CHAPTER II

THEORY AND LITTERATURE REVIEW

2.1 Soybean oil

Soybean oil is the most important vegetable oil produced in the world because of its high quality and low cost. Favorable agronomic characteristics, reasonable returns to the farmer and processor, high-quality protein and edible oil products, and plentiful, dependable supply of soybeans available at a competitive price will enable soybean oil to maintain its dominance in both domestic and world markets.

The advantages include: (1) a high level of unsaturation is present; (2) the oil remains liquid over a relatively wide temperature range; (3) it can be hydrogenated selectively for blending with semisolid or liquid oils; (4) when partially hydrogenated, it can be used as a operable, semisolid oil; (5) phosphatides, trace metals, and soaps in soybean oil can be removed without much difficulty to obtain a high-quality product; and (6) the presence of naturally occurring antioxidants[9].

2.1.1 Used of soybean oil [10]

Soybean oil is widely used in the chemicals industry. Firstly, in the drying oil industry it is used mainly in alkyd resin. The skipped methylene unsaturated acid can be conjugated by the action of catalysts, e.g. nickel, sulphur dioxide or iodine, to produce faster-drying coatings.

Second, the epoxidation of soybean oil is carrier out with performic acid, peracetic acid or hydrogen peroxide in the presence of a strong acid or ion exchange resin. Then epoxidized oil is used both as a stabilizer and plasticizer.

Moreover, the dimerized fatty acids of soybean oil are used in adhesives, coating, lubricants, printing inks and iron corrosion inhibitors. They are produced by condensing two unsaturated fatty acids in the presence of a catalyst. They are further converted to polyamides for applications in adhesives, coating and inks.

2.1.2 Composition and physical properties

Because of their linoleic acid contents, unhydrogenated and partially hydrogenated soybean oil are good sources of this essential fatty acid. Soybean oil is the principal vegetable oil consumed and is partially hydrogenated to impart high temperature stability to cooking oil, extend shelf life, and improve flavor stability and physical and plastic property [11]. Linoleic and linolenic acid contents of soybean oil are reduced by hydrogenation, but more important from a nutritional viewpoint are migration of double bonds up and down the carbon chain and the conversion of cis to trans isomers.

Physical properties of soybean oil depend on climate and variety. Processing the oil has an influence on the presence and level of the many minor constituents. The more important physical properties of soybean oil are summarized in Table 2.1 [12]. The values reported are not absolute because the composition and many of the properties depend on degree of unsaturation and other factor.

Properties	Value
Specific gravity at 25°C	0.922-0.927
Refractive index at 25°C	1.4765-1.4775
Iodine value (Wijs)	120-141
Viscosity, centipoises at 25°C	50.9
Solidification point (°C)	-10 to -16
Saponification number	189-195
Unsaponifiable matter, %	not over 105

Table 2.1 The ranges of physical properties of soybean oil

Except for minor amounts of impurities, some of which are removable by refining treatment, fats and oils consist of triglycerides, or esters of glycerol and high-molecular or long-chain aliphatic acids, both saturated and unsaturated, known as fatty acids. Because the fatty acid radicals constitute the greater part (usually about 95% by weight) of the glyceride molecule, and also the reactive portion, the chemical and physical properties of a fat or oil are determined largely by the properties of the component fatty acids [13].

Owning to the complexity of glyceride structure and present impossibility of accurately determining the composition of fat in terms of all the component glycerides, chemical analyses of fats usually show only the relative proportions of the different fatty acids. The soybean oil consists of a complex mixture of glycerides which containing several different fatty acids. The composition of soybean oil is shown in Table 2.2 [14].

Table 2.2 Fatty acid composition of soybean oil

Component acids	Amount of fatty acid composition (wt%)
Saturated	
Palmitic	10-11
Stearic	3-6
Unsaturated	
Oleic	20-31
Linoleic	55-61
Linolenic	6-9

Due to glycerides present being mixed, which the glyceride containing two or three different acid radicals, the physical and chemical properties of soybean oil are depend on the nature of the fatty acid moiety.

The greater the degree of unsaturation in the fatty acids of the fat molecules the softer the fat will be, and the lower its melting point will be. When there is a considerable degree of unsaturation, the fat will be liquid at room temperature and will be called oil [15].

