sdn. Bhd.** Batu 9, Jalan Banting – Klang, 42500 Telok Panglima Garang Industrial Estate, Selangor.	Date : 5th October 2004 Time : 11.00 a.m –1.00 pm

Afterwards, experimental rigs using rotavapor were set up according to optimum operating conditions of degumming and bleaching processes. During this stage, DOE study was also conducted in order to determine optimum number of experiments to be run. There are 20 set of experiments need to be run with 6 quality checking parameter experiments each for both samples; crude palm oil (CPO) and degummed bleached palm oil (DBPO).

Based on the experimental data, 2 studies were conducted:

(i) Experimental data findings analysis

(ii) ANN models development

In this study, 3 models of ANN were developed, and performance of each model is evaluated in term of error generated and model fitness.

Finally, based on the deduction from experimental findings analysis and ANN model, a conclusion will be derived which at the end will suggest some improvements to the degumming and bleaching practices in Malaysia’s refineries. Figure 5.1 shows the overall methodology of the research study.

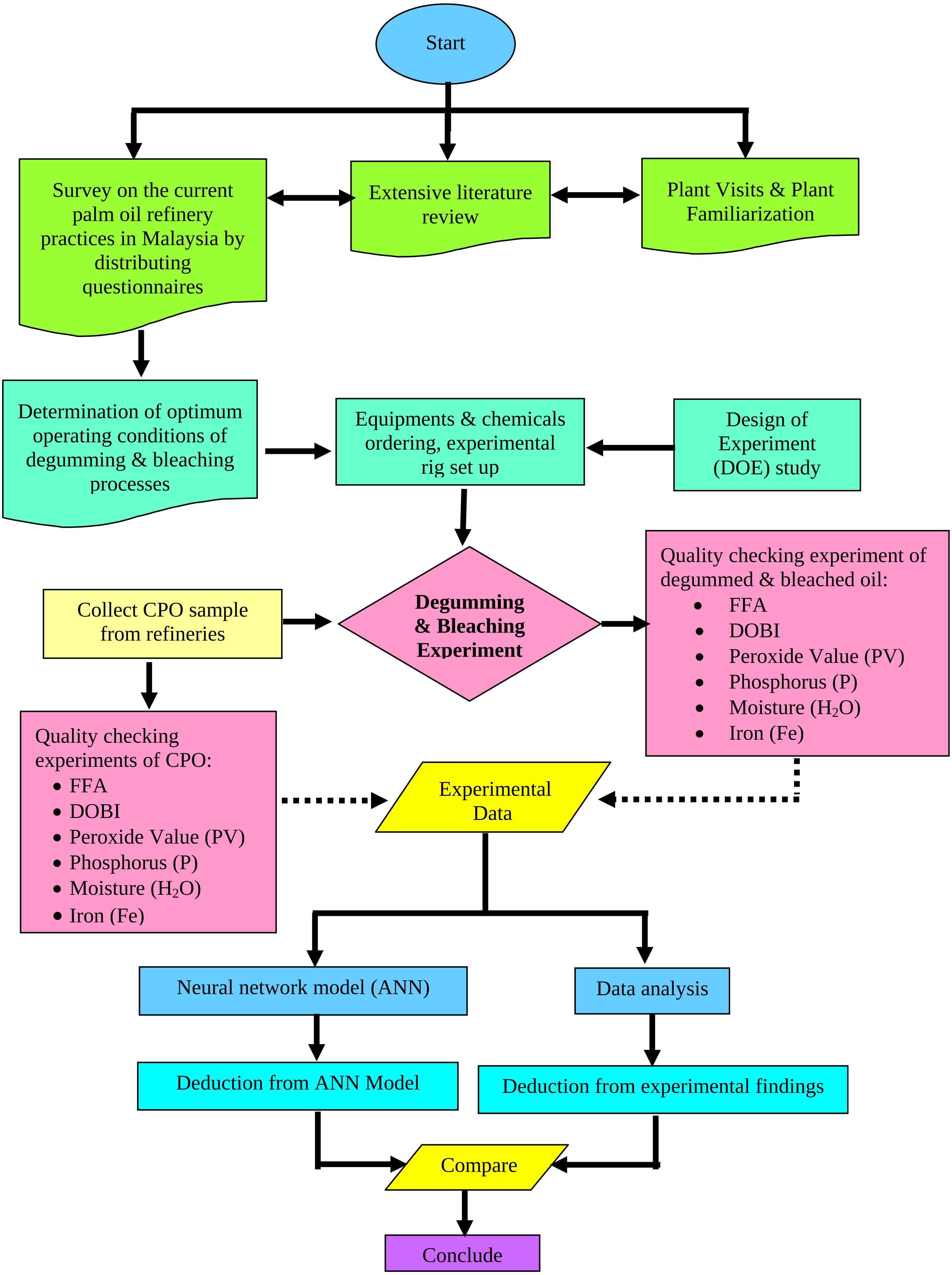


Figure 5.1: Overall methodology of the research.

5.2 Operational Conditions

One of the main parts of this research study is to identify the optimal operating parameters for bleaching and degumming processes. There are various operating conditions for degumming and bleaching processes of palm refining. Based on preliminary studies through surveys and literature it is found that the optimal operating conditions for a typical configuration of palm oil refinery are identified as per shown in Table 5.1 below;

Table 5.1: Optimum operating condition for a typical configuration of palm oil refinery

Parameters	**Operating Conditions**
Temperature	100^{o}C
Pressure	Vacuum (50 torr)
Contact time	30 minutes

5.2.1 Operating Temperature

Higher temperature encourages a better adsorption process between the bleaching earth and impurities inside the degummed oil and promotes a better bleaching process. But, at temperature higher than 150 ^{o}C, changes in structure of fatty acids might occur and isomerization might also start (Bockish, 1993). Thus, in order to maintain the efficiency of bleaching process, it is safer to maintain an upper limit of 110 ^{o}C for bleaching operating temperature because it will minimizes the probability of chemical or physical changes in triglycerides (Patterson, 1992). And based on the preliminary survey studies, most of refineries in Malaysia operate bleaching process at temperature of 100^{o}C. Therefore, it can be concluded that, a typical configuration of palm oil refining process, 100 ^{o}C is identified as the most optimal operating temperature for bleaching and degumming processes.

5.2.2 Pressure

It is found out that from the preliminary survey, the bleaching and degumming processes is conducted under vacuum condition of about 50 torr. Operating under vacuum condition is desired in the operation of bleaching process while removing all the unwanted gums (from precipitation during degumming), trace metals, peroxides and some volatile and oxidized products in order to keep the moisture level low. It is important in order to keep the moisture level low as high level of moisture must be avoided since it might hydrolyzed the triglycerides causing more formation of free fatty acids and thus affecting the stability of oil.

5.2.3 Contact Time

From the preliminary studies survey, 30 minutes of contact time is the normal time cycles used for bleaching process by the refiners in Malaysia. Although, higher efficiency of bleaching process can be achieved by increasing the bleaching time but by increasing the contact time between bleaching earth and the degummed oil, the daily production of a refinery will be affected, as the overall refinery operating time will be increased. And consequently affect the profit of a refinery, since it will reduce the daily production. Therefore, a commercial equilibrium balance should be considered between the lower cycle times and higher daily production. Thus, it can be concluded that the optimal bleaching and degumming time for a typical configuration of palm oil refinery is 30 minutes.

5.2.4 CPO Samples Quality

The crude palm oil (CPO) samples used in this research study were obtained from Pandamaran Delima Oil, Klang (for high FFA content) and Golden Jomalina Food Ind., Sdn Bhd, Banting (for low FFA content). Due to the inconsistent content of FFA content in CPO samples obtained, the samples were divided into 3 main categories namely;

(1) Category A: CPO with FFA content less than 2.5%
(2) Category B: CPO with FFA content between 2.6 –3.5%
(3) Category C: CPO with FFA content more than 3.6%

5.2.5 Bleaching Earth and Phosphoric Acid Quality

Bleaching earth and phosphoric acid used were obtained from Pandamaran Delima Oil, Klang Selangor. Bleaching earth used is acid activated clays and phosphoric acid is 85% concentrated.

5.2.6 Quality Checking Parameters

Based on preliminary studies, there are 6 important parameters that are usually being checked by the analysis lab of the palm oil refineries either it is on daily basis, weekly basis or monthly basis. The parameters are;

(i) Free Fatty Acid (FFA) content
(ii) Deterioration of Bleachability Index (DOBI) value
(iii) Peroxide Value (PV)
(iv) Phosphorus Content
(v) Moisture Content
(vi) Iron Content

All these experiments except iron content were conducted at CLEAR (Center Lipids Engineering & Applied Research).

The ranges of these parameters are set as per tabulated in Table 3.1 below. These ranges are identified based on the maximum acceptable and allowable values used by the refiners.

Table 5.2: Ranges of parameters

Parameters		Range
Dosage of phosphoric acid		Maximum : 1.00 % (per 1MT of CPO)
Dosage of bleaching earth		Maximum : 2.00% (per 1MT of CPO)
Quality of CPO	FFA	Maximum: 5 %
	Moisture	Maximum : 2 %
	DOBI	2-3.5
	Peroxide Value	1.5-5 meq/kg
	Phosphorus	10-18 ppm
	Iron (Fe)	4-10 ppm

5.3 Experimental Rigs of Degumming and Bleaching

Experimental rigs are designed and set up, according to the optimal conditions for a typical a typical configuration of palm oil refinery, which are 100^{o}C, under vacuum of 50 torr and contact time of 30 minutes;

5.3.1 Equipment

Rotavapor unit is used to carried out the experiment of degumming and bleaching processes, Figure 5.2 shows the image of rotavapor used in this study.

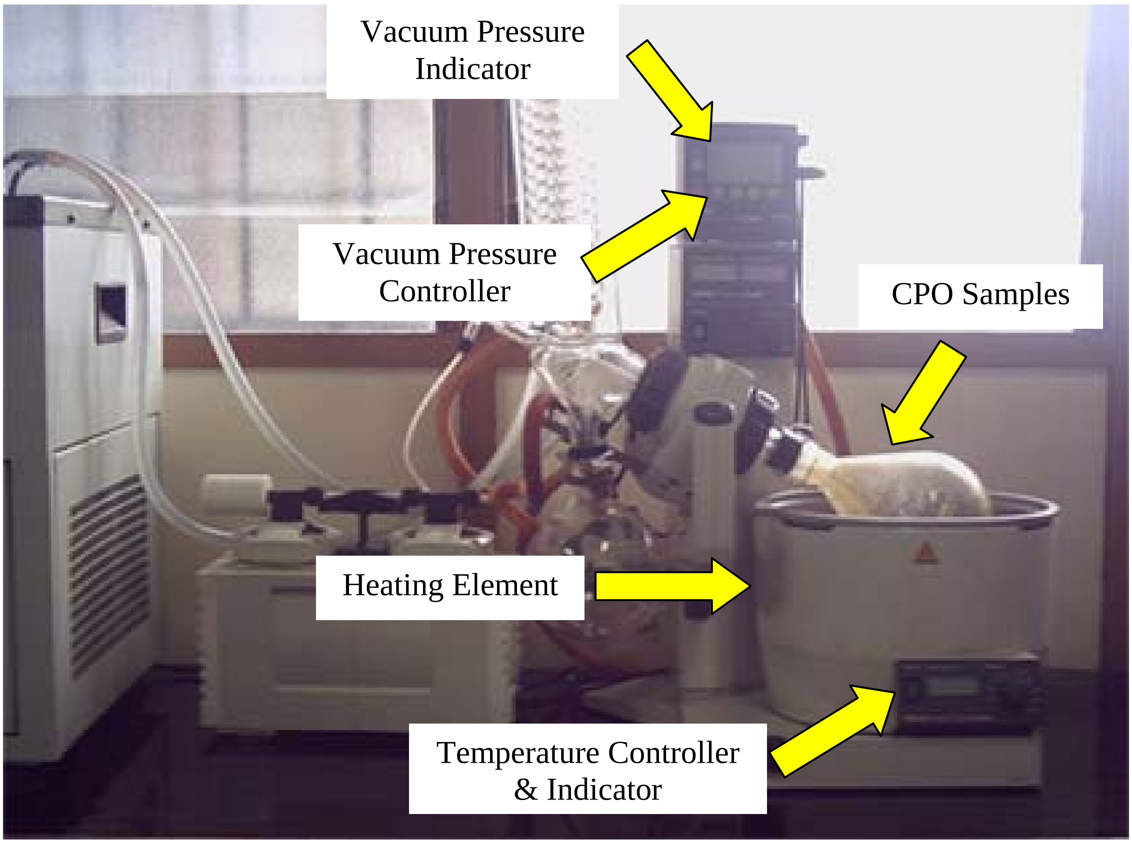


Figure 5.2: Rotavapor unit used for deguming and bleaching process

5.3.2 Experimental Procedure for Degumming and Bleaching Processes

In this study, the CPO sample used for degumming and bleaching processes is 500g for each run. The CPO sample, bleaching earth and phosphoric acid are put into a conical flask before it is attached to rotavapor unit.

Heating element used in this experiment is silicon oil. The silicon oil is initially heated up to $100^{o}C$ and vacuum is set to 50 torr before attaching the conical flask containing the CPO sample, certain dosages of bleaching earth and phosphoric acid.

The dosages of bleaching earth and phosphoric acid are added accordingly based on the experimental arrangement given by the DOE as shown later in Table 5.3, in section 5.3.

When $100^{o}C$ temperature and 50 torr vacuum are achieved, the bleaching and degumming processes will be carried out for 30 minutes and the mixture will be rotated continuously in order to have complete mixing between CPO, phospohoric acid and bleaching earth.

After 30 minutes, the degummed and bleached oil (DBPO) will be filtered with Whatman filter paper under vacuum as soon as possible in order to prevent any undesirable oxidation. Thereafter, the further quality checking experiments will be carried out for analysis.

5.4 Quality Checking Parameters Experiment & Experimental Procedures

The experiments for CPO and DBPO qualities are conducted based on the standard method by PORAM and AOCS.

5.4.1 FFA Experiment

Free fatty acids content in the crude palm oil or degummed and bleached oil can be determine by using titration method according to the PORIM Test Methods (1995). The experimental procedures for the FFA content determination are as follow;

(a) Prepare the sample for the analysis by melting the sample at 60 °C to 70 °C and thoroughly homogenise it before sampling
(b) Determine the size of sample from the following table

Table 5.3: Size of sample for palm oil acidity test

Acidity	Weight of sample (+/- 10 %), grams	Weighing accuracy (grams)
0 to 1	20	0.05
1 to 4	10	0.02
4 to 15	5	0.01
15 to 75	2.5	0.01
75 and over	0.5	0.001

(c) Weigh the specified amount of sample into a Erlemeyer flask
(d) Add 50 ml of neutralised solvent and place the flask on the hot plate and regulate the temperature of about 40 °C
(e) Shake the sample gently while titrating with standard alkali (sodium hydroxide) to the first permanent pink colour. The colour must persist for 30 seconds.

(f) The results can be expressed as below:

$$\textit{\textbf{FFA \% as palmitic acid (for palm oil and fractions)}} = \frac{25.6 \times N \times V}{W}$$

where N = normality of NaOH solution

V = volume of NaOH solution used in ml

W = weight of sample

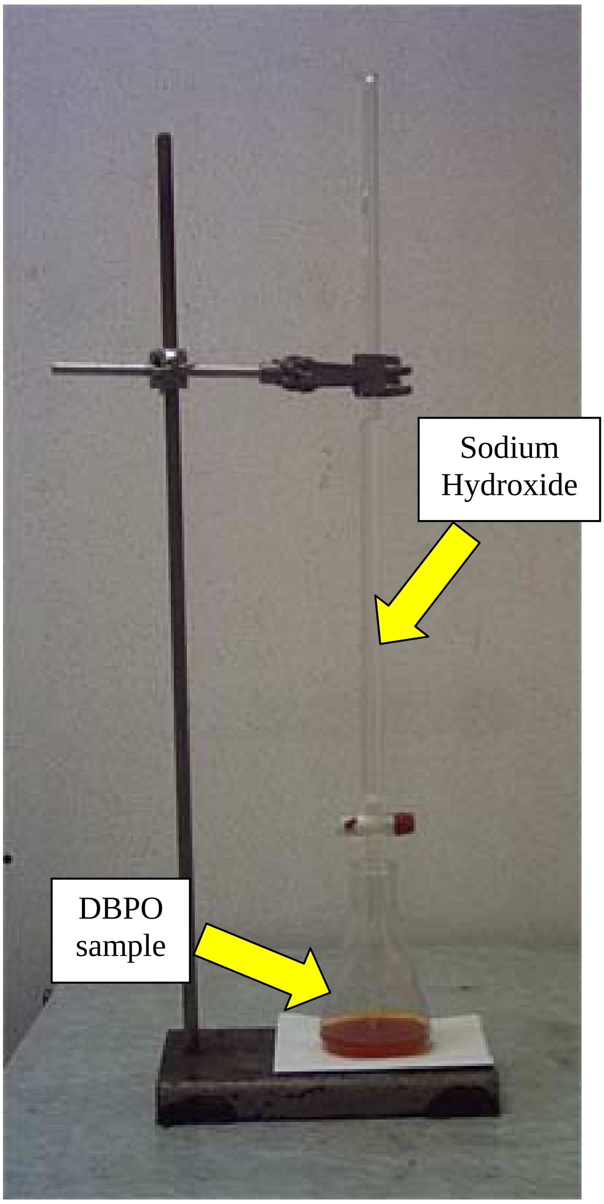


Figure 5.3: Titration method for FFA experiment

5.4.2 DOBI Experiment

DOBI can be measured using UV-visible spectrophotometer and it is a numerical ratio of spectrophotometric absorbance at the wavelength at 446 nm to absorbance at 269 nm. The measurement is carried out on 1% concentration solution of palm oil in isooctane.

The experimental procedures for the determination for DOBI value are as follows:

(a). Weigh about 0.1 g of completely melted and homogenised palm oil sample into 25 ml volumetric flask. Dissolve in so-octane or n-hexane (0.5 –1.0 % concentration) and make-up to the desired volume.

(b). Fill a 10 mm cuvettes with oil solution and measure its absorbances at 269 nm and 446 nm against pure solvent using spectrophotometer (Figure 5.4)

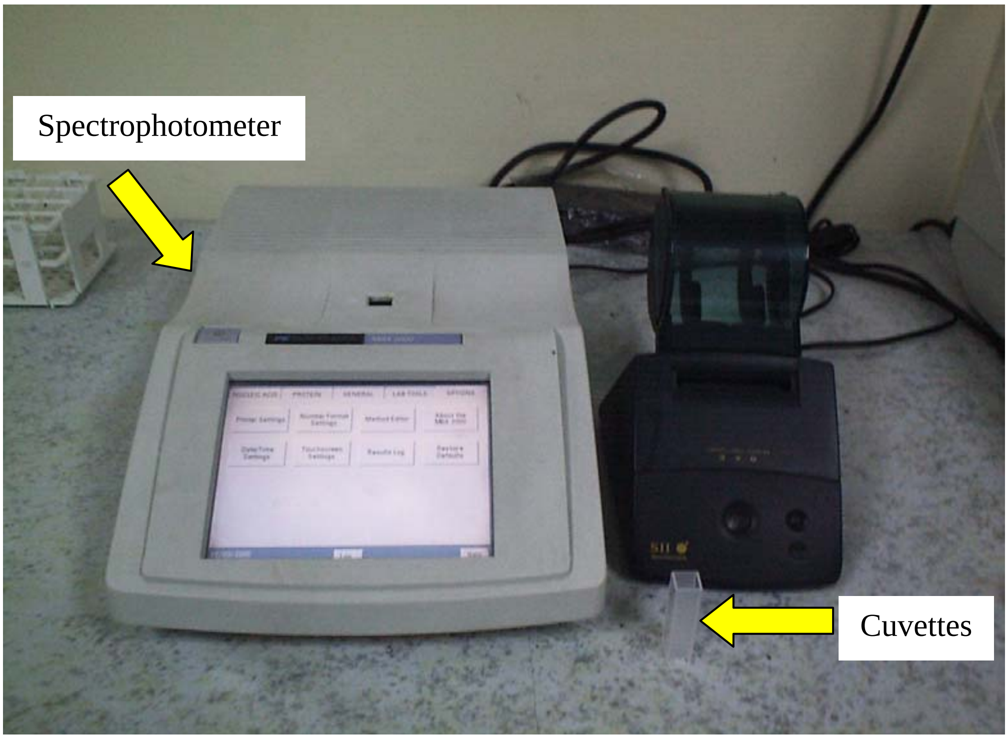


Figure 5.4: Spectrophotometer for determination of DOBI value

(c). Calculate the DOBI value using the equation below;

$$\mathit{DOBI} = \frac{\mathit{Abs\ 446}}{\mathit{Abs\ 269}} \textit{ in a 10mm quartz cell}$$

5.4.3 Peroxide Value Experiment

The peroxide is a measure of those substances in a sample, expresses in terms of milliequivalents of active oxygen per kilogram which oxidize potassium iodide under conditions test

The procedures of this experiment are according to PORIM Test Methods (1995) and are described as follows:

(a) Sample collected should be analysed as soon as possible or should kept in a cool dark place before analysis
(b) Weigh to the nearest 0.1 mg 5.00 +/- 0.05 g of the sample into the 250 ml flask
(c) Add 30 ml of acetic acid –chloroform solution. Swirl the flask until sample is dissolved in the solution.
(d) Add 0.5 ml of saturated potassium iodide with graduated pipette. Swirl for 1 minute and then add 30 ml of distilled water. For freshly produced oil, add a few drops of starch solution
(e) Titrate the solution with 0.01 N sodium thiosulphate solution adding it gradually and with constant vigorous shaking. Add thiosulphate solution dropwise until the blue color just disappears.
(f) Carry out a blank test with determination. The blank titration must not exceed 0.1 ml of the 0.01 N sodium thiosulphate solution
(g) The peroxide value content can be calculated as (the results is expressed in milliequivalent of active oxygen per kilogram of sample) :

$$PV = \frac{(Vs - Vb)\, N \times 1000}{W}$$

where ;

Vs = volume in ml of sodium thiosulphate solution of normality N used for the determination

Vb = volume in ml of sodium thiosulphate solution of normality N used for the blank test.

W = weight in grams of the test portion

N = normality of sodium thiosulphate solution

5.4.4 Phosphorus Content Experiment

This method determines total phosphorus content by charring and ashing in the presence of magnesium oxide followed by colorimetric measurement as phosphovanadomolybdic complex. The procedures are described as below:

(a) Prepare the sample by melting the palm oil at 60 °C to 70 °C and thoroughly homogenise it before taking a test portion.

(b) Weigh to within 1 mg exactly 0.1 g of magnesium oxide into crucible or porcelain dish (Figure 5.6).

(c) Weigh about 5 g of fat into the same crucible (or according to its presumed phosphorus content)

(d) Burn off the fat by ignite it to white ash on the furnace (Figure 5.5) at 800 °C to 900 °C for 2 hours

Figure 5.5: Furnace used for the determination of phosphorus content experiment

Mixture of CPO & magnesium oxide

Figure 5.6: Crucible porcelain containing CPO sample and magnesium oxide

(e) Dissolve the magnesium-containing ashing exactly 5 ml of the aqueous nitric acid solution.

(f) Add exactly 20 ml of a mixture of 10 ml of aqueous ammonium vanadate solution and 10 ml of acidic aqueous ammonium molybdate solution. Mix and allow stand for 20 minutes.

(g) Prepare blank test, not containing the fat exactly the same conditions

(h) Transfer the test solution into the cell of spectrophotometer (Figure 5.4). Measure extinction at 400 nm against blank solution. Read the absorbance. The ppm phosphorus in the fat can be calculated as:

$$\textit{ppm Phosphorus} = \frac{CF \times 25 \times Abs}{\textit{weight of oil (g)}}$$

where CF = calibration factor

Abs = Absorbance value

5.4.5 Moisture Content Experiment

For this experiment, the moisture content experiment will be conducted according to AOCS Recommended Practice Ca 2f-93 method. The experimental procedures are as follows:

(a) Weigh accurately about 15-20 g of well-mixed palm oil sample into tared filter flask containing a magnetic bar as a part of tared weight.

(b) Add 5 ml acetone, using graduated cylinder. Stoppered the flask and placed in glycerol bath which is heated by means of electric hot plate.

(c) The flask is placed under vacuum and with continuous stirring, heated to 100 ^{o}C for 20 min

(d) Remove the flask from the hot bath and cooled at the room temperature while maintaining the vacuum.

(e) Carefully release the vacuum and dry the flask. Place the flask in desiccator (Figure 5.6) for a few minutes and weighed.

Figure 5.6: Desiccator for moisture content experiment

(f) The moisture content can be calculated by using equation below:

$$\textit{Moisture and volatile matter \%} = \frac{\textit{loss in mass, g}}{\textit{Mass of sample, g}} \times 100$$

5.4.6 Iron Content Experiment

For iron content experiment, we sent the sample to AOTD (Analytical Oleochemical Technology Division), Bangi, Selangor for testing. This is because the experiment requires the use of atomic absorption spectrophotometer (AAS) but the equipment is not available at CLEAR.

The experiment measures iron in palm oil dissolved in methyl isobutyl ketone (MIBK). The method used will be based on the PORIM Test Methods (1995). The principal of this experiment is analysing the iron by direct aspiration. The experimental procedures are divided into 2 parts:

Preparation of Standards 100 ppm Stock Standard in Oil

(a) Weigh exactly 250.00 gn of RBD palm oil in a clean 500 ml round bottom flask
(b) Weigh 0.1581 g of Ferric acetlacetone in a clean 50 ml beaker
(c) Dissolve the salt in approximately 25 ml ethanol and transfer quantitatively to the flask. Wash the beaker thoroughly with a further 25 ml ethanol into the flask.
(d) Evaporate all the ethanol from the oil using rotary evaporator under vacuum at 60 ^oC until constant weight.

Working Standard in Oil

(a) Prepare working standards of 1,2,3,4,5,6,8 and 10 ppm by diluting (w/w) the 100 ppm stock standard with 'metal-free' RBD palm oil
(b) Weigh 2.50 g of melted samples and RBDPO containing working standards into 25 ml volumetric flasks. Dilute with MIBK and mix. Keep the solutions in an oven maintained at 40 ^oC
(c) Set up instrument for analysis of iron with air-acetylene-organic solvent flame at wavelength 248.3 nm and slit width of 0.2 nm
(d) Aspirate 'metal free' RBDPO solution, allow the absorbance meter reading to be stable and set absorbance to be zero. The zero setting may

fluctuate within ± 0.005 absorbance unit. The instrument must be recalibrated if values outside this limit are obtained.

(e) Aspirate the working standards and samples and record the absorbance readings. Take the average of three absorbance readings.

(f) Concentration of iron can be determined by plotting the calibration curve versus absorbance of standard solution and read the concentration of iron sample from the graph.

5.5 Design of Experiment Application in Estimating Optimum Number of Experiments

Design of Experiment (DOE) was applied to arrange the number of experiments run for the study. With the help of DOE method, there are 20 set of experiment were arranged. All the 20 set of experiments with 6 quality checking experiments each, has been determined through Central Composite Design (CCD) technique via Response Surface Method (RSM) using Minitab 14 environment.

5.5.1 Optimum Number of Experiment

Table 5.4 below, shows the arrangement of experimental sequence given DOE method. In total, there are 120 numbers of experiments to be run, for each CPO (crude palm oil) and (DBPO) degummed bleached palm oil with 3 repetitions for each experiment in order to get a good result.

Table 5.4: Arrangement of experiment sequence by DOE

Run Order	**Bleaching Earth Dosage (wt%)**	**Phosphoric Acid Dosage (wt%)**	*** FFA Content**	**FFA Category**
1	1	0.5	3	B
2	1	0.5	1	A
3	0	1.0	1	A
4	2	0.5	3	B
5	2	1.0	5	C
6	2	1.0	1	A
7	1	0.5	3	B
8	1	0.0	3	B
9	2	0.0	1	A
10	1	0.5	3	B
11	0	0.0	1	A
12	1	0.5	3	B
13	1	0.5	3	B
14	1	0.5	3	B

15	0	0.0	5	C
16	0	1.0	5	C
17	1	1.0	3	B
18	0	0.5	3	B
19	2	0.0	5	C
20	1	0.5	5	C

* FFA content 1: Category A of CPO

FFA content 2: Category B of CPO

FFA content 3: Category C of CPO

5.6 Model Development

Using MATLAB Neural Network Toolbox and MATLAB environment, a feed forward neural network was designed and back-propagation training algorithm. In this study, 3 models of back-propagation feed forward network were developed in MATLAB 7.0 environment. The models are MISO 1, MISO 2 and MIMO. The details on each model will be discussed later in; Neural network modeling section.

5.6.1 Selection of Input and Output variables

The most important task in developing a NN model is to select the most significant variables as estimator inputs. In the process of degumming and beaching of palm oil, there are a lot of variables that can affect the efficiency of system. Therefore, in order to simplify the model structure, input variables that gave dominant impact to the process outputs were selected while the others were neglected. The independent experimental variables selected are FFA content, DOBI, Peroxide Value (PV), Phosphorus (P) content, moisture content and iron (Fe) content for each CPO and DBPO. The outputs were phosphoric acid and bleaching earth dosages.

5.6.2 Neural Network Modelling

From the 20 set experiments arranged by response surface method, 14 experimental data are employed for model development (training) data set and 6 experimental data are employed for validation (generalization) data set, based on rule of thumb of ratio 70:30.

The developments of neural network are accomplished by following the all the process phases as discussed previously in section 4.2.5: Neural Network Development. The overall steps of neural network modelling are shown in the flowcharts (Figure 5.7) below;

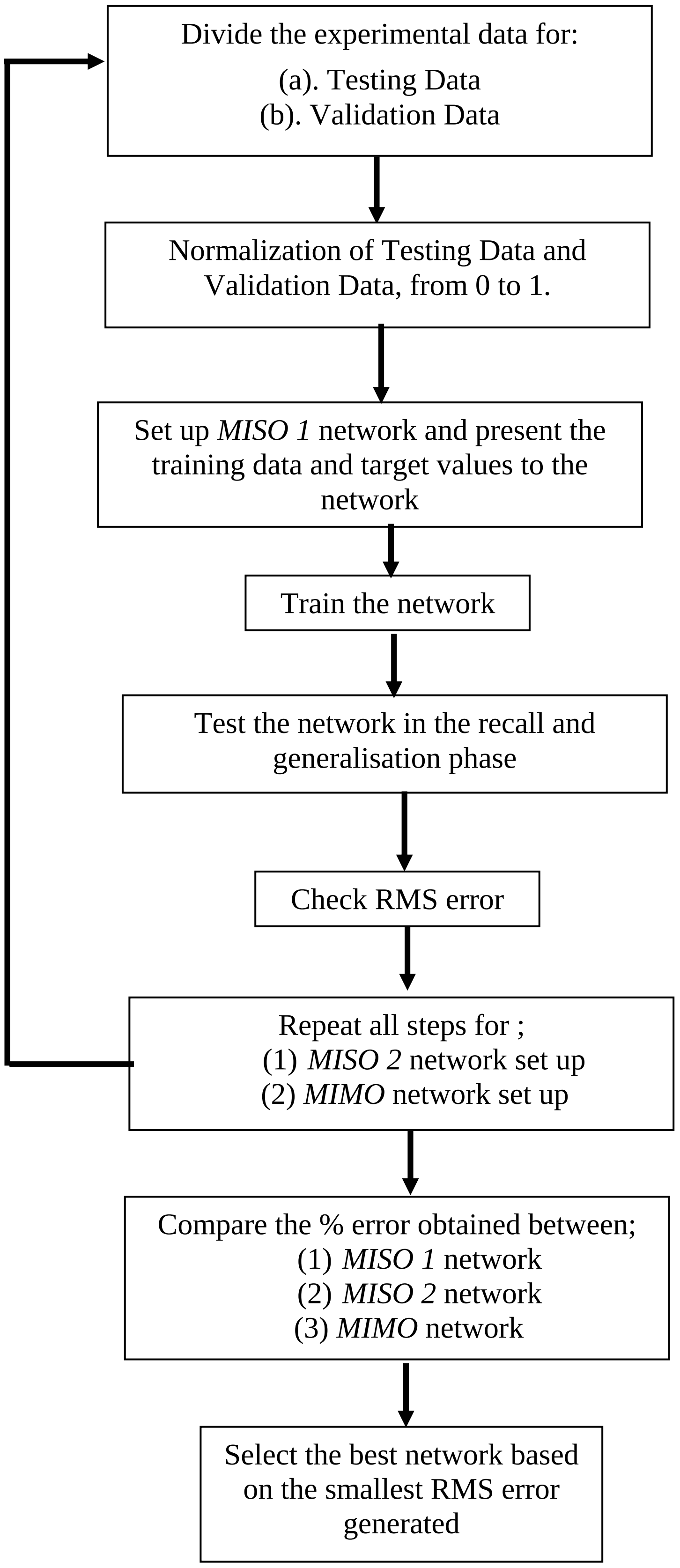


Figure 5.7: Modelling a network methodology

In this study, there are 12 independent experimental variables identified because it involved 6 quality parameters for each CPO and DBPO for degumming and bleaching processes. The independent experimental variables are as per listed in Table 5.5 below;

Table 5.5: List of independent variables

Independent Variables	**Symbol**
FFA_{CPO}	X_1
FFA_{DBPO}	X_2
$DOBI_{CPO}$	X_3
$DOBI_{DBPO}$	X_4
PV_{CPO}	X_5
PV_{DBPO}	X_6
P_{CPO}	X_7
P_{DBPO}	X_8
$Moisture_{CPO}$	X_9
$Moisture_{DBPO}$	X_{10}
Fe_{CPO}	X_{11}
Fe_{DBPO}	X_{12}

* P: Phosphorus
Fe: Iron

The response factors or outputs were phosphoric acid dosage (Y_1) and bleaching earth dosage (Y_2).

In modelling a neural network, after dividing the experimental data for training and validation, the next step is to do normalization of experimental data. This is because the process inputs for neural network models were inconsistent in term of their magnitudes. Therefore, these input data were scaled to be within a consistent range (from 0 to 1), before introducing them to the input layer of the network to ensure that each data was given a fair contribution in determining the network output. The data scaling method employed in this research is shown as follows:

$$X_{is} = \frac{X_i - X_{i\min}}{X_{i\max} - X_{i\min}}$$

where, X_{is} is scaled input and X_i is the actual input before scaling whereas $X_{i\min}$ and $X_{i\max}$ are the minimum and maximum values of the inputs respectively. The maximum and minimum values of the input were selected based on training data.

As mention previously, there are two types of networks were developed and studied namely;

(i) Multi-Input Single-Output (MISO) network
(ii) Multi-Input Multi-Output (MIMO) network.

Two MISO (MISO 1 and MISO2) and one MIMO networks were developed, as per shown in Figure 5.7. A back-propagation feed forward neural network was employed for both networks.

Figure 5.7 :

The selection criterion is dependent on the efficiency of the two networks in predicting the process output. Therefore, the performance of both networks were tested and compared. The best network with smallest error was chosen to represent the process.

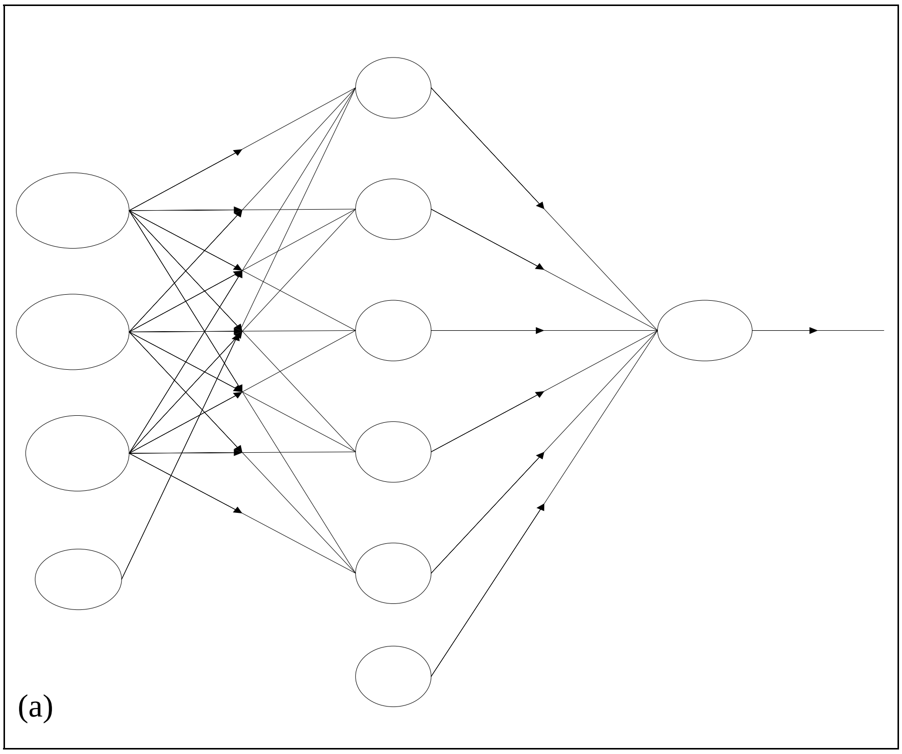
The M-file for MISO network of each response factors (phosphoric acid dosage (Y_1) and bleaching earth dosage (Y_2) are shown in Appendix C and D. Whereas, the M-File for MIMO network is shown in Apendix E.

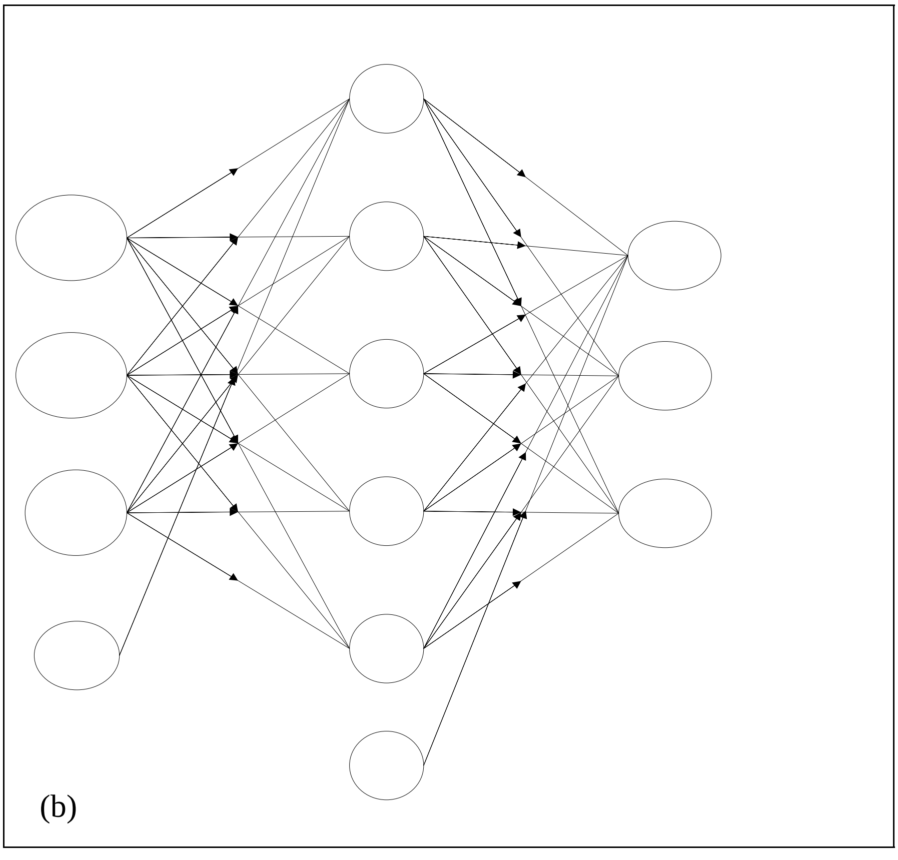
Neural Network toolbox in MATLAB 7 was used to design and simulate the networks. The transfer functions employed in the networks were log-sigmoid transfer function and purelin transfer function and the networks were trained using Levenberg-Marquardt learning algorithm (Trainlm).

In order to select the best network topology/architecture a systematic trial and error was utilized where each of the studied neural network topology was carried out for 10 times runs. The information was then propagated forward to the output layer where the output was calculated and compared to the actual value in order to calculate the prediction error.

The network with smallest root mean sum of squares of the errors (RMSE) was selected to represent the process. The values of weights and biases associated to each connection between neurons of adjacent layers of chosen network were obtained. The RMSE is defined as

$$RMSE = \sqrt{\frac{\sum\left(Observed - \Pr edicted\right)^2}{No.\,of\ \ Data}}$$


(a)


(b)

Input

Ten

Rat

Figure 3.4 Feed Forward Neural Networks Topology (a) MISO Neural Networks (b) MIMO neural Networks

5.6.2.1 MISO network model

5.6.2.2 MIMO network model

5.7 Design of Experiment Application in Estimating Optimum Number of Experiments

There were 22 experimental data used to develop the neural networks models where 16 experimental data arranged by response surface methodology were employed as training data and six experimental data were used as generalized data or validation data

5.8 Model Development
5.8.1 Selection of Input and Output variables
5.8.2 Neural Network Modelling
5.8.2.1 MISO network model
5.8.2.2 MIMO network model
5.8.3 Model Comparisons

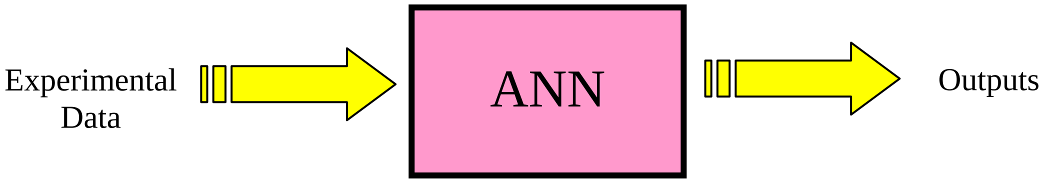
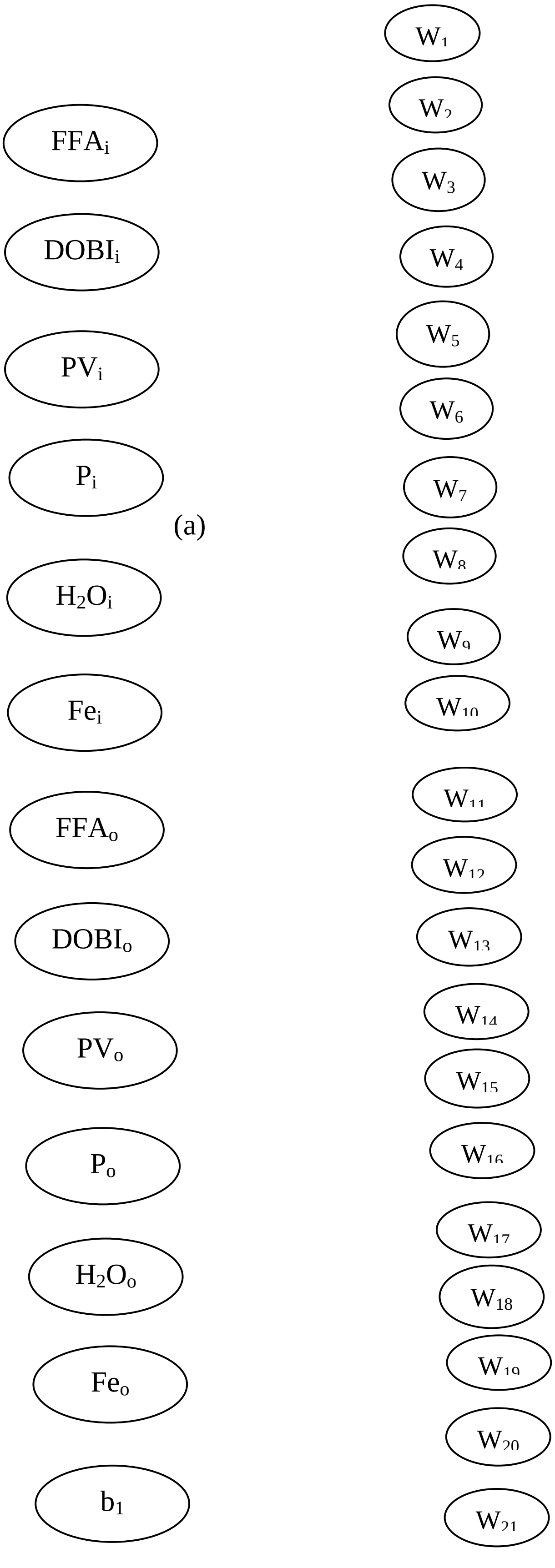


Figure 5.5 : General scheme of neural network architecture


W_1
W_2
FFA_i
W_3
$DOBI_i$
W_4
W_5
PV_i
W_6
P_i
W_7
(a)
W_8
H_2O_i
W_9
W_{10}
Fe_i
W_{11}
FFA_o
W_{12}
$DOBI_o$
W_{13}
W_{14}
PV_o
W_{15}
W_{16}
P_o
W_{17}
H_2O_o
W_{18}
W_{19}
Fe_o
W_{20}
b_1
W_{21}


Bias 1
W22
W23
W24

CHAPTER 6

RESULTS AND DISCUSSION

6.1 Results Overview

In this research, there are two methods in the determining the suitable ratio of phosphoric acid and bleaching earth dosage to be added to degumming and bleaching process of palm oil refining, which is via experimental works and through modelling of ANN.