2.1.3 Chemical reaction of soybean oil [16]

The chemical reactions of fats and fatty acids are important because they are employed in the manufacture of commercial products. Fats and fatty acids are organic chemicals and thus are subject to the numerous reactions available to the synthetic organic chemist. Because of double bonds and ester linkages in soybean oil provide reaction sites for the preparation of many useful derivatives. Chemical reactions commercially used to create important soybean oil derivatives are as shown in Table 2.3

Part of structure	Nature of reaction	Type of products
Ester linkage	(1) Hydrolysis	Fatty acids
		Glycerol
	(2) Esterification	Esters
	(3) Alcoholysis	Mono- and diglycerides,
		Monoglycerol. etc.
	(4) Reduction	Alcohols
	(5) Saponification	Soluble soaps
		Insoluble soaps
	(6) Halogenation	Fatty acid halogens
Double bond	(7) Oxidative	Polymerized oils
	polymerization	
	(8) Hydrogenation	Saturated oil
	(9) Halogenation	Halogenated oils
	(10) Epoxidation	Epoxidized oils
	(11) Sulfonation	Sulfonated oils

2.2 Hydrogenation of oil [17]

The process hydrogenation is the reduction of fatty acid double bonds of unsaturation triglycerides by the addition of gaseous hydrogen, with the aid of a nickel catalyst. It is done to increase the solidification oil of the oil to convert a liquid vegetable oil into margarine or plastic shortening. Shortening derived from vegetable oil is need, as there is insufficient animal fat to satisfy the demands of the baking industry and domestic users. Hydrogenation is also important to increase the oil oxidation stability by reducing double bonds, which are the sites of peroxide formation and subsequent off-flavor development. Oxidative stability can be improve by partial hydrogenation which reduces only some of the double bonds in the oil, although fully hydrogenation oil would be most stable.

2.2.1 Mechanism and selectivity [18]

The basic chemical equation for hydrogenation of an unsaturated carboncarbon double bond is shown below.

$$-CH = CH - + H_2 \longrightarrow -CH_2 - CH_2 - \dots \dots (1)$$

While chemically and structurally simple, the reaction can be extremely complex in practice. As previously noted and as equation 1 indicates, hydrogenation can take place only when the liquid unsaturated oil, the solid catalyst, and the gaseous hydrogen have been brought together.

Each unsaturated group of the fatty ester chain can transfer back and forth between the main body of the oil and the bulk surface of the catalyst. These unsaturated groups can be adsorbed on the catalyst surface. Each adsorbed unsaturated group can react with a hydrogen atom to form an unstable complex that is a partially hydrogenated double bond. Some of the complexes may react with another hydrogen atom to complex the saturation of the double bond. If the complex does not react with another atom of hydrogen, hydrogen is removed from the adsorbed molecule and the "new" unsaturated bond is desorbed. Both the saturated and the unsaturated bonds are desorbed from the catalyst surface and diffused into the main body of the oil. Thus not only are some of the bonds saturated but some are also isomerized to new positions or new geometric forms. The addition of a single hydrogen atom allows free rotation around the carbon-carbon single bond.

The term selectivity as used in the edible oils and fats industrial has had two meanings as applied to the hydrogenation reaction and products. The term originally was defined as the conversion of a diene to a monomer compared with the conversion of a monomer to a saturate. Another type of selectivity was applied to catalysts. If a catalyst had "selectivity", it produced an oil of softer consistency or lower melting point at a given iodine value. The model (equation 2) shown below could be used to measure the relative reaction rate constants for each hydrogenation step during batch hydrogenation of soybean oils.

2.2.2 Hydrogenation Catalyst [19]

Dry nickel is the preferred catalyst, but Raney nickel has been used without any great competitive advantage. The poisoning of nickel catalyst by sulfur not only reduce the catalyst activity but also promotes the formation of trans isomers. Copper is better than nickel in selectively reducing linolenic acid but must be used at high pressure, which is uneconomical. The use of precious metals such as palladium, platinum, and ruthenium as alternatives to conventional catalysts offers the potential to improve the control of trans isomer formation by enabling hydrogenation to proceed at reduced temperatures.

2.3 Clay mineral [20-26]

2.3.1 Introduction

Clay minerals have long been recognized as efficient material for the promotion of many organic reactions. They are highly versatile, affording both industrial (petroleum, etc.) and laboratory catalysts with excellent product-, regio- and stereo-selectivity. Clay catalysts have distinct advantages over homogenous catalyst as the work- up of the reaction mixture is often very sample, i.e. the clay is removed by filtration.

Clays are powerful adsorbents with extremely high surface areas, and industrially they are used in such diverse applications as the co-reactant for carbonless copying paper development and the decolorizing of vegetable oils.