The degumming and bleaching experiment were carried out at optimum operating conditions, with different dosage of phosphoric acid (PA) and bleaching earth (BE), which were specified by design of experiment (DOE) method. The category of crude palm oil (CPO) are divided into category A, B and C. The parameters that are being monitored in this experiment are FFA content, DOBI value, peroxide value (PV), phosphorus content, moisture content and iron content.

For ANN modelling part, the experimental data obtained are also then be used as the input vectors to the network. Three NN models (two MISO and one MIMO) were developed and compared in which the best model will be selected best on the smallest error generated.

These results will at the end gives some valuable information that would suggest some improvements to the degumming and bleaching practices in Malaysia’s palm oil refineries.

6.2 Experimental Results Analysis

In this section, experimental results will be analysed and discussed based on each parameter involved. Degumming and bleaching processes was done at optimum temperature (100^{o}C), pressure (vacuum – 50 torr) and contact time (30 minutes) in order to study the effects of phosphorus acid and bleaching earth dosages on each parameter studied namely; FFA content, DOBI value, PV value, phosphorus content, moisture content and iron content. During experiment, each sample was run 3 times in order to get good average results.

The graphs are plotted based on varying the bleaching earth dosage at constant phosphoric acid dosage at 0.5 wt% and 1.0 wt% for each parameter involved.

6.2.1 Effects of Phosphoric Acid and Bleaching Earth Dosage on FFA

Category	Before Degumming	After Degumming
B	2.84	3.28
A	1.21	1.42
B	2.9	3.14
C	3.74	3.97
B	3.05	3.4

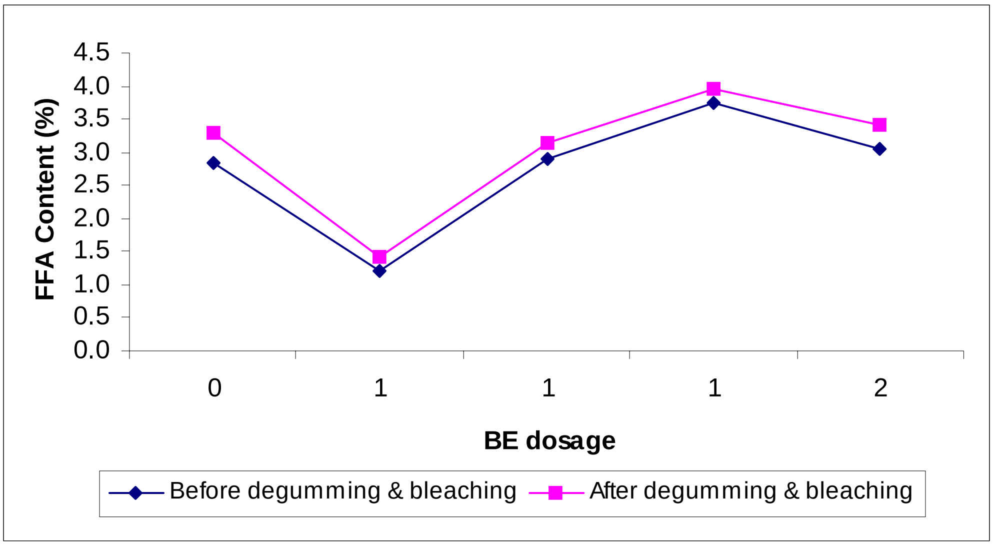


Figure 6.1: FFA Content of CPO Before & After Degumming and Bleaching Processes at 0.5 wt % of Phosphoric Acid

Category	Before Degumming	After Degumming
A	1.48	2.3
C	3.69	4.48
B	3.04	3.11
A	1.35	1.42
C	3.81	4.07

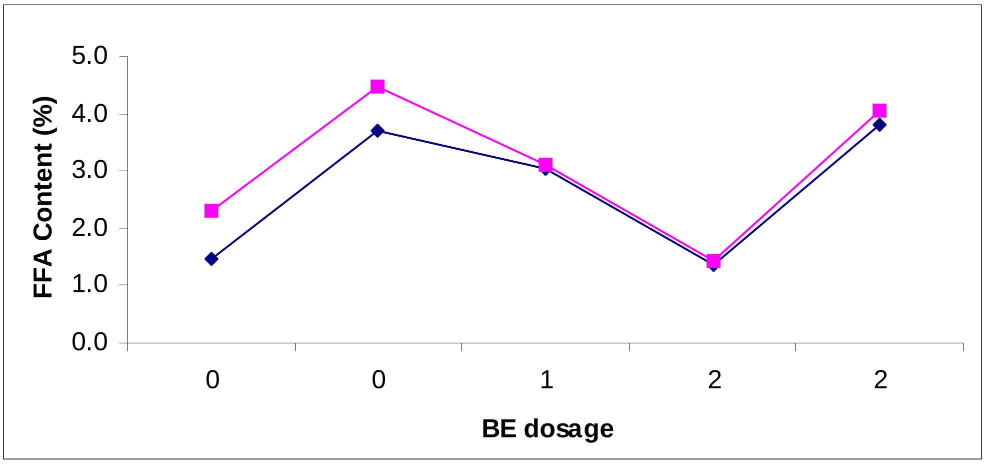


Figure 6.2: FFA Content of CPO Before & After Degumming and Bleaching Processes at 1.0 wt % of Phosphoric Acid

FFA is the amount of fatty acid occurring naturally or produced during storage or processing that exists in edible oils as a distinct chemical unit in an uncombined state (Wan and Wakelyn, 1997). The quantity of FFA present is a good measure of the quality of the crude oil.

FFA levels in crude oils can be adversely affected by bleaching earth conditions and acidity of the adsorbent (bleaching earth) as well as nature properties of the phosphoric acid used. Small changes in FFA levels can have a great impact on oil loss. Mathematical models show that a FFA rise of 0.1%for a refinery with a production rate of 1000 tonnes per day will result in an annual loss of 365 tonnes (Brooks, 1999).

Higher FFA means lower triglyceride (TG) since FFA is derived from triglycerides (TG). In addition, high FFA not only infers lower extractable oil but also corresponds to the higher content of emulsifiers namely diglycerides (DG) and monoglyceride (MG) and significant presence of DG and MG has shown effects on crystallization of oil during fractionation process (Abdul Azis, 2000).

Figure 6.1 and 6.2 show the effects on FFA content on all categories of CPO (A, B and C) by varying the bleaching earth dosage at 0.5 wt% and 1.0 wt% phosphoric acid dosage respectively.

Generally, the graph in Figure 6.1 shows the content of FFA is found higher after degumming and bleaching processes, for all points due to acidic nature of phosphoric acid added and due to splitting or hydrolysis of triglyceride by heating. The reaction is shown as below, (Gapor and Chong, 1985);

$$\text{Triglycerides} + H_2O \longrightarrow \text{FFA} + \text{Glycerides}. \ldots\ldots 6.1$$

From the graph, when phosphoric acid is added at 0.5 wt % but with no bleaching earth the increment of FFA content is highest about 0.44 %. This is because no adsorption process occurs due to the absence of bleaching earth.

However, even though bleaching earth are added during the degumming and bleaching process there is still some rise (about 0.2 - 0.35%) of the FFA content in degummed oil. Traces of phosphoric acid remaining in the oil after degumming treatment are said to be responsible for such situation to occur (Rossi et al. 2003). For this graph, the lowest FFA increment detected is for category A of oil at 1.0 wt% of bleaching earth with 0.5 wt% phosphoric acid which is about 0.21% Figure 61.

Figure 6.2 shows the effect on FFA content at various dosage of bleaching earth at 1.0 wt% of phosphoric acid. From the graph, it can be observed that similar trend with the previous graph, where when there is no bleaching earth added to the degumming and bleaching treatment (means adsorption process), the rise in FFA content is quite high, about 0.8% in average. This rise of FFA is also attributed to the higher acidity of phosphoric used during the treatment which is 1.0 wt%.

However at high dosage of bleaching earth of 1-2 wt%, the increment is quite insignificant. This is because the increase in FFA content is apparently being adsorbed by the bleaching earth and with that amount of bleaching earth, it does not allow further splitting of triglycerides to occur. In addition, during adsorption process impurities such as FFA are retained on the pore surface of the bleaching earth, however when molecules are not adsorb triglycerides escapes. Gradually, the concentration of the of the impurities on available surface of the bleaching earth and the concentration remaining in the oil is in equilibrium, so further exchange is negligible (Patterson, 1992). It is said optimum condition is achieved.

Based on the both graphs it can be deduced that the optimal dosage of bleaching earth and phosphoric acid for all categories of CPO (A,B and C) is 1.0 – 2.0 wt% and 0.5 –1.0 wt% respectively

6.2.2 Effects of Phosphoric Acid and Bleaching Earth Dosage on DOBI

Category	Before Degumming	After Degumming
B	2.43	1.98
A	3.84	1.61
B	3.00	1.80
C	3.00	2.21
B	2.35	1.62

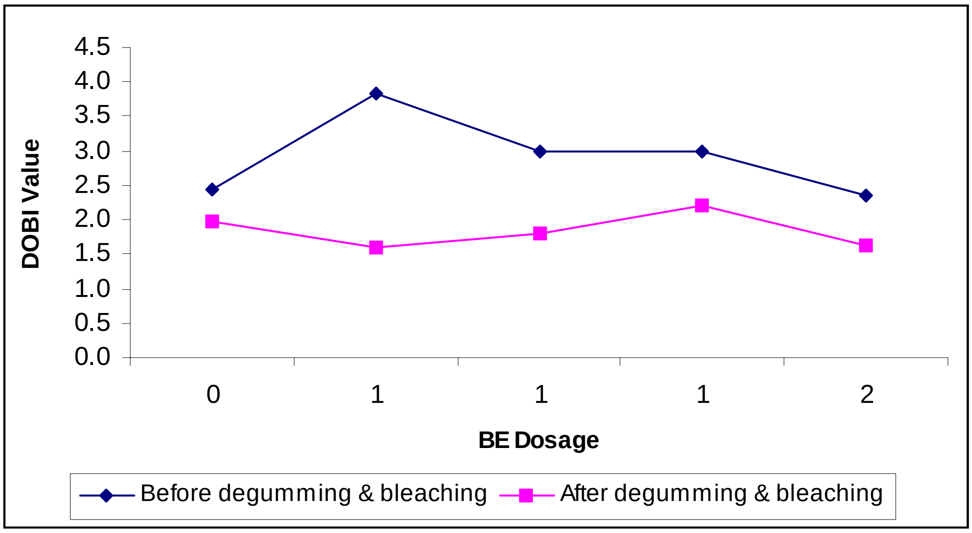


Figure 6.3: DOBI Value of CPO Before & After Degumming and Bleaching Processes at 0.5 wt % of Phosphoric Acid

Category	Before Degumming	After Degumming
A	2.38	1.97
C	2.39	2.05
B	3.17	1.99
A	3.20	1.92
C	2.22	1.58

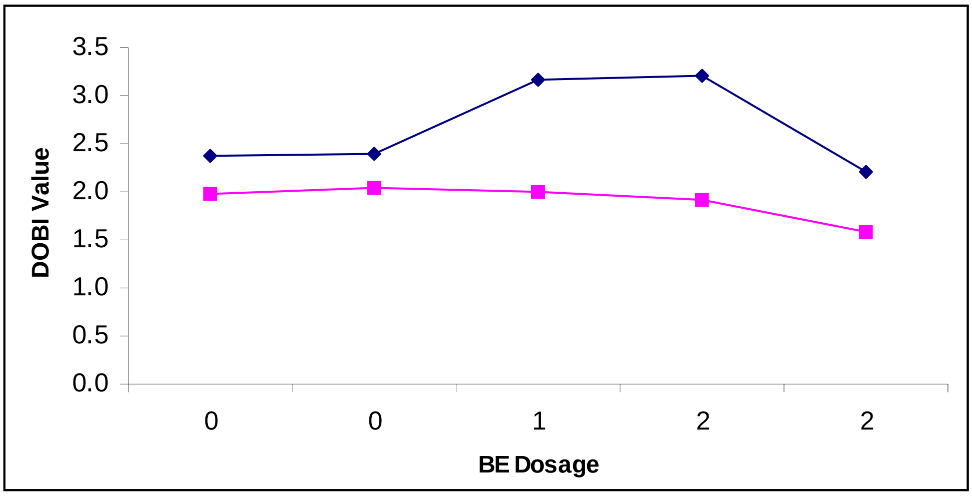


Figure 6.4: DOBI Value of CPO Before & After Degumming and Bleaching Processes at 1.0 wt % of Phosphoric Acid

According to Abdul Azis (2000), all refineries will undoubtedly prefer crude palm oil (CPO) exhibiting good DOBI value which is minimum of 2.3 and the value will equate nicely with reduced bleaching earth requirement. Low DOBI value relates to high oxidation products. High oxidation products require high dosage of bleaching earth.

Therefore, with high oxidation products (low DOBI) and with increase of FFA, the cost of utilities on a per tonne basis of RBDPO production will subsequently increased. The deodorization conditions may have to be reformatted to ensure complete removal of stubborn volatiles (oxidized products, moisture, carotene and its derivatives) and all remnants of FFA. These measures will associate with higher utility requirements (Abdul Azis, 2000).

Figure 6.3 and 6.4 show the effects on DOBI value on all categories of CPO (A, B and C) by varying the bleaching earth dosage at 0.5 wt% and 1.0 wt% phosphoric acid dosage respectively.

Generally for the graph in Figure 6.3, all the CPO samples used exhibit a good DOBI value which is higher than 2.3. This indicates that all the samples used have low oxidation products. After degumming and bleaching processes, the DOBI values are generally reduced. However, the reduction in DOBI value is quite small, when there is no bleaching earth is added to the process. Thus, no adsorption process of oxidized products can take place due to the absence of bleaching earth. But, it seems that an effective degumming and bleaching process happen for category A of oil when the bleaching earth added is at 1.0 wt% where the reduction of DOBI value is more than half of its initial value (from 3.84 to 1.61).

Whereas for the samples used for the second graph in Figure 6.4, all the samples except for the last sample (category C) of oil, exhibit good DOBI values. An effective degumming and bleaching process seems to happen for category B and A of oil when the bleaching earth added is at 1.0 wt% and 2.0 wt% respectively. The reduction of DOBI values for both categories is about 1.2.

6.2.3 Effects of Phosphoric Acid and Bleaching Earth Dosage on PV

Category	Before Degumming	After Degumming
B	1.78	1.53
A	2.4	0
B	1.97	0
C	3.41	0
B	2.11	0

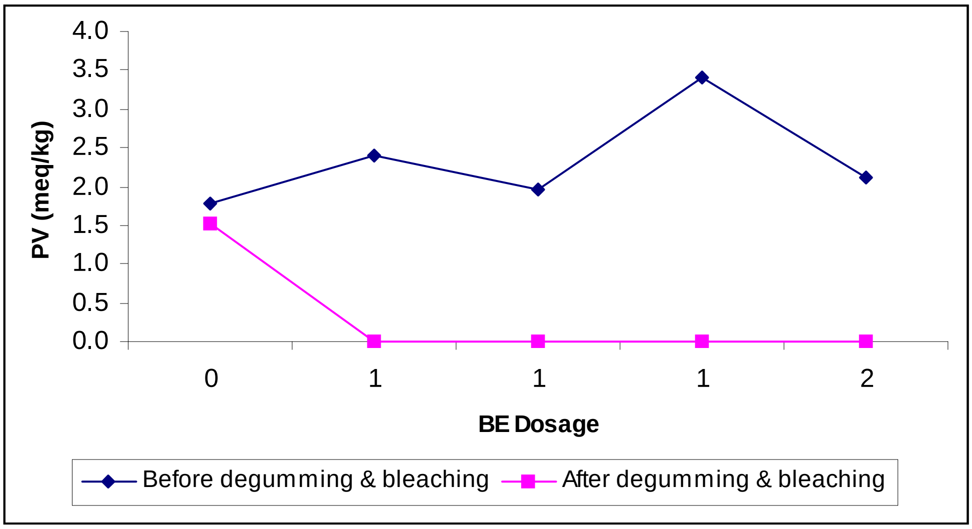


Figure 6.5: PV of CPO Before & After Degumming and Bleaching Processes at 0.5 wt % of Phosphoric Acid

Category	Before Degumming	After Degumming
A	2.9	2.83
C	3.38	3.14
B	1.46	0
A	1.4	0
C	3.29	0

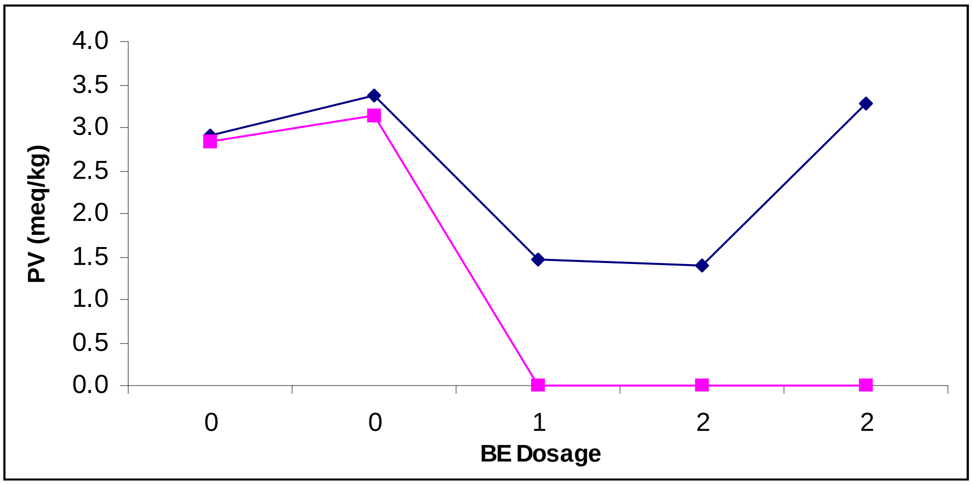


Figure 6.6: PV of CPO Before & After Degumming and Bleaching Processes at 1.0 wt % of Phosphoric Acid

Peroxide value (PV) is an indicator of primary oxidation of the oil. PV is already reduced by degumming treatment and it is further lowered after the bleaching process (Rossi et al., 2003). During bleaching stage, peroxides are removed by adsorptive effect of the bleaching earth.

The efficiency of adsorption of oxidation products such as peroxides is dependant on the starting quality of crude oils. If the content of oxidation products is relatively high, a greater dosage of clays is needed (Wei et al., 2003)Bleaching earth clays can adsorb peroxides as well as catalyze peroxide degradation into secondary oxidation products (Rossi et al., 2003).

Figure 6.5 and 6.6 show the effects on peroxide value (PV) on all categories of CPO (A, B and C) by varying the bleaching earth dosage at 0.5 wt% and 1.0 wt% phosphoric acid dosage respectively.

Unlike FFA, the variations in bleaching earth and phosphoric acid dosage affecting values of PV, the graphs will be discussed together. Overall, for both graphs the peroxide value (PV) decreases after degumming and bleaching processes. For both graphs, the decrease in PV is quite small when no bleaching earth is added at 0.5 wt% of phosphoric acid. This proves that degumming treatment by itself reduces only slightly the PV value.

At high bleaching earth dosages, which are 1 – 2 wt%, the PV value is undetectable due to high adsorption capacity of bleaching earth and due to double actions of degumming and bleaching processes. This observation were supported by Wei et al., (2003), based on their research work, where they also claimed that the PV value is reduced to undetectable value with the bleaching earth dosages greater than 0.8wt%.

According to Er (1985), it is desired to have no PV in degummed and bleached palm oil (DBPO) but this cannot be achieved with degumming treatment alone with the absence of bleaching earth in the processes. This indicates that both degumming treatment and bleaching process take places effectively and accomplished desired value of PV in DBPO. It can be concluded that the reduction of PV is mainly attributed to bleaching

6.2.2 Effects of Phosphoric Acid and Bleaching Earth Dosage on Phosphorus

Category	Before Degumming	After Degumming
B	11.05	9.87
A	10.3	4.18
B	10.18	4.14
C	14.2	4.1
B	10.72	3.52

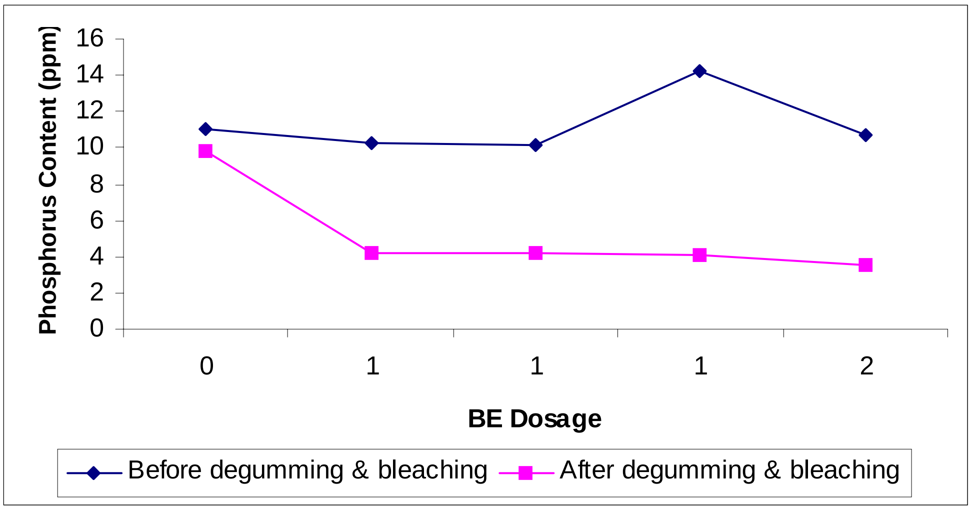


Figure 6.7: Phosphorus Content of CPO Before & After Degumming and Bleaching Processes at 0.5 wt % of Phosphoric Acid

Category	Before Degumming	After Degumming
A	12.02	10.5
C	18.2	16.7
B	11.38	3.54
A	10.46	3.14
C	16.84	3.1

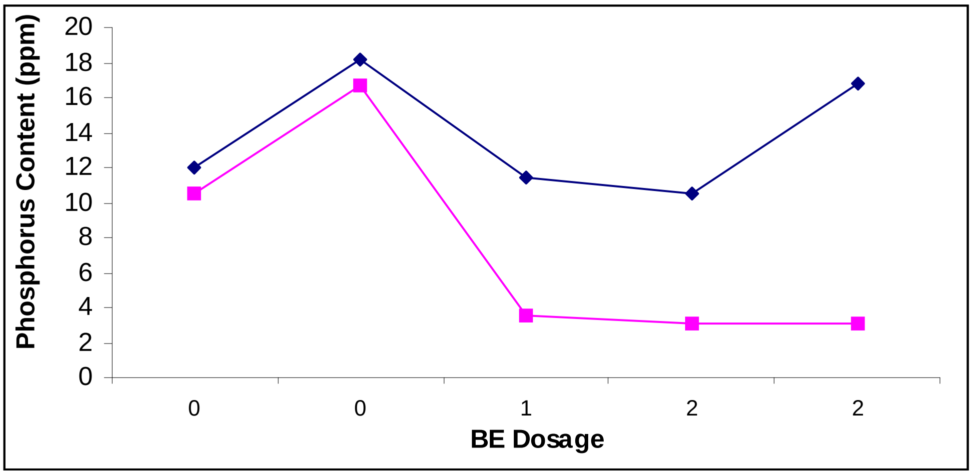


Figure 6.8: Phosphorus Content of CPO Before & After Degumming and Bleaching Processes at 1.0 wt % of Phosphoric Acid

The total phosphorus content is a measurement of total phosphatides, such as phospholipids and some inorganic phosphates present in the oil (Wei, 2003). Phosphorus in oil has been considered as an oxidative impurity and a catalyst poison (Gee et al., 1985).