2.3.2 The structure of clay Minerals

Clay minerals are the most abundant sedimentary mineral group. They predominate in the colloidal fractions of soils, sediments, rocks and waters and are classified as hydrous aluminosilicates.

In geology the word clay is used in two ways: firstly as a rock classification which generally implies an earthy, fine-grained material that develops plasticity on mixing a limited amount water. Secondly, it is used as a particle size term, which describes clay as materials which have particle sizes <4 μ m, although the modern tendency is to define clays as having particle sizes <2 μ m as factions of this size generally give the very pure mineral.

Clay minerals have been shown by X-ray diffraction studies to be crystalline, even in their finest particles, although the presence of traces of amorphous materials have been verified in some clay sample. Most of the important clay minerals are made up of combinations of two basic types of layers, the first consists of sheet of $[SiO_4]^{4-}$ tetrahedra arranged in hexagonal ring sub-units, show in Figure 2.1. Each silica ring is combined with the two adjacent ring so as to form anther 12-membereg ring

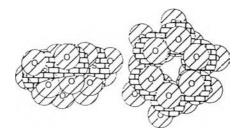


Figure 2.1 Silica sub-units of the tetrahedral sheets.

The second type of layer is usually composed of ether gibbsite $[Al_2(OH)_6]$ or brucite $[Mg_3(OH)_6]$ units which form octahedrally coordinated sheets, with the octahedral arranged so that each oxygen atom is shared with two neighboring metal atoms, show in Figure 2.2.

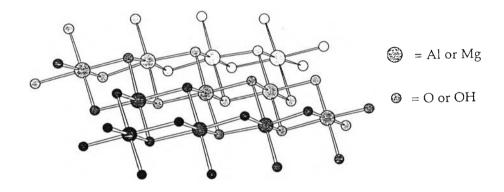


Figure 2.2 The arrangement of atoms in a dioctahedral layer.

2.3.3 Ion exchange of clay

Cation exchange clay

To a greater or lesser degree, many clays have the ability to adsorb and exchange cation from solution and it is this cation 'storage' that makes clays such an important of many soils. A typical montmorillonite can exchange over 100 millimoles of M^+ cations per 100 g of clay, whilst a kaolinite only adsorbs a few tens of millimoles.

The ideal structures of the clay minerats depicted above are deviated prom in a number of ways which introduce charge imbalances into the structure. The main causes of these charge imbalances are isomorphous exchange of Al³⁺ cations for Si⁴⁺ in the tetrahedral layers, and of Li⁺ for Mg²⁺ or Mg²⁺ for Al³⁺ cation in the octahedral layer and crytal defect, usually at the crystallite edge. Natural minerals will usually combine each of these types of exchange sites. The layers therefore have an overall negative charge which is balanced by adsorption of metal cations into the interlayer region of the clay mineral.

Anion exchange clays

A series of clay minerals of lesser importance, whose charge imbalance or layer substitution pattern has give them positively charge layers, are known. Such minerals as hydrotalcite ($Mg_{4.5}AlO_{7.5}$) and xonotlite ($Ca_6Si_6O_{17}(OH)_2$) can be used as silid 'carriers' of hydroxy and *t*-butoxy anions, respectively.

2.3.4 Acidity of Clays

Several measurement of the acidity of clay surfaces have been carried out using surface conductivity, nuclear magnetic resonance and Hammett indicator methods. These experiments show that the effective pH of water at the mineral surface is usually between 1.5 and 3; which is 4 to 5 units more acidic than of bulk water. Clay minerals show both Bronsted and Lewis acidity. Structural and environmental factors show govern the degree to which is present and normally one type predominates for a given set conditions.

A further source of acidity is associated with the –OH groups of the octahedral layer which protrude into the interlayer region *via* the holes of the ring. The incidence of these protons may be increase by preparing a 'proton-exchanged' clay. This is achieved either a simply exchanging the clay with dilute acid, or less destructively to exchanging the clay with ammonium ions and calcining at 200-300 °C expel ammonia. The exchanged proton can migrate into vacancies on the octahedral layer of diotahedral clays where they protonate bridging oxygen