It is absolute necessary to obtain a good quality of degummed bleached palm oil for physical refining. It is proposed that the very secure standard that the phosphorus content of the crude degummed oil should not exceed 20 ppm and the phosphorus content of pretreated oil immediately prior to physical refining should not exceed 5 ppm (Patterson, 1992).

The use of high dosage of phosphoric acid results in a higher phosphorus content in the oil, which cannot then be reduced by bleaching clay treatment at a reasonable level (Zschau, 1983). According to Wei et al.,(2003), the reduction of phosphorus is proportionate to the dosage of clay used. Kheok and Lim (1982), suggested that the mechanism for phosphors reduction was adsorption of phosphorus ions on the lattice structure of the clay.

Figure 6.7 and 6.8 show the effects on phosphorus content on all categories of CPO (A, B and C) by varying the bleaching earth dosage at 0.5 wt% and 1.0 wt% phosphoric acid dosage respectively.

In general, for both graphs, the phosphorus content is higher before the degumming and bleaching processes. After the degumming and bleaching process, the phosphorus content is generally reduced. However, for both graphs when there is no bleaching earth added, the amount of phosphorus reduction is considered small about 1-2 ppm. This is because no adsorption of phosphors can occur due to the absence of bleaching earth

However, when high dosage of bleaching earth is added during degumming and bleaching, noticeable reduction of phosphorus content is observed. The phosphorus content is reduced up to acceptable level of DBPO, which is less than 5 ppm.

From the graph in Figure 6.7, an effective degumming and bleaching processing for phosphorus reduction is discovered at bleaching earth of 1.0 wt% with 0.5 wt% of phosphoric acid, where the reduction is about 10 ppm.

Whereas, for the graph in Figure 6.8, the reduction of phosphorus occurred efficiently at bleaching earth of 2 wt % with 1.0 wt% of phosphoric acid. This proves the deduction made by Wei et al., (2003), that the reduction of phosphorus is proportional to the bleaching earth dosage; higher bleaching earth dosage the better the reduction of phosphorus in oil.

6.2.2 Effects of Phosphoric Acid and Bleaching Earth Dosage on Moisture

Category	Before Degumming	After Degumming
B	1.87	2.38
A	0.92	0.16
B	2.11	0.17
C	0.42	0.19
B	0.75	0.19

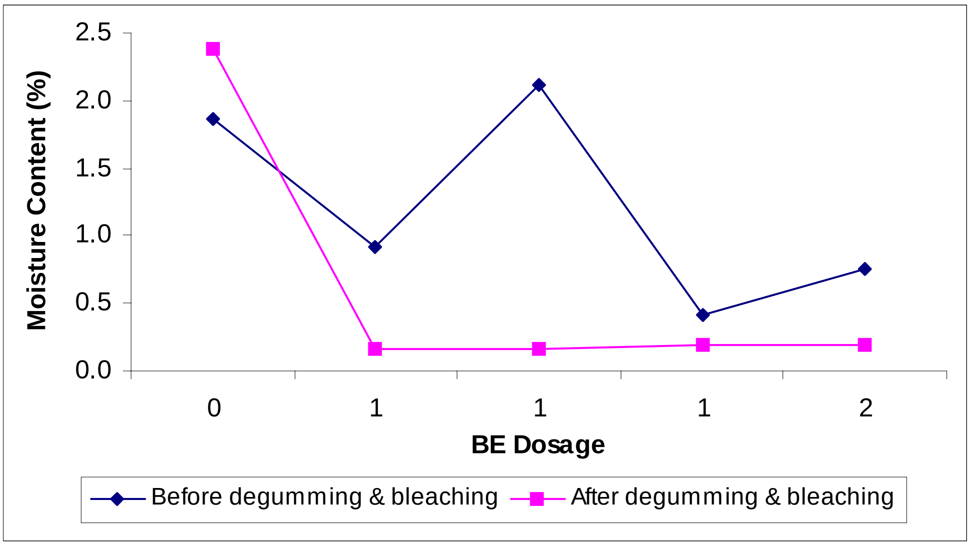


Figure 6.9: Moisture Content of CPO Before & After Degumming and Bleaching Processes at 0.5 wt % of Phosphoric Acid

Category	Before Degumming	After Degumming
A	0.1	0.98
C	2.78	3.57
B	2.05	0.14
A	2.26	0.19
C	2.51	0.20

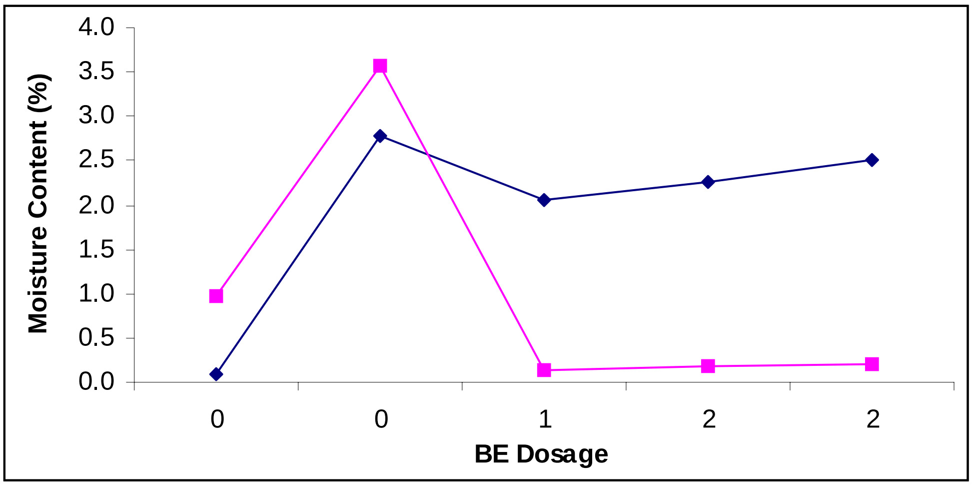


Figure 6.10: Moisture Content of CPO Before & After Degumming and Bleaching Processes at 1.0 wt % of Phosphoric Acid

The main reactions that cause the quality deterioration of palm oil are hydrolysis and oxidation. Among the factors that causing these reactions are the presence of moisture and trace metals content (Gapor and Chong, 1985).

The presence of moisture could cause the hydrolysis of palm oil triglycerides resulting in the formation of free fatty acids (FFA) and partial glycerides (Gapor and Chong, 1985) and the reaction is as per shown in equation 6.1 earlier.

One of the vital roles of bleaching earth is to adsorb undesirable impurities such as moisture content (Hamilton, 1995). Theoretically, solely hydrolytic deterioration can be prevented if the oil is as dry as possible. It is important to consider the effect of hydrolysis because apart from increasing the FFA content and associated with refining losses; hydrolysis also results in formation partial glycerides, which would significantly influence the crystallization behaviour and stability of emulsion (Jacobsberg, 1983).

However, moisture content has shown opposite effects on the oxidation reactions. It has been reported that dissolved water could affect the oxidation process hydration of trace metals, hence decreasing their catalytic activity, promoting the recombination of free radicals and by hydration of hydroperoxides, thus slowing down their breakdown (Gapor and Chong, 1985).

Therefore, a compromise in moisture level (final product RBDPO) for storage and transportation has been suggested by Jacobsberg, (1983), where he concluded that 0.1 % level are optimal for reducing the chemical splitting rate at normal storage and transportation temperature. Thus, to ensure that this optimum level are obtainable in RBDPO, an efficient degumming and bleaching process is important to assure that moisture in degummed and bleaching oil (DBPO) is within the acceptable range level of 0.2% (Er, 1985).

Figure 6.9 and 6.10 shows the effects of phosphoric acid and bleaching earth dosage on moisture content during degumming and bleaching processes for all categories of CPO (A, B and C).

From Figure 6.9 it can be observed that there is a significant increase in moisture content after degumming and bleaching processes when phosphoric acid added at 0.5 wt% but no bleaching earth. This is due to moisture content already present in phosphoric acid added and there is no adsorption process of moisture occurred due to the absence of bleaching earth.

When the bleaching earth dosage is added at 1wt% and 2 wt%, the moisture content is reduced up to acceptable level of DBPO between 0.16 – 0.19 %. From Figure 6.9, it seems the moisture adsorption process on bleaching earth surface occurred effectively at the dosage of 0.5 wt% phosphoric acid and 1wt % bleaching earth, as it reduces the level of moisture content from 2.11 % to 0.27%.

Same trend are observed in Figure 6.10, where the moisture level becomes higher after degumming and bleaching processes when 1.0 wt% phosphoric acid are added but with absence of bleaching earth. Nevertheless, the increase of moisture content is relatively higher (0.7 - 0.9 %) compared to when 0.5 wt% of phosphoric acid (0.5 %) with no bleaching earth are added to the CPO. This is because higher moisture content in 1wt% of phosphoric acid compared to 0.5 wt% of phosphoric acid.

6.2.2 Effects of Phosphoric Acid and Bleaching Earth Dosage on Iron

Category	Before Degumming	After Degumming
B	0.28	0.15
A	0.54	0.28
B	0.15	0.11
C	4.56	0.63
B	0.23	0.12

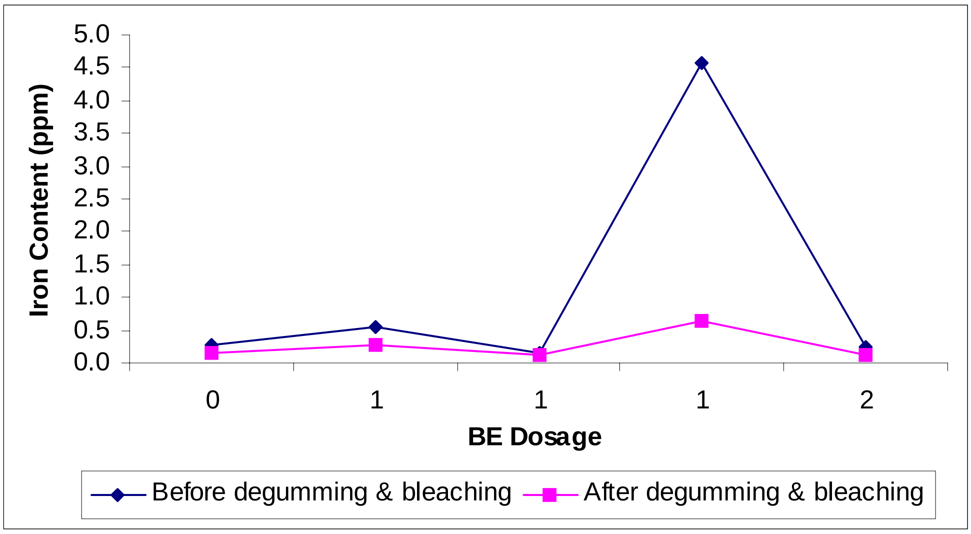


Figure 6.11: Iron Content of CPO Before & After Degumming and Bleaching Processes at 0.5 wt % of Phosphoric Acid

Category	Before Degumming	After Degumming
A	0.65	0.04
C	3.96	0.44
B	0.44	0.29
A	0.34	0.24
C	4.59	2.07

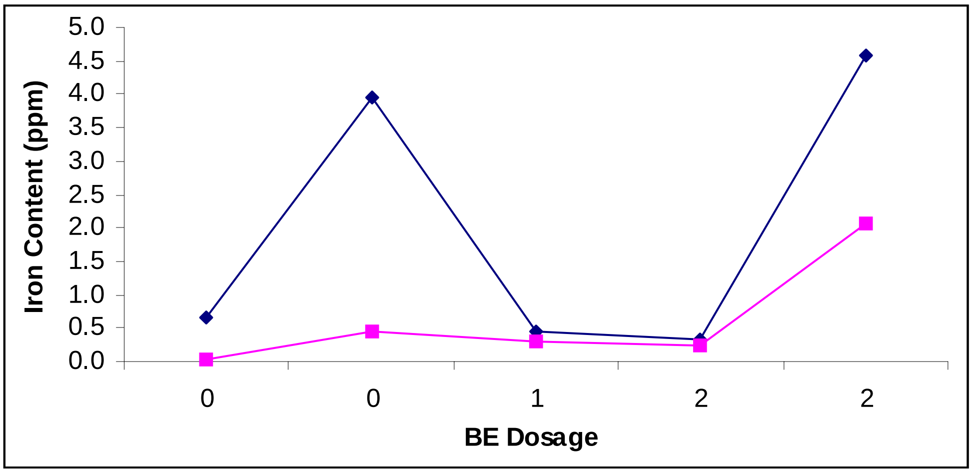


Figure 6.12: Iron Content of CPO Before & After Degumming and Bleaching Processes at 1.0 wt % of Phosphoric Acid

Trace metals such as iron and copper, present in palm oil are chiefly present in the form of metal soaps produced by the action of free fatty acids on storage tanks and other metallic equipment (Shaw and Tribe, 1983). It is believed that, these metals exhibit pro-oxidant effect on the oil by being catalyst to the decomposition of hydroperoxides. Equation 6.2 below shows the decomposition of hydroperoxides due to iron;

$$Fe^{II} + ROOH \longrightarrow Fe^{III} + OH^{-} + RO^{\bullet} \quad \ldots\ldots 6.2$$

A concentration as low as 0.03 ppm of iron content is capable of producing noticeable oxidative effect is (Higuchi, 1983).

It can be envisaged that two possible mechanisms exists for iron and copper removal (Shaw and Tribe, 1983);

- adsorption of these metal complexes by the bleaching earth or
- metal exchange by double decomposition between bleaching earth clay and the metal complex

According to Shaw and Tribe, (1983) treatment of palm oil with bleaching earth with phosphoric acid followed by bleaching earth gave residual iron levels much lower than bleaching earth alone or phosphoric acid alone. Thus, it is important to have combination process of degumming and bleaching to give optimum performance, in order to obtain the desired level of iron in DBPO and to produce an excellent decolourised and purified palm oil with good stability towards autoxidation.

Figure 6.11 and 6.12 shows the effects phosphoric acid and bleaching earth dosage on iron content during degumming and bleaching processes for all categories of CPO (A, B and C).

From the graph in Figure 6.11, when there is no bleaching earth are added in the process, the reduction in iron content is not much significant as the removal process of iron is due to degumming treatment alone. However, when both degumming and bleaching processes occurred at 1.0 –2.0 wt% of bleaching earth and 0.5 wt % of phosphoric acid the removal process of iron is effective.

Similar trend can be observed, in Figure 6. 12, but by looking at category C of oil with 0 wt % of bleaching earth and 1 wt % of phosphoric acid the reduction in iron content is quite significant. This is maybe due to higher dosage of phosphoric acid used at this particular point. Effective removal of iron also can be observed when both degumming and bleaching processes occurred at 1.0 –2.0 wt% of bleaching earth with 1.0 wt % of phosphoric acid.

6.3 Conclusion on Experimental Data Analysis

Based on the experimental data analysis done, it can be concluded that the removal or reduction level of moisture, phosphorus, peroxide value (PV) and DOBI are mainly due to bleaching treatment process. Whereas degumming treatment is responsible for influencing the increment in FFA level in CPO and DBPO. For iron, both degumming and bleaching processes are essential to ensure the removal process run effectively.

Based on the analysis, the range of suitable dosage of phosphoric acid to be used is about 0.5 - 1.0wt% and bleaching earth dosage is about 1.0 - 2.0 wt%.

In general, the contributions of both degumming and bleaching treatment processes are more preferred rather than being run separately since it will save a lots of time and save more costs.

6.4 ANN Modeling Results Analysis

In this study, three layers of MIMO and MISO network were developed the model for prediction of phosphoric acid and bleaching earth dosage.

6.4.1 Models Performance and Comparisons

The performance of the networks can also be improved or even worsen by constructing the network to be MISO and MIMO networks. The comparison among these networks are based on training and validation error generated. In this study, two MISO network and one MIMO network of each response factor combination were evaluated.

6.4.2 Models Evaluation

From Table 6.1, it can be observed that the validation error increased as the number of nodes in hidden layer increased. For example, in MISO 1 network, the validation error increased from 0.6305 to 1.1924 when number of nodes in hidden layer increased from 24 to 26.

Same trend was also observed for MISO 2 and MIMO network. In MIMO network, the validation error increased from 0.9991 to 1.0950 when the number of nodes in hidden layer increased from 24 to 26 nodes. As a result, it can be concluded that the higher the number of nodes in hidden layer, higher validation error was achieved in MIMO and MISO network.

It is most likely caused by overfitting. When the number of nodes increased, the network became more complicated due to the increasing number of weight and biases in the network that linked to each other. That is the reason why validation error increased as number of nodes in hidden layer increased.

From Table 6.1, it also can be observed that, the smallest validation errors of MISO 1, MISO 2 and MIMO networks were observed when 24 nodes available in the hidden layer with 0.6305, 0.0026 and 0.9991 respectively.

However, MIMO network performance was different. The training error fluctuate as the number of nodes increased. As the number of nodes increased from 24 to 25 the training error is increased from 2.8760e-004 to 0.0026 but as the number of nodes increased from 25 to 26 the training error dropped from 0.0026 to 0.0012. But still the smallest error is generated by the MIMO network with 24 nodes.

In addition, the value of validation error of MIMO network was larger than MISO 1 and MISO 2 network. This was most likely caused by the over trained network due to the complexity of the network that allow too many iterations.

From the analyses done, it can be concluded that, the optimum hidden layer nodes for MIMO and MISO network is 24 nodes. Interesting observation was obtained in which the validation error and training error increased as the number of nodes increased after the network reached its optimum network for MISO networks. This is perhaps due to the result of over trained network which occur when the capacity of the ANN for training is too great to allow too many training iterations (Yin *et al.*, 2003)

Table 6.1: Training and Validation Error of MISO and MIMO networks for various number of nodes in hidden layer.

No. of Nodes	**24**		**25**		**25**	
Network Structure	**Training Error**	**Validation Error**	**Training Error**	**Validation Error**	**Training Error**	**Validation Error**
MISO 1	**3.0057e-005**	**0.6305**	4.5975e-005	0.8826	6.7970e-005	1.1924
MISO 2	**1.0193 e-005**	**0.0026**	2.7973e-004	0.5553	4.6238e-005	0.9472
MIMO	**2.8760e-004**	**0.9991**	0.0026	0.9996	0.0012	1.0950

6.4.3 Model Selection

The criterion to select the most optimum network among the networks tested is based on the smallest cross-validation errors produced. Thus, from the result in Table 6.1, the optimum MISO network for response factor of phosphoric acid dosage and bleaching earth dosage prediction were selected from topologies with 12 input nodes, 24 hidden layer nodes and one output node. The selection of the network was based on the smallest validation error among other hidden layer nodes.

6.4.4 Weight and Bias

The model equation of a single neuron can be written as follows:

$$y_{pi}^{l} = \sum_{i=1}^{n} \left(w_{ij} a_{ij} \right) + b_j \qquad \text{……………………………………..6.3}$$

where w_{ij}, a_{ij} and b_j represent weight, input and bias of i row in j layer. Once the node was calculated, it passed the result to the transfer function, $f(y)$. The functions used in this study were sigmoidal function and purelin function because the normalization value of training and validation data for this simulation were in the range of 0 to 1. Thus, the complete node calculation for a sigmoidal function was:

$$f(y) = \frac{1}{1+e^{-y}} \qquad \text{…………………………………..6.4}$$

and for purelin function was:

$$f(y) = y \qquad \text{……………………………………..6.5}$$

As a result, the model equation of each response factor was presented in a value of weight of each layer.

Therefore, the input weight, layer weight and biases for all the input namely FFA, DOBI, PV, Phosphorus, moisture, and iron are presented in Table 6.2 for optimum MISO network of 12 input nodes, 24 hidden layer nodes and one output node with phosphoric acid dosage and bleaching earth dosage respectively. The initial value of weights and biases were set to 0 for all MISO networks.

In addition, Table 6.5 shows the input weight, layer weight and biases for MIMO network to predict hydrogen selectivity and H_2/CO ratio. The initial value of weights and biases were also set to 0 for MIMO network.

Table 6.2: The value of biases and weight for back-propagation model for MISO 1 and MISO 2 network

	Input weight	**Input biases [1 x 24]**	**Layer weight [24 x 1]**	**Layer bias**
Phosphoric Acid Dosage (MISO 1)	Refer to Table 6.3	3.8076 -3.2642 -3.0170 -2.7032 2.3636 -2.2543 -1.7443 -1.5053 1.1024 -0.8244 0.7833 -0.3874 -0.1838 0.3723 -0.9117 1.1216 -1.2967 -1.8235 -2.0356 -2.6445 2.5204 3.0226 -3.2656 3.5141	Columns 1 through 9 0.8476 0.0923 0.2623 0.0686 -0.6295 -0.0477 0.0030 0.2455 0.4980 Columns 10 through 18 -0.9316 -0.2888 1.2398 0.3311 -0.5409 1.0028 -0.2718 -0.0698 1.0064 Columns 19 through 24 0.8068 -0.2802 -1.4940 -0.2896 -1.1511 - 0.6698	0.9276

Bleaching Earth Dosage (MIMO 2)	Refer to Table 6.4	3.7843 3.3615 3.1318 -2.6985 -2.4426 2.1195 -1.4090 -1.4335 -1.1400 -0.8856 0.8565 -0.1284 0.1057 0.5493 0.7458 -1.2919 -1.4247 1.8070 2.0467 -2.4080 2.7698 -2.9673 -3.3607 -3.6404	Columns 1 through 9 -0.5640 -0.0027 -0.8642 0.0691 -0.3999 0.7023 1.3788 -0.7725 0.5487 Columns 10 through 18 -0.8471 -1.0427 -0.3500 -0.1677 -0.6376 0.3788 -0.0418 0.7541 0.8467 Columns 19 through 24 0.3356 0.2461 0.5496 0.8463 0.9447 0.3717	-0.7506

Table 6.3: Initial weight values for MISO 1

Input weight	-1.6567	0.7457	0.4149	-1.31	0.3496	0.8274	0.9474	1.2256	0.88	1.3469	0.8047	1.3556
	1.7544	1.037	0.8621	-0.6345	0.8843	1.718	0.8107	-0.7724	0.5217	0.7824	1.6136	-0.377
	1.4493	1.1046	-1.429	0.3825	-0.7679	0.1984	1.2193	1.3727	0.2202	-1.4927	-1.2004	0.1121
	0.3466	0.5758	-0.2428	-1.0961	-0.798	-1.6295	-0.9711	-1.3313	-0.8546	-1.4456	-1.2704	-1.065
	-1.0007	-1.2545	-0.5194	0.7495	1.1472	0.5503	-0.148	-1.4932	1.5043	-1.6587	1.3063	0.2439
	1.2495	-0.5733	-1.2435	-1.1301	0.9608	1.0781	-1.5311	-0.8079	-0.9555	-1.2022	0.7806	-0.0822
	0.0513	0.4545	1.2006	0.316	1.7568	1.7249	1.2697	1.5581	0.914	0.0559	0.5472	0.5755
	1.0708	0.2247	1.6425	-0.0005	-0.1681	-1.2209	0.1709	-2.1207	0.8449	-0.1239	1.2252	0.8401
	-0.3072	-1.7456	-0.1903	-0.1563	1.5735	0.7946	-0.71	-0.9088	-1.1213	0.7952	0.6568	1.8125
	1.5357	-1.4586	1.5659	0.0767	-0.0386	0.0869	-0.3727	1.8196	0.414	0.3122	1.4072	-1.0003
	-1.2322	-0.8338	-1.0361	1.6503	0.9969	1.3029	1.3413	-1.0051	0.2831	0.0089	-0.675	-1.2371
	0.1545	-0.3529	1.7727	-1.0874	1.3574	1.2331	-0.8683	-0.1195	0.8776	-2.1219	-0.4833	-0.0253
	-0.763	-0.0896	0.8867	-0.299	-1.1695	-0.9255	-1.5598	-0.7997	-1.1648	-1.0854	-1.0349	1.6885
	1.3369	1.6293	-0.1038	-0.8871	-0.4742	-0.2997	-1.0915	0.7711	0.5109	-0.5683	-1.8235	-1.7235
	-0.4865	0.1466	0.1147	-0.2996	-0.2517	-1.1269	-1.8556	1.5388	-1.1326	-0.5793	-1.4749	1.2503
	0.6014	-0.5697	1.5629	-0.7337	0.7553	1.6655	0.1793	0.9022	0.1099	0.9686	1.2081	-1.6769
	-2.2206	-0.0248	0.8853	-0.5531	0.3267	-0.8633	-1.8088	0.762	0.4954	-1.2167	-0.9778	0.0228
	-0.6982	0.4993	-1.1478	-0.0081	-0.2217	-2.0029	0.0043	-1.8058	1.0095	0.7705	-0.1169	-1.4448
	0.0022	-1.2377	-0.6005	0.8018	-0.1781	0.929	-1.3598	-1.5295	0.5991	1.7143	1.4813	0.6739
	-0.286	1.2876	-1.2324	-0.5166	-1.1096	1.4393	0.9272	-1.4463	1.4122	0.1203	-0.7209	-0.593
	-0.0989	1.81	0.1095	-1.4032	-0.3115	-0.9999	-0.5032	-0.3343	-1.7492	1.3459	1.0503	1.7691
	0.5383	0.4956	-0.4399	-0.1924	-0.6205	-1.1745	1.4725	1.4778	-0.8654	1.5576	0.7542	-1.6433
	-1.0637	-1.4128	0.1726	0.0027	-0.6313	1.3303	-1.2969	1.724	0.9342	1.3452	1.2329	0.4253
	1.2495	1.1547	0.5046	-1.5873	1.028	-1.1684	0.3248	-1.263	-0.5957	-1.3201	-0.1206	1.5126

Table 6.4: Initial weight values for MISO 2

Input weight	-1.2845	1.9211	0.8418	-1.0545	-0.1736	-1.4542	0.1363	0.3052	0.8041	0.0221	-0.0187	1.8057
	-1.3545	0.1571	-1.2698	0.3053	-1.8491	-0.1139	-0.5626	-0.8984	1.0779	1.2813	1.4905	-0.2534
	-0.603	0.0174	0.8144	2.1803	1.5369	0.4509	-0.9895	0.7374	-0.0471	-0.7793	1.2632	1.0373
	1.6548	1.5711	-0.5478	0.4088	-0.1311	-1.0396	-0.4274	0.5144	-1.4933	0.8904	0.6096	1.6472
	1.6281	-0.9379	0.5305	-1.4496	1.0161	0.1241	0.7338	1.4883	0.2807	-1.066	-0.5722	-1.398
	-1.2763	-0.7118	0.1165	-1.2838	1.3403	0.6116	1.2424	-0.7909	1.3262	1.1618	-0.7465	-1.3493
	0.9874	1.103	1.4139	-0.7306	0.7502	0.2754	-1.4638	-1.8554	-0.262	-1.4025	1.1286	0.581
	0.05	1.406	-1.5171	1.0991	-0.5003	0.5477	-0.3304	-1.3809	1.1996	1.5264	-0.4637	-1.229
	0.5453	0.5548	-0.2707	-1.8244	-1.3503	-0.4453	-0.7656	1.6226	1.2284	0.165	-1.2608	-1.5033
	1.5111	-0.3449	1.357	-1.5764	0.5141	0.7969	1.329	-0.3882	0.6019	-0.1205	1.5476	-1.0946
	-1.6761	1.3617	0.0545	-1.2584	-1.6574	-1.2073	0.8451	-0.1959	1.3976	-0.838	0.6656	0.1457
	1.4523	0.8545	1.078	-0.189	-0.4095	0.6548	1.2031	-1.4698	0.8372	1.5971	0.3872	-1.3358
	0.7989	0.8457	-0.9474	0.9449	0.0907	1.2707	-1.4718	1.6473	-0.7802	0.7849	1.4075	0.6738
	1.5587	0.7188	1.5397	-1.0779	0.7716	-1.3317	1.4042	1.6623	-0.0889	0.0569	-0.2108	0.6772
	0.1028	1.8551	1.1812	-1.0316	-1.5239	-0.2876	0.1122	-0.3821	1.7928	0.2023	0.824	1.1461
	-0.7109	-1.4798	1.4704	0.2912	1.175	0.9636	1.3765	0.9377	-1.0545	-0.6769	-0.5868	1.2511
	-0.5725	1.5055	-0.8804	-0.5986	1.3724	1.1875	-0.152	0.719	-1.5786	0.7594	1.54	0.7421
	0.2734	-0.7969	1.8669	-1.0559	-1.591	0.9419	0.5943	0.2302	-0.7334	0.3311	0.3362	-1.829
	1.1379	1.2909	0.6285	1.5511	-0.6078	-1.3762	-0.0369	1.4864	-0.526	-1.5601	-0.2705	-0.5676
	-0.8796	-0.7366	-0.16	-0.7869	0.8761	-1.3573	1.5735	1.3129	-1.537	0.9678	-0.9008	-0.5984
	0.2181	0.9123	0.6171	-0.3502	0.0993	-1.5719	1.3115	-1.3089	-0.6539	-1.3321	1.5347	0.9842
	-0.5823	0.8943	-1.7538	-1.2649	0.4946	1.2428	-0.294	1.4481	-0.9649	-1.1994	0.9452	0.6755
	-0.1889	-1.3159	-1.3838	0.4747	0.7957	0.9406	0.9631	0.3606	-1.6267	-1.8313	0.2699	-1.0248
	-0.4934	-1.9808	1.205	0.9834	0.6658	0.6883	-0.0484	-0.988	-0.8976	-0.0118	1.8441	0.8577

Table 6.5: The value of biases and weight of back-propagation model for MIMO network

	Input weight	**Input biases**	**Layer weight**	**Layer bias**
Phosphoric Acid and Bleaching Earth Dosage	Refer to Table 6.6	-3.6636 -3.3429 3.2057 -2.4122 -2.5010 -1.8897 1.4573 1.3680 -1.2361 -0.7666 -0.6790 0.0179 -0.2228 0.1630 -0.6295 1.1435 -1.1639 1.7737 2.0150 2.3490 2.7152 -3.1045 -3.5458 3.3361	Columns 1 through 9 0.0817 -0.3220 0.3446 -1.0607 0.4677 0.2858 -1.3240 0.5639 -0.7723 Columns 10 through 18 0.4105 -0.6183 0.6813 0.0767 1.7221 - 1.5932 -0.0777 0.4103 0.3883 Columns 19 through 24 -1.1388 0.3304 -0.0875 0.4984 -0.2473 1.1032	-0.6958

Table 6.6: Initial weight values for MIMO

Input weight	0.8421	1.0898	0.5823	1.251	-0.6398	0.3796	-0.2826	0.3678	-1.7508	-0.0186	-1.7559	1.604
	1.5517	0.4854	0.9002	1.3432	-0.5298	-0.433	0.179	0.2982	1.8816	-0.3507	1.959	0.0331
	-1.7561	0.3265	0.7549	0.6214	1.5738	-0.1337	-0.7204	1.0178	-0.4598	0.5464	1.6162	1.0032
	0.5632	-0.4156	-0.5648	-0.9678	-0.6254	-1.7659	1.0667	1.3197	-1.1697	1.2999	1.0634	-1.6055
	1.2162	0.1569	-0.7474	-1.8219	0.8057	-0.9951	0.9135	0.6311	1.838	1.3271	-0.2569	-0.0621
	0.8845	0.7198	-1.1873	-1.2438	-1.5267	-1.0829	1.1972	0.8615	0.5755	-1.5894	-0.5916	-0.1037
	-0.6966	0.8782	1.0854	2.091	1.2435	-0.7068	1.1489	0.2214	-0.3061	-1.1691	1.2779	1.2177
	-1.1521	-1.6973	0.3412	-1.3405	1.3299	-0.9281	-0.2076	0.1061	-1.4279	1.0322	1.1855	0.1897
	0.5182	1.538	-0.8803	0.4301	1.3102	-1.8408	-0.6124	0.1872	0.3032	1.0544	0.6228	1.4917
	1.7336	0.8325	0.1129	1.9424	0.4046	0.2672	-0.3883	-1.0583	1.0081	-1.2227	1.2439	0.5695
	1.7173	-1.2133	1.1573	-1.3668	0.1972	0.8203	0.4381	-0.1938	-1.4275	-0.0926	0.3925	2.0075
	-0.812	0.2156	-1.5441	-0.4929	-1.7589	-0.5852	-0.8771	0.6547	-0.4494	-0.9445	1.8766	0.6674
	0.437	-1.8999	-0.9557	0.9491	0.1497	0.6255	1.201	0.2423	1.9211	0.3239	1.5883	-1.1475
	1.2533	0.5641	-0.4919	-0.6743	-1.5464	0.5986	-0.8889	-0.423	-1.003	-2.1441	-0.7213	1.5739
	-2.2203	0.5275	1.7057	-0.3229	-1.821	-0.5086	0.363	-1.2057	0.9396	-0.3769	0.9679	-1.5467
	1.3789	-0.3477	-0.695	-1.5084	-0.7181	0.8689	-1.541	-1.5639	0.071	-0.9286	-1.2246	-0.7018
	-1.0196	-1.4675	1.8415	0.7059	-0.7507	-1.3863	1.6635	-0.9306	-0.5871	0.3062	-0.6851	-1.224
	0.1792	0.3237	0.8088	0.7392	1.6735	0.1378	-0.3425	-1.5372	-0.0723	-1.2723	-1.9184	1.262
	1.8813	-1.3963	-0.8964	0.0533	1.2412	-1.7608	-0.5555	-1.2308	-0.0606	-0.0714	-0.704	-0.1993
	0.5708	1.2072	0.7805	1.0265	0.5159	1.5579	-0.9761	1.0355	0.3306	-0.2906	-1.865	-0.7993
	0.4639	-1.6887	0.7967	0.8796	-1.9485	-0.8025	-0.1436	0.2138	-0.8579	1.5689	-1.0017	0.3036
	-1.573	0.368	0.0005	1.4588	0.8769	1.7625	-0.236	-1.2315	0.4217	0.5327	-0.2484	1.3516
	-0.7059	0.9768	-1.8318	0.5529	-1.763	0.8606	0.47	0.8686	-0.3061	1.0494	-0.226	0.9819
	1.0846	-1.944	1.5007	0.9901	-1.1079	-0.2605	1.3611	-0.883	1.9582	0.4276	-0.233	-0.2145

6.5 Conclusion on ANN Model Analysis

In this study three ANN models were developed, which are two MISO networks and one MIMO network. The criterion of selection for the best model is based on the least validation error generated among these network. Thus, the optimum network for response factor of phosphoric acid dosage and bleaching earth dosage prediction were selected from topologies of MISO network with 12 input nodes, 24 hidden layer nodes and one output node.

CHAPTER 7

CONCLUSIONS AND RECOMMENDATIONS

6.1 Conclusions

From observations of this study the following principles can be concluded;

1. The optimal operating conditions for these process operation of degumming and bleaching processes is about 100 ^{o}C , under vacuum (~50 torr) and 30 minutes of contact time for a palm oil refinery that operating using physical refining method.

2. The optimum number of experiment to be run for the study was determined through Design of Experiment (DOE) method. There are 20 set of experiment were arranged. All the 20 set of experiments with 6 quality checking experiments each, has been determined through Central Composite Design (CCD) technique via Response Surface Method (RSM) using Minitab 14 environment.

3. The removal or reduction level of moisture, phosphorus, peroxide value (PV) and DOBI are mainly due to bleaching treatment process, whereas degumming treatment is responsible for influencing the increment in FFA

level in CPO and DBPO. For iron, both degumming and bleaching processes are essential to ensure the removal process run effectively.

4. Based on the experimental analysis, the range of suitable dosage of phosphoric acid to be used is about 0.5 - 1.0wt% and bleaching earth dosage is about 1.0 - 2.0 wt%.

5. The optimum network for response factor of phosphoric acid dosage and bleaching earth dosage prediction were selected from topologies of MISO network with 12 input nodes, 24 hidden layer nodes and one output node.

6.2 Recommendations

Based on the results, discussions and experimental data analysis done, these recommendations can be taken into considerations to upgrade future studies of degummming and bleaching processes;

1. A further study can be conducted in developing a more complex neural network model such as stacked-neural network in order to obtain more accurate model in predicting the amount of phosphoric acid and bleaching earth to be added in degumming and bleaching processes of palm il refining.

APPENDIX A

MISO 1: MULTIPLE INPUT AND SINGLE OUPUT (PHOSPHORIC ACID DOSAGE)

```
clear
clc

% ------------------------------------------------------------------------------------------------------
% INITIAL VALUES OF WEIGHT AND BIAS SET UP
%------------------------------------------------------------------------------------------------------
net.IW{1,1} = [0 0 0 0 0 0 0 0 0 0 0 0; 0 0 0 0 0 0 0 0 0 0 0 0; 0 0 0 0 0 0 0 0 0 0 0 0; 0 0 0 0 0 0 0
0 0 0 0 0;0 0 0 0 0 0 0 0 0 0 0 0;0 0 0 0 0 0 0 0 0 0 0 0;0 0 0 0 0 0 0 0 0 0 0 0; 0 0 0 0 0 0 0 0 0 0 0
0; 0 0 0 0 0 0 0 0 0 0 0 0; 0 0 0 0 0 0 0 0 0 0 0 0; 0 0 0 0 0 0 0 0 0 0 0 0;0 0 0 0 0 0 0 0 0 0 0 0;0 0
0 0 0 0 0 0 0 0 0 0];
net.b{1,1}= [ 0; 0; 0; 0;0; 0;0; 0; 0; 0; 0;0; 0];
net.b{2,1}=[0];
net.LW{2,1}= [ 0 0 0 0 0 0 0  0 0 0 0 0 0];

%------------------------------------------------------------------------------------------------------
% OBJECTIVE: TO CREATE DATA FOR TRAINING
%------------------------------------------------------------------------------------------------------
ffa=[ 3.05 1.21 1.35 2.90 3.69 1.48 3.12 3.23 1.36 3.09 2.90 3.76 3.81 3.65];  % input FFA in;

dobi = [3.000 3.835 2.375 2.345 2.218 3.203 2.980 2.222 3.038 2.986 2.977 2.380 2.390 3.001];
% input DOBI in;

moisture = [2.11 0.92 0.1 0.75 2.51 2.26 2.08 0.94 2.54 2.05 2.10 2.99 2.78 0.41]; % input
moisture in;

peroxide = [1.97 2.40 2.90 2.11 3.29 1.40 2.04 1.17 2.84 1.84 2.17  3.63 3.38 3.43]; % input
peroxide value in;

phosphorus = [10.18 10.30 12.02 10.72 16.84 10.46 11.14 10.46 11.06 10.51 11.03 16.00 18.2
13.44]; % input phosphorus in;

iron = [0.15 0.54 0.65 0.23 4.59 0.34 0.17 0.38 0.78 0.17 0.21 3.2 3.96 4.09]; % input iron in;

ffao=[3.40 1.42 1.42 3.14 4.48 2.30 3.32 3.84 1.39 3.46 3.30 3.97 4.07 3.87];  % input FFA out;

dobio = [1.798 1.610 1.972 1.622 1.578 1.920 1.810 2.030 3.808 1.813 1.805 2.788 2.047 1.820];
% input DOBI out;

moistureo = [0.27 0.16 0.98 0.19 0.25 0.39 0.25 0.63 2.46 0.24 0.29 2.82 3.57 0.15]; % input
moisture out;

peroxideo = [0 0 2.83 0 0 0 0 0 2.64 0 0 3.32 3.14 0]; % input peroxide value out;

phosphoruso = [4.14 4.18 10.5 3.52 3.1 3.14 4.21 10.24 10.18 4.21 4.11 15.4 16.7 11.88]; % input
phosphorus out;
```

```
irono= [0.11 0.28 0.04 0.12 2.07 0.24 0.13 0.13 0.49 0.12 0.18 3.19 0.44 1.31]; % input iron out;


pa = [0.5 0.5 1 0.5 1 1 0.5 0 0 0.5 0.5 0 1 0]; % input bleaching earth;


%------------------------------------------------------------------------------------------------------
% OBJECTIVE: SCALED UP THE TRAINING DATA IN A RANGE OF 0 TO 1
%-------------------------------------------------------------------------------------------------------
% minffai=1.21;
% maxffai=3.81;
[ffas,minffai,maxffai]=premnmx(ffa);

% mindobii=2.222;
% maxdobii=3.835;
[dobis,mindobii,maxdobii]=premnmx(dobi);

% minmoisturei=0.10;
% maxmoisturei=2.99;
[moistures,minmoisturei,maxmoisturei]=premnmx(moisture);

% minperoxidei=1.36;
% maxperoxidei=3.63;
[peroxides,minperoxidei,maxperoxidei]=premnmx(peroxide);

% minphosphorusi=10.18;
% maxphosphorusi=18.2;
[phosphoruss,minphosphorusi,maxphosphorusi]=premnmx(phosphorus);

% minironi=0.15;
% maxironi=4.59;
[irons,minironi,maxironi]=premnmx(iron);

% min ffaoi=1.39;
% max ffaoi= 4.48;
[ffaos,minffaoi,maxffaoi]=premnmx(ffao);

% mindobioi=1.610;
% maxdobioi=3.808;
[dobios,mindobioi,maxdobioi]=premnmx(dobio);

% minmoistureoi=0.15;
% maxmoistureoi=3.57;
[moistureos,minmoistureoi,maxmoistureoi]=premnmx(moistureo);

% minperoxideoi=0;
% maxperoxideoi=3.32;
[peroxideos,minperoxideoi,maxperoxideoi]=premnmx(peroxideo);

% minphosphorusoi=3.1;
```

```
% maxphosphorusoi=16.7;
[phosphorusos,minphosphorusoi,maxphosphorusoi]=premnmx(phosphoruso);

% minironoi=0.04;
% maxironoi=2.07;
[ironos,minironoi,maxironoi]=premnmx(irono);

% minpai=0;
% maxpai=1;
[pas,minpai,maxpai]=premnmx(pa);



M=14;

    tinput(1,1:M)=ffas(1,1:M);     % input FFA in
    tinput(2,1:M)=dobis(1,1:M);    % input DOBI in
    tinput(3,1:M)=moistures(1,1:M);     % input moisture in
    tinput(4,1:M)=peroxides(1,1:M);       % input peroxide in
    tinput(5,1:M)=phosphoruss(1,1:M);    % input phosphorus in
    tinput(6,1:M)=irons(1,1:M);    % input phosphorus


    tinput(7,1:M)=ffaos(1,1:M);       % input FFA out
    tinput(8,1:M)=dobios(1,1:M);    % input DOBI out
    tinput(9,1:M)=moistureos(1,1:M);     % input moisture out
    tinput(10,1:M)=peroxideos(1,1:M);       % input peroxide out
    tinput(11,1:M)=phosphorusos(1,1:M);    % input phosphorus out
    tinput(12,1:M)=ironos(1,1:M);    % input iron out
    toutput(1,1:M)=pas(1,1:M);      % input bleaching earth


%----------------------------------------------------------------------------------------------------------
% OBJECTIVE: TO CREATE DATA FOR VALIDATION
%-----------------------------------------------------------------------------------------------------------
ffav =[1.25 2.94 3.10 3.04 2.84 3.74];  % input FFA in;

dobiv = [2.851 3.030 3.019 3.172 2.43 3.000]; % input DOBI in;

moisturev = [0.34 2.14 2.12 2.05 1.87 0.42]; % input moisture in;

peroxidev = [2.33 2.24 2.09 1.46 1.78 3.41];  % input peroxide value;

phosphorusv = [10.52 11.24 11.17 11.38 11.05 14.2]; % input phosphorus in;

ironv=[0.68 0.19 0.25 0.44 0.28 4.56]; % input iron in;

ffaov=[1.40 3.37 3.50 3.11 3.28 3.97];  % input FFA out;

dobiov =[1.938 1.779 1.910 1.994 1.978 2.214 ]; % input DOBI out;
```

```
moistureov = [0.18 0.31 0.26 0.64 2.38 0.19]; % input moisture out;

peroxideov = [0 0 0 0 1.53 0]; % input peroxide value out;

phosphorusov = [8.16 3.87 4.19 3.54 9.87 4.1]; % input phosphorus out;
ironov=[0.28 0.16 0.21 0.29 0.15 0.63]; % input iron out;



pav = [0 0.5 0.5 1 0.5 0.5]; % input bleaching earth;
bev = [2 1 1 1 0 1]; % input bleaching earth

%----------------------------------------------------------------------------------------------------
% OBJECTIVE: SCALED UP THE VALIDATION.DATA IN A RANGE OF 0 TO 1
%----------------------------------------------------------------------------------------------------
% minffaiv=1.25;
% maxffaiv=3.74;
[ffavs,minffaiv,maxffaiv]=premnmx(ffav);

% mindobiiv=2.43;
% maxdobiiv=3.172;
[dobivs,mindobiiv,maxdobiiv]=premnmx(dobiv);

% minmoistureiv=0.34;
% maxmoistureiv=2.14;
[moisturevs,minmoistureiv,maxmoistureiv]=premnmx(moisturev);

% minperoxideiv=1.46;
% maxperoxideiv=3.41;
[peroxidevs,minperoxideiv,maxperoxideiv]=premnmx(peroxidev);

% minphosphorusiv=10.52;
% maxphosphorusiv=14.20;
[phosphorusvs,minphosphorusiv,maxphosphorusiv]=premnmx(phosphorusv);


% minironiv=0.25;
% maxironiv=4.56;
[ironvs,minironiv,maxironiv]=premnmx(ironv);


% min ffaoiv=1.40;
% max ffaoiv= 3.97;
[ffaovs,minffaoiv,maxffaoiv]=premnmx(ffaov);

% mindobioiv=1.779;
% maxdobioiv=2.214;
[dobiovs,mindobioiv,maxdobioiv]=premnmx(dobiov);

% minmoistureoiv=0.18;
% maxmoistureoiv=2.38;
```

```
[moistureovs,minmoistureoiv,maxmoistureoiv]=premnmx(moistureov);

% minperoxideoiv=0;
% maxperoxideoiv=1.53;
[peroxideovs,minperoxideoiv,maxperoxideoiv]=premnmx(peroxideov);


% minphosphorusoiv=4.1;
% maxphosphorusoiv=9.87;
[phosphorusovs,minphosphorusoiv,maxphosphorusoiv]=premnmx(phosphorusov);


% minironiv=0.15;
% maxironiv=0.63;
[ironovs,minironoiv,maxironoiv]=premnmx(ironv);



% minpaiv=0;
% maxpaiv=1;
[pavs,minpaiv,maxpaiv]=premnmx(pav);



X=6;

   vinput(1,1:X)=ffavs(1,1:X);    % input FFA in
   vinput(2,1:X)=dobivs(1,1:X);   % input DOBI in
   vinput(3,1:X)=moisturevs(1,1:X);    % input moisture in
   vinput(4,1:X)=peroxidevs(1,1:X);      % input peroxide in
   vinput(5,1:X)=phosphorusvs(1,1:X);   % input phosphorus in
   vinput(6,1:X)=ironvs(1,1:X);   % input iron in



   vinput(7,1:X)=ffaovs(1,1:X);      % input FFA out
   vinput(8,1:X)=dobiovs(1,1:X);   % input DOBI out
   vinput(9,1:X)=moistureovs(1,1:X);    % input moisture out
   vinput(10,1:X)=peroxideovs(1,1:X);      % input peroxide out
   vinput(11,1:X)=phosphorusovs(1,1:X);   % input phosphorus out
    vinput(12,1:X)=ironovs(1,1:X);   % input iron out
   voutput(1,1:X)=pavs(1,1:X);     % input bleaching earth

%---------------------------------------------------------------------------------------------
% OBJECTIVE: NETWORK SETUP
%---------------------------------------------------------------------------------------------
S1=24; % Number of nodes
net=newff(minmax(tinput),[S1 1],{'logsig' 'purelin'},'trainlm');
net.trainparam.epochs=500;                  % Max epoch number
net.trainParam.goal=1e-5;
net.trainParam.show=10;
```

```
net.trainParam.max_fail=10;
net=init(net);

%------------------------------------------------------------------
% Objective: to train, error calculation and predict the output for validation and training
% an= predicted data for training
%van=predicted data for validation
% error= predicted data- onserved data
% trainrmse= root mean square training error
% valmse= root mean square validation error
%-------------------------------------------------------------------
[net,tr]=train(net,tinput,toutput);

initIW=net.IW{1,1};
initB=net.b{1,1};
initLW=net.LW{2,1};

an=sim(net,tinput);
error=an-toutput;
trainmse=sumsqr(error)/M;
trainerr=sumsqr(error);
trainrmse=sqrt(trainerr/M);

van=sim(net,vinput);
verror=van-voutput;
valmse=sumsqr(verror)/X;
valerr=sumsqr(verror);
valrmse=sqrt(valerr/X);
```