2.3.5 Some clays in Thailand [27-29]

Ball clay

These clays are named from the cubes or balls in which the clay was cut. They are found from many resources in Thailand, scattering from northern, middle to southern parts of the country. Northern ball clays are Lampang ball clay from Ban jae Korn and brown ball clay from Ban Mae Than which is the largest clay production area. Ball clays in the middle and eastern parts of Thailand are in Pracheanburi and Chantaburi Provinces. Southern ball clays were found in the areas of Ampohur Nasarn, Viensah, Kanchanadith, Suraj Thaini Province and in Nakoronsrithammarat Province at Amphur Chwang, Pipoon, Lansaka, Ron Phibun and Sichon. Ball clays of Thailand have high plasticity and fine grain size and often high content of organic material. Although the clay mineral is essentially kaolinite, it is much finer than that found in china clay, giving ball clays greater plasticity and drystrength than china clays. They contain a greater quantity and variety of impurity than china clays. Iron and titanium impurities give the clay a fired colour which may vary from off white to dark brown depending on the amounts, and fluxing impurities reduce the refractoriness of the clays. The advantages of ball clays are their high plasticity and dry-strength; their disadvantages are fired colour and low refractoriness.

China clay

These are residual clays, and are essentially composed of the clay mineral kaolinite contaminated with silica, mica, feldspar and partly decomposed feldspar, all from the rock which the clay was formed. They are found in Northern parts; Lampang, Changmai and southern parts: Ranong, Nakornsrithammarat.

China clay is much finer than the contaminating materials, and thus is relatively easily purified by sedimentation, giving a white clay which is also white burning due to its low content of colouring impurities, particularly iron and titanium compounds. Large quantities of china clay are used in paper manufacture as well as in the ceramics industry.

Diatomite

Diatomite (diatomaceous earth, kieselguhr) is a sedimentary rock of marine or tacustrine deposition. It consists mainly of accumulated shells or frustules of hydrous silica secreted by diatoms, microscopic one-celled flowerless plants of the class Bacillarieae. Chemically, diatomite consists primary of silicon dioxide, and is essential inert. Impurities are other aquatic fossils, sand clay, volcanic ash, calcium carbonate. The colour of pure diatomite is white, or near white. Diatomite products are available, generally grouped according to use as follows: filter aids, fillers or extenders, thermal insulation, absorbents, catalyst carriers and insecticide carriers.

In Thailand there are several reserves of diatomite. Most of them are found in Lampang, a province of northern Thailand. The main deposits were found at Amphoe Ko Kha, Amphoe Muang and Amphoe Mae Tha. Diatomite in Lampang has a microstruvture of diatoms, namely <u>Melosira granulata</u> which is a fresh-water diatom.

2.4 Theory of catalyst [30-33]

2.4.1 Definition of catalyst

The fact that a catalyst cannot initiate a reaction does not mean that its introduction into a reacting system may not influence the composition of the reaction products or that different catalysts may not yield different products. In many cases, reaction follows a number of alternative courses. Hence, the composition of the final products will depend on the relative rate of the various alternative reactions. Where a number of different reactions occur together, the addition of a catalyst to the system may accelerate some of them to a far greater extent than others.

Catalysts increase the reaction rate through their influence on the activation energy. A catalyst breaks the reaction up into two successive steps: the combination of catalyst and reactants to form an unstable intermediate compound, and the breakdown of this compound to yield a new product and the free catalyst. This has the

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effect of permitting the energy barrier to be surmounted in two small steps, rather than one large one (Figure 2.3). In other words, two reactions with relatively low activation energies are substituted for a single reaction with high activation energy.

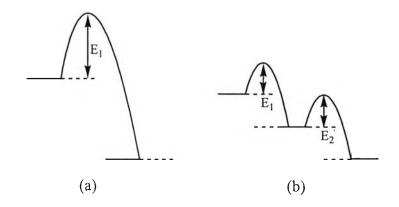


Figure 2.3 Graphical representation of the activation energy factor in (a) uncatalyzed and (b) catalyzed reaction

2.4.2 Classification of catalysts

The numerous catalysts known today can be classified according to various criteria: structure, composition, area of application, or state of aggregation. Here it shall classify the catalysts according to the state of aggregation in which they act. There are two large groups: heterogeneous catalysts (solid-state catalysts) and homogeneous catalysts. There are also intermediate forms such as homogeneous catalysts attached to solids (supported catalysts), also known as immobilized catalyst. The well known biocatalysts (enzymes) also belong to this class.

Whereas for heterogeneous catalysts, phase boundaries are always present between the catalyst and the reactants, in homogeneous catalysis, catalyst, starting materials, and products are present in the same phase. Homogeneous catalysts have a higher degree of dispersion than heterogeneous catalysts since in theory each individual atom can be catalytically active. In heterogeneous catalysts only the surface atoms are active.