APPENDIX B

MISO 2: MULTIPLE INPUT AND SINGLE OUPUT (BLEACHING EARTH DOSAGE)

```
clear
clc

% ---------------------------------------------------------------------------------------------------
% INITIAL VALUES OF WEIGHT AND BIAS SET UP
%----------------------------------------------------------------------------------------------------
net.IW{1,1} = [0 0 0 0 0 0 0 0 0 0 0 0; 0 0 0 0 0 0 0 0 0 0 0 0; 0 0 0 0 0 0 0 0 0 0 0 0; 0 0 0 0 0 0 0
0 0 0 0 0;0 0 0 0 0 0 0 0 0 0 0 0;0 0 0 0 0 0 0 0 0 0 0 0;0 0 0 0 0 0 0 0 0 0 0 0; 0 0 0 0 0 0 0 0 0 0 0
0; 0 0 0 0 0 0 0 0 0 0 0 0; 0 0 0 0 0 0 0 0 0 0 0 0; 0 0 0 0 0 0 0 0 0 0 0 0;0 0 0 0 0 0 0 0 0 0 0 0;0 0
0 0 0 0 0 0 0 0 0 0];
net.b{1,1}= [ 0; 0; 0; 0;0; 0;0; 0; 0; 0; 0;0; 0];
net.b{2,1}=[0];
net.LW{2,1}= [ 0 0 0 0 0 0 0  0 0 0 0 0 0];

%----------------------------------------------------------------------------------------------------
% OBJECTIVE: TO CREATE DATA FOR TRAINING
%-----------------------------------------------------------------------------------------------------
ffa=[ 3.05 1.21 1.35 2.90 3.69 1.48 3.12 3.23 1.36 3.09 2.90 3.76 3.81 3.65];  % input FFA in;

dobi = [3.000 3.835 2.375 2.345 2.218 3.203 2.980 2.222 3.038 2.986 2.977 2.380 2.390 3.001];
% input DOBI in;

moisture = [2.11 0.92 0.1 0.75 2.51 2.26 2.08 0.94 2.54 2.05 2.10 2.99 2.78 0.41]; % input
moisture in;

peroxide = [1.97 2.40 2.90 2.11 3.29 1.40 2.04 1.17 2.84 1.84 2.17  3.63 3.38 3.43]; % input
peroxide value in;

phosphorus = [10.18 10.30 12.02 10.72 16.84 10.46 11.14 10.46 11.06 10.51 11.03 16.00 18.2
13.44]; % input phosphorus in;

iron = [0.15 0.54 0.65 0.23 4.59 0.34 0.17 0.38 0.78 0.17 0.21 3.2 3.96 4.09]; % input iron in;

ffao=[3.40 1.42 1.42 3.14 4.48 2.30 3.32 3.84 1.39 3.46 3.30 3.97 4.07 3.87];  % input FFA out;

dobio = [1.798 1.610 1.972 1.622 1.578 1.920 1.810 2.030 3.808 1.813 1.805 2.788 2.047 1.820];
% input DOBI out;

moistureo = [0.27 0.16 0.98 0.19 0.25 0.39 0.25 0.63 2.46 0.24 0.29 2.82 3.57 0.15]; % input
moisture out;

peroxideo = [0 0 2.83 0 0 0 0 0 2.64 0 0 3.32 3.14 0]; % input peroxide value out;
```

```
phosphoruso = [4.14 4.18 10.5 3.52 3.1 3.14 4.21 10.24 10.18 4.21 4.11 15.4 16.7 11.88]; % input phosphorus out;

irono= [0.11 0.28 0.04 0.12 2.07 0.24 0.13 0.13 0.49 0.12 0.18 3.19 0.44 1.31]; % input iron out;


be = [1 1 0 2 2 2 1 1 0 1 1 0 0 2]; % input bleaching earth;


%-----------------------------------------------------------------------------------------------------
% OBJECTIVE: SCALED UP THE TRAINING DATA IN A RANGE OF 0 TO 1
%-----------------------------------------------------------------------------------------------------
% minffai=1.21;
% maxffai=3.81;
[ffas,minffai,maxffai]=premnmx(ffa);

% mindobii=2.222;
% maxdobii=3.835;
[dobis,mindobii,maxdobii]=premnmx(dobi);

% minmoisturei=0.10;
% maxmoisturei=2.99;
[moistures,minmoisturei,maxmoisturei]=premnmx(moisture);

% minperoxidei=1.36;
% maxperoxidei=3.63;
[peroxides,minperoxidei,maxperoxidei]=premnmx(peroxide);

% minphosphorusi=10.18;
% maxphosphorusi=18.2;
[phosphoruss,minphosphorusi,maxphosphorusi]=premnmx(phosphorus);

% minironi=0.15;
% maxironi=4.59;
[irons,minironi,maxironi]=premnmx(iron);

% min ffaoi=1.39;
% max ffaoi= 4.48;
[ffaos,minffaoi,maxffaoi]=premnmx(ffao);

% mindobioi=1.610;
% maxdobioi=3.808;
[dobios,mindobioi,maxdobioi]=premnmx(dobio);

% minmoistureoi=0.15;
% maxmoistureoi=3.57;
[moistureos,minmoistureoi,maxmoistureoi]=premnmx(moistureo);

% minperoxideoi=0;
% maxperoxideoi=3.32;
[peroxideos,minperoxideoi,maxperoxideoi]=premnmx(peroxideo);
```

```
% minphosphorusoi=3.1;
% maxphosphorusoi=16.7;
[phosphorusos,minphosphorusoi,maxphosphorusoi]=premnmx(phosphoruso);

% minironoi=0.04;
% maxironoi=2.07;
[ironos,minironoi,maxironoi]=premnmx(irono);

% minbei=0;
% maxbei=2;
[bes,minbei,maxbei]=premnmx(be);



M=14;

   tinput(1,1:M)=ffas(1,1:M);     % input FFA in
   tinput(2,1:M)=dobis(1,1:M);    % input DOBI in
   tinput(3,1:M)=moistures(1,1:M);     % input moisture in
   tinput(4,1:M)=peroxides(1,1:M);       % input peroxide in
   tinput(5,1:M)=phosphoruss(1,1:M);    % input phosphorus in
   tinput(6,1:M)=irons(1,1:M);    % input phosphorus


   tinput(7,1:M)=ffaos(1,1:M);       % input FFA out
   tinput(8,1:M)=dobios(1,1:M);    % input DOBI out
   tinput(9,1:M)=moistureos(1,1:M);     % input moisture out
   tinput(10,1:M)=peroxideos(1,1:M);       % input peroxide out
   tinput(11,1:M)=phosphorusos(1,1:M);    % input phosphorus out
   tinput(12,1:M)=ironos(1,1:M);    % input iron out
   toutput(1,1:M)=bes(1,1:M);      % input bleaching earth


%----------------------------------------------------------------------------------------------------------
% OBJECTIVE: TO CREATE DATA FOR VALIDATION
%-----------------------------------------------------------------------------------------------------------
ffav =[1.25 2.94 3.10 3.04 2.84 3.74];  % input FFA in;

dobiv = [2.851 3.030 3.019 3.172 2.43 3.000]; % input DOBI in;

moisturev = [0.34 2.14 2.12 2.05 1.87 0.42]; % input moisture in;

peroxidev = [2.33 2.24 2.09 1.46 1.78 3.41];  % input peroxide value;

phosphorusv = [10.52 11.24 11.17 11.38 11.05 14.2]; % input phosphorus in;

ironv=[0.68 0.19 0.25 0.44 0.28 4.56]; % input iron in;

ffaov=[1.40 3.37 3.50 3.11 3.28 3.97];  % input FFA out;
```

```
dobiov =[1.938 1.779 1.910 1.994 1.978 2.214 ]; % input DOBI out;

moistureov = [0.18 0.31 0.26 0.64 2.38 0.19]; % input moisture out;

peroxideov = [0 0 0 0 1.53 0]; % input peroxide value out;

phosphorusov = [8.16 3.87 4.19 3.54 9.87 4.1]; % input phosphorus out;
ironov=[0.28 0.16 0.21 0.29 0.15 0.63]; % input iron out;


bev = [2 1 1 1 0 1]; % input bleaching earth;

%-----------------------------------------------------------------------------------------------------
% OBJECTIVE: SCALED UP THE VALIDATION.DATA IN A RANGE OF 0 TO 1
%------------------------------------------------------------------------------------------------------
% minffaiv=1.25;
% maxffaiv=3.74;
[ffavs,minffaiv,maxffaiv]=premnmx(ffav);

% mindobiiv=2.43;
% maxdobiiv=3.172;
[dobivs,mindobiiv,maxdobiiv]=premnmx(dobiv);

% minmoistureiv=0.34;
% maxmoistureiv=2.14;
[moisturevs,minmoistureiv,maxmoistureiv]=premnmx(moisturev);

% minperoxideiv=1.46;
% maxperoxideiv=3.41;
[peroxidevs,minperoxideiv,maxperoxideiv]=premnmx(peroxidev);

% minphosphorusiv=10.52;
% maxphosphorusiv=14.20;
[phosphorusvs,minphosphorusiv,maxphosphorusiv]=premnmx(phosphorusv);


% minironiv=0.25;
% maxironiv=4.56;
[ironvs,minironiv,maxironiv]=premnmx(ironv);


% min ffaoiv=1.40;
% max ffaoiv= 3.97;
[ffaovs,minffaoiv,maxffaoiv]=premnmx(ffaov);

% mindobioiv=1.779;
% maxdobioiv=2.214;
[dobiovs,mindobioiv,maxdobioiv]=premnmx(dobiov);

% minmoistureoiv=0.18;
% maxmoistureoiv=2.38;
```

```
[moistureovs,minmoistureoiv,maxmoistureoiv]=premnmx(moistureov);

% minperoxideoiv=0;
% maxperoxideoiv=1.53;
[peroxideovs,minperoxideoiv,maxperoxideoiv]=premnmx(peroxideov);


% minphosphorusoiv=4.1;
% maxphosphorusoiv=9.87;
[phosphorusovs,minphosphorusoiv,maxphosphorusoiv]=premnmx(phosphorusov);


% mindironiv=0.15;
% maxironiv=0.63;
[ironovs,minironoiv,maxironoiv]=premnmx(ironv);



% minbeiv=0;
% maxbeiv=2;
[bevs,minbeiv,maxbeiv]=premnmx(bev);



X=6;

   vinput(1,1:X)=ffavs(1,1:X);     % input FFA in
   vinput(2,1:X)=dobivs(1,1:X);    % input DOBI in
   vinput(3,1:X)=moisturevs(1,1:X);     % input moisture in
   vinput(4,1:X)=peroxidevs(1,1:X);       % input peroxide in
   vinput(5,1:X)=phosphorusvs(1,1:X);    % input phosphorus in
   vinput(6,1:X)=ironvs(1,1:X);    % input iron in



   vinput(7,1:X)=ffaovs(1,1:X);       % input FFA out
   vinput(8,1:X)=dobiovs(1,1:X);    % input DOBI out
   vinput(9,1:X)=moistureovs(1,1:X);     % input moisture out
   vinput(10,1:X)=peroxideovs(1,1:X);       % input peroxide out
   vinput(11,1:X)=phosphorusovs(1,1:X);    % input phosphorus out
    vinput(12,1:X)=ironovs(1,1:X);    % input iron out
   voutput(1,1:X)=bevs(1,1:X);      % input bleaching earth

%----------------------------------------------------------------------------------------------------
% OBJECTIVE: NETWORK SETUP
%----------------------------------------------------------------------------------------------------
S1=24; % Number of nodes
net=newff(minmax(tinput),[S1 1],{'logsig' 'purelin'},'trainlm');
net.trainparam.epochs=500;                    % Max epoch number
net.trainParam.goal=1e-5;
net.trainParam.show=10;
```

```
net.trainParam.max_fail=10;
net=init(net);

%-------------------------------------------------------------------
% Objective: to train, error calculation and predict the output for validation and training
% an= predicted data for training
%van=predicted data for validation
% error= predicted data- onserved data
% trainrmse= root mean square training error
% valmse= root mean square validation error
%--------------------------------------------------------------------
[net,tr]=train(net,tinput,toutput);

initIW=net.IW{1,1};
initB=net.b{1,1};
initLW=net.LW{2,1};

an=sim(net,tinput);
error=an-toutput;
trainmse=sumsqr(error)/M;
trainerr=sumsqr(error);
trainrmse=sqrt(trainerr/M);

van=sim(net,vinput);
verror=van-voutput;
valmse=sumsqr(verror)/X;
valerr=sumsqr(verror);
valrmse=sqrt(valerr/X);
```