 Table 2.4 Comparison of homogeneous and heterogeneous catalysts

	Homogeneous		Heterogeneous
1.	Catalyst and reactants are in the	1.	Catalyst and reactants are in different
	same phase.		phase separated by a phase boundary.
2.	Catalyst is uniformly distributed	2.	Catalyst is not uniformly distributed
	throughout the system and the		throughout the system and the
	mobility of catalyst is same as that		mobility of catalyst is different from
	of the other components.		that of the other components.
3.	Catalyst acts by it mass and the rate	3.	Catalyst acts by its surface and the
	of reaction is proportional to the		rate of reaction is proportional to the
	concentration of catalyst.		catalyst surface area exposed for the
			reaction and the concentration on the
			catalyst surface of various adsorbed
			species.
4.	High cost of catalyst losses.	4.	Low cost of catalyst losses.

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The major disadvantage of homogeneous transition metal catalysts is the difficulty of separating the catalyst from the product. Heterogeneous catalysts are either automatically removed in the process, or they can be separated by simple methods such as filtration or centrifugation. Table 2.4 summarizes the difference of the two classes of catalyst. Heterogeneous catalysts are the most important in industry generally, and in edible oil hydrogenation they are used exclusively. The hydrogenation of soybean oil, heterogeneous catalysts was used in this experiment.

2.4.3 Heterogeneous catalysis

The type of catalysis that operates the fat hydrogenation is the heterogeneous catalysis. By definition, a heterogeneous system is one in which the catalyst and the reactants exist in different physical states. Heterogeneous catalysis is to be somewhat sharply distinguished from homogeneous catalysis, in which the catalyst and the reactants comprise a single phase. In homogeneous catalysis the catalyst functions in the form of individual molecules, which are uniformly distributed through the reaction system. Thus the question of catalyst structure or of surface phenomena does not enter. On the other hand, in heterogeneous catalysis it is the question surface, which performs the catalytic function; hence the nature of the surface is of extreme importance. A catalyst operating in a homogeneous system is defined simply in terms of its chemical constitution and its concentration in the system. With all other factors controlled, the effect of a homogeneous catalyst of definite composition is exactly predictable upon the basis of its concentration. If the catalyst is a solid, however, its behavior will depend not only both the nature and extent of its surface. The fact that

so largely the submicroscopic character of its surface determines the characteristics of a solid catalyst renders the study and control of such catalysts very difficult. Apparently similar catalysts may differ enormously in their activity and specific action.

In the catalytic heterogeneous reaction, Figure 2.4, there is a reaction occurring between fluid and porous solid catalysts. In order for the reaction to occur, the reactants in the fluid must first be transported to the outer surface of the solid, and then they must diffuse through the pores of the solid to catalytically active site. At least one of the reactant species must usually be chemisorbs onto the surface of the solid. Subsequently, the reaction occurs among chemisorbed species or between a chemisorbed species and another species that is either physically adsorbed or that collides with the chemisorbed species directly from the fluid phase. After the reaction, products are desorbed and diffuse out through the pores various steps, which respond in a different way to experimental variables such as pressure, temperature. For bulk-fluid velocity, and chemical and physical structure of the catalyst, it is convenient to classify then as follows:

1. External mass transfer of reactants from the bulk fluid to the external surface of the catalyst particle.

2. Internal diffusion of reactants to active sites within the particle.

3. Adsorption of reactants on active sites.

4. Surface reaction.

5. Desorption of products.

6. Internal diffusion of products to surface of particle.

7. External mass transfer of products to bulk fluid.

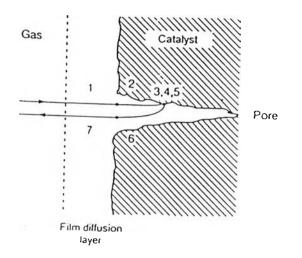


Figure 2.4 Individual steps of a heterogeneously catalyzed gas-phase reaction

2.4.4 Preparation of heterogeneous catalysts [34-36]

Preparation of the catalyst is a very important step in the study of heterogeneous catalysts. Heterogeneous catalysts are necessary to keep the properties of high reactivity, stability, and prevention of side reactions. These properties depend on the preparation technique. In the past, preparation of catalyst was an art. Properties of the catalyst may not be the same in each batch of preparation even if the same technique is used. It is according to difficulty in the control of the reaction condition. Testing instruments of catalyst give reproducibility of reaction.