APPENDIX C

MIMO 1: MULTIPLE INPUT MULTIPLE OUPUT

```
clear
clc

% -----------------------------------------------------------------------------------------------------
% INITIAL VALUES OF WEIGHT AND BIAS SET UP
%------------------------------------------------------------------------------------------------------
net.IW{1,1} = [0 0 0 0 0 0 0 0 0 0 0 0; 0 0 0 0 0 0 0 0 0 0 0 0; 0 0 0 0 0 0 0 0 0 0 0 0; 0 0 0 0 0 0 0
0 0 0 0 0;0 0 0 0 0 0 0 0 0 0 0 0;0 0 0 0 0 0 0 0 0 0 0 0;0 0 0 0 0 0 0 0 0 0 0 0; 0 0 0 0 0 0 0 0 0 0 0
0; 0 0 0 0 0 0 0 0 0 0 0 0; 0 0 0 0 0 0 0 0 0 0 0 0; 0 0 0 0 0 0 0 0 0 0 0 0;0 0 0 0 0 0 0 0 0 0 0 0;0 0
0 0 0 0 0 0 0 0 0 0; 0 0 0 0 0 0 0 0 0 0 0 0];
net.b{1,1}= [ 0; 0; 0; 0;0; 0;0; 0; 0; 0; 0;0; 0;0];
net.b{2,1}=[0];
net.LW{2,1}= [ 0 0 0 0 0 0 0  0 0 0 0 0 0 0];

%-------------------------------------------------------------------------------------------------------
% OBJECTIVE: TO CREATE DATA FOR TRAINING
%--------------------------------------------------------------------------------------------------------
ffa=[ 3.05 1.21 1.35 2.90 3.69 1.48 3.12 3.23 1.36 3.09 2.90 3.76 3.81 3.65];  % input FFA in;

dobi = [3.000 3.835 2.375 2.345 2.218 3.203 2.980 2.222 3.038 2.986 2.977 2.380 2.390 3.001];
% input DOBI in;

moisture = [2.11 0.92 0.1 0.75 2.51 2.26 2.08 0.94 2.54 2.05 2.10 2.99 2.78 0.41]; % input
moisture in;

peroxide = [1.97 2.40 2.90 2.11 3.29 1.40 2.04 1.17 2.84 1.84 2.17  3.63 3.38 3.43]; % input
peroxide value in;

phosphorus = [10.18 10.30 12.02 10.72 16.84 10.46 11.14 10.46 11.06 10.51 11.03 16.00 18.2
13.44]; % input phosphorus in;

iron = [0.15 0.54 0.65 0.23 4.59 0.34 0.17 0.38 0.78 0.17 0.21 3.2 3.96 4.09]; % input iron in;

ffao=[3.40 1.42 1.42 3.14 4.48 2.30 3.32 3.84 1.39 3.46 3.30 3.97 4.07 3.87];  % input FFA out;

dobio = [1.798 1.610 1.972 1.622 1.578 1.920 1.810 2.030 3.808 1.813 1.805 2.788 2.047 1.820];
% input DOBI out;

moistureo = [0.27 0.16 0.98 0.19 0.25 0.39 0.25 0.63 2.46 0.24 0.29 2.82 3.57 0.15]; % input
moisture out;

peroxideo = [0 0 2.83 0 0 0 0 0 2.64 0 0 3.32 3.14 0]; % input peroxide value out;
```

```
phosphoruso = [4.14 4.18 10.5 3.52 3.1 3.14 4.21 10.24 10.18 4.21 4.11 15.4 16.7 11.88]; % input phosphorus out;

irono= [0.11 0.28 0.04 0.12 2.07 0.24 0.13 0.13 0.49 0.12 0.18 3.19 0.44 1.31]; % input iron out;


pa = [0.5 0.5 1 0.5 1 1 0.5 0 0 0.5 0.5 0 1 0]; % input phosphoric acid;

be = [1 1 0 2 2 2 1 1 0 1 1 0 0 2]; % input bleaching earth;

%----------------------------------------------------------------------------------------------------
% OBJECTIVE: SCALED UP THE TRAINING DATA IN A RANGE OF 0 TO 1
%-----------------------------------------------------------------------------------------------------
% minffai=1.21;
% maxffai=3.81;
[ffas,minffai,maxffai]=premnmx(ffa);

% mindobii=2.222;
% maxdobii=3.835;
[dobis,mindobii,maxdobii]=premnmx(dobi);

% minmoisturei=0.10;
% maxmoisturei=2.99;
[moistures,minmoisturei,maxmoisturei]=premnmx(moisture);

% minperoxidei=1.36;
% maxperoxidei=3.63;
[peroxides,minperoxidei,maxperoxidei]=premnmx(peroxide);

% minphosphorusi=10.18;
% maxphosphorusi=18.2;
[phosphoruss,minphosphorusi,maxphosphorusi]=premnmx(phosphorus);

% minironi=0.15;
% maxironi=4.59;
[irons,minironi,maxironi]=premnmx(iron);

% min ffaoi=1.39;
% max ffaoi= 4.48;
[ffaos,minffaoi,maxffaoi]=premnmx(ffao);

% mindobioi=1.610;
% maxdobioi=3.808;
[dobios,mindobioi,maxdobioi]=premnmx(dobio);

% minmoistureoi=0.15;
% maxmoistureoi=3.57;
[moistureos,minmoistureoi,maxmoistureoi]=premnmx(moistureo);

% minperoxideoi=0;
% maxperoxideoi=3.32;
```

```
[peroxideos,minperoxideoi,maxperoxideoi]=premnmx(peroxideo);

% minphosphorusoi=3.1;
% maxphosphorusoi=16.7;
[phosphorusos,minphosphorusoi,maxphosphorusoi]=premnmx(phosphoruso);

% minironoi=0.04;
% maxironoi=2.07;
[ironos,minironoi,maxironoi]=premnmx(irono);

% minpai=0;
% maxpai=1;
[pas,minpai,maxpai]=premnmx(pa);

% minbei=0;
% maxbei=2;
[bes,minbei,maxbei]=premnmx(be);

M=14;

   tinput(1,1:M)=ffas(1,1:M);     % input FFA in
   tinput(2,1:M)=dobis(1,1:M);    % input DOBI in
   tinput(3,1:M)=moistures(1,1:M);     % input moisture in
   tinput(4,1:M)=peroxides(1,1:M);       % input peroxide in
   tinput(5,1:M)=phosphoruss(1,1:M);    % input phosphorus in
   tinput(6,1:M)=irons(1,1:M);    % input phosphorus


   tinput(7,1:M)=ffaos(1,1:M);       % input FFA out
   tinput(8,1:M)=dobios(1,1:M);    % input DOBI out
   tinput(9,1:M)=moistureos(1,1:M);     % input moisture out
   tinput(10,1:M)=peroxideos(1,1:M);       % input peroxide out
   tinput(11,1:M)=phosphorusos(1,1:M);    % input phosphorus out
   tinput(12,1:M)=ironos(1,1:M);    % input iron out
   toutput(1,1:M)=pas(1,1:M);      % input phosphoric acid
   toutput(1,1:M)=bes(1,1:M);      % input bleaching earth

%---------------------------------------------------------------------------------------------------
% OBJECTIVE: TO CREATE DATA FOR VALIDATION
%---------------------------------------------------------------------------------------------------
ffav =[1.25 2.94 3.10 3.04 2.84 3.74];  % input FFA in;

dobiv = [2.851 3.030 3.019 3.172 2.43 3.000]; % input DOBI in;

moisturev = [0.34 2.14 2.12 2.05 1.87 0.42]; % input moisture in;

peroxidev = [2.33 2.24 2.09 1.46 1.78 3.41];  % input peroxide value;

phosphorusv = [10.52 11.24 11.17 11.38 11.05 14.2]; % input phosphorus in;

ironv=[0.68 0.19 0.25 0.44 0.28 4.56]; % input iron in;
```

```
ffaov=[1.40 3.37 3.50 3.11 3.28 3.97];  % input FFA out;

dobiov =[1.938 1.779 1.910 1.994 1.978 2.214 ]; % input DOBI out;

moistureov = [0.18 0.31 0.26 0.64 2.38 0.19]; % input moisture out;

peroxideov = [0 0 0 0 1.53 0]; % input peroxide value out;

phosphorusov = [8.16 3.87 4.19 3.54 9.87 4.1]; % input phosphorus out;
ironov=[0.28 0.16 0.21 0.29 0.15 0.63]; % input iron out;



pav = [0 0.5 0.5 1 0.5 0.5]; % input phosphoric acid;

bev = [1 1 1 1 0 1]; % input bleaching earth;

%------------------------------------------------------------------------------------------------------
% OBJECTIVE: SCALED UP THE VALIDATION.DATA IN A RANGE OF 0 TO 1
%------------------------------------------------------------------------------------------------------
% minffaiv=1.25;
% maxffaiv=3.74;
[ffavs,minffaiv,maxffaiv]=premnmx(ffav);

% mindobiiv=2.43;
% maxdobiiv=3.172;
[dobivs,mindobiiv,maxdobiiv]=premnmx(dobiv);

% minmoistureiv=0.34;
% maxmoistureiv=2.14;
[moisturevs,minmoistureiv,maxmoistureiv]=premnmx(moisturev);

% minperoxideiv=1.46;
% maxperoxideiv=3.41;
[peroxidevs,minperoxideiv,maxperoxideiv]=premnmx(peroxidev);

% minphosphorusiv=10.52;
% maxphosphorusiv=14.20;
[phosphorusvs,minphosphorusiv,maxphosphorusiv]=premnmx(phosphorusv);


% minironiv=0.25;
% maxironiv=4.56;
[ironvs,minironiv,maxironiv]=premnmx(ironv);


% min ffaoiv=1.40;
% max ffaoiv= 3.97;
[ffaovs,minffaoiv,maxffaoiv]=premnmx(ffaov);
```

```
% mindobioiv=1.779;
% maxdobioiv=2.214;
[dobiovs,mindobioiv,maxdobioiv]=premnmx(dobiov);

% minmoistureoiv=0.18;
% maxmoistureoiv=2.38;
[moistureovs,minmoistureoiv,maxmoistureoiv]=premnmx(moistureov);

% minperoxideoiv=0;
% maxperoxideoiv=1.53;
[peroxideovs,minperoxideoiv,maxperoxideoiv]=premnmx(peroxideov);


% minphosphorusoiv=4.1;
% maxphosphorusoiv=9.87;
[phosphorusovs,minphosphorusoiv,maxphosphorusoiv]=premnmx(phosphorusov);


% minironiv=0.15;
% maxironiv=0.63;
[ironovs,minironoiv,maxironoiv]=premnmx(ironv);


% minpaiv=0;
% maxpaiv=1;
[pavs,minpaiv,maxpaiv]=premnmx(pav);

% minbeiv=0;
% maxbeiv=2;
[bevs,minbeiv,maxbeiv]=premnmx(bev);


X=6;

   vinput(1,1:X)=ffavs(1,1:X);     % input FFA in
   vinput(2,1:X)=dobivs(1,1:X);    % input DOBI in
   vinput(3,1:X)=moisturevs(1,1:X);     % input moisture in
   vinput(4,1:X)=peroxidevs(1,1:X);       % input peroxide in
   vinput(5,1:X)=phosphorusvs(1,1:X);    % input phosphorus in
   vinput(6,1:X)=ironvs(1,1:X);    % input iron in



   vinput(7,1:X)=ffaovs(1,1:X);       % input FFA out
   vinput(8,1:X)=dobiovs(1,1:X);    % input DOBI out
   vinput(9,1:X)=moistureovs(1,1:X);     % input moisture out
   vinput(10,1:X)=peroxideovs(1,1:X);       % input peroxide out
   vinput(11,1:X)=phosphorusovs(1,1:X);    % input phosphorus out
    vinput(12,1:X)=ironovs(1,1:X);    % input iron out
   voutput(1,1:X)=pavs(1,1:X);      % input phosphoric acid
   voutput(1,1:X)=bevs(1,1:X);      % input bleaching earth
```

```
%-----------------------------------------------------------------------------------------------------
% OBJECTIVE: NETWORK SETUP
%-----------------------------------------------------------------------------------------------------
S1=20; % Number of nodes
net=newff(minmax(tinput),[S1 1],{'logsig' 'purelin'},'trainlm');
net.trainparam.epochs=500;                    % Max epoch number
net.trainParam.goal=1e-5;
net.trainParam.show=10;
net.trainParam.max_fail=10;
net=init(net);

%--------------------------------------------------------------------
% Objective: to train, error calculation and predict the output for validation and training
% an= predicted data for training
%van=predicted data for validation
% error= predicted data- onserved data
% trainrmse= root mean square training error
% valmse= root mean square validation error
%---------------------------------------------------------------------
[net,tr]=train(net,tinput,toutput);

initIW=net.IW{1,1};
initB=net.b{1,1};
initLW=net.LW{2,1};

an=sim(net,tinput);
error=an-toutput;
trainmse=sumsqr(error)/M;
trainerr=sumsqr(error);
trainrmse=sqrt(trainerr/M);

van=sim(net,vinput);
verror=van-voutput;
valmse=sumsqr(verror)/X;
valerr=sumsqr(verror);
valrmse=sqrt(valerr/X);
```

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MISO 2: MULTIPLE INPUT AND SINGLE OUPUT (BLEACHING EARTH DOSAGE)

```
clear
clc

% ----------------------------------------------------------------------------------------------------
% INITIAL VALUES OF WEIGHT AND BIAS SET UP
%-----------------------------------------------------------------------------------------------------
net.IW{1,1} = [0 0 0 0 0 0 0 0 0 0 0 0; 0 0 0 0 0 0 0 0 0 0 0 0; 0 0 0 0 0 0 0 0 0 0 0 0; 0 0 0 0 0 0 0
0 0 0 0 0;0 0 0 0 0 0 0 0 0 0 0 0;0 0 0 0 0 0 0 0 0 0 0 0;0 0 0 0 0 0 0 0 0 0 0 0; 0 0 0 0 0 0 0 0 0 0 0
0; 0 0 0 0 0 0 0 0 0 0 0 0; 0 0 0 0 0 0 0 0 0 0 0 0; 0 0 0 0 0 0 0 0 0 0 0 0;0 0 0 0 0 0 0 0 0 0 0 0;0 0
0 0 0 0 0 0 0 0 0 0];
net.b{1,1}= [ 0; 0; 0; 0;0; 0;0; 0; 0; 0; 0;0; 0];
net.b{2,1}=[0];
net.LW{2,1}= [ 0 0 0 0 0 0 0  0 0 0 0 0 0];

%-----------------------------------------------------------------------------------------------------
% OBJECTIVE: TO CREATE DATA FOR TRAINING
%------------------------------------------------------------------------------------------------------
ffa=[ 3.05 1.21 1.35 2.90 3.69 1.48 3.12 3.23 1.36 3.09 2.90 3.76 3.81 3.65];  % input FFA in;

dobi = [3.000 3.835 2.375 2.345 2.218 3.203 2.980 2.222 3.038 2.986 2.977 2.380 2.390 3.001];
% input DOBI in;

moisture = [2.11 0.92 0.1 0.75 2.51 2.26 2.08 0.94 2.54 2.05 2.10 2.99 2.78 0.41]; % input
moisture in;

peroxide = [1.97 2.40 2.90 2.11 3.29 1.40 2.04 1.17 2.84 1.84 2.17  3.63 3.38 3.43]; % input
peroxide value in;

phosphorus = [10.18 10.30 12.02 10.72 16.84 10.46 11.14 10.46 11.06 10.51 11.03 16.00 18.2
13.44]; % input phosphorus in;

iron = [0.15 0.54 0.65 0.23 4.59 0.34 0.17 0.38 0.78 0.17 0.21 3.2 3.96 4.09]; % input iron in;

ffao=[3.40 1.42 1.42 3.14 4.48 2.30 3.32 3.84 1.39 3.46 3.30 3.97 4.07 3.87];  % input FFA out;

dobio = [1.798 1.610 1.972 1.622 1.578 1.920 1.810 2.030 3.808 1.813 1.805 2.788 2.047 1.820];
% input DOBI out;

moistureo = [0.27 0.16 0.98 0.19 0.25 0.39 0.25 0.63 2.46 0.24 0.29 2.82 3.57 0.15]; % input
moisture out;

peroxideo = [0 0 2.83 0 0 0 0 0 2.64 0 0 3.32 3.14 0]; % input peroxide value out;

phosphoruso = [4.14 4.18 10.5 3.52 3.1 3.14 4.21 10.24 10.18 4.21 4.11 15.4 16.7 11.88]; % input
phosphorus out;

irono= [0.11 0.28 0.04 0.12 2.07 0.24 0.13 0.13 0.49 0.12 0.18 3.19 0.44 1.31]; % input iron out;
```

```
be = [1 1 0 2 2 2 1 1 0 1 1 0 0 2]; % input bleaching earth;


%---------------------------------------------------------------------------------------------------------
% OBJECTIVE: SCALED UP THE TRAINING DATA IN A RANGE OF 0 TO 1
%----------------------------------------------------------------------------------------------------------
% minffai=1.21;
% maxffai=3.81;
[ffas,minffai,maxffai]=premnmx(ffa);

% mindobii=2.222;
% maxdobii=3.835;
[dobis,mindobii,maxdobii]=premnmx(dobi);

% minmoisturei=0.10;
% maxmoisturei=2.99;
[moistures,minmoisturei,maxmoisturei]=premnmx(moisture);

% minperoxidei=1.36;
% maxperoxidei=3.63;
[peroxides,minperoxidei,maxperoxidei]=premnmx(peroxide);

% minphosphorusi=10.18;
% maxphosphorusi=18.2;
[phosphoruss,minphosphorusi,maxphosphorusi]=premnmx(phosphorus);

% minironi=0.15;
% maxironi=4.59;
[irons,minironi,maxironi]=premnmx(iron);

% min ffaoi=1.39;
% max ffaoi= 4.48;
[ffaos,minffaoi,maxffaoi]=premnmx(ffao);

% mindobioi=1.610;
% maxdobioi=3.808;
[dobios,mindobioi,maxdobioi]=premnmx(dobio);

% minmoistureoi=0.15;
% maxmoistureoi=3.57;
[moistureos,minmoistureoi,maxmoistureoi]=premnmx(moistureo);

% minperoxideoi=0;
% maxperoxideoi=3.32;
[peroxideos,minperoxideoi,maxperoxideoi]=premnmx(peroxideo);

% minphosphorusoi=3.1;
% maxphosphorusoi=16.7;
[phosphorusos,minphosphorusoi,maxphosphorusoi]=premnmx(phosphoruso);
```

```
% mini ronoi=0.04;
% maxironoi=2.07;
[ironos,minironoi,maxironoi]=premnmx(irono);

% minbei=0;
% maxbei=2;
[bes,minbei,maxbei]=premnmx(be);



M=14;

    tinput(1,1:M)=ffas(1,1:M);     % input FFA in
    tinput(2,1:M)=dobis(1,1:M);    % input DOBI in
    tinput(3,1:M)=moistures(1,1:M);     % input moisture in
    tinput(4,1:M)=peroxides(1,1:M);       % input peroxide in
    tinput(5,1:M)=phosphoruss(1,1:M);    % input phosphorus in
    tinput(6,1:M)=irons(1,1:M);    % input phosphorus


    tinput(7,1:M)=ffaos(1,1:M);       % input FFA out
    tinput(8,1:M)=dobios(1,1:M);    % input DOBI out
    tinput(9,1:M)=moistureos(1,1:M);     % input moisture out
    tinput(10,1:M)=peroxideos(1,1:M);       % input peroxide out
    tinput(11,1:M)=phosphorusos(1,1:M);    % input phosphorus out
    tinput(12,1:M)=ironos(1,1:M);    % input iron out
    toutput(1,1:M)=bes(1,1:M);      % input bleaching earth


%-------------------------------------------------------------------------------------------------------
% OBJECTIVE: TO CREATE DATA FOR VALIDATION
%--------------------------------------------------------------------------------------------------------
ffav =[1.25 2.94 3.10 3.04 2.84 3.74];  % input FFA in;

dobiv = [2.851 3.030 3.019 3.172 2.43 3.000]; % input DOBI in;

moisturev = [0.34 2.14 2.12 2.05 1.87 0.42]; % input moisture in;

peroxidev = [2.33 2.24 2.09 1.46 1.78 3.41];  % input peroxide value;

phosphorusv = [10.52 11.24 11.17 11.38 11.05 14.2]; % input phosphorus in;

ironv=[0.68 0.19 0.25 0.44 0.28 4.56]; % input iron in;

ffaov=[1.40 3.37 3.50 3.11 3.28 3.97];  % input FFA out;

dobiov =[1.938 1.779 1.910 1.994 1.978 2.214 ]; % input DOBI out;

moistureov = [0.18 0.31 0.26 0.64 2.38 0.19]; % input moisture out;

peroxideov = [0 0 0 0 1.53 0]; % input peroxide value out;
```

```
phosphorusov = [8.16 3.87 4.19 3.54 9.87 4.1]; % input phosphorus out;
ironov=[0.28 0.16 0.21 0.29 0.15 0.63]; % input iron out;


bev = [2 1 1 1 0 1]; % input bleaching earth;

%----------------------------------------------------------------------------------------------------
% OBJECTIVE: SCALED UP THE VALIDATION.DATA IN A RANGE OF 0 TO 1
%-----------------------------------------------------------------------------------------------------
% minffaiv=1.25;
% maxffaiv=3.74;
[ffavs,minffaiv,maxffaiv]=premnmx(ffav);

% mindobiiv=2.43;
% maxdobiiv=3.172;
[dobivs,mindobiiv,maxdobiiv]=premnmx(dobiv);

% minmoistureiv=0.34;
% maxmoistureiv=2.14;
[moisturevs,minmoistureiv,maxmoistureiv]=premnmx(moisturev);

% minperoxideiv=1.46;
% maxperoxideiv=3.41;
[peroxidevs,minperoxideiv,maxperoxideiv]=premnmx(peroxidev);

% minphosphorusiv=10.52;
% maxphosphorusiv=14.20;
[phosphorusvs,minphosphorusiv,maxphosphorusiv]=premnmx(phosphorusv);


% minironiv=0.25;
% maxironiv=4.56;
[ironvs,minironiv,maxironiv]=premnmx(ironv);


% min ffaoiv=1.40;
% max ffaoiv= 3.97;
[ffaovs,minffaoiv,maxffaoiv]=premnmx(ffaov);

% mindobioiv=1.779;
% maxdobioiv=2.214;
[dobiovs,mindobioiv,maxdobioiv]=premnmx(dobiov);

% minmoistureoiv=0.18;
% maxmoistureoiv=2.38;
[moistureovs,minmoistureoiv,maxmoistureoiv]=premnmx(moistureov);

% minperoxideoiv=0;
% maxperoxideoiv=1.53;
[peroxideovs,minperoxideoiv,maxperoxideoiv]=premnmx(peroxideov);
```

```
% minphosphorusoiv=4.1;
% maxphosphorusoiv=9.87;
[phosphorusovs,minphosphorusoiv,maxphosphorusoiv]=premnmx(phosphorusov);


% mintroniv=0.15;
% maxironiv=0.63;
[ironovs,minironoiv,maxironoiv]=premnmx(ironv);



% minbeiv=0;
% maxbeiv=2;
[bevs,minbeiv,maxbeiv]=premnmx(bev);



X=6;

    vinput(1,1:X)=ffavs(1,1:X);     % input FFA in
    vinput(2,1:X)=dobivs(1,1:X);    % input DOBI in
    vinput(3,1:X)=moisturevs(1,1:X);     % input moisture in
    vinput(4,1:X)=peroxidevs(1,1:X);       % input peroxide in
    vinput(5,1:X)=phosphorusvs(1,1:X);    % input phosphorus in
    vinput(6,1:X)=ironvs(1,1:X);    % input iron in



    vinput(7,1:X)=ffaovs(1,1:X);       % input FFA out
    vinput(8,1:X)=dobiovs(1,1:X);    % input DOBI out
    vinput(9,1:X)=moistureovs(1,1:X);     % input moisture out
    vinput(10,1:X)=peroxideovs(1,1:X);       % input peroxide out
    vinput(11,1:X)=phosphorusovs(1,1:X);    % input phosphorus out
     vinput(12,1:X)=ironovs(1,1:X);    % input iron out
    voutput(1,1:X)=bevs(1,1:X);      % input bleaching earth

%-----------------------------------------------------------------------------------------------------
% OBJECTIVE: NETWORK SETUP
%-----------------------------------------------------------------------------------------------------
S1=20; % Number of nodes
net=newff(minmax(tinput),[S1 1],{'logsig' 'purelin'},'trainlm');
net.trainparam.epochs=500;                    % Max epoch number
net.trainParam.goal=1e-5;
net.trainParam.show=10;
net.trainParam.max_fail=10;
net=init(net);

%---------------------------------------------------------------------
% Objective: to train, error calculation and predict the output for validation and training
```

```
% an= predicted data for training
%van=predicted data for validation
% error= predicted data- onserved data
% trainrmse= root mean square training error
% valmse= root mean square validation error
%--------------------------------------------------------------------
[net,tr]=train(net,tinput,toutput);

initIW=net.IW{1,1};
initB=net.b{1,1};
initLW=net.LW{2,1};

an=sim(net,tinput);
error=an-toutput;
trainmse=sumsqr(error)/M;
trainerr=sumsqr(error);
trainrmse=sqrt(trainerr/M);

van=sim(net,vinput);
verror=van-voutput;
valmse=sumsqr(verror)/X;
valerr=sumsqr(verror);
valrmse=sqrt(valerr/X);
```