There are many catalyst preparation techniques used in industry. Each catalyst has some selectivity on the preparation method. The most popular technique is impregnation.

Impregnation is the most popular and the easiest technique. A support, such as alumina, was added into metal salt solution such as nitrate salt. After dispersion of the metal salt in order to equilibrium, heating is applied to convert metal salt on the surface of support to metal oxide.

Impregnation can be done by two techniques.

1. Wet Impregnation

The support was added into an excess amount of metal salt solution. The amount of metal salt can be calculated from the different amount of metal salt in solution before and after the impregnation process. This process is too complicated and inconvenient to carry out in the laboratory.

2. Dry Impregnation

This is the most convenient technique for laboratory scale work. In this technique, it is necessary to prepare the solution, which the amount of metal salt is equal to the surface of support.

Dry impregnated catalysts became commercially available in the hydrogenation reaction. Their combination of performance characteristics (i.e., good activity, excellent selectivity, and superior filterability compared with wet impregnation catalysts) made them attractive to edible oil hydrogenators. They currently constitute essentially 100% of commercial catalysts consumed worldwide in the edible oil industry. And in this experiment, dry impregnation was used for preparing catalysts because this method is a convenient technique and easy to work in laboratory.

2.5 Literature reviews

The invention related to the catalytic hydrogenation of soybean oil and other substances, are the followings:

In 1979, Cahen [38] studied the process for partially and selectively hydrogenating soybean oil, which comprises the step of treating soybean oil in the presence of nickel catalyst on kieselguhr and a mixture of aliphatic amines. The hydrogenation of soybean oil was carried out at 140 °C and a hydrogen flow rate of 4 m³/hour was maintained during the reaction. After the reaction, liquid hydrogenation products were obtained and the formation of trans-isomers or conjugated dienes or saturated compounds was substantially reduced.

In 1990, Echeverria [6] prepared nickel-kieselguhr catalyst for hydrogenation of vegetable oil by impregnation from basic (pH 11) solution and precipitationdeposition at 80 °C from high pH (11) and low pH (6) solution. The operation conditions were carried out at 180 °C temperature, atmospheric pressure, 300 ml./min hydrogenation flow rate, 1600 rpm agitation speed, 0.02 catalyst oil ratio. The best method for the preparation of catalyst was precipitation-deposition from low pH. It had high activity but it was a difficult method of catalyst preparation and used many chemical reagents. In 1992, Rodrigo et al. [39] studied activity and selectivity for hydrogenation of sunflower seed oil by nickel supported on four natural clays in Spain. The catalysts were prepared using two different methods: precipitation and dry impregnation. The reaction was operated at a temperature of 180°C, atmospheric pressure, 200 cm³/min. hydrogen flow, 1400 rpm. agitation speed and 0.05 % w/w nickel per oil. The nickel on sepiolite catalyst had consistently more activity and selectivity than the other catalysts. Catalyst prepared by precipitation show a larger metal area and activity than those prepared by impregnation.

In 1992, Rodrigo et al. [40] prepared catalysts to be used for selective hydrogenation of sunflower seed oil. The catalyst nickel was supported on sepiolite and prepared by the precipitation/deposition method. The catalyst was used for the hydrogenation of sunflower seed oil by variable factors such as active phase concentration, reaction temperature and agitation speed. The best condition for the production of oleic was at a temperature of 180°C, agitation speed of 1200 rpm. and 0.05 % w/w nickel per oil.

In 1998, Jovanovic et al. [41] prepared catalyst to be used for selective hydrogenation of vegetable oil. The catalyst was a nickel deposited on a natural silicate support (diatomite). This catalyst was used in hydrogenation of soybean oil and sunflower oil. The reaction was operated at a temperature of 195 °C, 1.5 Mpa. hydrogen pressure and 180 min. process duration time. This catalyst has good catalytic properties and high selectivity but it is difficult to regenerate.

In 2000, Pruckchartsiri [42] studied the selective hydrogenation of soybean oil by two transition metal catalysts. The catalysts were 5 %Ni -5 %W, 5 %Ni -10 %W, 5 %Co -5 %W and 5 %Co -10 %W supported on alumina support. This research was found that the best catalyst was 5 %Ni -10 %W on alumina. The suitable condition to produce oleic acid was at 150° C temperature, 150 psig. hydrogen pressure, 2 hour time, 500 rpm. agitation speed and catalyst concentration of 10 % by weight of oil. This catalyst had high selectivity but it was expensive.