MISO 1: MULTIPLE INPUT AND SINGLE OUPUT (PHOSPHORIC ACID DOSAGE)

```
clear
clc

% ---------------------------------------------------------------------------------------------------
% INITIAL VALUES OF WEIGHT AND BIAS SET UP
%---------------------------------------------------------------------------------------------------
net.IW{1,1} = [0 0 0 0 0 0 0 0 0 0 0 0; 0 0 0 0 0 0 0 0 0 0 0 0; 0 0 0 0 0 0 0 0 0 0 0 0; 0 0 0 0 0 0 0
0 0 0 0 0;0 0 0 0 0 0 0 0 0 0 0 0;0 0 0 0 0 0 0 0 0 0 0 0;0 0 0 0 0 0 0 0 0 0 0 0; 0 0 0 0 0 0 0 0 0 0 0
0; 0 0 0 0 0 0 0 0 0 0 0 0; 0 0 0 0 0 0 0 0 0 0 0 0; 0 0 0 0 0 0 0 0 0 0 0 0;0 0 0 0 0 0 0 0 0 0 0 0;0 0
0 0 0 0 0 0 0 0 0 0];
net.b{1,1}= [ 0; 0; 0; 0;0; 0;0; 0; 0; 0; 0;0; 0];
net.b{2,1}=[0];
net.LW{2,1}= [ 0 0 0 0 0 0 0  0 0 0 0 0 0];

%---------------------------------------------------------------------------------------------------
% OBJECTIVE: TO CREATE DATA FOR TRAINING
%---------------------------------------------------------------------------------------------------
ffa=[ 3.05 1.21 1.35 2.90 3.69 1.48 3.12 3.23 1.36 3.09 2.90 3.76 3.81 3.65];  % input FFA in;

dobi = [3.000 3.835 2.375 2.345 2.218 3.203 2.980 2.222 3.038 2.986 2.977 2.380 2.390 3.001];
% input DOBI in;

moisture = [2.11 0.92 0.1 0.75 2.51 2.26 2.08 0.94 2.54 2.05 2.10 2.99 2.78 0.41]; % input
moisture in;

peroxide = [1.97 2.40 2.90 2.11 3.29 1.40 2.04 1.17 2.84 1.84 2.17  3.63 3.38 3.43]; % input
peroxide value in;

phosphorus = [10.18 10.30 12.02 10.72 16.84 10.46 11.14 10.46 11.06 10.51 11.03 16.00 18.2
13.44]; % input phosphorus in;

iron = [0.15 0.54 0.65 0.23 4.59 0.34 0.17 0.38 0.78 0.17 0.21 3.2 3.96 4.09]; % input iron in;

ffao=[3.40 1.42 1.42 3.14 4.48 2.30 3.32 3.84 1.39 3.46 3.30 3.97 4.07 3.87];  % input FFA out;

dobio = [1.798 1.610 1.972 1.622 1.578 1.920 1.810 2.030 3.808 1.813 1.805 2.788 2.047 1.820];
% input DOBI out;

moistureo = [0.27 0.16 0.98 0.19 0.25 0.39 0.25 0.63 2.46 0.24 0.29 2.82 3.57 0.15]; % input
moisture out;

peroxideo = [0 0 2.83 0 0 0 0 0 2.64 0 0 3.32 3.14 0]; % input peroxide value out;

phosphoruso = [4.14 4.18 10.5 3.52 3.1 3.14 4.21 10.24 10.18 4.21 4.11 15.4 16.7 11.88]; % input
phosphorus out;

irono= [0.11 0.28 0.04 0.12 2.07 0.24 0.13 0.13 0.49 0.12 0.18 3.19 0.44 1.31]; % input iron out;
```

```
pa = [0.5 0.5 1 0.5 1 1 0.5 0 0 0.5 0.5 0 1 0]; % input bleaching earth;


%----------------------------------------------------------------------------------------------------
% OBJECTIVE: SCALED UP THE TRAINING DATA IN A RANGE OF 0 TO 1
%-----------------------------------------------------------------------------------------------------
% minffai=1.21;
% maxffai=3.81;
[ffas,minffai,maxffai]=premnmx(ffa);

% mindobii=2.222;
% maxdobii=3.835;
[dobis,mindobii,maxdobii]=premnmx(dobi);

% minmoisturei=0.10;
% maxmoisturei=2.99;
[moistures,minmoisturei,maxmoisturei]=premnmx(moisture);

% minperoxidei=1.36;
% maxperoxidei=3.63;
[peroxides,minperoxidei,maxperoxidei]=premnmx(peroxide);

% minphosphorusi=10.18;
% maxphosphorusi=18.2;
[phosphoruss,minphosphorusi,maxphosphorusi]=premnmx(phosphorus);

% minironi=0.15;
% maxironi=4.59;
[irons,minironi,maxironi]=premnmx(iron);

% min ffaoi=1.39;
% max ffaoi= 4.48;
[ffaos,minffaoi,maxffaoi]=premnmx(ffao);

% mindobioi=1.610;
% maxdobioi=3.808;
[dobios,mindobioi,maxdobioi]=premnmx(dobio);

% minmoistureoi=0.15;
% maxmoistureoi=3.57;
[moistureos,minmoistureoi,maxmoistureoi]=premnmx(moistureo);

% minperoxideoi=0;
% maxperoxideoi=3.32;
[peroxideos,minperoxideoi,maxperoxideoi]=premnmx(peroxideo);

% minphosphorusoi=3.1;
% maxphosphorusoi=16.7;
[phosphorusos,minphosphorusoi,maxphosphorusoi]=premnmx(phosphoruso);
```

```
% mironoi=0.04;
% maxironoi=2.07;
[ironos,minironoi,maxironoi]=premnmx(irono);

% minpai=0;
% maxpai=1;
[pas,minpai,maxpai]=premnmx(pa);



M=14;

   tinput(1,1:M)=ffas(1,1:M);     % input FFA in
   tinput(2,1:M)=dobis(1,1:M);    % input DOBI in
   tinput(3,1:M)=moistures(1,1:M);     % input moisture in
   tinput(4,1:M)=peroxides(1,1:M);       % input peroxide in
   tinput(5,1:M)=phosphoruss(1,1:M);    % input phosphorus in
   tinput(6,1:M)=irons(1,1:M);    % input phosphorus


   tinput(7,1:M)=ffaos(1,1:M);       % input FFA out
   tinput(8,1:M)=dobios(1,1:M);    % input DOBI out
   tinput(9,1:M)=moistureos(1,1:M);     % input moisture out
   tinput(10,1:M)=peroxideos(1,1:M);       % input peroxide out
   tinput(11,1:M)=phosphorusos(1,1:M);    % input phosphorus out
   tinput(12,1:M)=ironos(1,1:M);    % input iron out
   toutput(1,1:M)=pas(1,1:M);      % input bleaching earth


%----------------------------------------------------------------------------------------------------
% OBJECTIVE: TO CREATE DATA FOR VALIDATION
%-----------------------------------------------------------------------------------------------------
ffav =[1.25 2.94 3.10 3.04 2.84 3.74];  % input FFA in;

dobiv = [2.851 3.030 3.019 3.172 2.43 3.000]; % input DOBI in;

moisturev = [0.34 2.14 2.12 2.05 1.87 0.42]; % input moisture in;

peroxidev = [2.33 2.24 2.09 1.46 1.78 3.41];  % input peroxide value;

phosphorusv = [10.52 11.24 11.17 11.38 11.05 14.2]; % input phosphorus in;

ironv=[0.68 0.19 0.25 0.44 0.28 4.56]; % input iron in;

ffaov=[1.40 3.37 3.50 3.11 3.28 3.97];  % input FFA out;

dobiov =[1.938 1.779 1.910 1.994 1.978 2.214 ]; % input DOBI out;

moistureov = [0.18 0.31 0.26 0.64 2.38 0.19]; % input moisture out;

peroxideov = [0 0 0 0 1.53 0]; % input peroxide value out;
```

```
phosphorusov = [8.16 3.87 4.19 3.54 9.87 4.1]; % input phosphorus out;
ironov=[0.28 0.16 0.21 0.29 0.15 0.63]; % input iron out;



pav = [0 0.5 0.5 1 0.5 0.5]; % input bleaching earth;
bev = [2 1 1 1 0 1]; % input bleaching earth

%----------------------------------------------------------------------------------------------------
% OBJECTIVE: SCALED UP THE VALIDATION.DATA IN A RANGE OF 0 TO 1
%----------------------------------------------------------------------------------------------------
% minffaiv=1.25;
% maxffaiv=3.74;
[ffavs,minffaiv,maxffaiv]=premnmx(ffav);

% mindobiiv=2.43;
% maxdobiiv=3.172;
[dobivs,mindobiiv,maxdobiiv]=premnmx(dobiv);

% minmoistureiv=0.34;
% maxmoistureiv=2.14;
[moisturevs,minmoistureiv,maxmoistureiv]=premnmx(moisturev);

% minperoxideiv=1.46;
% maxperoxideiv=3.41;
[peroxidevs,minperoxideiv,maxperoxideiv]=premnmx(peroxidev);

% minphosphorusiv=10.52;
% maxphosphorusiv=14.20;
[phosphorusvs,minphosphorusiv,maxphosphorusiv]=premnmx(phosphorusv);


% minironiv=0.25;
% maxironiv=4.56;
[ironvs,minironiv,maxironiv]=premnmx(ironv);


% min ffaoiv=1.40;
% max ffaoiv= 3.97;
[ffaovs,minffaoiv,maxffaoiv]=premnmx(ffaov);

% mindobioiv=1.779;
% maxdobioiv=2.214;
[dobiovs,mindobioiv,maxdobioiv]=premnmx(dobiov);

% minmoistureoiv=0.18;
% maxmoistureoiv=2.38;
[moistureovs,minmoistureoiv,maxmoistureoiv]=premnmx(moistureov);

% minperoxideoiv=0;
```

```
% maxperoxideoiv=1.53;
[peroxideovs,minperoxideoiv,maxperoxideoiv]=premnmx(peroxideov);


% minphosphorusoiv=4.1;
% maxphosphorusoiv=9.87;
[phosphorusovs,minphosphorusoiv,maxphosphorusoiv]=premnmx(phosphorusov);


% minironiv=0.15;
% maxironiv=0.63;
[ironovs,minironoiv,maxironoiv]=premnmx(ironv);



% minpaiv=0;
% maxpaiv=1;
[pavs,minpaiv,maxpaiv]=premnmx(pav);



X=6;

   vinput(1,1:X)=ffavs(1,1:X);     % input FFA in
   vinput(2,1:X)=dobivs(1,1:X);    % input DOBI in
   vinput(3,1:X)=moisturevs(1,1:X);     % input moisture in
   vinput(4,1:X)=peroxidevs(1,1:X);       % input peroxide in
   vinput(5,1:X)=phosphorusvs(1,1:X);    % input phosphorus in
   vinput(6,1:X)=ironvs(1,1:X);    % input iron in



   vinput(7,1:X)=ffaovs(1,1:X);       % input FFA out
   vinput(8,1:X)=dobiovs(1,1:X);    % input DOBI out
   vinput(9,1:X)=moistureovs(1,1:X);     % input moisture out
   vinput(10,1:X)=peroxideovs(1,1:X);       % input peroxide out
   vinput(11,1:X)=phosphorusovs(1,1:X);    % input phosphorus out
    vinput(12,1:X)=ironovs(1,1:X);    % input iron out
   voutput(1,1:X)=pavs(1,1:X);      % input bleaching earth

%-----------------------------------------------------------------------------------------------------
% OBJECTIVE: NETWORK SETUP
%-----------------------------------------------------------------------------------------------------
S1=20; % Number of nodes
net=newff(minmax(tinput),[S1 1],{'logsig' 'purelin'},'trainlm');
net.trainparam.epochs=500;                    % Max epoch number
net.trainParam.goal=1e-5;
net.trainParam.show=10;
net.trainParam.max_fail=10;
net=init(net);
```

```
%----------------------------------------------------------------------
% Objective: to train, error calculation and predict the output for validation and training
% an= predicted data for training
%van=predicted data for validation
% error= predicted data- onserved data
% trainrmse= root mean square training error
% valmse= root mean square validation error
%-----------------------------------------------------------------------
[net,tr]=train(net,tinput,toutput);

initIW=net.IW{1,1};
initB=net.b{1,1};
initLW=net.LW{2,1};

an=sim(net,tinput);
error=an-toutput;
trainmse=sumsqr(error)/M;
trainerr=sumsqr(error);
trainrmse=sqrt(trainerr/M);

van=sim(net,vinput);
verror=van-voutput;
valmse=sumsqr(verror)/X;
valerr=sumsqr(verror);
valrmse=sqrt(valerr/X);
```

MIMO 1: MULTIPLE INPUT MULTIPLE OUPUT

```
clear
clc

% ----------------------------------------------------------------------------------------------------
% INITIAL VALUES OF WEIGHT AND BIAS SET UP
%----------------------------------------------------------------------------------------------------
net.IW{1,1} = [0 0 0 0 0 0 0 0 0 0 0 0; 0 0 0 0 0 0 0 0 0 0 0 0; 0 0 0 0 0 0 0 0 0 0 0 0; 0 0 0 0 0 0 0
0 0 0 0 0;0 0 0 0 0 0 0 0 0 0 0 0;0 0 0 0 0 0 0 0 0 0 0 0;0 0 0 0 0 0 0 0 0 0 0 0; 0 0 0 0 0 0 0 0 0 0 0
0; 0 0 0 0 0 0 0 0 0 0 0 0; 0 0 0 0 0 0 0 0 0 0 0 0; 0 0 0 0 0 0 0 0 0 0 0 0;0 0 0 0 0 0 0 0 0 0 0 0;0 0
0 0 0 0 0 0 0 0 0 0; 0 0 0 0 0 0 0 0 0 0 0 0];
net.b{1,1}= [ 0; 0; 0; 0;0; 0;0; 0; 0; 0; 0;0; 0;0];
net.b{2,1}=[0];
net.LW{2,1}= [ 0 0 0 0 0 0 0  0 0 0 0 0 0 0];

%-----------------------------------------------------------------------------------------------------
% OBJECTIVE: TO CREATE DATA FOR TRAINING
%-----------------------------------------------------------------------------------------------------
ffa=[ 3.05 1.21 1.35 2.90 3.69 1.48 3.12 3.23 1.36 3.09 2.90 3.76 3.81 3.65];  % input FFA in;

dobi = [3.000 3.835 2.375 2.345 2.218 3.203 2.980 2.222 3.038 2.986 2.977 2.380 2.390 3.001];
% input DOBI in;

moisture = [2.11 0.92 0.1 0.75 2.51 2.26 2.08 0.94 2.54 2.05 2.10 2.99 2.78 0.41]; % input
moisture in;

peroxide = [1.97 2.40 2.90 2.11 3.29 1.40 2.04 1.17 2.84 1.84 2.17  3.63 3.38 3.43]; % input
peroxide value in;

phosphorus = [10.18 10.30 12.02 10.72 16.84 10.46 11.14 10.46 11.06 10.51 11.03 16.00 18.2
13.44]; % input phosphorus in;

iron = [0.15 0.54 0.65 0.23 4.59 0.34 0.17 0.38 0.78 0.17 0.21 3.2 3.96 4.09]; % input iron in;

ffao=[3.40 1.42 1.42 3.14 4.48 2.30 3.32 3.84 1.39 3.46 3.30 3.97 4.07 3.87];  % input FFA out;

dobio = [1.798 1.610 1.972 1.622 1.578 1.920 1.810 2.030 3.808 1.813 1.805 2.788 2.047 1.820];
% input DOBI out;

moistureo = [0.27 0.16 0.98 0.19 0.25 0.39 0.25 0.63 2.46 0.24 0.29 2.82 3.57 0.15]; % input
moisture out;

peroxideo = [0 0 2.83 0 0 0 0 0 2.64 0 0 3.32 3.14 0]; % input peroxide value out;

phosphoruso = [4.14 4.18 10.5 3.52 3.1 3.14 4.21 10.24 10.18 4.21 4.11 15.4 16.7 11.88]; % input
phosphorus out;

irono= [0.11 0.28 0.04 0.12 2.07 0.24 0.13 0.13 0.49 0.12 0.18 3.19 0.44 1.31]; % input iron out;
```

```
pa = [0.5 0.5 1 0.5 1 1 0.5 0 0 0.5 0.5 0 1 0]; % input phosphoric acid;

be = [1 1 0 2 2 2 1 1 0 1 1 0 0 2]; % input bleaching earth;

%----------------------------------------------------------------------------------------------------
% OBJECTIVE: SCALED UP THE TRAINING DATA IN A RANGE OF 0 TO 1
%----------------------------------------------------------------------------------------------------
% minffai=1.21;
% maxffai=3.81;
[ffas,minffai,maxffai]=premnmx(ffa);

% mindobii=2.222;
% maxdobii=3.835;
[dobis,mindobii,maxdobii]=premnmx(dobi);

% minmoisturei=0.10;
% maxmoisturei=2.99;
[moistures,minmoisturei,maxmoisturei]=premnmx(moisture);

% minperoxidei=1.36;
% maxperoxidei=3.63;
[peroxides,minperoxidei,maxperoxidei]=premnmx(peroxide);

% minphosphorusi=10.18;
% maxphosphorusi=18.2;
[phosphoruss,minphosphorusi,maxphosphorusi]=premnmx(phosphorus);

% minironi=0.15;
% maxironi=4.59;
[irons,minironi,maxironi]=premnmx(iron);

% min ffaoi=1.39;
% max ffaoi= 4.48;
[ffaos,minffaoi,maxffaoi]=premnmx(ffao);

% mindobioi=1.610;
% maxdobioi=3.808;
[dobios,mindobioi,maxdobioi]=premnmx(dobio);

% minmoistureoi=0.15;
% maxmoistureoi=3.57;
[moistureos,minmoistureoi,maxmoistureoi]=premnmx(moistureo);

% minperoxideoi=0;
% maxperoxideoi=3.32;
[peroxideos,minperoxideoi,maxperoxideoi]=premnmx(peroxideo);

% minphosphorusoi=3.1;
% maxphosphorusoi=16.7;
[phosphorusos,minphosphorusoi,maxphosphorusoi]=premnmx(phosphoruso);
```

```
% minironoi=0.04;
% maxironoi=2.07;
[ironos,minironoi,maxironoi]=premnmx(irono);

% minpai=0;
% maxpai=1;
[pas,minpai,maxpai]=premnmx(pa);

% minbei=0;
% maxbei=2;
[bes,minbei,maxbei]=premnmx(be);

M=14;

   tinput(1,1:M)=ffas(1,1:M);     % input FFA in
   tinput(2,1:M)=dobis(1,1:M);    % input DOBI in
   tinput(3,1:M)=moistures(1,1:M);     % input moisture in
   tinput(4,1:M)=peroxides(1,1:M);       % input peroxide in
   tinput(5,1:M)=phosphoruss(1,1:M);    % input phosphorus in
   tinput(6,1:M)=irons(1,1:M);    % input phosphorus


   tinput(7,1:M)=ffaos(1,1:M);       % input FFA out
   tinput(8,1:M)=dobios(1,1:M);    % input DOBI out
   tinput(9,1:M)=moistureos(1,1:M);     % input moisture out
   tinput(10,1:M)=peroxideos(1,1:M);       % input peroxide out
   tinput(11,1:M)=phosphorusos(1,1:M);    % input phosphorus out
   tinput(12,1:M)=ironos(1,1:M);    % input iron out
   toutput(1,1:M)=pas(1,1:M);      % input phosphoric acid
   toutput(1,1:M)=bes(1,1:M);      % input bleaching earth

%---------------------------------------------------------------------------------------------------------
% OBJECTIVE: TO CREATE DATA FOR VALIDATION
%----------------------------------------------------------------------------------------------------------
ffav =[1.25 2.94 3.10 3.04 2.84 3.74];  % input FFA in;

dobiv = [2.851 3.030 3.019 3.172 2.43 3.000]; % input DOBI in;

moisturev = [0.34 2.14 2.12 2.05 1.87 0.42]; % input moisture in;

peroxidev = [2.33 2.24 2.09 1.46 1.78 3.41];  % input peroxide value;

phosphorusv = [10.52 11.24 11.17 11.38 11.05 14.2]; % input phosphorus in;

ironv=[0.68 0.19 0.25 0.44 0.28 4.56]; % input iron in;

ffaov=[1.40 3.37 3.50 3.11 3.28 3.97];  % input FFA out;

dobiov =[1.938 1.779 1.910 1.994 1.978 2.214 ]; % input DOBI out;
```

```
moistureov = [0.18 0.31 0.26 0.64 2.38 0.19]; % input moisture out;

peroxideov = [0 0 0 0 1.53 0]; % input peroxide value out;

phosphorusov = [8.16 3.87 4.19 3.54 9.87 4.1]; % input phosphorus out;
ironov=[0.28 0.16 0.21 0.29 0.15 0.63]; % input iron out;



pav = [0 0.5 0.5 1 0.5 0.5]; % input phosphoric acid;

bev = [1 1 1 1 0 1]; % input bleaching earth;

%-----------------------------------------------------------------------------------------------------
% OBJECTIVE: SCALED UP THE VALIDATION.DATA IN A RANGE OF 0 TO 1
%-----------------------------------------------------------------------------------------------------
% minffaiv=1.25;
% maxffaiv=3.74;
[ffavs,minffaiv,maxffaiv]=premnmx(ffav);

% mindobiiv=2.43;
% maxdobiiv=3.172;
[dobivs,mindobiiv,maxdobiiv]=premnmx(dobiv);

% minmoistureiv=0.34;
% maxmoistureiv=2.14;
[moisturevs,minmoistureiv,maxmoistureiv]=premnmx(moisturev);

% minperoxideiv=1.46;
% maxperoxideiv=3.41;
[peroxidevs,minperoxideiv,maxperoxideiv]=premnmx(peroxidev);

% minphosphorusiv=10.52;
% maxphosphorusiv=14.20;
[phosphorusvs,minphosphorusiv,maxphosphorusiv]=premnmx(phosphorusv);


% minironiv=0.25;
% maxironiv=4.56;
[ironvs,minironiv,maxironiv]=premnmx(ironv);


% min ffaoiv=1.40;
% max ffaoiv= 3.97;
[ffaovs,minffaoiv,maxffaoiv]=premnmx(ffaov);

% mindobioiv=1.779;
% maxdobioiv=2.214;
[dobiovs,mindobioiv,maxdobioiv]=premnmx(dobiov);

% minmoistureoiv=0.18;
```

```
% maxmoistureoiv=2.38;
[moistureovs,minmoistureoiv,maxmoistureoiv]=premnmx(moistureov);

% minperoxideoiv=0;
% maxperoxideoiv=1.53;
[peroxideovs,minperoxideoiv,maxperoxideoiv]=premnmx(peroxideov);


% minphosphorusoiv=4.1;
% maxphosphorusoiv=9.87;
[phosphorusovs,minphosphorusoiv,maxphosphorusoiv]=premnmx(phosphorusov);


% minironiv=0.15;
% maxironiv=0.63;
[ironovs,minironoiv,maxironoiv]=premnmx(ironv);


% minpaiv=0;
% maxpaiv=1;
[pavs,minpaiv,maxpaiv]=premnmx(pav);

% minbeiv=0;
% maxbeiv=2;
[bevs,minbeiv,maxbeiv]=premnmx(bev);


X=6;

    vinput(1,1:X)=ffavs(1,1:X);     % input FFA in
    vinput(2,1:X)=dobivs(1,1:X);    % input DOBI in
    vinput(3,1:X)=moisturevs(1,1:X);     % input moisture in
    vinput(4,1:X)=peroxidevs(1,1:X);       % input peroxide in
    vinput(5,1:X)=phosphorusvs(1,1:X);    % input phosphorus in
    vinput(6,1:X)=ironvs(1,1:X);    % input iron in



    vinput(7,1:X)=ffaovs(1,1:X);       % input FFA out
    vinput(8,1:X)=dobiovs(1,1:X);    % input DOBI out
    vinput(9,1:X)=moistureovs(1,1:X);     % input moisture out
    vinput(10,1:X)=peroxideovs(1,1:X);       % input peroxide out
    vinput(11,1:X)=phosphorusovs(1,1:X);    % input phosphorus out
     vinput(12,1:X)=ironovs(1,1:X);    % input iron out
    voutput(1,1:X)=pavs(1,1:X);      % input phosphoric acid
    voutput(1,1:X)=bevs(1,1:X);      % input bleaching earth

%-----------------------------------------------------------------------------------------------------
% OBJECTIVE: NETWORK SETUP
%-----------------------------------------------------------------------------------------------------
S1=20; % Number of nodes
```

```
net=newff(minmax(tinput),[S1 1],{'logsig' 'purelin'},'trainlm');
net.trainparam.epochs=500;                  % Max epoch number
net.trainParam.goal=1e-5;
net.trainParam.show=10;
net.trainParam.max_fail=10;
net=init(net);

%-------------------------------------------------------------------
% Objective: to train, error calculation and predict the output for validation and training
% an= predicted data for training
%van=predicted data for validation
% error= predicted data- onserved data
% trainrmse= root mean square training error
% valmse= root mean square validation error
%--------------------------------------------------------------------
[net,tr]=train(net,tinput,toutput);

initIW=net.IW{1,1};
initB=net.b{1,1};
initLW=net.LW{2,1};

an=sim(net,tinput);
error=an-toutput;
trainmse=sumsqr(error)/M;
trainerr=sumsqr(error);
trainrmse=sqrt(trainerr/M);

van=sim(net,vinput);
verror=van-voutput;
valmse=sumsqr(verror)/X;
valerr=sumsqr(verror);
valrmse=sqrt(valerr/X);
```